



**LABORATORY DATA CONSULTANTS, INC.**

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

August 15, 2008

SUBJECT: BRC Tronox Parcel G, Data Validation

Dear Ms. Barajas-Albalawi

Enclosed are the final validation reports for the fractions listed below. This SDG was received on July 28, 2008. Attachment 1 is a summary of the samples that were reviewed for each analysis.

**LDC Project # 19188:**

<b><u>SDG #</u></b>	<b><u>Fraction</u></b>
F8F120180	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 G

**Collection Date:** June 11, 2008

**LDC Report Date:** August 7, 2008

**Matrix:** Soil/Water

**Parameters:** Volatiles

**Validation Level:** EPA Level III & IV

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8F120180

TSB-GJ-09-10'

TSB-GJ-09-20'\*\*

TSB-GJ-09-30'

TSB-GJ-09-40'

TB-2 6/11/08

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 4 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 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 an EPA Level IV review. An 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
6/9/08	Ethanol	0.00221 ( $\geq 0.05$ )	All soil samples in SDG F8F120180	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	All water samples in SDG F8F120180	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 F8F120180	J+ (all detects)	A
5/28/08 (LICV9881)	2-Hexanone	25.04476	All water samples in SDG F8F120180	J- (all detects) UJ (all non-detects)	A

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
6/16/08 (FCAL1777)	Ethanol	0.00209 ( $\geq 0.05$ )	All soil samples in SDG F8F120180	J (all detects) UJ (all non-detects)	A

## V. Blanks

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

Sample TB-2 6/11/08 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 6/11/08	6/11/08	Dichloromethane	0.47 ug/L	All soil samples in SDG F8F120180

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
8172125MB	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. Percent recoveries (%R) and relative percent differences (RPD) were not within QC limits. Since there were no associated samples, no data were qualified.

## VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Although the percent recoveries (%R) and relative percent difference (RPD) for some compounds in the LCS/LCSD were not within QC limits, the 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.

## XI. Target Compound Identifications

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 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. Tentatively Identified Compounds (TICs)**

Tentatively identified compounds were not reported by the laboratory.

### **XIV. System Performance**

The system performance was acceptable for samples on which an 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 are summarized at the end of this report if data has been qualified.

### **XVI. Field Duplicates**

No field duplicates were identified in this SDG.



**BRC Tronox Parcel G  
Volatiles - Data Qualification Summary - SDG F8F120180**

SDG	Sample	Compound	Flag	A or P	Reason
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	Ethanol	J (all detects) UJ (all non-detects)	A	Initial calibration (RRF)
F8F120180	TB-2 6/11/08	Iodomethane	J+ (all detects)	A	Continuing calibration (%D)
F8F120180	TB-2 6/11/08	Iodomethane	J+ (all detects)	A	Continuing calibration (ICV %D)
F8F120180	TB-2 6/11/08	2-Hexanone	J- (all detects) UJ (all non-detects)	A	Continuing calibration (ICV %D)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	Ethanol	J (all detects) UJ (all non-detects)	A	Continuing calibration (RRF)

**BRC Tronox Parcel G  
Volatiles - Laboratory Blank Data Qualification Summary - SDG F8F120180**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel G  
Volatiles - Field Blank Data Qualification Summary - SDG F8F120180**

No Sample Data Qualified in this SDG

LDC #: 19188A1  
 SDG #: F8F120180  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 8/5/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: \_\_\_\_\_

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/11/08
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	W	FSD. 12
IV.	Continuing calibration/ICV	W	ICV/S 2570
V.	Blanks	A	
VI.	Surrogate spikes	W	
VII.	Matrix spike/Matrix spike duplicates	W	TSB-GJ-08-10' - No sp/ass'd. No eval
VIII.	Laboratory control samples	W	LCS/D
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)	N	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	W	TB = 5

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-GJ-09-10'	S	11	8170291MB	21	(S)	31
2	TSB-GJ-09-20**		12	8172125MB	22	W	32
3	TSB-GJ-09-30'		13	8172361MB	23	(N)	33
4	TSB-GJ-09-40'		14		24		34
5	TB-2 6/11/08	W	15		25		35
6			16		26		36
7			17		27		37
8			18		28		38
9			19		29		39
10			20		30		40

LDC #: 19188A1  
 SDG #: See COM

**VALIDATION FINDINGS CHECKLIST**

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 Reviewer: [Signature]  
 2nd Reviewer: \_\_\_\_\_

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

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>				
Were the BFB performance results reviewed and found to be within the specified criteria?	/			
Were all samples analyzed within the 12 hour clock criteria?	/			
<b>III. Initial calibration</b>				
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 $\geq 0.990$ ?	/			
Were all percent relative standard deviations (%RSD) $\leq 30\%$ and relative response factors (RRF) $> 0.05$ ?		/		
<b>IV. Continuing calibration</b>				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?	/			
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?	/			
Were all percent differences (%D) $\leq 25\%$ and relative response factors (RRF) $\geq 0.05$ ?		/		
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	/			
Was a method blank analyzed at least once every 12 hours for each matrix and concentration?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/		
<b>VI. Surrogate spikes</b>				
Were all surrogate %R within QC limits?	/	/		
If the percent recovery (%R) for one or more surrogates was out of QC limits, was a reanalysis performed to confirm samples with %R outside of criteria?		/		
<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?	/			

LDC #: 19188A1  
 SDG #: See EDW/Ver

**VALIDATION FINDINGS CHECKLIST**

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

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 checked="" type="checkbox"/>	<input 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 checked="" type="checkbox"/>	<input 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. Target 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. Compound quantitation/CRQLs</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. Tentatively identified compounds (TICs)</b>				
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were relative intensities of the major ions within $\pm$ 20% between the sample and the reference spectra?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" 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 type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>XIV. System performance</b>				
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XV. 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>XVI. 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>XVII. 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	JJJ. 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. <i>z,z-Dimethylpentanol</i>
M. 2-Butanone	GG. Xylenes, total	AAA. 1,3,5-Trimethylbenzene	UUU. 1,2-Dichlorotetrafluoroethane	OOOO. <i>Dimethyl disulfide</i>
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	AAAA. Ethyl tert-butyl ether	UUUU.
T. Dibromochloromethane	NN. Methyl ethyl ketone	HHH. 1,4-Dichlorobenzene	BBBB. tert-Amyl methyl ether	VVVV.

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

# VALIDATION FINDINGS WORKSHEET

## Initial Calibration

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

LDC #: 19188A  
 SDG #: See column

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

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

- N  A Did the laboratory perform a 5 point calibration prior to sample analysis?
- Y  N  A Were percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCC's and SPCC's?
- Y  N  A Was a curve fit used for evaluation? If yes, what was the acceptance criteria used for evaluation? \_\_\_\_\_
- Y  N  A Did the initial calibration meet the acceptance criteria?
- Y  N  A Were all %RSDs and RRFs within the validation criteria of  $\leq 30$  %RSD and  $\geq 0.05$  RRF?

#	Date	Standard ID	Compound	Finding %RSD (Limit: $\leq 30.0\%$ )	Finding RRF (Limit: $\geq 0.05$ )	Associated Samples	Qualifications
	<u>6/2/18</u>	<u>1CAL</u>	<u>N/W</u>		<u>0.00221</u>	<u>M501's. 8170-91MB</u>	<u>✓UN/A</u>

VALIDATION FINDINGS WORKSHEET  
 Continuing Calibration

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

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

N N/A Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?

N N/A Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's?

Y (N) N/A Were all %D and RRFs within the validation criteria of  $\leq 25\%$  %D and  $\geq 0.05$  RRF?

#	Date	Standard ID	Compound	Finding %D (Limit: $\leq 25.0\%$ )	Finding RRF (Limit: $\geq 0.05$ )	Associated Samples	Qualifications
	5/28/08	LCV988 (ICV)	1,4-dichlorobenzene Z	31.67573 25.04476		5.817>175MB	10/28/08 1/21/09
	6/16/08	FCA1777	N,N,N		0.00309	M/SOIS 8170291MB	1/21/09
	6/19/08	LCA40317	1,4-dichlorobenzene	67.71684		5.817>175MB	10/28/08

**VALIDATION FINDINGS WORKSHEET**  
**Field Blanks**

Page: 6 of 7  
 Reviewer: \_\_\_\_\_  
 2nd Reviewer: \_\_\_\_\_

LDC #: 19188A  
 SDG #: SEEDW

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

Y/N/N/A Were field blanks identified in this SDG?  
 Y/N/N/A Were target compounds detected in the field blanks?  
 Blank units: ppb Associated sample units: ppb  
 Sampling date: 5/11/08  
 Field blank type: (circle one) Field Blank / Rinsate / Trip Blank / Other: Field Blank Associated Samples: All soils

Compound	Blank ID	Sample Identification									
	<u>5</u>										
Methylene chloride											
Acetone											
Chloroform											
<u>Dichloromethane</u>	<u>0.47</u>										
CRQL											

Blank units: \_\_\_\_\_ Associated sample units: \_\_\_\_\_  
 Sampling date: \_\_\_\_\_  
 Field blank type: (circle one) Field Blank / Rinsate / Trip Blank / Other: \_\_\_\_\_ Associated Samples: \_\_\_\_\_

Compound	Blank ID	Sample Identification									
Methylene chloride											
Acetone											
Chloroform											
CRQL											

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:  
 Common contaminants such as Methylene chloride, Acetone, 2-Butanone and Carbon disulfide that were detected in samples within ten times the associated field blank concentration were qualified as not detected, "U". Other contaminants within five times the field blank concentration were also qualified as not detected, "U".



# VALIDATION FINDINGS WORKSHEET

## Surrogate Spikes

LDC #: 19188A  
 SDG #: See above

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

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

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".  
 Were all surrogate %R within QC limits? N/A  
 If the percent recovery (%R) for one or more surrogates was out of QC limits, was a reanalysis performed to confirm samples with %R out of outside of criteria? N/A

#	Date	Sample ID	Surrogate	%Recovery (Limits)	Qualifications
		8170125 MB	BFB	117 ( ) ( )	+ dete P
				( ) ( )	
				( ) ( )	
				( ) ( )	
				( ) ( )	
				( ) ( )	
				( ) ( )	
				( ) ( )	
				( ) ( )	
				( ) ( )	
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				( ) ( )	
				( ) ( )	
				( ) ( )	
				( ) ( )	

SMC1 (TOL) = Toluene-d8  
 SMC2 (BFB) = Bromofluorobenzene  
 SMC3 (DCE) = 1,2-Dichloroethane-d4  
 SMC4 (DFM) = Dibromofluoromethane

QC Limits (Soil)  
 81-117  
 74-121  
 80-120  
 80-120

QC Limits (Water)  
 88-110  
 86-115  
 80-120  
 86-118

SUR-1SB

# VALIDATION FINDINGS WORKSHEET

## Laboratory Control Samples (LCS)

LDC #: 19188A

Page: 1 of 2

Reviewer: [Signature]

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

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

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

N N/A Was a LCS required?

Y (N) N/A Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?

#	Date	LCS/LCSD ID	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		8172/25LC9	NN	293 (42-140)	( )	112 (≤ 20)	5. 8172/25NB	No dual
		7	1,2-dichloroethane	181 (45-140)	( )	( )		✓ (N's / NSD m)

LDC #: 9108A  
 SDG #: See com

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

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

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

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

$RRF = (A_x)(C_b)/(A_b)(C_x)$   
 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  
 $X$  = Mean of the RRFs  
 $A_b$  = Area of associated internal standard  
 $C_b$  = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				RRF (std)	(S)	RRF (std)	(S)	Average RRF (initial)	%RSD	Average RRF (initial)	%RSD
1	1CAL (F)	6/9/08	K (1st internal standard)	0.50971	0.50971	0.50831	0.50831	7.04091	7.0405		
			AA (2nd internal standard)	0.25572	0.25572	0.29404	0.29404	12.7450	12.7450		
			DDD (3rd internal standard)	3.57047	3.57047	3.42599	3.42599	8.25563	8.2556		
2	1CAL (F)	6/9/08	NNNN (1st internal standard)	0.74129	0.74129	0.73871	0.73871	2.56798	2.5677		
			OOOO (2nd internal standard)	0.59203	0.59203	0.55366	0.55366	13.47144	13.4718		
			OOO (3rd internal standard)	1.11568	1.11568	1.11150	1.11150	2.41699	2.4169		
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.

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

**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 =  $100 \cdot (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$   
 $\text{RRF} = (A_x)(C_b) / (A_b)(C_x)$

Where: ave. RRF = initial calibration average RRF  
 RRF = continuing calibration RRF  
 $A_x$  = Area of compound,  
 $C_x$  = Concentration of compound,  
 $A_b$  = Area of associated internal standard  
 $C_b$  = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference internal Standard)	Average RRF (Initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	FEA-1778	6/16/08	K	0.50831	0.57288	0.89949	0.57288	0.89949
	AA		0.31216	6.16168	0.31216	6.16168	0.31216	6.16168
	DDD		3.42599	6.59782	3.65203	6.59782	3.65203	6.59782
2	FEA-1777	6/16/08	NNNN (1st internal standard)	0.73871	0.72154	2.32442	0.72154	2.32442
	OOO (2nd internal standard)		0.55366	3.59757	0.57358	3.59757	0.57358	3.59757
	OOO (3rd internal standard)		1.11152	0.61152	1.10470	0.61152	1.10470	0.61152
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 #: 19188A1  
 SDG #: see down

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

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

**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	45.0289	90	90	0
Bromofluorobenzene	↓	42.1691	84	84	↓
1,2-Dichloroethane-d4	↓	45.1855	90	90	↓
Dibromofluoromethane	↓	44.0752	88	88	↓

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 #: 19185A  
 SDG #: See above

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

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

**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 =  $100 * SSC/SA$       Where: SSC = Spiked sample concentration  
 SA = Spike added  
 RPD =  $100 * |LCS - LCSDC| / 2 * (LCS + LCSDC)$       LCSC = Laboratory control sample concentration    LCSDC = Laboratory control sample duplicate concentration

LCS ID: 8170291

Compound	Spike Added (ug/L)		Spiked Sample Concentration (ug/L)		LCS		LCSD		Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
1,1-Dichloroethene	50	NA	47.8	NA	96	96								
Trichloroethene			49.5		99	99								
Benzene			49.6		99	99								
Toluene			50.2		100	100								
Chlorobenzene			49.9		100	100								

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.

LCCLC.15B

LDC #: 19188A  
 SDG #: SPCC01V

## VALIDATION FINDINGS WORKSHEET

### Sample Calculation Verification

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

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

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?

$$\text{Concentration} = \frac{(A_x)(I_s)(DF)}{(A_s)(RRF)(V_o)(\%S)}$$

- $A_x$  = Area of the characteristic ion (EICP) for the compound to be measured
- $A_s$  = Area of the characteristic ion (EICP) for the specific internal standard
- $I_s$  = Amount of internal standard added in nanograms (ng)
- RRF = Relative response factor of the calibration standard.
- $V_o$  = Volume or weight of sample pruged in milliliters (ml) or grams (g).
- Df = Dilution factor.
- %S = Percent solids, applicable to soils and solid matrices only.

Example:

Sample I.D. 2 , NO:

$$\text{Conc.} = \frac{(\quad)(\quad)(\quad)}{(\quad)(\quad)(\quad)(\quad)}$$

=

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

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

**Project/Site Name:** BRC Tronox Parcel G  
**Collection Date:** June 11, 2008  
**LDC Report Date:** August 6, 2008  
**Matrix:** Soil  
**Parameters:** Semivolatiles  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8F120180

**Sample Identification**

TSB-GJ-09-10'  
TSB-GJ-09-20'\*\*  
TSB-GJ-09-30'  
TSB-GJ-09-40'

\*\*Indicates sample underwent EPA Level IV review



## Introduction

This data review covers 4 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

## III. Initial Calibration

Initial calibration was performed using required standard concentrations.

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

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	0.01422 ( $\geq 0.05$ )	All samples in SDG F8F120180	J (all detects) UJ (all non-detects)	A
	N-(Hydroxymethyl)phthalimide	0.04408 ( $\geq 0.05$ )		J (all detects) UJ (all non-detects)	

## 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	Phthalic acid	25.06878	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	J- (all detects) UJ (all non-detects)	A

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 ( $\geq 0.05$ )	8168439MB	J (all detects) UJ (all non-detects)	A
	N-(Hydroxymethyl)phthalimide	0.04331 ( $\geq 0.05$ )		J (all detects) UJ (all non-detects)	
6/19/08	Phthalic acid	0.01066 ( $\geq 0.05$ )	TSB-GJ-09-10' TSB-GJ-09-20'**	J (all detects) UJ (all non-detects)	A
	N-(Hydroxymethyl)phthalimide	0.04523 ( $\geq 0.05$ )	TSB-GJ-09-30' TSB-GJ-09-40'	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 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-GJ-09-10'	Perylene-d12	198321 (281395-1125580)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) UJ (all non-detects)	A
TSB-GJ-09-20'***	Perylene-d12	191974 (281395-1125580)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) UJ (all non-detects)	A
TSB-GJ-09-30'	Perylene-d12	206248 (281395-1125580)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) UJ (all non-detects)	A
TSB-GJ-09-40'	Perylene-d12	212988 (281395-1125580)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) UJ (all non-detects)	A

## **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)**

Tentatively identified compounds were not reported by the laboratory.

## **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 G  
Semivolatiles - Data Qualification Summary - SDG F8F120180**

SDG	Sample	Compound	Flag	A or P	Reason
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'*** TSB-GJ-09-30' TSB-GJ-09-40'	Phthalic acid N-(Hydroxymethyl)phthalimide	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A	Initial calibration (RRF)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'*** TSB-GJ-09-30' TSB-GJ-09-40'	Phthalic acid	J- (all detects) UJ (all non-detects)	A	Continuing calibration (%D)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'*** TSB-GJ-09-30' TSB-GJ-09-40'	Phthalic acid N-(Hydroxymethyl)phthalimide	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A	Continuing calibration (RRF)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'*** TSB-GJ-09-30' TSB-GJ-09-40'	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) UJ (all non-detects)	A	Internal standards (area)

**BRC Tronox Parcel G  
Semivolatiles - Laboratory Blank Data Qualification Summary - SDG F8F120180**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel G  
Semivolatiles - Field Blank Data Qualification Summary - SDG F8F120180**

No Sample Data Qualified in this SDG

LDC #: 19188A2  
 SDG #: F8F120180  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 8/5/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: \_\_\_\_\_

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/11/08
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	SW	
IV.	Continuing calibration/ICV	SW	ICV = 2570
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	A	TSB-GJ-08-10'
VIII.	Laboratory control samples	SW	LOS
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	SW	
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)	N	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  
 N = Not provided/applicable  
 SW = See worksheet  
 ND = No compounds detected  
 R = Rinsate  
 FB = Field blank  
 D = Duplicate  
 TB = Trip blank  
 EB = Equipment blank

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

1	TSB-GJ-09-10'	5	11	8168439MB	21		31
2	TSB-GJ-09-20**	↓	12		22		32
3	TSB-GJ-09-30'		13		23		33
4	TSB-GJ-09-40'	↓	14		24		34
5			15		25		35
6			16		26		36
7			17		27		37
8			18		28		38
9			19		29		39
10			20		30		40



**VALIDATION FINDINGS CHECKLIST**

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

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. GC/MS instrument performance check</b>				
Were the DFTPP 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>III. 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"/>	
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 checked="" type="checkbox"/>	
Did the initial calibration meet the curve fit acceptance criteria of $\geq 0.990$ ?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were all percent relative standard deviations (%RSD) $\leq 30\%$ and relative response factors (RRF) $> 0.05$ ?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Continuing calibration</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) $\leq 25\%$ and relative response factors (RRF) $\geq 0.05$ ?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>V. 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>VI. Surrogate spikes</b>				
Were all surrogate %R within QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
If 2 or more base neutral or acid surrogates were 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>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.	<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>VIII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

LDC #: 19188A2  
 SDG #: see cover

**VALIDATION FINDINGS CHECKLIST**

Page: 2 of 2  
 Reviewer: 9  
 2nd Reviewer: \_\_\_\_\_

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"/>	
<b>IX. Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input 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 checked="" type="checkbox"/>	<input checked="" 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"/>	
<b>XI. Target 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. Compound quantitation/CRQLs</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. Tentatively identified compounds (TICs)</b>				
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were relative intensities of the major ions within ± 20% between the sample and the reference spectra?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" 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 type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>XIV. System performance</b>				
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XV. 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>XVI. 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>XVII. Field blanks</b>				
Field blanks were identified in this SDG.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" 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 8270)

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**	<i>Acetophenone</i>
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	UUU.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	<i>1,2,4,5-Tetra(nobenzene)</i>

xxx. *N-(hydroxymethyl)phthalimide*.  
 AAAA. *1-chlorophenyl sulfone*  
 COMPNDL2S

yyy. *Phenyl sulfide*.  
 zzz. *Phenyl disulfide*

www. *Benzenethiol*  
*4-chlorobenzenethiol*

**VALIDATION FINDINGS WORKSHEET**

Initial Calibration

LDC #: 19188A  
 SDG #: See copy  
 METHOD: GC/MS BNA (EPA SW 846 Method 8270)  
 Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".  
 Y  N  N/A Did the laboratory conduct an acceptable 5 point calibration prior to sample analysis?  
 Y  N  N/A Were percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCC's and SPCC's?  
 Y  N  N/A Was a curve fit used for evaluation? If yes, what was the acceptance criteria used for evaluation? \_\_\_\_\_  
 Y  N  N/A Did the initial calibration meet the acceptance criteria?  
 Y  N  N/A Were all %RSDs and RRFs within the validation criteria of ≤30 %RSD and ≥ 0.05 RRF ?

#	Date	Standard ID	Compound	Finding %RSD (Limit: ≤30.0%)	Finding RRF (Limit: ≥0.05)	Associated Samples	Qualifications
	6/13/08	16A	Phthalic acid		0.01422	M1 + Bk S	N/A
			XXX		0.04408		A

LDC #: 19185A2

SDG #: ~~222~~ CDUN

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

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

Was a continuing calibration standard analyzed at least once every 12 hours of sample analysis for each instrument?

N  N/A

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

Y  N  N/A

Were all %D and RRFs within the validation criteria of ≤25 %D and ≥0.05 RRF ?

VALIDATION FINDINGS WORKSHEET

Continuing Calibration

#	Date	Standard ID	Compound	Finding %D (Limit: ≤25.0%)	Finding RRF (Limit: ≥0.05)	Associated Samples	Qualifications
	6/18/08	NCA-5197	Phthalic acid		0.01330	BAK	<del>Y</del> <del>N</del> <del>N/A</del>
			XXX		0.04331		
	6/19/08	NCA-5229	Phthalic acid	25.0818	0.01066	M+NB	<del>Y</del> <del>N</del> <del>N/A</del>
			XXX		0.04523		<del>Y</del> <del>N</del> <del>N/A</del>

**VALIDATION FINDINGS WORKSHEET**  
**Laboratory Control Samples (LCS)**

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

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

#	Date	LCS/LCSD ID	Compound	LCS %R (Limits)	LCS D %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		8168A-392es	HH	1.9 (5-150)	( ) ( )	( ) ( )	MTBF	<del>None</del> No Qual (MS/MSD in)

LDC #: 19188A  
SDG #: 2000000

### VALIDATION FINDINGS WORKSHEET Internal Standards

Page: 6 of 1  
Reviewer: [Signature]  
2nd Reviewer:

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

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

Were all internal standard area counts within -50 to +100 of the associated calibration standard?

Were the retention times of the internal standards within +/- 30 seconds of the retention times of the associated calibration standard?

#	Date	Sample ID	Internal Standard	Area (Limits)	RT (Limits)	Qualifications
		1	PRY	198321 (281395 - 1125580)		N/A
		2	PRY	191974 ( )		
		3	PRY	206248 ( )		
		4	PRY	212988 ( )		(FFF - 442)

\* QC limits are advisory  
IS1 (DCB) = 1,4-Dichlorobenzene-d4  
IS2 (NPT) = Naphthalene-d8  
IS3 (ANT) = Acenaphthene-d10

IS4 (PHN) = Phenanthrene-d10  
IS5 (CRY) = Chrysene-d12  
IS6 (PRY) = Perylene-d12

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

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

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

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				RRF (S std)	(S std)	RRF (S std)	(S std)	Average RRF (Initial)	%RSD	Average RRF (Initial)	%RSD
1	ICAZ	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)	1.41178	1.41178	1.41229	1.41229	0.573	0.573	0.573	0.573
			Pentachlorophenol (4th internal standard)	0.20260	0.20260	0.19634	0.19634	10.265	10.265	10.265	10.265
			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	ICAR	6/18/08	Phenol (1st internal standard)	0.57976	0.57976	0.51274	0.51274	0.71511	0.71511	0.71511	0.71511
			Naphthalene (2nd internal standard)	1.20177	1.20177	1.18223	1.18223	0.93662	0.93662	0.93662	0.93662
			Fluorene (3rd internal standard)								
			Pentachlorophenol (4th internal standard)								
			Bis(2-ethylhexyl)phthalate (5th internal standard)								
			Benzo(a)pyrene (6th internal standard)								
3	ICAZ	6/18/08	Phenol (1st internal standard)	1.60116	1.60116	1.57590	1.57590	2.64232	2.64232	2.64232	2.64232
			Naphthalene (2nd internal standard)	0.33910	0.33910	0.33002	0.33002	4.49570	4.49570	4.49570	4.49570
			Fluorene (3rd internal standard)	1.03639	1.03639	1.02385	1.02385	1.22192	1.22192	1.22192	1.22192
			Pentachlorophenol (4th internal standard)	0.38651	0.38651	0.36637	0.36637	8.75397	8.75397	8.75397	8.75397
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.39110	0.39110	0.39265	0.39265	2.58826	2.58826	2.58826	2.58826
			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.



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

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

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	JCA527	6/19/08	Phenol (1st internal standard)	1.85537	1.80162	2.89731	1.80162	2.89731
			Naphthalene (2nd internal standard)	1.10901	1.08712	1.97399	1.08712	1.97399
			Fluorene (3rd internal standard)	1.41229	1.40878	0.24855	1.40878	0.24855
			Pentachlorophenol (4th internal standard)	0.19634	0.20730	5.58529	0.20730	5.58529
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.86343	0.87788	0.97842	0.87788	0.97842
			Benzo(a)pyrene (6th internal standard)	1.11182	1.12694	1.36062	1.12694	1.36062
2	JCA528	6/19/08	Phenol (1st internal standard)	0.57574	0.51326	0.10062	0.51326	0.10062
			Naphthalene (2nd internal standard)	1.18223	1.20702	2.09666	1.20702	2.09666
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
3	JCA529	6/19/08	Phenol (1st internal standard)	1.57590	1.59157	0.99462	1.59157	0.99462
			Naphthalene (2nd internal standard)	0.33002	0.33954	2.88719	0.33954	2.88719
			Fluorene (3rd internal standard)	1.02385	1.01788	0.58319	1.01788	0.58319
			Pentachlorophenol (4th internal standard)	0.36637	0.38045	3.84416	0.38045	3.84416
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.39265	0.39958	1.76341	0.39958	1.76341
			Benzo(a)pyrene (6th 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.

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

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

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	1CA2595	6/18/08	Phenol (1st internal standard)	1.85537	1.87174	0.88210	0.8822	
			Naphthalene (2nd internal standard)	1.10901	1.10135	0.69070	0.6909	
			Fluorene (3rd internal standard)	1.41229	1.39801	1.01058	1.0108	
			Pentachlorophenol (4th internal standard)	0.19634	0.20370	3.74980	3.7476	
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.86343	0.87088	0.86222	0.8628	
			Benzo(a)pyrene (6th internal standard)	1.1182	1.11507	0.29280	0.2924	
2	1CA2596	6/18/08	Phenol (1st internal standard)	0.51274	0.52185	1.77632	1.7767	
			Naphthalene (2nd internal standard)	1.18223	1.17316	0.76746	0.7673	
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
3	1CA2597	6/18/08	Phenol (1st internal standard)	1.67590	1.60400	1.78300	1.7832	
			Naphthalene (2nd internal standard)	0.33002	0.33744	2.25045	2.2490	
			Fluorene (3rd internal standard)	1.02385	1.03316	0.9094	0.9099	
			Pentachlorophenol (4th internal standard)	0.26637	0.38274	4.46802	4.4672	
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.39265	0.39671	1.03290	1.0333	
			Benzo(a)pyrene (6th 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 #: 19188A2  
 SDG #: See CONW

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

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

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

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: \_\_\_\_\_

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5		33.9134	65		
2-Fluorobiphenyl		35.9420	68		
Terphenyl-d14		39.8482	78		
Phenol-d5		52.0537	66		
2-Fluorophenol		50.9295	65		
2,4,6-Tribromophenol		52.8840	69		
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID: 2

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	50	32.5367	65	65	0
2-Fluorobiphenyl	↓	33.9843	68	68	↓
Terphenyl-d14	↓	38.7625	78	78	↓
Phenol-d5	75	49.6403	66	66	↓
2-Fluorophenol	↓	49.0421	65	65	↓
2,4,6-Tribromophenol	↓	52.0744	69	69	↓
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					





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

**Project/Site Name:** BRC Tronox, Parcel G  
**Collection Date:** June 11, 2008  
**LDC Report Date:** August 6, 2008  
**Matrix:** Soil  
**Parameters:** Chlorinated Pesticides  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8F120180

**Sample Identification**

TSB-GJ-09-10'  
TSB-GJ-09-20'\*\*  
TSB-GJ-09-30'  
TSB-GJ-09-40'

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 4 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.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. GC/ECD Instrument Performance Check

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

## III. Initial Calibration

Initial calibration of single compounds were 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 selected compounds.

A curve fit, based on the initial calibration, was established for quantitation for selected compounds. The coefficient of determination ( $r^2$ ) was greater than or equal to 0.990 .

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	KCAL092	A	Toxaphene	15.2	TSB-GJ-09-30' TSB-GJ-09-40'	J+ (all detects)	A
6/18/08	KCAL095	A	2,4'-DDD	22.6	TSB-GJ-09-30' TSB-GJ-09-40'	J+ (all detects)	P

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.

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

## **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 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 G  
Chlorinated Pesticides - Data Qualification Summary - SDG F8F120180**

<b>SDG</b>	<b>Sample</b>	<b>Compound</b>	<b>Flag</b>	<b>A or P</b>	<b>Reason</b>
F8F120180	TSB-GJ-09-30' TSB-GJ-09-40'	Toxaphene	J+ (all detects)	A	Continuing calibration (%D)
F8F120180	TSB-GJ-09-30' TSB-GJ-09-40'	2,4'-DDD	J+ (all detects)	P	Continuing calibration (%D)

**BRC Tronox, Parcel G  
Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG  
F8F120180**

No Sample Data Qualified in this SDG

**BRC Tronox, Parcel G  
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG  
F8F120180**

No Sample Data Qualified in this SDG

LDC #: 19188A3a  
 SDG #: F8F120180  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**

Level III/IV

Date: 8/11/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: \_\_\_\_\_

**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	A	Sampling dates: 6/11/08
II.	GC/ECD Instrument Performance Check	A	
III.	Initial calibration	A	RSD. 12
IV.	Continuing calibration/ICV	W	ICV = 1570
V.	Blanks	A	
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	A	Not reviewed for Level III validation.
XII.	Compound quantitation and reported CRQLs	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

1	TSB-GJ-09-10'	↙	11	8168164 MB	21		31
2	TSB-GJ-09-20**	↓	12		22		32
3	TSB-GJ-09-30'		13		23		33
4	TSB-GJ-09-40'	↓	14		24		34
5			15		25		35
6			16		26		36
7			17		27		37
8			18		28		38
9			19		29		39
10			20		30		40

LDC #: 19188A39  
 SDG #: See Cont

**VALIDATION FINDINGS CHECKLIST**

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

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? <u>✓</u> %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%.0 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"/>	

LDC #: 19188A39  
 SDG #: 5000W1

VALIDATION FINDINGS CHECKLIST

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

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"/>	
<b>IX. Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Were the performance evaluation (PE) samples within the acceptance limits?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>X. Target compound identification</b>				
Were the retention times of reported detects within the RT windows?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<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?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XII. System performance</b>				
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XIII. 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>XIV. Field duplicates</b>				
Were field duplicate pairs identified in this SDG?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Were target compounds detected in the field duplicates?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>XV. Field blanks</b>				
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:** 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 #: 19188A39  
 SDG #: 2000W

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

Page: 1 of 1  
 Reviewer: 9  
 2nd Reviewer: \_\_\_\_\_

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 (0.05 std)	CF (0.05 std)	Average CF (Initial)	Average CF (Initial)	%RSD	%RSD		
1	1CA	6/12/08	2.4'-DDE (ch A) ↓ F (ch A)	37366840 21121700 655423200	37366844 21121700 655423200	373785260 203802798 681332132	373785260 203802798 681332132	1.30108 2.78044 2.76188	1.3011 2.7804 2.7619		
2	1CA	6/16/08	0 (ch A) ↓ F (ch B) 0	282338600 363591000 120815600	282338600 363591000 120815600	301269612 380182886 133202078	301269612 380182886 133202078	4.112425 2.83696 8.79750	1.1124 2.8370 8.7975		
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 #: 19188A39  
 SDG #: 2000000

**VALIDATION FINDINGS WORKSHEET**  
Continuing Calibration Results Verification

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

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 (cal)/ CCV Conc.	Reported		Recalculated	
					CF/Conc. CCV	%D	CF/Conc. CCV	%D
1	<del>KA1064</del>	6/18/08	F (cal A) ↓ 2,4-DDE (cal A)	0.025 ↓ 0.025		0.0257 0.0251 0.0258	2.6 0.5 3.2	2.6 0.52 3.2
2	<del>KA1080</del>	6/18/08	F (cal A) ↓ 2,4-DDE (cal A)	0.025 ↓ 0.025		0.0255 0.0257 0.0255	1.8 8.7 2.2	1.8 2.7 2.2
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 #: 19188A39  
 SDG #: 22222222

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

Page: 1 of 1  
 Reviewer: 9  
 2nd reviewer: \_\_\_\_\_

METHOD: ✓ GC     HPLC

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

% Recovery: SF/SS \* 100  
 Where: SF = Surrogate Found  
 SS = Surrogate Spiked

Sample ID: 2

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery		Percent Difference
				Reported	Recalculated	
TCM X	CA A	0.020	0.01860	93	93	0
DCB3	✓	✓	0.01958	98	98	0

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

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

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

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

LDC #: 1928239  
 SDG #: See below

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

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

METHOD:  GC  HPLC

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 SC = Concentration  
 SA = Spike added  
 RPD =  $100 * |SSCLCS - SSCLCSD| / (SSCLCS + SSCLCSD)$  LCS = Laboratory control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples: 8168164

Compound	Spike Added		Spiked Sample Concentration		LCS		LCSD		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	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)												
F	16.7	NA	15.0	NA	90	90						
D	↓	↓	16.8	↓	101	101						

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 G  
**Collection Date:** June 11, 2008  
**LDC Report Date:** August 6, 2008  
**Matrix:** Soil  
**Parameters:** Polychlorinated Biphenyls  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** F8F120180

**Sample Identification**

TSB-GJ-09-10'  
TSB-GJ-09-20'\*\*  
TSB-GJ-09-30'  
TSB-GJ-09-40'

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 4 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 G  
Polychlorinated Biphenyls - Data Qualification Summary - SDG F8F120180**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel G  
Polychlorinated Biphenyls - Laboratory Blank Data Qualification Summary - SDG  
F8F120180**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel G  
Polychlorinated Biphenyls - Field Blank Data Qualification Summary - SDG  
F8F120180**

No Sample Data Qualified in this SDG

LDC #: 19188A3b  
 SDG #: F8F120180  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 8/4/08  
 Page: bf/1  
 Reviewer: [Signature]  
 2nd Reviewer: \_\_\_\_\_

**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	A	Sampling dates: 6/11/08
II.	GC/ECD Instrument Performance Check	N	
III.	Initial calibration	A	
IV.	Continuing calibration/ICV	A	ICV = 1570
V.	Blanks	A	
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	A	Not reviewed for Level III validation.
XII.	Compound quantitation and reported CRQLs	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

1	TSB-GJ-09-10'	5	11	3168762 MB	21		31
2	TSB-GJ-09-20**		12		22		32
3	TSB-GJ-09-30'		13		23		33
4	TSB-GJ-09-40'		14		24		34
5			15		25		35
6			16		26		36
7			17		27		37
8			18		28		38
9			19		29		39
10			20		30		40

LDC #: 19188A3b  
 SDG #: 2200W

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2  
 Reviewer: 9  
 2nd Reviewer: \_\_\_\_\_

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 type="checkbox"/>	<input checked="" 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? <u>  </u> %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%.0 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 type="checkbox"/>	<input checked="" 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"/>	

LDC #: 19188A3b  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

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

Validation Area	Yes	No	NA	Findings/Comments
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 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>				
Were field duplicate pairs identified in this SDG?		/		
Were target compounds detected in the field duplicates?			/	
<b>XV. Field blanks</b>				
Were field blanks identified in this SDG?		/		
Were target compounds 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 #: 181033A36  
 SDG #: See below

**VALIDATION FINDINGS WORKSHEET**  
Initial Calibration Calculation Verification

Page: 1 of 1  
 Reviewer: 9  
 2nd Reviewer: \_\_\_\_\_

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 (50 std)	CF (50 std)	Average CF (initial)	Average CF (initial)	%RSD	%RSD		
1	10A ✓	5/31/08	BB (RX-24Post)	33154	33154	27977	27977	12.0	12.0		
			BB ( ↓ II )	45676	45676	39164	39164	9.582	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 #: 1985Aab  
 SDG #: See down

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

Page: 1 of 1  
 Reviewer: 9  
 2nd Reviewer: \_\_\_\_\_

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	PKA089	6/18/08	BB (RTX - cufest)	1000	952.1902	4.8	952.2	4.8
			<del>BB (RTX - cufest)</del>	<del>1000</del>				
2	PKA100	6/18/08	BB (RTX - cufest)	1000	937.3342	6.3	937.3	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.

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

LDC #: 1988A36  
 SDG #: 52000N  
 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		Percent Difference
				Reported	Recalculated	
<u>DCP</u>	<u>ChA</u>	<u>20</u>	<u>20.4758</u>	<u>102</u>	<u>102</u>	<u>0</u>

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

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

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

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

METHOD: GC\_HPLC

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 SC = Concentration  
 SA = Spike added

RPD =  $100 * |SSCLCS - SSCLCSD| / ((SSCLCS + SSCLCSD) / 2)$  LCS = Laboratory control sample percent recovery  
 LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples: 8168762

Compound	Spike Added		Spiked Sample Concentration		LCS		LCSD		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	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)												
<u>BB</u>	<u>167</u>	<u>NA</u>	<u>171</u>	<u>NA</u>	<u>103</u>	<u>102</u>						

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 G  
**Collection Date:** June 11, 2008  
**LDC Report Date:** August 8, 2008  
**Matrix:** Soil  
**Parameters:** Metals  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** F8F120180

**Sample Identification**

TSB-GJ-09-10'  
TSB-GJ-09-20'\*\*  
TSB-GJ-09-30'  
TSB-GJ-09-40'

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 4 water 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
ICB/CCB	Antimony Thallium Tungsten Vanadium Lithium Mercury	1.3 ug/L 1.1 ug/L 1.4 ug/L 2.7 ug/L 8.0 ug/L 0.1 ug/Kg	All samples in SDG F8F120180

Sample concentrations were compared to concentrations detected in the method blanks as required by the QAPP. No sample data was qualified with the following exceptions:

Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-GJ-09-10'	Lithium	6.7 mg/Kg	26.6U mg/Kg
TSB-GJ-09-40'	Lithium Mercury	111 mg/Kg 22.0 ug/Kg	157U mg/Kg 52.4U ug/Kg

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-GJ-08-10'MS/MSD (All samples in SDG F8F120180)	Sulfur Phosphorus	140.1 (75-125) 134.8 (75-125)	135.4 (75-125) -	- -	J+ (all detects) J+ (all detects)	A
TSB-GJ-08-10'MS/MSD (All samples in SDG F8F120180)	Antimony Copper Silicon Vanadium Lithium Nickel Tungsten Zinc	55.2 (75-125) 72.5 (75-125) 65.4 (75-125) 68.4 (75-125) - - - -	39.4 (75-125) 60.9 (75-125) 44.6 (75-125) 56.0 (75-125) 69.8 (75-125) 71.1 (75-125) 60.6 (75-125) 62.2 (75-125)	- - - - - - - -	J- (all detects) UJ (all non-detects)	A
TSB-GJ-08-10'MS/MSD (All samples in SDG F8F120180)	Niobium	-	29.7 (75-125)	-	J- (all detects) R (all non-detects)	A

## VI. Duplicate Sample Analysis

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

## VII. Laboratory Control Samples (LCS)

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

## VIII. Internal Standards (ICP-MS)

All internal standard percent recoveries (%R) were within QC limits for samples on which a Level IV review was performed. 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-GJ-08-10'L	Iron	10.4 (≤10)	All samples in SDG F8F120180	J (all detects)	A

### **XI. Sample Result Verification**

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

### **XII. Overall Assessment of Data**

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

### **XIII. Field Duplicates**

No field duplicates were identified in this SDG.

**BRC Tronox Parcel G  
Metals - Data Qualification Summary - SDG F8F120180**

SDG	Sample	Analyte	Flag	A or P	Reason
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	Sulfur Phosphorus	J+ (all detects) J+ (all detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	Antimony Copper Silicon Vanadium Lithium Nickel Tungsten Zinc	J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	Niobium	J- (all detects) R (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	Iron	J (all detects)	A	ICP serial dilution (%D)

**BRC Tronox Parcel G  
Metals - Laboratory Blank Data Qualification Summary - SDG F8F120180**

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8F120180	TSB-GJ-09-10'	Lithium	26.6U mg/Kg	A
F8F120180	TSB-GJ-09-40'	Lithium Mercury	157U mg/Kg 52.4U ug/Kg	A

**BRC Tronox Parcel G  
Metals - Field Blank Data Qualification Summary - SDG F8F120180**

No Sample Data Qualified in this SDG

LDC #: 19188A4  
 SDG #: F8F120180  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 8/4/08  
 Page: 1 of 1  
 Reviewer: my  
 2nd Reviewer: Q

**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: 6/11/08
II.	Calibration	A	
III.	Blanks	SW	
IV.	ICP Interference Check Sample (ICS) Analysis	A	
V.	Matrix Spike Analysis	SW	3MS/MSD
VI.	Duplicate Sample Analysis	N	
VII.	Laboratory Control Samples (LCS)	A	LCS
VIII.	Internal Standard (ICP-MS)	A	not reviewed for level 3
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: \*\* Indicates sample underwent Level IV validation

1	TSB-GJ-09-10'	11		21		31	
2	TSB-GJ-09-20'	12		22		32	
3	TSB-GJ-09-30'	13		23		33	
4	TSB-GJ-09-40'	14		24		34	
5	PB	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 #: 19188A4  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

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

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 #: 19188/14  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: WJ  
 2nd Reviewer: C

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?	/			
Were all percent differences (%Ds) < 10%?		/		
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.		/		
<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?	/			
If the %Rs were outside the criteria, was a reanalysis performed?			/	
<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. Sample Result Verification</b>				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
<b>XI. Overall assessment of data</b>				
Overall assessment of data was found to be acceptable.	/			
<b>XII. Field duplicates</b>				
Field duplicate pairs were identified in this SDG.		/	/	
Target analytes were detected in the field duplicates.			/	
<b>XIII. Field blanks</b>				
Field blanks were identified in this SDG.		/	/	
Target analytes were detected in the field blanks.			/	









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?  
 D N/A Were ICP serial dilution percent differences (%D) ≤10%?  
 N 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-GJ-0610	Soil	Fe	10.4	A4	JLT/A

Comments: Max. Y < 100X [Signature]

LDC #: 19188AY  
 SDG #: See cover

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

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

**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	
<del>ICV</del>	ICP (Initial calibration)	S	42700	40000	106.8	106.8	106.8		Y
	GFAA (Initial calibration)								
ICV	CVAA (Initial calibration)	Hg	2.33	2.50	93.2	93.2	93.2		Y
CCV	ICP (Continuing calibration)	Li	4754	5000	95.1	95.1	95.1		Y
	GFAA (Continuing calibration)								
CCV	CVAA (Continuing calibration)	Hg	4.98	5.0	99.6	99.6	99.6		Y
ICV	ICP/MS (Initial calibration)	U	1028.4	1000	102.8	102.8	102.8		Y
CCV	ICP/MS (Continuing calibration)	As	908.3	1000	90.8	90.8	90.8		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 #: 19188A4  
 SDG #: 5-11-0000

Page: 1 of 1  
 Reviewer: AW  
 2nd Reviewer: Q

**VALIDATION FINDINGS WORKSHEET**  
Level IV Recalculation Worksheet

**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		Reported		Acceptable (Y/N)
					%R / RPD / %D	%R / RPD / %D			
TSAB	ICP interference check	Zn	104.0	100	104	104	104		Y
LC3	Laboratory control sample	Sb	96.8	127	96.2	96.2	96.2		
TSB-GJ-08-10	Matrix spike	B (SSR-SR)	164.2	107.09	91.3	91.3	91.3		
J	Duplicate	Cu	20.05	20.26	1.0	1.0	1.0		Y
	ICP serial dilution	Pb	16112	16568	2.8	2.8	2.8		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 #: 1918874  
 SDG #: See cover

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

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

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

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

- 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:

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

Recalculation:

$$S = \frac{8486 \text{ mg/L} \times 0.05 \text{ L} \times 5 \times 1000 \text{ g/kg}}{0.5 \text{ g} \times 0.9961} = 53299 \text{ mg/kg}$$

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

Sample ID	Analyte	Reported Concentration (mg/kg)	Calculated Concentration (mg/kg)	Acceptable (Y/N)
2	S	53300	53300	Y
	Al	10100	10100	
	As	27.6	27.6	
	Ba	64.6	64.6	
	Be	0.64	0.64	
	Ca	75800	75800	
	Cr	22.2	22.2	
	Co	5.7	5.6	
	Cu	13.5	13.5	
	Fe	13200	13200	
	Pb	7.1	7.1	
	Mg	18200	18200	
	Mn	170	170	
	Ni	14.7	14.6	
	Pd	1.1	1.1	
	P	528	528	
	K	2710	2710	
	Si	549	549	
	Ag	0.14	0.14	
	Na	944	943	
	Sr	505	505	
	Ti	558	557	✓

LDC #: 191887A  
 SDG #: Sel case

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

Page: 2 of 2  
 Reviewer: mu  
 2nd reviewer: v

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:

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

Recalculation:

$$V = \frac{45.91 \mu g / L \times 0.1 L \times 5}{0.5 g \times 0.9961} = 57.67 \text{ mg/kg}$$

Sample ID	Analyte	Reported Concentration (mg/kg)	Calculated Concentration (mg/kg)	Acceptable (Y/N)
2	U	3.7	3.7	Y
	V	57.7	57.7	Y
	Zn	91.2	91.2	Y
	Zr	31.7	31.6	Y

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

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

**Collection Date:** June 11, 2008

**LDC Report Date:** August 7, 2008

**Matrix:** Soil

**Parameters:** Wet Chemistry

**Validation Level:** EPA Level III & IV

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8F120180

**Sample Identification**

TSB-GJ-09-10'  
TSB-GJ-09-20'\*\*  
TSB-GJ-09-30'  
TSB-GJ-09-40'

\*\*Indicates sample underwent EPA Level IV review



## **Introduction**

This data review covers 4 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
ICB/CCB	Orthophosphate as P	0.102 mg/L	All samples in SDG F8F120180

Sample concentrations were compared to concentrations detected in the method blanks as required by the QAPP. No sample data was qualified with the following exceptions:

Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-GJ-09-20**	Orthophosphate as P	1.5 mg/Kg	6.3U mg/Kg

No field blanks were identified in this SDG.

## IV. 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 with the following exceptions:

<b>Spike ID (Associated Samples)</b>	<b>Analyte</b>	<b>MS (%R) (Limits)</b>	<b>MSD (%R) (Limits)</b>	<b>RPD (Limits)</b>	<b>Flag</b>	<b>A or P</b>
TSB-CJ-09-0'MS/MSD (All samples in SDG F8F120180)	Oil and grease	63 (75-125)	63 (75-125)	-	J- (all detects) UJ (all non-detects)	A

## **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 G  
Wet Chemistry - Data Qualification Summary - SDG F8F120180**

SDG	Sample	Analyte	Flag	A or P	Reason
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'*** TSB-GJ-09-30' TSB-GJ-09-40'	Oil and grease	J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)

**BRC Tronox Parcel G  
Wet Chemistry - Laboratory Blank Data Qualification Summary - SDG F8F120180**

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8F120180	TSB-GJ-09-20'**	Orthophosphate as P	6.3U mg/Kg	A

**BRC Tronox Parcel G  
Wet Chemistry - Field Blank Data Qualification Summary - SDG F8F120180**

No Sample Data Qualified in this SDG

LDC #: 19188A6  
 SDG #: F8F120180  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 8/4/08  
 Page: 1 of 1  
 Reviewer: MJ  
 2nd Reviewer: Q

**METHOD: (Analyte) Bromide, Bromine, Chlorate, Chloride, Chlorine, Fluoride, Nitrate-N, Nitrite-N, 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: 6/11/08
IIa.	Initial calibration	A	
IIb.	Calibration verification	A	
III.	Blanks	SW	
IV	Matrix Spike/Matrix Spike Duplicates	SW	3 MS/MSD / Dup
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      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-GJ-09-10'	11		21		31	
2	TSB-GJ-09-20**	12		22		32	
3	TSB-GJ-09-30'	13		23		33	
4	TSB-GJ-09-40'	14		24		34	
5	PB	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 #: 19188A6  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

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

Method: Inorganics (EPA Method 300.0) *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. Method 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 Duplicate, and Duplicate</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 #: 19188A6  
 SDG #: fuel com

VALIDATION FINDINGS CHECKLIST

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

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>Overall assessment of data was found to be acceptable.</b>				
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.			/	









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

Method: Inorganics, Method See cover

The correlation coefficient (r) for the calibration of Br 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	Br	s1	250	0.02	0.99997	0.99997			Y
		s2	500	0.039					
		s3	1000	0.076					
		s4	2500	0.196					
		s5	5000	0.396					
CCV Calibration verification	ClO <sub>2</sub>	4000	3904		98	NR		Y	
CCV Calibration verification	F	1000	949.5		94.95	94.95		↓	
CCV Calibration verification	o-py-p	8000	7856		98.20	98.20		↓	

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 #: 19188A6

SDG #: See curve

VALIDATION FINDINGS WORKSHEET  
Level IV Recalculation Worksheet

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

METHOD: Inorganics, Method See Curve

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		Acceptable (Y/N)
					%R / RPD	%R / RPD	
LCS	Laboratory control sample	$NO_2-N$	1.59	1.60	98	98	Y
TSB-CJ-09-01	Matrix spike sample	OTG	880 (SSR-SR)	1390	63	63	Y
↓	Duplicate sample	SO4	1500	1480	1.3	1.3	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 #: 19188/16  
 SDG #: see cover

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

Page: 1 of 1  
 Reviewer: MM  
 2nd reviewer: CC

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 =  $\frac{Area \times 40ml}{0.099 \times 48 \times 10^{-5} \times 1.1}$       Recalculation:  $\frac{0.033 \times 40}{0.099 \times 48 \times 0.996} = 3.66 \text{ mg/ug}$

#	Sample ID	Analyte	Reported Concentration (ug/ug)	Calculated Concentration (mg/kg)	Acceptable (Y/N)
1	2	O-P04 - P	1.5	1.5	Y
		Chloride	3.7	3.7	
		Cl	244	244	
		Cl <sub>2</sub>	488	488	
		F	0.58	0.59	
		NO <sub>3</sub> -N	5.3	5.3	
		SO <sub>4</sub>	11600	11600	

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

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

**Project/Site Name:** BRC Tronox Parcel G  
**Collection Date:** June 11, 2008  
**LDC Report Date:** August 6, 2008  
**Matrix:** Soil  
**Parameters:** Gasoline Range Organics  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** F8F120180

**Sample Identification**

TSB-GJ-09-10'  
TSB-GJ-09-20'\*\*  
TSB-GJ-09-30'  
TSB-GJ-09-40'

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 4 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. Percent recoveries (%R) and relative percent differences (RPD) 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 G  
Gasoline Range Organics - Data Qualification Summary - SDG F8F120180**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel G  
Gasoline Range Organics - Laboratory Blank Data Qualification Summary - SDG  
F8F120180**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel G  
Gasoline Range Organics - Field Blank Data Qualification Summary - SDG  
F8F120180**

No Sample Data Qualified in this SDG

LDC #: 19188A7  
 SDG #: F8F120180  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**

Level III/IV

Date: 8/4/08  
 Page: 6 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: \_\_\_\_\_

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/11/08
IIa.	Initial calibration	A	
IIb.	Calibration verification/ICV	A	ICV = 1570
III.	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	NA	field spiked - TSB-GJ-08-10'
IVc.	Laboratory control samples	A	LCS/D
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

1	TSB-GJ-09-10'	5	11	8165269MB	21		31	
2	TSB-GJ-09-20**		12		22		32	
3	TSB-GJ-09-30'		13		23		33	
4	TSB-GJ-09-40'		14		24		34	
5			15		25		35	
6			16		26		36	
7			17		27		37	
8			18		28		38	
9			19		29		39	
10			20		30		40	

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

LDC #: 19128A7  
 SDG #: 2a epuv

VALIDATION FINDINGS CHECKLIST

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

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 type="checkbox"/>	<input checked="" 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>IV. Continuing calibration</b>				
What type of continuing calibration calculation was performed? <u>    </u> %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%.0 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>V. 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 type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>VI. 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>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.	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Was a MS/MSD analyzed every 20 samples of each matrix?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>VIII. 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"/>	

LDC #: 19188AT  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

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

Validation Area	Yes	No	NA	Findings/Comments
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 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>				
Were field duplicate pairs identified in this SDG?		/		
Were target compounds idetected in the field duplicates?			/	
<b>XV. Field blanks</b>				
Were field blanks identified in this SDG?		/		
Were target compounds detected in the field blanks?			/	

LDC #: 191887  
 SDG #: See COM

**VALIDATION FINDINGS WORKSHEET**  
Initial Calibration Calculation Verification

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

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 (0.1/ std)	CF (0.1/ std)	Average CF (initial)	Average CF (initial)	%RSD	%RSD		
1	10A2	5/27/08	GRD	18352700	18352700	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.

LDC #: 19188A7  
 SDG #: 281 CONN

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

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

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 =  $100 \times (\text{ave. CF} - \text{CF}) / \text{ave. CF}$  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 (cal)/ CCV Conc.	Reported		Recalculated	
					CF/Conc. CCV	%D	CF/Conc. CCV	%D
1	<u>1CAZ398B</u>	<u>6/13/08</u>	<u>GRO</u>	<u>1.0</u>	<u>0.9982</u>	<u>0.2</u>	<u>0.9982</u>	<u>0.2</u>
2	<u>1CAZ406B</u>	<u>6/14/08</u>	<u>GRO</u>	<u>1.0</u>	<u>0.9824</u>	<u>1.8</u>	<u>0.9824</u>	<u>1.8</u>
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 #: 1908A7  
SDG #: see count

# VALIDATION FINDINGS WORKSHEET

## Surrogate Results Verification

Page: 9 of 7  
Reviewer: \_\_\_\_\_  
2nd reviewer: \_\_\_\_\_

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		Percent Difference
				Reported	Recalculated	
<u>IFT</u>	<u>NS</u>	<u>0.04</u>	<u>0.03383</u>	<u>85</u>	<u>85</u>	<u>0</u>

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

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

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

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

LDC #: 19188A  
 SDG #: Sec 001

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

Page: 1 of 7  
 Reviewer: [Signature]  
 2nd Reviewer: \_\_\_\_\_

METHOD: GC HPLC

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 =  $100 \times (SSC-SC)/SA$  Where: SSC = Spiked sample concentration SC = Concentration  
 SA = Spike added  
 RPD =  $100 \times |SSCLCS - SSCLCSD| / (SSCLCS + SSCLCSD)$  LCS = Laboratory control sample percent recovery  
 LCS/LCSD samples: 8165269 LCS/LCSD = Laboratory control sample duplicate percent recovery

Compound	Spike Added		Spiked Sample Concentration		LCS		LCS/LCSD		LCS		LCS/LCSD		LCS/LCSD	
	LCS	LCS/LCSD	LCS	LCS/LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)	1.0	1.0	1.0	0.944	100	100	94	94	6.2	5.8				
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 G  
**Collection Date:** June 11, 2008  
**LDC Report Date:** August 6, 2008  
**Matrix:** Soil  
**Parameters:** Diesel Range Organics  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** F8F120180

**Sample Identification**

TSB-GJ-09-10'  
TSB-GJ-09-20'\*\*  
TSB-GJ-09-30'  
TSB-GJ-09-40'

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 4 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.
- 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**

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 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 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 G  
Diesel Range Organics - Data Qualification Summary - SDG F8F120180**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel G  
Diesel Range Organics - Laboratory Blank Data Qualification Summary - SDG  
F8F120180**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel G  
Diesel Range Organics - Field Blank Data Qualification Summary - SDG  
F8F120180**

No Sample Data Qualified in this SDG



LDC #: 19188A8  
 SDG #: F8F120180  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 8/4/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: \_\_\_\_\_

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/11/08
IIa.	Initial calibration	A	
IIb.	Calibration verification/ICV	A	ICV = 1570
III.	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	KA	Matrix spike performed TSB GJ-08-10
IVc.	Laboratory control samples	A	LCS
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

1	TSB-GJ-09-10'	S	11	8465291 MD	21		31
2	TSB-GJ-09-20**		12	8170312 MB	22		32
3	TSB-GJ-09-30'		13		23		33
4	TSB-GJ-09-40'		14		24		34
5			15		25		35
6			16		26		36
7			17		27		37
8			18		28		38
9			19		29		39
10			20		30		40

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

LDC #: 19188A8  
 SDG #: see cover

**VALIDATION FINDINGS CHECKLIST**

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

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 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>IV. Continuing calibration</b>				
What type of continuing calibration calculation was performed? <u>    </u> %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%.0 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>V. 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>VI. 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>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.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Was a MS/MSD analyzed every 20 samples of each matrix?	<input type="checkbox"/>	<input checked="" 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 type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>VIII. 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"/>	

LDC #: 19188A8  
 SDG #: See column

**VALIDATION FINDINGS CHECKLIST**

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

Validation Area	Yes	No	NA	Findings/Comments
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 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>				
Were field duplicate pairs identified in this SDG?		/		
Were target compounds detected in the field duplicates?			/	
<b>XV: Field blanks</b>				
Were field blanks identified in this SDG?		/		
Were target compounds detected in the field blanks?			/	

LDC #: 191088A8  
 SDG #: 22222222

**VALIDATION FINDINGS WORKSHEET**  
Initial Calibration Calculation Verification

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

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 (100std)	CF (100std)	Average CF (initial)	Average CF (initial)	%RSD	%RSD		
1	10A2	3/16/08	DRO	15394	15394	16023	16023	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 #: 19108AD  
 SDG #: 2000000

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

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

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 = 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	SCAL535	4/17/08	DRO	1000	996.5312	0.3	996.53	0.3
2	SCAL537	6/17/08	DRO	1000	1034.567	3.5	1034.55	3.5
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.

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

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		Percent Difference
				Reported	Recalculated	
TPH	NS	25.0	21.2901	85	85	0

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

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

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

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

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

METHOD: GC HPLC

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 SC = Concentration  
 RPD =  $|SSCLCS - SSCLCSD| * 2 / (SSCLCS + SSCLCSD)$  SA = Spike added LCS = Laboratory control sample percent recovery LCS = Laboratory control sample duplicate percent recovery  
 LCS/LCSD samples: 8165291

Compound	Spike Added (WTS)		Spiked Sample Concentration (WTS)		LCS		LCSD		LCS/LCSD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)										
Diesel (8015)	83.3	NA	68.9	NA	83	83				
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.

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

LDC #: 9108A  
SDG #: see down

Page: 6 of 7  
Reviewer: [Signature]  
2nd Reviewer: \_\_\_\_\_

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 agree within 10% of the reported results?

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

Example:  
Sample ID: 2 Compound Name: ND

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

- 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

#	Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications

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



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

**Project/Site Name:** BRC Tronox Parcel G  
**Collection Date:** June 11, 2008  
**LDC Report Date:** August 8, 2008  
**Matrix:** Soil  
**Parameters:** Polynuclear Aromatic Hydrocarbons  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** F8F120180

**Sample Identification**

TSB-GJ-09-10'  
TSB-GJ-09-20'\*\*  
TSB-GJ-09-30'  
TSB-GJ-09-40'

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 4 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.
- 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

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-GJ-09-10' TSB-GJ-09-20'*	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	16.6	All samples in SDG F8F120180	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 G  
 Polynuclear Aromatic Hydrocarbons - Data Qualification Summary - SDG F8F120180**

SDG	Sample	Compound	Flag	A or P	Reason
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'***	Benzo(g,h,i)perylene	J+ (all detects)	A	Continuing calibration (%D)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'*** TSB-GJ-09-30' TSB-GJ-09-40'	Benzo(k)fluoranthene	J+ (all detects)	A	Continuing calibration (ICV %D)

**BRC Tronox Parcel G  
 Polynuclear Aromatic Hydrocarbons - Laboratory Blank Data Qualification Summary  
 - SDG F8F120180**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel G  
 Polynuclear Aromatic Hydrocarbons - Field Blank Data Qualification Summary -  
 SDG F8F120180**

No Sample Data Qualified in this SDG

LDC #: 19188A9  
 SDG #: F8F120180  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 8/4/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: \_\_\_\_\_

**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: 6/11/08
IIa.	Initial calibration	A	
IIb.	Calibration verification/ICV	W	ICV = 1570
III.	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	A	TSB-GJ-08-10'
IVc.	Laboratory control samples	A	LC 9
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

1	TSB-GJ-09-10'	S	11	8168158MB	21		31	
2	TSB-GJ-09-20**		12		22		32	
3	TSB-GJ-09-30'		13		23		33	
4	TSB-GJ-09-40'		14		24		34	
5			15		25		35	
6			16		26		36	
7			17		27		37	
8			18		28		38	
9			19		29		39	
10			20		30		40	

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



LDC #: 19188A9  
 SDG #: 3a EDW

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2  
 Reviewer: 9  
 2nd Reviewer: \_\_\_\_\_

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"/>			
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>			
<b>II. Initial calibration</b>				
Did the laboratory perform a 5 point calibration prior to sample analysis?	<input checked="" type="checkbox"/>			
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?	<input checked="" type="checkbox"/>			
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?		<input checked="" type="checkbox"/>		
Did the initial calibration meet the curve fit acceptance criteria?	<input checked="" type="checkbox"/>		<input checked="" type="checkbox"/>	
Were the RT windows properly established?	<input checked="" type="checkbox"/>			
<b>IV. Continuing calibration</b>				
What type of continuing calibration calculation was performed? <u>    </u> %D or %R	<input checked="" type="checkbox"/>			
Was a continuing calibration analyzed daily?	<input checked="" type="checkbox"/>			
Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?		<input checked="" type="checkbox"/>		
Were all the retention times within the acceptance windows?	<input checked="" type="checkbox"/>			
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>			
Was a method blank analyzed for each matrix and concentration?	<input checked="" type="checkbox"/>			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		<input checked="" type="checkbox"/>		
<b>VI. Surrogate spikes</b>				
Were all surrogate %R within the QC limits?	<input checked="" type="checkbox"/>			
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?			<input checked="" type="checkbox"/>	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?			<input checked="" type="checkbox"/>	
<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.	<input checked="" type="checkbox"/>			
Was a MS/MSD analyzed every 20 samples of each matrix?	<input checked="" type="checkbox"/>			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input checked="" type="checkbox"/>			
<b>VIII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>			
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>			

LDC #: 19188A9  
 SDG #: 3220000

VALIDATION FINDINGS CHECKLIST

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

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 checked="" type="checkbox"/>	<input 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 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 detected 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-O	X. EPN	EE. Ethyl Benzene
D. Benzo(a)anthracene	D. 1,3-Dinitrobenzene	D. 2,4,5-TP	D. Demeton-S	Y. Azinphos-methyl	SSS. O-Xylene
E. Benzo(a)pyrene	E. Tetryl	E. Dinoseb	E. Ethoprop	Z. Coumaphos	RRR. MP-Xylene
F. Benzo(b)fluoranthene	F. Nitrobenzene	F. Dichlorprop	F. 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	HH. Tetrachlorvinphos	
N. Naphthalene	N. 4-Nitrotoluene		N. Malathion	II. Sulprofos	
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:



LDC #: 19188A9  
 SDG #: See COM

**VALIDATION FINDINGS WORKSHEET**  
Initial Calibration Calculation Verification

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

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 (std)	CF (std)	Average CF (initial)	Average CF (initial)	%RSD	%RSD		
1	<u>10A1</u>	<u>6/4/08</u>	<u>C</u>	<u>807269</u>	<u>807269</u>	<u>806710</u>	<u>806710</u>	<u>1.82</u>	<u>1.82</u>		
			<u>F</u>	<u>67485</u>	<u>67485</u>	<u>62608</u>	<u>62608</u>	<u>17.820</u>	<u>17.820</u>		
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 #: 17188A9  
 SDG #: See below

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

Page: 6 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: \_\_\_\_\_

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 = 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	80A2862	6/16/08	C	0.50	0.5344	6.9	0.5344	6.9
			P	✓	0.4834	3.3	0.4834	3.3
2	80A2873	6/16/08	C	0.50	0.5307	6.1	0.5307	6.1
			P	✓	0.4984	0.3	0.4984	0.3
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.

LDC #: 17088A9  
 SDG #: See Column

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

Page: 6 of 1  
 Reviewer: [Signature]  
 2nd reviewer: \_\_\_\_\_

METHOD: GC / HPLC

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

% Recovery: SF/SS \* 100  
 Where: SF = Surrogate Found  
 SS = Surrogate Spiked

Sample ID: 2

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery		Percent Difference
				Reported	Recalculated	
7PH	NS	25.0	18.9528	73	73	0

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

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

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

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

METHOD: GC  HPLC

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

RPD =  $100 * (SSCLCS - SSCLCSD) / ((SSCLCS + SSCLCSD) / 2)$

LCS = Laboratory control sample percent recovery  
LCS = Laboratory control sample percent recovery

SC = Concentration

LCS = Laboratory control sample duplicate percent recovery

LCS/LCSD samples: 2168158

Compound	Spike Added Concentration		Spiked Sample Concentration		LCS		LCS		LCS		LCS		LCS	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)														
Diesel (8015)														
Benzene (8021B)														
Methane (RSK-175)														
2,4-D (8151)														
Dinoseb (8151)														
Naphthalene (8310) $\Phi$	66.7	NA	52.6	NA	79	79								
Anthracene (8310)	↓	↓	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 G  
**Collection Date:** June 11, 2008  
**LDC Report Date:** August 8, 2008  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** F8F120180

**Sample Identification**

TSB-GJ-09-10'  
TSB-GJ-09-20'\*\*  
TSB-GJ-09-30'  
TSB-GJ-09-40'

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 4 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.
- UU Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- 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 with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
7/7/08	<sup>13</sup> C-2,3,7,8-TCDF	37.2	TSB-GJ-09-40'	J+ (all detects)	P

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 with the following exceptions:

LCS ID	Compound	%R (Limits)	Associated Samples	Flag	A or P
8170493LCS	1,2,3,7,8,9-HxCDD OCDD	137 (71-129) 154 (74-144)	TSB-GJ-09-10' TSB-GJ-09-30' TSB-GJ-09-40' 8170493MB	J+ (all detects) J+ (all detects)	P

**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
TSB-GJ-09-30'	<sup>13</sup> C-2,3,7,8-TCDF <sup>13</sup> C-1,2,3,7,8-PeCDF <sup>13</sup> C-1,2,3,7,8-PeCDD <sup>13</sup> C-1,2,3,4,7,8-HxCDF <sup>13</sup> C-1,2,3,6,7,8-HxCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD	38 (40-135) 26 (40-135) 27 (40-135) 18 (40-135) 21 (40-135) 11 (40-135) 16 (40-135) 9.7 (40-135)	1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 2,3,7,8-TCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	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 G  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG F8F120180**

SDG	Sample	Compound	Flag	A or P	Reason
F8F120180	TSB-GJ-09-40'	2,3,7,8-TCDF	J+ (all detects)	P	Routine calibration (%D)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-30' TSB-GJ-09-40'	1,2,3,7,8,9-HxCDD OCDD	J+ (all detects) J+ (all detects)	P	Laboratory control samples (%R)
F8F120180	TSB-GJ-09-30'	1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 2,3,7,8-TCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R)

**BRC Tronox Parcel G  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG F8F120180**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel G  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG F8F120180**

No Sample Data Qualified in this SDG



LDC #: 19188A21  
 SDG #: F8F120180  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 5/11/08  
 Page: 1 of 1  
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 2nd Reviewer: \_\_\_\_\_

**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: <u>5/11/08</u>
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	SW	
V.	Blanks	A	
VI.	Matrix spike/Matrix spike duplicates	N	<u>client specified</u>
VII.	Laboratory control samples	SW	<u>LC9</u>
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	<del>A</del>	Not reviewed for Level III validation.
XI.	Compound quantitation and CRQLs	<del>A</del>	Not reviewed for Level III validation.
XII.	System performance	<del>A</del>	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

1	TSB-GJ-09-10'	S	11	8170493 MB	21	31
2	TSB-GJ-09-20**		12	8171591 MB	22	32
3	TSB-GJ-09-30'		13		23	33
4	TSB-GJ-09-40'		14		24	34
5			15		25	35
6			16		26	36
7			17		27	37
8			18		28	38
9			19		29	39
10			20		30	40

Notes: \_\_\_\_\_  
 \_\_\_\_\_  
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LDC #: 19188A21  
 SDG #: See down

VALIDATION FINDINGS CHECKLIST

Page: 1 of 3  
 Reviewer: 9  
 2nd Reviewer: \_\_\_\_\_

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?	/			

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VALIDATION FINDINGS CHECKLIST

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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"/>	
<b>VIII. Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Were the performance evaluation (PE) samples within the acceptance limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>IX. Internal standards</b>				
Were internal standard recoveries within the 40-135% criteria?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Was the minimum S/N ratio of all internal standard peaks $\geq 10$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>X. Target compound identification</b>				
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?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
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?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
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?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Did compound spectra contain all characteristic ions listed in the table attached?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Was the signal to noise ratio for each target compound and labeled standard $\geq 2.5$ ?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Does the maximum intensity of each specified characteristic ion coincide within $\pm 2$ seconds (includes labeled standards)?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
For PCDF identification, was any signal ( $S/N \geq 2.5$ , at $\pm$ seconds RT) detected in the corresponding PCDPE channel?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Was an acceptable lock mass recorded and monitored?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>XI. Compound quantitation/CRQLs</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>XII. System performance</b>				
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XIII. 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>XIV. Field duplicates</b>				
Field duplicate pairs were identified in this SDG.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	

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SDG #: see cover

VALIDATION FINDINGS CHECKLIST

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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:









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

**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:

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (initial)	Average RRF (initial)	RRF (ES3 std)	RRF (ES3 std)	%RSD	%RSD		
1	12A2	6/17/08	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	12.70	
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.913	0.913	0.93	0.93	10.2	10.2	10.3	
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	0.821	0.821	0.87	0.87	13.9	13.9	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	
			OCDF ( <sup>13</sup> C-OCDD)	1.721	1.721	1.86	1.86	16.2	16.2	16.2	
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-OCDD)								
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 Initial Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

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 SDG #: See copy

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

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

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

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	AN 08405	6/29/08	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.798	0.85	5.9	0.85	5.9
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.913	0.82	10.3	0.82	10.3
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	0.821	0.92	11.6	0.92	11.6
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.844	0.89	5.2	0.89	5.2
			OCDF ( <sup>13</sup> C-OCDD)	1.721	1.65	4.3	1.65	4.3
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-OCDD)					
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.

LDC #: 19188A21  
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**VALIDATION FINDINGS WORKSHEET**  
**Laboratory Control Sample Results Verification**

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

RPD =  $|\text{LCS} - \text{LCSD}| \cdot 2 / (\text{LCS} + \text{LCSD})$       LCS = Laboratory control sample percent recovery      LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 871591

Compound	Spike Added (pg/g)		Spiked Sample Concentration		LCS		LCSD		Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.		
2,3,7,8-TCDD	20.0	NA	19.2	NA	96	96								
1,2,3,7,8-PeCDD	100		104		104	104								
1,2,3,4,7,8-HxCDD	✓		126		126	126								
1,2,3,4,7,8,9-HpCDF	✓		90.8		91	91								
OCDF	200	✓	238	✓	119	119								

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>(b)</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>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDF		
	305.8987	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	TCDF		409.7788	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF		
	315.9419	M	<sup>13</sup> 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 <sub>35</sub> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDF (S)		
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF (S)		419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> ClO	HpCDF		
	319.8965	M	C <sub>12</sub> H <sub>35</sub> Cl <sub>4</sub> O <sub>2</sub>	TCDD		423.7767	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>6</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	321.8936	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD		425.7737	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	331.9368	M	<sup>13</sup> C <sub>12</sub> H <sub>4</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 <sub>35</sub> Cl <sub>6</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	333.9338	M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD (S)		437.8140	M+4	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> ClO <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	HxCDFE		479.7165	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>7</sub> <sup>37</sup> ClO	NCDFE		
	[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> Cl <sub>3</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>7</sub> <sup>37</sup> ClO	OCDF
		341.8567	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		443.7399	M+4	C <sub>12</sub> H <sub>35</sub> 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> Cl <sub>3</sub> <sup>37</sup> ClO		PeCDF (S)		457.7377	M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
353.8970		M+4	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>4</sub> <sup>37</sup> ClO	PeCDF (S)	459.7348	M+4		<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>6</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD		
355.8546		M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	469.7780	M+2		C <sub>12</sub> H <sub>35</sub> 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> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD	471.7750	M+4		<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD (S)		
367.8949		M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	513.6775	M+4		<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	DCDFE		
369.8919		M+4	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <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> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDFE							
[354.9792]		LOCK	C <sub>9</sub> F <sub>13</sub>	PFK							
3		373.8208	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>3</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>6</sub>	HxCDF (S)						
	385.8610	M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>3</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> Cl <sub>2</sub> O <sub>2</sub>	HxCDD							
	401.8559	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</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>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDFE							
	[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

