



**LABORATORY DATA CONSULTANTS, INC.**

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

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

August 15, 2008

SUBJECT: BRC Tronox Parcel C, 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 # 19191:**

<b><u>SDG #</u></b>	<b><u>Fraction</u></b>
F8F130140	Volatiles, Semivolatiles, Chlorinated Pesticides, Polychlorinated Biphenyls, Metals, Wet Chemistry, Gasoline Range Organics, Diesel Range Organics, Polynuclear Aromatic Hydrocarbons, Dioxins/Dibenzofurans

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

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

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

Sincerely,

Erlinda T. Rauto  
Operations Manager/Senior Chemist

### LDC #19191 (ERM-Sacramento / BRC Tronox Parcel C)

80/20

LDC	SDG#	DATE REC'D	(3) DATE DUE	VOA (8260B)		SVOA (8270C)		Pest. (8081A)		PCBs (8082)		Metals (SW846)		GRO (8015)		DRO (8015)		PAHs (8310)		Dioxins (8290)		Bromide/Chloride Bromine/Chlorine Chlorate/Fluoride		NO <sub>3</sub> NO <sub>2</sub> O-OP <sub>4</sub>		SO <sub>4</sub> (300.0)		O&G (9071B)	
				W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S
A	F8F130140	07/28/08	08/18/08	4	1	1	1	1	2	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
A	F8F130140	07/28/08	08/18/08	0	1	0	1	0	1	0	1	0	1	0	1	0	1	0	1	0	1	0	1	0	1	0	1	0	1
Total	T/LR			4	2	1	2	1	3	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2

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

**Project/Site Name:** BRC Parcel C  
**Collection Date:** June 12, 2008  
**LDC Report Date:** August 6, 2008  
**Matrix:** Soil/Water  
**Parameters:** Volatiles  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8F130140

RINSATE-2  
TB-2  
TSB-CJ-09-0  
TSB-CJ-09-10\*\*  
TB-1 6/12/08  
TB-2 6/12/08  
RINSATE-2MS  
RINSATE-2MSD  
TSB-CJ-09-0MS  
TSB-CJ-09-0MSD

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 4 soil samples and 6 water samples 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.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

## III. Initial Calibration

Initial calibration was performed using required standard concentrations.

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

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

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

Average relative response factors (RRF) for all 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 F8F130140	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	Iodomethane	67.71684	All water samples in SDG F8F130140	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	Iodomethane	31.67513	All water samples in SDG F8F130140	J+ (all detects)	A
5/28/08	2-Hexanone	25.04476	All water samples in SDG F8F130140	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	Ethanol	0.00209 (≥0.05)	All soil samples in SDG F8F130140	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.

Samples TB-2, TB-1 6/12/08, and TB-2 6/12/08 were identified as trip blanks. No volatile contaminants were found in these blanks with the following exceptions:

Trip Blank ID	Sampling Date	Compound	Concentration	Associated Samples
TB-2	6/12/08	Acetone Chloroform	2.8 ug/L 0.14 ug/L	RINSATE-2
TB-1 6/12/08	6/12/08	Chloroform Dichloromethane	0.11 ug/L 0.41 ug/L	All soil samples in SDG F8F130140

Trip Blank ID	Sampling Date	Compound	Concentration	Associated Samples
TB-2 6/12/08	6/12/08	Acetone	1.7 ug/L	RINSATE-2

Sample RINSATE-2 was identified as a rinsate. No volatile contaminants were found in this blank with the following exceptions:

Rinsate ID	Sampling Date	Compound	Concentration	Associated Samples
RINSATE-2	6/12/08	Chloromethane Dichloromethane Toluene	0.25 ug/L 2.8 ug/L 0.22 ug/L	All soil samples in SDG F8F130140

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 with the following exceptions:

Sample	Compound	Reported Concentration	Modified Final Concentration
TSB-CJ-09-0	Toluene	0.49 ug/Kg	5.2U ug/Kg

## 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
RINSATE-2	Bromofluorobenzene	117 (79-115)	Nonanal Dimethyl disulfide	J+ (all detects) J+ (all detects)	A

## VII. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Although the MS/MSD percent recoveries (%R) or and relative percent difference (RPD) were not within QC limits for some compounds, the MS, MSD, or LCS percent recovery (%R) was within QC limits and no data were qualified.



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

Laboratory control samples were reviewed for each matrix as applicable. Although the 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 the report if data has been qualified.

## **XVI. Field Duplicates**

No field duplicates were identified in this SDG.

**BRC Parcel C**  
**Volatiles - Data Qualification Summary - SDG F8F130140**

SDG	Sample	Compound	Flag	A or P	Reason
F8F130140	TSB-CJ-09-0 TSB-CJ-09-10**	Ethanol	J (all detects) UJ (all non-detects)	A	Initial calibration (RRF)
F8F130140	RINSATE-2 TB-2 TB-1 6/12/08 TB-2 6/12/08	Iodomethane	J+ (all detects)	A	Continuing calibration (%D)
F8F130140	RINSATE-2 TB-2 TB-1 6/12/08 TB-2 6/12/08	Iodomethane	J+ (all detects)	A	Continuing calibration (ICV %D)
F8F130140	RINSATE-2 TB-2 TB-1 6/12/08 TB-2 6/12/08	2-Hexanone	J- (all detects) UJ (all non-detects)	A	Continuing calibration (ICV %D)
F8F130140	TSB-CJ-09-0 TSB-CJ-09-10**	Ethanol	J (all detects) UJ (all non-detects)	A	Continuing calibration (RRF)
F8F130140	RINSATE-2	Nonanal Dimethyl disulfide	J+ (all detects) J+ (all detects)	A	Surrogate recovery (%R)

**BRC Parcel C**  
**Volatiles - Laboratory Blank Data Qualification Summary - SDG F8F130140**

No Sample Data Qualified in this SDG

**BRC Parcel C**  
**Volatiles - Field Blank Data Qualification Summary - SDG F8F130140**

SDG	Sample	Compound	Modified Final Concentration	A or P
F8F130140	TSB-CJ-09-0	Toluene	5.2U ug/Kg	A

LDC #: 19191A1

## VALIDATION COMPLETENESS WORKSHEET

SDG #: F8F130140

Level III/IV

Laboratory: Test America

Date: 8/5/08

Page: 1 of 1

Reviewer: [Signature]

2nd Reviewer: [Signature]

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: 6/12/08
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	W	PSD. 12
IV.	Continuing calibration/ICV	W	ICV = 2570
V.	Blanks	A	
VI.	Surrogate spikes	W	
VII.	Matrix spike/Matrix spike duplicates	W	
VIII.	Laboratory control samples	W	CCS 7
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	R=1. TB=2. S. 6

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

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

D = Duplicate  
TB = Trip blank  
EB = Equipment blank

Validated Samples:

\*\* Indicates sample underwent Level IV validation

1 1/3	RINSATE-2	W	11	8170291MB	21	S	31
2 1/2	TB-2	↓	12	8172125MB	22	W	32
3	TSB-CJ-09-0	S	13	8172361MB	23	W (N)	33
4	TSB-CJ-09-10**	↓	14	8172539MB	24	V (N.D)	34
5 1/2	TB-1 6/12/08	↓	15		25		35
6 1/2	TB-2 6/12/08	↓	16		26		36
7	RINSATE-2MS	↓	17		27		37
8	RINSATE-2MSD	↓	18		28		38
9	TSB-CJ-09-0MS	S	19		29		39
10	TSB-CJ-09-0MSD	↓	20		30		40

LDC #: 19191A1  
 SDG #: See cover

**VALIDATION FINDINGS CHECKLIST**

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

**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.	<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 BFB performance results reviewed and found to be within the specified criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all samples analyzed within the 12 hour clock criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>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 type="checkbox"/>	
Did the initial calibration meet the curve fit acceptance criteria of > 0.990?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent relative standard deviations (%RSD) ≤ 30% and relative response factors (RRF) > 0.05?	<input 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) ≤ 25% and relative response factors (RRF) ≥ 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 at least once every 12 hours for each matrix and concentration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>VI. Surrogate spikes</b>				
Were all surrogate %R within QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
If the percent recovery (%R) for one or more surrogates was out of QC limits, was a reanalysis performed to confirm samples with %R outside of criteria?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>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 type="checkbox"/>	<input checked="" 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 #: PP1A1  
 SDG #: see cover

VALIDATION FINDINGS CHECKLIST

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

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per analytical batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>IX. Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?	<input type="checkbox"/>	<input 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 ± 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	JJ. 1,2-Dichlorobenzene	DDDD. Isopropyl alcohol
C. Vinyl chloride**	W. trans-1,3-Dichloropropene	QQ. 1,1-Dichloropropene	KKK. 1,2,4-Trichlorobenzene	EEEE. Acetonitrile
D. Chloroethane	X. Bromoform*	RR. Dibromomethane	LLL. Hexachlorobutadiene	FFFF. Acrolein
E. Methylene chloride	Y. 4-Methyl-2-pentanone	SS. 1,3-Dichloropropane	MMM. Naphthalene	GGGG. Acrylonitrile
F. Acetone	Z. 2-Hexanone	TT. 1,2-Dibromoethane	NNN. 1,2,3-Trichlorobenzene	HHHH. 1,4-Dioxane
G. Carbon disulfide	AA. Tetrachloroethene	UU. 1,1,1,2-Tetrachloroethane	OOO. 1,3,5-Trichlorobenzene	IIII. Isobutyl alcohol
H. 1,1-Dichloroethene**	BB. 1,1,2,2-Tetrachloroethane*	VV. Isopropylbenzene	PPP. trans-1,2-Dichloroethene	JJJJ. Methacrylonitrile
I. 1,1-Dichloroethane*	CC. Toluene**	WW. Bromobenzene	QQQ. cis-1,2-Dichloroethene	KKKK. Propionitrile
J. 1,2-Dichloroethene, total	DD. Chlorobenzene*	XX. 1,2,3-Trichloropropane	RRR. m,p-Xylenes	LLLL. Ethyl ether
K. Chloroform**	EE. Ethylbenzene**	YY. n-Propylbenzene	SSS. o-Xylene	MMMM. Benzyl chloride
L. 1,2-Dichloroethane	FF. Styrene	ZZ. 2-Chlorotoluene	TTT. 1,1,2-Trichloro-1,2,2-trifluoroethane	NNNN. <i>2,2-Dimethyl pentanol</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

**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".

- Y  N  N/A Did the laboratory perform a 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
	<u>6/9/08</u>	<u>1CAL</u>	<u>WNW</u>		<u>0.00227</u>	<u>M/Salts. 8170-91 MB</u>	<u>[Signature]</u>

**VALIDATION FINDINGS WORKSHEET**  
Continuing Calibration

LDC #: 19191A  
SDG #: See Copy

**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".

Y N N/A Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?  
Y 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	11CV9881 (1CV)	1,1-dichloroethane	31.67513		A11H203	$\rightarrow$ + <u>data</u> / A
			Z	25.04476		8172125MB	$\rightarrow$ / <u>data</u> / A
	6/16/08	FEAL1777	N/N		0.00209	A115013	$\rightarrow$ / <u>data</u> / A
						8170291MB	
	6/19/08	LCAL0217	1,1-dichloroethane	67.71684		A11H203	$\rightarrow$ + <u>data</u> / A
						8172125MB	



VALIDATION FINDINGS WORKSHEET  
Field Blanks

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

LDC #: 19191A  
SDG #: Rec 00101

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

N/A N/A Were field blanks identified in this SDG?  
 N/A N/A Were target compounds detected in the field blanks?  
 Blank units: [Signature] Associated sample units: [Signature]  
 Sampling date: 6/12/18  
 Field blank type: (circle one) Field Blank / Rinsate / Trip Blank / Other: MI soils

Compound	Blank ID	Sample Identification
	5	
Methylene chloride		
Acetone		
Chloroform	0.11	
Dichloromethane	0.4	
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  
Field Blanks

Page: 4 of 9  
Reviewer: [Signature]  
2nd Reviewer: [Signature]

LDC #: 1919A  
SDG #: See above

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

Were field blanks identified in this SDG? Y  
Were target compounds detected in the field blanks? N  
Blank units: None Associated sample units: None  
Sampling date: 6/12/18  
Field blank type: (circle one) Field Blank (Rinsate) / Trip Blank / Other: all soils

Compound	Blank ID	Sample Identification
Methylene chloride	1	
Acetone		
Chloroform		
A	0.75	
Dichloromethane	2.8	
CC	0.22	
CRQL	0.49/5.0	

Blank units: None Associated sample units: None  
Sampling date: 6/12/18  
Field blank type: (circle one) Field Blank (Rinsate) / Trip Blank / Other: None

Compound	Blank ID	Sample Identification
Methylene chloride	2	
Acetone		
Chloroform		
	6	
	1.7	
	0.14	
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".

LDC #: PP1A  
SDG #: 200201

VALIDATION FINDINGS WORKSHEET  
Surrogate Spikes

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

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".

Y  N  N/A

Were all surrogate %R within QC limits?  
Y  N  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?

#	Date	Sample ID	Surrogate	%Recovery (Limits)	Qualifications
		<u>072125MB</u>	<u>BFB</u>	<u>117</u> ( <u>79-115</u> )	<u>1+ Lots / P</u>
		<u>1</u>	<u>BFB</u>	<u>117</u> ( <u>79-115</u> )	<u>1+ Lots / A (Nonanal, 0000)</u>
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

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

LDC #: 1919A  
 SDG #: Decker

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

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

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".  
 Y  N  N/A Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.  
 Y  N  N/A Was a MS/MSD analyzed every 20 samples of each matrix?  
 Y  N  N/A Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		<u>9/8/10</u>	<u>N</u>	<u>73 (81-127)</u>	<u>77 (81-127)</u>	( )	<u>3</u>	<u>No Anal (CAS in)</u>
		<u>7/8</u>	<u>1,1-dichloroethane</u>	( )	( )	<u>22 (≤ 20)</u>	<u>1</u>	<u>No Anal (70 R in)</u>
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		

Compound	QC Limits (Soil)	RPD (Soil)	QC Limits (Water)	RPD (Water)
H. 1,1-Dichloroethane	59-172%	≤ 22%	61-145%	≤ 14%
S. Trichloroethane	62-137%	≤ 24%	71-120%	≤ 14%
V. Benzene	66-142%	≤ 21%	76-127%	≤ 11%
CC. Toluene	59-139%	≤ 21%	76-125%	≤ 13%
DD. Chlorobenzene	60-133%	≤ 21%	75-130%	≤ 13%

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

LDC #: 1991A1  
SDG #: see above

Page: 6/1  
Reviewer: g  
2nd Reviewer: l

**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".

Y N/A Was a LCS required?  
Y 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
		<u>8172/25 LCS</u>	<u>NN</u>	<u>293 (42-140)</u>	<u>( )</u>	<u>112 ( ≤ 20 )</u>	<u>M1+0's</u>	<u>No Qual</u>
		<u>10</u>	<u>iodomethane</u>	<u>166 (45-140)</u>	<u>181 (45-140)</u>	<u>( )</u>	<u>8172/25 M1's</u>	<u>(NS/NSD in)</u>

LDC #: PA1A1  
 SDG #: See Com

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

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

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

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

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				RRF (std)	RRF (std)	RRF (std)	Average RRF (Initial)	Average RRF (Initial)	%RSD	%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)	0.2592	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	13.4744	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 =  $100 \cdot (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$       Where: ave. RRF = initial calibration average RRF  
 $\text{RRF} = (A_s)(C_i) / (A_s)(C_s)$       RRF = continuing calibration RRF  
 $A_s$  = Area of compound,       $A_w$  = Area of associated internal standard  
 $C_s$  = Concentration of compound,       $C_w$  = 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-117D	6/16/08	K (1st internal standard)	0.50831	0.57288	0.89949	0.57288	0.899
			AA (2nd internal standard)	0.29404	0.31216	6.16168	0.31216	6.1612
			DDP (3rd internal standard)	3.42599	3.65203	6.59782	3.65203	6.5978
2	FEA-117T	6/16/08	NNNN (1st internal standard)	0.73871	0.72154	2.32412	0.72154	2.3244
			OOO (2nd internal standard)	0.55366	0.57358	3.59757	0.57358	3.5985
			PPP (3rd internal standard)	1.11150	1.10470	0.61152	1.10470	0.6118
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 #: 19191A  
 SDG #: See CDW

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

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

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

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

% Recovery:  $SF/SS * 100$

Where: SF = Surrogate Found  
 SS = Surrogate Spiked

Sample ID: 4

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8	50	45.0916	90	90	0
Bromofluorobenzene	↓	42.0950	84	84	↓
1,2-Dichloroethane-d4	↓	48.4101	97	97	↓
Dibromofluoromethane	↓	43.2435	86	86	↓

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					



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

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

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

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

RPD =  $|MSC - MSC | * 2 / (MSC + MSDC)$       MSC = Matrix spike concentration      MSDC = Matrix spike duplicate concentration

MS/MSD sample: 9/10

Compound	Spike Added (1/45)		Sample Concentration (1/45)	Spiked Sample Concentration (1/45)		Matrix Spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		MS/MSD RPD	
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
1,1-Dichloroethene	52.4	58.0	ND	41.6	42.8	79	79	82	82	2.9	2.8
Trichloroethene			↓	49.1	49.9	94	94	96	96	1.5	1.6
Benzene			0.49	46.9	47.6	90	90	91	91	1.4	1.5
Toluene			ND	47.0	47.8	89	89	91	91	1.8	1.7
Chlorobenzene			ND	46.4	47.0	89	89	90	90	1.2	1.3

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



LDC #: 19191A  
SDG #: see cover

### VALIDATION FINDINGS WORKSHEET

#### Sample Calculation Verification

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

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

- Y  N  N/A Were all reported results recalculated and verified for all level IV samples?  
 Y  N  N/A 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. 4 , 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 Parcel C  
**Collection Date:** June 12, 2008  
**LDC Report Date:** August 6, 2008  
**Matrix:** Soil/Water  
**Parameters:** Semivolatiles  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8F130140

**Sample Identification**

RINSATE-2  
TSB-CJ-09-0  
TSB-CJ-09-10\*\*  
RINSATE-2MS  
RINSATE-2MSD  
TSB-CJ-09-0MS  
TSB-CJ-09-0MSD

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 4 soil samples and 3 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

## III. Initial Calibration

Initial calibration was performed using required standard concentrations.

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

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

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

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

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
6/18/08	Phthalic acid	0.01422 ( $\geq 0.05$ )	All samples in SDG F8F130140	J (all detects)	A
	N-(Hydroxymethyl)phthalimide	0.04408 ( $\geq 0.05$ )		UJ (all non-detects)	
				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	All samples in SDG F8F130140	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
6/19/08	Phthalic acid	0.01066 ( $\geq 0.05$ )	All samples in SDG F8F130140	J (all detects) UJ (all non-detects)	A
	N-(Hydroxymethyl)phthalimide	0.04523 ( $\geq 0.05$ )		J (all detects) UJ (all non-detects)	

## V. Blanks

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

Sample RINSATE-2 was identified as a rinsate. No semivolatile contaminants were found in this blank.

## 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. Although the percent recovery (%R) for one compound in the LCS was 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 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 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 Parcel C  
Semivolatiles - Data Qualification Summary - SDG F8F130140**

SDG	Sample	Compound	Flag	A or P	Reason
F8F130140	RINSATE-2 TSB-CJ-09-0 TSB-CJ-09-10**	Phthalic acid N-(Hydroxymethyl)phthalimide	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A	Initial calibration (RRF)
F8F130140	RINSATE-2 TSB-CJ-09-0 TSB-CJ-09-10**	Phthalic acid	J- (all detects) UJ (all non-detects)	A	Continuing calibration (%D)
F8F130140	RINSATE-2 TSB-CJ-09-0 TSB-CJ-09-10**	Phthalic acid N-(Hydroxymethyl)phthalimide	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A	Continuing calibration (RRF)

**BRC Parcel C  
Semivolatiles - Laboratory Blank Data Qualification Summary - SDG F8F130140**

No Sample Data Qualified in this SDG

**BRC Parcel C  
Semivolatiles - Field Blank Data Qualification Summary - SDG F8F130140**

No Sample Data Qualified in this SDG

LDC #: 19191A2  
 SDG #: F8F130140  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**

Level III/IV

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

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/12/08
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	SW	
IV.	Continuing calibration/ICV	W	ICV ≤ 25%
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	W	LC9
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	ND	R=1. [Signature]

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	RINSATE-2	W	11	8168351MB	21	W	31
2	TSB-CJ-09-0	S	12	8170309MB	22	S	32
3	TSB-CJ-09-10**	↓	13		23		33
4	RINSATE-2MS	W	14		24		34
5	RINSATE-2MSD	↓	15		25		35
6	TSB-CJ-09-0MS	S	16		26		36
7	TSB-CJ-09-0MSD	↓	17		27		37
8	<del>RINSATE-1</del>	W	18		28		38
9			19		29		39
10			20		30		40

LDC #: 1919182  
 SDG #: 78FT30140

VALIDATION FINDINGS CHECKLIST

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

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.	/			
Cooler temperature criteria was met.	/			
<b>II. GC/MS Instrument performance check</b>				
Were the DFTPP 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 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 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?			/	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?			/	
<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 #: 19191A2  
 SDG #: FS720140

VALIDATION FINDINGS CHECKLIST

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

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 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 type="checkbox"/>	<input checked="" 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 checked="" type="checkbox"/>	<input type="checkbox"/>	
Target compounds were detected in the field blanks.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input 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. yyy. Phenyl sulfide. zzz. Phenyl disulfide*  
*AAAA. 4-chlorophenyl sulfone*  
*Benzenethiol*  
*4-CH(nobenzenthio)*

# VALIDATION FINDINGS WORKSHEET

## Initial Calibration

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

LDC #: 1919A-2  
SDG #: 322044  
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  $\leq 30$  %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>6/18/08</u>	<u>1cA</u>	<u>Phthalic acid</u> <u>xxx</u>		<u>0.01422</u> <u>0.04408</u>	<u>M1 + Pk's</u>	<u>Y N / A</u>

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

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

#	Date	Standard ID	Compound	Finding %D (Limit: ≤25.0%)	Finding RRF (Limit: ≥0.05)	Associated Samples	Qualifications
	6/19/08	NCAL529	Phthalic acid	25.0878		MU + Bks	J-UN/A
			↓		0.01266		J-UN/A
			XXX		0.04523		↓



VALIDATION FINDINGS WORKSHEET  
 Laboratory Control Samples (LCS)

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 Was a LCS required?  
 Y 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
		8170309205	HH	36 (57-90)	( ) ( )	( ) ( )	MS/IS.	No Qual
					( ) ( )	( ) ( )	8170309MB	(MS/MSD)
					( ) ( )	( ) ( )		
					( ) ( )	( ) ( )		
					( ) ( )	( ) ( )		
					( ) ( )	( ) ( )		
					( ) ( )	( ) ( )		
					( ) ( )	( ) ( )		
					( ) ( )	( ) ( )		
					( ) ( )	( ) ( )		
					( ) ( )	( ) ( )		
					( ) ( )	( ) ( )		
					( ) ( )	( ) ( )		
					( ) ( )	( ) ( )		
					( ) ( )	( ) ( )		
					( ) ( )	( ) ( )		
					( ) ( )	( ) ( )		
					( ) ( )	( ) ( )		
					( ) ( )	( ) ( )		
					( ) ( )	( ) ( )		

**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:

$RRF = (A_x)(C_w)/(A_w)(C_x)$   
 average RRF = sum of the RRF's/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  
 $A_w$  = Area of associated internal standard  
 $C_w$  = Concentration of internal standard  
 $S$  = Standard deviation of the RRFs,  $X$  = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				RRF (SD std)	RRF (SD std)	RRF (SD std)	RRF (SD std)	Average RRF (Initial)	%RSD	Average RRF (Initial)	%RSD
1	1CAZ	6/2/08	Phenol (1st internal standard)	1.87853	1.87853	1.85537	1.85537	1.070	1.070	1.070	1.070
			Naphthalene (2nd internal standard)	1.09438	1.09438	1.10901	1.10901	1.328	1.328	1.328	1.328
			Fluorene (3rd internal standard)	1.41778	1.41778	1.4229	1.4229	0.573	0.573	0.573	0.573
			Pentachlorophenol (4th internal standard)	0.20260	0.20260	0.19634	0.19634	10.255	10.255	10.255	10.255
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.90763	0.90763	0.86343	0.86343	9.524	9.524	9.524	9.524
			Benzo(a)pyrene (6th internal standard)	1.13808	1.13808	1.1182	1.1182	6.486	6.486	6.486	6.486
2	1CAZ	6/18/08	Phenol (1st internal standard)	0.51976	0.51976	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	1CAZ	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	0.33062	0.33062	4.49540	4.49540	4.49540	4.49540
			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)	0.39770	0.39770	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.

LDC #: 19191A-2  
SDG #: See copy

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

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

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	VCA527	6/19/08	Phenol (1st internal standard)	1.85537	1.80162	2.89731	2.89731	2.89731
			Naphthalene (2nd internal standard)	1.10901	1.08712	1.97399	1.97399	1.97399
			Fluorene (3rd internal standard)	1.41229	1.40878	1.24855	1.24855	0.2488
			Pentachlorophenol (4th internal standard)	0.19634	0.20730	5.58529	5.58529	5.5830
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.86343	0.87788	0.97842	0.97842	0.97842
			Benzo(a)pyrene (6th internal standard)	1.11183	1.12694	1.30062	1.30062	1.30062
2	VCA528	6/19/08	Phenol (1st internal standard)	0.51574	0.51326	0.10062	0.10062	0.1010
			Naphthalene (2nd internal standard)	1.18223	1.20702	2.09666	2.09666	2.09669
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
3	VCA529	6/19/08	Phenol (1st internal standard)	1.57590	1.59157	0.99462	0.99462	0.9947
			Naphthalene (2nd internal standard)	0.33002	0.33954	2.88719	2.88719	2.8858
			Fluorene (3rd internal standard)	1.02385	1.01788	0.58319	0.58319	0.5839
			Pentachlorophenol (4th internal standard)	0.36637	0.38045	3.84416	3.84416	3.8431
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.39265	0.39958	1.76341	1.76341	1.7639
			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 #: 19191A2  
 SDG #: Secon

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

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

**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: 3

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	50	34.9805	70	70	0
2-Fluorobiphenyl	✓	36.6753	73	73	↓
Terphenyl-d14	✓	33.4531	67	67	
Phenol-d5	75	53.9595	72	72	
2-Fluorophenol	↓	52.1505	70	70	
2,4,6-Tribromophenol	↓	59.7343	80	80	
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

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

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

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

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

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

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

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

RPD =  $|MSC - MSC| * 2 / (MSC + MSC)$       MSC = Matrix spike concentration      MSDC = Matrix spike duplicate concentration

MS/MSD samples: 3170309

Compound	Spike Added		Sample Concentration	Spiked Sample Concentration		Matrix Spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		MS/MSD RPD	
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
Phenol	3480	3430	ND	2640	2400	76	76	70	70	9.4	9.5
N-Nitroso-di-n-propylamine				2840	2580	82	82	75	75	9.4	9.6
4-Chloro-3-methylphenol				2870	2670	83	83	78	78	7.1	7.2
Acenaphthene				2780	2590	80	80	75	75	7.0	7.1
Pentachlorophenol				2590	2410	75	75	70	70	7.4	7.2
Pyrene				2490	2340	72	72	68	68	6.0	6.2

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

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

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

% Recovery =  $100 * (SC/SA)$

Where: SSC = Spike concentration  
SA = Spike added

RPD =  $\frac{LCSC - LCSDC}{LCSC + LCSDC} * 100$

LCSC = Laboratory control sample concentration LCSDC = Laboratory control sample duplicate concentration

LCS/LCSD samples: 8170309

Compound	Spike Added (MMS)		Spike Concentration		LCS		LCSD		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenol	3330	NA	2370	NA	71	71						
N-Nitroso-di-n-propylamine			2570		77	77						
4-Chloro-3-methylphenol			2600		78	78						
Acenaphthene			2520		75	76						
Pentachlorophenol			2270		68	68						
Pyrene			2260		68	68						

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

LDC #: 19191A2  
SDG #: See Cover

# VALIDATION FINDINGS WORKSHEET

## Sample Calculation Verification

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

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

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?

$$\text{Concentration} = \frac{A_x(L)(V_i)(DF)(2.0)}{A_s(RRF)(V_o)(V_i)(\%S)}$$

- A<sub>x</sub> = Area of the characteristic ion (EICP) for the compound to be measured
- A<sub>s</sub> = Area of the characteristic ion (EICP) for the specific internal standard
- I<sub>s</sub> = Amount of internal standard added in nanograms (ng)
- V<sub>o</sub> = Volume or weight of sample extract in milliliters (ml) or grams (g).
- V<sub>i</sub> = Volume of extract injected in microliters (ul)
- V<sub>t</sub> = Volume of the concentrated extract in microliters (ul)
- Df = Dilution Factor.
- %S = Percent solids, applicable to soil and solid matrices only.
- 2.0 = Factor of 2 to account for GPC cleanup

Example:

Sample I.D. 3, NO:

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

=

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

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

**Project/Site Name:** BRC Tronox Parcel C  
**Collection Date:** June 12, 2008  
**LDC Report Date:** August 6, 2008  
**Matrix:** Soil/Water  
**Parameters:** Chlorinated Pesticides  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8F130140

**Sample Identification**

RINSATE-2  
TSB-CJ-09-0  
TSB-CJ-09-0DL  
TSB-CJ-09-10\*\*  
TSB-CJ-09-0MS  
TSB-CJ-09-0MSD

\*\*Indicates sample underwent EPA Level IV review



## Introduction

This data review covers 5 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 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 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.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UU Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. GC/ECD Instrument Performance Check

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

## III. Initial Calibration

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

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

Retention time windows were evaluated and considered technically acceptable for samples on which an 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
6/19/08	KCAL133	A	Methoxychlor	15.4	TSB-CJ-09-0 TSB-CJ-09-10** TSB-CJ-09-OMS TSB-CJ-09-OMSD 8170319MB	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.

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

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

## V. Blanks

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

Sample RINSATE-2 was identified as a rinsate. No chlorinated pesticide contaminants were found in this blank.

## 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	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
TSB-CJ-09-0	Not specified	Decachlorobiphenyl	314 (61-137)	All TCL compounds	J+ (all detects)	A
8169189MB	Not specified	Tetrachloro-m-xylene	58 (72-135)	All TCL compounds	J- (all detects) UJ (all non-detects)	P

## VII. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Although the MS/MSD percent recoveries (%R) and relative percent difference (RPD) were not within QC limits for one compound, the LCS percent recovery (%R) was within QC limits and no data were qualified.

## VIII. Laboratory Control Samples (LCS)

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

## IX. Regional Quality Assurance and Quality Control

Not applicable.

## X. Pesticide Cleanup Checks

### a. Florisil Cartridge Check

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

### b. GPC Calibration

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

## XI. Target Compound Identification

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

## XII. Compound Quantitation and Reported CRQLs

All compound quantitation and CRQLs were within validation criteria with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P
TSB-CJ-09-0	beta-BHC	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	A

The sample results for detected compounds from the two columns were within 40% percent difference (%D) with the following exceptions:

Sample	Compound	%D	Flag	A or P
TSB-CJ-09-10**	gamma-Chlordane	218.5	J (all detects)	A

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 C  
Chlorinated Pesticides - Data Qualification Summary - SDG F8F130140**

SDG	Sample	Compound	Flag	A or P	Reason
F8F130140	TSB-CJ-09-0 TSB-CJ-09-10**	Methoxychlor	J+ (all detects)	A	Continuing calibration (%D)
F8F130140	TSB-CJ-09-0	All TCL compounds	J+ (all detects)	A	Surrogate spikes (%R)
F8F130140	TSB-CJ-09-0	beta-BHC	J (all detects)	A	Compound quantitation and CRQLs
F8F130140	TSB-CJ-09-10**	gamma-Chlordane	J (all detects)	A	Compound quantitation and CRQLs (%D)

**BRC Tronox Parcel C  
Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG F8F130140**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel C  
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG F8F130140**

No Sample Data Qualified in this SDG

LDC #: 19191A3a  
 SDG #: F8F130140  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

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

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/12/08
II.	GC/ECD Instrument Performance Check	A	
III.	Initial calibration	A	PSD. 12
IV.	Continuing calibration/ICV	SW	LEV = 1570
V.	Blanks	A	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	A	LOG
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	SW	Not reviewed for Level III validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	ND	R = 1

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	RINSATE-2	W	11	8169189MB	21	W	31
2	TSB-CJ-09-0	S	12	8170319MB	22	S	32
3	TSB-CJ-09-ODL		13		23		33
4	TSB-CJ-09-10**		14		24		34
5	TSB-CJ-09-OMS		15		25		35
6	TSB-CJ-09-OMSD		16		26		36
7			17		27		37
8			18		28		38
9			19		29		39
10			20		30		40

# 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 #: 19191A3d  
 SDG #: SECOWW

**VALIDATION FINDINGS CHECKLIST**

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

Method:  GC  HPLC

Validation Area	Yes	No	NA	Findings/Comments
<b>Technical holding times:</b>				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>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>IV. Continuing calibration:</b>				
What type of continuing calibration calculation was performed? <input checked="" type="checkbox"/> %D or <input type="checkbox"/> %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 type="checkbox"/>	<input checked="" 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 checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>VII. Matrix spike/Matrix spike duplicate:</b>				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a MS/MSD analyzed every 20 samples of each matrix?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input 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"/>	
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

LDC #: 19191 D3a  
 SDG #: see COM

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: 9  
 2nd Reviewer: 4

Validation Area	Yes	No	NA	Findings/Comments
IX. Regional Quality Assurance and Quality Control 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"/>	
X. Target compound identification 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"/>	
XI. Compound quantitation/CRQLs Were the retention times of reported detects within the RT windows?	<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"/>	
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 checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Were target compounds detected in the field blanks?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	



LDC #: 1919A32  
 SDG #: See comm

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

**VALIDATION FINDINGS WORKSHEET**  
**Surrogate Recovery**

METHOD:  GC  HPLC

Are surrogates required by the method? Yes  or No

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

Were surrogates spiked into all samples and blanks?  N/A  N  Y  N/A

Did all surrogate recoveries (%R) meet the QC limits?  N/A  N  Y  N/A

#	Sample ID	Detector/Column	Surrogate Compound	%R (Limits)	Qualifications
2		MS	DCB	314	(61-137) <u>1+ detects / A</u>
3		[check]	All	DIC	( - ) <u>No equal</u>
81691894B		MS	TCMX	58	(72-135) <u>1/MS / P</u>
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

Surrogate Compound	Surrogate Compound	Surrogate Compound	Surrogate Compound	Surrogate Compound
A Chlorobenzene (CBZ)	G Octacosane	M Benzo(e)Pyrene	S 1-Chloro-3-Nitrobenzene	Y Tetrachloro-m-xylene
B 4-Bromofluorobenzene (BFB)	H Ortho-Terphenyl	N Terphenyl-D14	T 3,4-Dinitrotoluene	
C a,a-Trifluorotoluene	I Fluorobenzene (FBZ)	O Decachlorobiphenyl (DCB)	U Tripentyltin	
D Bromochlorobenzene	J n-Triacontane	P 1-methylmethylthalene	V Tri-n-propyltin	
E 1,4-Dichlorobutane	K Hexacosane	Q Dichlorophenyl Acetic Acid (DCAA)	W Tributyl Phosphate	
F 1,4-Difluorobenzene (DFB)	L Bromobenzene	R 4-Nitrophenol	X Triphenyl Phosphate	



VALIDATION FINDINGS WORKSHEET  
Compound Quantitation and Reported CRQLs

METHOD:  GC  HPLC

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".  
 Level: WHD Only  
 Y N / N/A  
 Y N / N/A  
 Y N / N/A

Were CRQLs adjusted for sample dilutions, dry weight factors, etc.?  
 Did the reported results for detected target compounds agree within 10.0% of the recalculated results?

#	Compound Name	Finding	Associated Samples	Qualifications
	B	exceeded cal-b range	2	lots / A

Comments: See sample calculation verification worksheet for recalculations

**VALIDATION FINDINGS WORKSHEET**  
**Compound Quantitation and Reported CRQLs**

METHOD:  GC  HPLC

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

Level I/IV Only

Y N N/A

Y N N/A

Y N N/A

Were CRQLs adjusted for sample dilutions, dry weight factors, etc.?

Did the reported results for detected target compounds agree within 10.0% of the recalculated results?

Did the percent difference of detected compounds between two columns/detectors  $\leq$ 40%?

If no, please see findings below.

#	Compound Name	Sample ID	%D Between Two Columns/Detectors Limit ( $\leq$ 40%)	Qualifications
	<u>Y</u>	<u>A</u>	<u>.218.5</u>	<u>Notes/A</u>

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

LDC #: 1919, A39  
 SDG #: Beccom

**VALIDATION FINDINGS WORKSHEET**  
Initial Calibration Calculation Verification

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

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 (% std)	σ <sub>CF</sub> (std)	CF	σ <sub>CF</sub> (std)	Average CF (Initial)	%RSD	Average CF (Initial)	%RSD
1	1CA2	6/12/08	2,4'-DDE (ch A) ✓	37366840	37366844	373785260	373785260	1.30108	1.3011	203802778	2.78044
			F (ch A)	21121720	21121720	681332132	681332132	2.76188	2.7619	301269612	1.12425
2	1CA2	6/16/08	0 (✓) F (ch B) 0 ✓	363591000	363591000	380182886	380182886	2.83696	2.8370	133202078	8.79725
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 #: 19191A39  
 SDG #: See CDW

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

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

METHOD: GC  HPLC

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

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

#	Standard ID	Calibration Date	Compound	Average CF (cal)/ CCV Conc.	Reported		Recalculated	
					CF/Conc. CCV	%D	CF/Conc. CCV	%D
1	KC4133	6/19/18	2,4'-DDDE (ch A) ↓ F (ch B) F (ch A)	0.025	0.0242	3.3	0.0242	3.3
					0.0248	0.8	0.0248	0.8
					0.0256	2.3	0.0256	2.3
2			0 ↓ F (ch B) D ↓		0.0283	13.1	0.0283	13.1
					0.0251	0.4	0.0251	0.4
					0.0269	7.4	0.0269	7.4
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 #: 1991A3a  
 SDG #: See column  
 METHOD: GC HPLC

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

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: 4

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery		Percent Difference
				Reported	Recalculated	
TMX	enB	0.020	0.01992	100	100	0
DEB	✓	✓	0.01705	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	

METHOD:  GC  HPLC

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

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

Where

SSC = Spiked sample concentration

SC = Sample concentration

SA = Spike added

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

MSD = Matrix spike duplicate

MS/MSD samples: 5/6

Compound	Spike Added (MS/MSD)		Spike Sample Concentration (MS/MSD)		Sample Conf. (MS/MSD)		Matrix spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		MS/MSD RPD	
	MS	MSD	MS	MSD	MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)												
Diesel (8015)												
Benzene (8021B)												
Methane (RSK-175)												
2,4-D (8151)												
Dinoseb (8151)												
Naphthalene (8310)												
Anthracene (8310)												
HMX (8330)												
2,4,6-Trinitrotoluene (8330)												
F	17.4	17.4	16.1	15.6	ND		92	92	90	90	3.1	3.2
0	↓	↓	23.2	22.4	6.6		96	95	91	91	3.8	3.5

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

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 =  $100 \times \frac{SSC-SC}{SSC-SA}$

Where: SSC = Spiked sample concentration  
SA = Spike added

RPD =  $100 \times \frac{SSC-LCS}{SSC}$

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples: 8170319

Compound	Spike Added		Spiked Sample Concentration		LCS		LCSD		Percent Recovery		Percent Recovery		RPD	
	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)														
Anthracene (8310)														
HMX (8330)														
2,4,6-Trinitrotoluene (8330)														
F	16.7	NA	15.1	NA	90	90								
D	17.0	✓	17.0	✓	102	102								

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.

METHOD:  GC  HPLC

Y N N/A Were all reported results recalculated and verified for all level IV samples?  
 Y N N/A Were all recalculated results for detected target compounds within 10% of the reported results?

Concentration =  $\frac{(A)(Fv)(Df)}{(RF)(Vs \text{ or } Ws)(\%S/100)}$   
Example: Sample ID: 4 Compound Name: T

$(8297556)$   
 $(\cancel{44470164})(10000)(1)$   
Concentration =  $(\cancel{311047242})(30.2)(0.913)$   
 $(565875360)$   
= 5.32 mg/kg

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 Parcel C  
**Collection Date:** June 12, 2008  
**LDC Report Date:** August 6, 2008  
**Matrix:** Soil/Water  
**Parameters:** Polychlorinated Biphenyls  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8F130140

**Sample Identification**

RINSATE-2  
TSB-CJ-09-0  
TSB-CJ-09-10\*\*  
RINSATE-2MS  
RINSATE-2MSD  
TSB-CJ-09-0MS  
TSB-CJ-09-0MSD

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 4 soil samples and 3 water 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 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.
- 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 were 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 an 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 difference (%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 an 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.

Sample RINSATE-2 was identified as a rinsate. No polychlorinated biphenyl contaminants were found in this blank.

## 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	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
TSB-CJ-09-0	Not specified	Decachlorobiphenyl	522 (57-150)	All TCL compounds	J+ (all detects)	A

## VII. Matrix Spike/Matrix Spike Duplicates

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

## VIII. Laboratory Control Samples (LCS)

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

## IX. Regional Quality Assurance and Quality Control

Not applicable.

## X. Pesticide Cleanup Checks

### a. Florisil Cartridge Check

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

### b. GPC Calibration

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

## XI. Target Compound Identification

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

## XII. Compound Quantitation and Reported CRQLs

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

### **XIII. Overall Assessment of Data**

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

### **XIV. Field Duplicates**

No field duplicates were identified in this SDG.

**BRC Parcel C  
Polychlorinated Biphenyls - Data Qualification Summary - SDG F8F130140**

<b>SDG</b>	<b>Sample</b>	<b>Compound</b>	<b>Flag</b>	<b>A or P</b>	<b>Reason</b>
F8F130140	TSB-CJ-09-0	All TCL compounds	J+ (all detects)	A	Surrogate spikes (%R)

**BRC Parcel C  
Polychlorinated Biphenyls - Laboratory Blank Data Qualification Summary - SDG  
F8F130140**

No Sample Data Qualified in this SDG

**BRC Parcel C  
Polychlorinated Biphenyls - Field Blank Data Qualification Summary - SDG  
F8F130140**

No Sample Data Qualified in this SDG

LDC #: 19191A3b  
 SDG #: F8F130140  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

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

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/12/08
II.	GC/ECD Instrument Performance Check	N	
III.	Initial calibration	A	
IV.	Continuing calibration/ICV	A	ICV = 1570
V.	Blanks	A	
VI.	Surrogate spikes	TW	
VII.	Matrix spike/Matrix spike duplicates	A	
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	ND	R = 1

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	RINSATE-2	W	11	8168237 MB	21		31
2	TSB-CJ-09-0	S	12	8170314 MB	22		32
3	TSB-CJ-09-10**	✓	13		23		33
4	RINSATE-2MS	W	14		24		34
5	RINSATE-2MSD	✓	15		25		35
6	TSB-CJ-09-0MS	S	16		26		36
7	TSB-CJ-09-0MSD	✓	17		27		37
8			18		28		38
9			19		29		39
10			20		30		40

LDC #: 19191A36  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

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

Method: GC HPLC

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times:</b>				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>II. Initial calibration:</b>				
Did the laboratory perform a 5 point calibration prior to sample analysis?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	
Did the initial calibration meet the curve fit acceptance criteria?	<input 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 type="checkbox"/>	<input checked="" 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 checked="" type="checkbox"/>	<input type="checkbox"/>	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input 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 #: 199143b  
 SDG #: see con

**VALIDATION FINDINGS CHECKLIST**

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

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 checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were target compounds detected in the field blanks?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input 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: \_\_\_\_\_



# VALIDATION FINDINGS WORKSHEET

## Surrogate Recovery

LDC #: 191A3b  
SDG #: See column

Page: 1 of 1  
Reviewer: 4  
2nd Reviewer: 9

METHOD:  GC  HPLC

Are surrogates required by the method? Yes  or No

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

N/A Were surrogates spiked into all samples and blanks?

N/A Did all surrogate recoveries (%R) meet the QC limits?

#	Sample ID	Detector/Column	Surrogate Compound	%R (Limits)	Qualifications
	<u>2</u>	<u>NS</u>	<u>DCB</u>	<u>522 (57-150)</u>	<u>+ dete / A</u>

Surrogate Compound	Surrogate Compound	Surrogate Compound	Surrogate Compound	Surrogate Compound	Surrogate Compound
A Chlorobenzene (CBZ)	G Octacosane	M Benzo(e)Pyrene	S 1-Chloro-3-Nitrobenzene	Y Tetrachloro-m-xylene	
B 4-Bromofluorobenzene (BFB)	H Ortho-Terphenyl	N Terphenyl-D14	T 3,4-Dinitrotoluene		
C a,a,a-Trifluorotoluene	I Fluorobenzene (FBZ)	O Decachlorobiphenyl (DCB)	U Tripentyltin		
D Bromochlorobenzene	J n-Triacotane	P 1-methylnaphthalene	V Tri-n-propyltin		
E 1,4-Dichlorobutane	K Hexacosane	Q Dichlorophenyl Acetic Acid (DCAA)	W Tributyl Phosphate		
F 1,4-Difluorobenzene (DFB)	L Bromobenzene	R 4-Nitrophenol	X Triphenyl Phosphate		

LDC #: 1991A36  
 SDG #: 201.com

**VALIDATION FINDINGS WORKSHEET**  
Initial Calibration Calculation Verification

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

METHOD: GC ✓ HPLC \_\_\_\_\_

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

CF = A/C  
 average CF = sum of the CF/number of standards  
 %RSD =  $100 \cdot (S/X)$   
 A = Area of compound,  
 C = Concentration of compound,  
 S = Standard deviation of the CF  
 X = Mean of the CFs

#	Standard ID	Calibration Date	Compound	Reported		Recalculated		Reported		Recalculated	
				CF (SD std)	CF (SD std)	CF (SD std)	Average CF (initial)	Average CF (initial)	%RSD	%RSD	
1	10A ✓	5/21/08	BB (RX-c-post)	33154	33154	27977	27977	12.0	12.0		
			BB ( V II)	45676	45676	39164	39164	9.58	9.58		
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 #: 191112  
 SDG #: See com

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

Page: 1 of 1  
 Reviewer: SS  
 2nd Reviewer: SS

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	PCA422	6/18/08	BB (cal A)	1000	1027.438	2.7	1027.40	2.7
2	PCA428	6/18/08	BB (cal A) V (cal B)	1000 1000	991.8768 1075.6662	0.8 7.6	992.0 1075.60	0.8 7.6
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 #: 19191Aab  
 SDG #: Se. equn  
 METHOD: GC HPLC

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

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: 3

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
<u>PCP</u>	<u>ch A</u>	<u>20.0</u>	<u>19.3366</u>	<u>97</u>	<u>97</u>	<u>0</u>

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

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

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

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

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

LDC #: 1919Azb  
 SDG #: See com

METHOD:  GC  HPLC

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

$\% \text{Recovery} = 100 * ((\text{SSC} - \text{SC}) / \text{SA})$       Where       $\text{SSC} = \text{Spiked sample concentration}$        $\text{SC} = \text{Sample concentration}$   
 $\text{RPD} = (((\text{SSCMS} - \text{SSCMSD}) * 2) / ((\text{SSCMS} + \text{SSCMSD})) * 100$        $\text{SA} = \text{Spike added}$        $\text{MSD} = \text{Matrix spike duplicate}$   
 $\text{MS} = \text{Matrix spike}$

MS/MSD samples: 6/7

Compound	Spike Added		Sample Comp. (MSD)	Spike Sample Concentration (MSD)		Matrix spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		MS/MSD RPD	
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)											
Diesel (8015)											
Benzene (8021B)											
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)											
Anthracene (8310)											
HMX (8330)											
2,4,6-Trinitrotoluene (8330)											
<u>BB</u>	173	174	ND	193	178	112	112	102	102	8.4	8.1

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

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 =  $100 * |SSCLCS - SSC| / SSC$       LCS = Laboratory control sample percent recovery      LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples: 8170315

Compound	Spike Added ( <u>1715</u> )		Spiked Sample Concentration ( <u>1715</u> )		LCS		LCSD		
	LCS	LCSD	LCS	LCSD	Percent Recovery		Percent Recovery		
	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>178</u>	<u>NA</u>	<u>107</u>	<u>107</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.

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

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

LDC #: 199846  
 SDG #: 22000

METHOD:  GC  HPLC

N/A N/A Were all reported results recalculated and verified for all level IV samples?  
N/A N/A 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: B Compound Name NO

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 C  
**Collection Date:** June 12, 2008  
**LDC Report Date:** August 7, 2008  
**Matrix:** Soil/Water  
**Parameters:** Metals  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8F130140

**Sample Identification**

RINSATE-2  
TSB-CJ-09-0  
TSB-CJ-09-10\*\*  
TSB-CJ-09-0MS  
TSB-CJ-09-0MSD

\*\*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 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 with the following exceptions:

Date	Lab. Reference/ID	Analyte	%R (Limits)	Associated Samples	Flag	A or P
6/25/08	CCV (22:30)	Thallium Uranium	113.4 (90-110) 115.8 (90-110)	All water samples in SDG F8F130140	J+ (all detects) J+ (all detects)	P

## III. Blanks

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

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
PB (prep blank)	Antimony Iron	0.89 ug/L 21.5 ug/L	All water samples in SDG F8F130140
ICB/CCB	Antimony Cadmium Vanadium	1.3 ug/L 0.1 ug/L 2.7 ug/L	All water samples in SDG F8F130140
ICB/CCB	Antimony Thallium Tungsten Vanadium Lithium Mercury	1.3 ug/L 1.1 ug/L 1.5 ug/L 2.7 ug/L 8.0 ug/L 0.1 ug/L	All soil samples in SDG F8F130140

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-CJ-09-0	Tungsten	0.54 mg/Kg	1.0U mg/Kg
TSB-CJ-09-10**	Thallium Tungsten Mercury	0.40 mg/Kg 1.1 mg/Kg 21.2 ug/Kg	0.44U mg/Kg 1.1U mg/Kg 36.5U ug/Kg

Sample RINSATE-2 was identified as a rinsate. No metal contaminants were found in this blank with the following exceptions:

Rinsate ID	Sampling Date	Analyte	Concentration	Associated Samples
RINSATE-2	6/12/08	Calcium Iron Magnesium Sodium Strontium Thallium	48.2 ug/L 59.1 ug/L 6.1 ug/L 11.0 ug/L 0.80 ug/L 1.5 ug/L	All soil samples in SDG F8F130140

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

Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-CJ-09-10**	Thallium	0.40 mg/Kg	0.44U mg/Kg

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

The frequency of analysis was met.

The criteria for analysis were met with the following exceptions:

ICS ID	Analyte	%R (Limits)	Associated Samples	Flag	A or P
ICSAB (6/17/08)	Sulfur	120.2 (80-120)	All water samples in SDG F8F130140	None	P

#### 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-CJ-09-0MS/MSD (All soil samples in SDG F8F130140)	Silicon Titanium Potassium Zinc	393.7 (75-125) 237.7 (75-125) - -	361.5 (75-125) 300.9 (75-125) 128.9 (75-125) 125.7 (75-125)	- - - -	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	A
TSB-CJ-09-0MS/MSD (All soil samples in SDG F8F130140)	Magnesium	64.6 (75-125)	161.1 (75-125)	-	J (all detects) UJ (all non-detects)	A
TSB-CJ-09-0MS/MSD (All soil samples in SDG F8F130140)	Antimony Mercury Strontium Niobium	53.5 (75-125) 52.6 (75-125) - 42.1 (75-125)	55.4 (75-125) - 74.8 (75-125) 46.5 (75-125)	- - - -	J- (all detects) UJ (all non-detects)	A

## VI. Duplicate Sample Analysis

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

## VII. Laboratory Control Samples (LCS)

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

LCS ID	Analyte	%R (Limits)	Associated Samples	Flag	A or P
LCS	Palladium	81.0 (85-115)	All water samples in SDG F8F130140	J- (all detects) UJ (all non-detects)	P

## VIII. Internal Standards (ICP-MS)

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

Sample	Internal Standard	%R (Limits)	Analyte	Flag	A or P
TSB-CJ-09-10**	Scandium-45	129.434 (30-120)	Silicon  Strontium	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A

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

## IX. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

## X. ICP Serial Dilution

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

Diluted Sample	Analyte	%D (Limits)	Associated Samples	Flag	A or P
TSB-GJ-08-10'L	Iron	10.4 ( $\leq 10$ )	All soil samples in SDG F8F130140	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 C  
Metals - Data Qualification Summary - SDG F8F130140**

SDG	Sample	Analyte	Flag	A or P	Reason
F8F130140	RINSATE-2	Thallium Uranium	J+ (all detects) J+ (all detects)	P	Calibration (CCV %R)
F8F130140	RINSATE-2	Sulfur	None	P	ICP interference check sample analysis (%R)
F8F130140	TSB-CJ-09-0 TSB-CJ-09-10**	Silicon Titanium Potassium Zinc	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8F130140	TSB-CJ-09-0 TSB-CJ-09-10**	Magnesium	J (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8F130140	TSB-CJ-09-0 TSB-CJ-09-10**	Antimony Mercury Strontium Niobium	J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8F130140	RINSATE-2	Palladium	J- (all detects) UJ (all non-detects)	P	Laboratory control samples (%R)
F8F130140	TSB-CJ-09-10**	Silicon  Strontium	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A	Internal standards (%R)
F8F130140	TSB-CJ-09-0 TSB-CJ-09-10**	Iron	J (all detects)	A	ICP serial dilution (%D)

**BRC Tronox Parcel C  
Metals - Laboratory Blank Data Qualification Summary - SDG F8F130140**

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8F130140	TSB-CJ-09-0	Tungsten	1.0U mg/Kg	A
F8F130140	TSB-CJ-09-10**	Thallium Tungsten Mercury	0.44U mg/Kg 1.1U mg/Kg 36.5U ug/Kg	A

**BRC Tronox Parcel C  
Metals - Field Blank Data Qualification Summary - SDG F8F130140**

<b>SDG</b>	<b>Sample</b>	<b>Analyte</b>	<b>Modified Final Concentration</b>	<b>A or P</b>
F8F130140	TSB-CJ-09-10**	Thallium	0.44U mg/Kg	A



LDC #: 19191A4  
 SDG #: F8F130140  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 8/5/08  
 Page: 1 of 1  
 Reviewer: MY  
 2nd Reviewer: 9

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/12/08
II.	Calibration	SW	
III.	Blanks	SW	
IV.	ICP Interference Check Sample (ICS) Analysis	SW	
V.	Matrix Spike Analysis	SW	3MS/MSD
VI.	Duplicate Sample Analysis	N	
VII.	Laboratory Control Samples (LCS)	SW	LCS
VIII.	Internal Standard (ICP-MS)	SW	N.t reviewed for level 3
IX.	Furnace Atomic Absorption QC	N	N.t 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	SW	R=1

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	RINSATE-2	A2	11	PB	21		31
2	TSB-CJ-09-0	S <sub>2</sub> -1	12		22		32
3	TSB-CJ-09-10**		13		23		33
4	TSB-CJ-09-0MS		14		24		34
5	TSB-CJ-09-0MSD		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 #: 19191A4  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

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

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 #: 19/91 A/C  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: MM  
 2nd Reviewer: 9

Validation Area	Yes	No	NA	Findings/Comments
<b>VII: ICP Serial Dilution</b>				
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the IDL?	<input checked="" type="checkbox"/>			> 100x MOC for 20/1/13
Were all percent differences (%Ds) < 10%?		<input checked="" type="checkbox"/>		
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.		<input checked="" type="checkbox"/>		
<b>VIII: Internal Standards (EPA SW-846 Method 8020)</b>				
Were all the percent recoveries (%R) within the 30-120% of the intensity of the internal standard in the associated initial calibration?	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>		
If the %Rs were outside the criteria, was a reanalysis performed?		<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>IX: Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?			<input checked="" type="checkbox"/>	
Were the performance evaluation (PE) samples within the acceptance limits?			<input checked="" type="checkbox"/>	
<b>X: Sample Result Verification</b>				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>			
<b>XI: Overall assessment of field</b>				
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>			
<b>XII: Field duplicates</b>				
Field duplicate pairs were identified in this SDG.		<input checked="" type="checkbox"/>		
Target analytes were detected in the field duplicates.			<input checked="" type="checkbox"/>	
<b>XIII: Field blanks</b>				
Field blanks were identified in this SDG.	<input checked="" type="checkbox"/>			
Target analytes were detected in the field blanks.	<input checked="" type="checkbox"/>			

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

All circled elements are applicable to each sample.

Sample ID	Matrix	Target Analyte List (TAL)
1-3	As/soil	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
m4-5	soil	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
1-3	As/soil	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
m4-5	soil	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
<b>Analysis Method</b>		
ICP		Li, S,
ICP-MS		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
ICP-MS		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Zr,
GFAA		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN

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

LDC #: 1919/46  
 SDG #: See over

# VALIDATION FINDINGS WORKSHEET

## Calibration

Page: 1 of 1  
 Reviewer: dy  
 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 Were all instruments calibrated daily, each set-up time, and were the proper number of standards used?  
 Y  N/A Were all initial and continuing calibration verification percent recoveries (%R) within the control limits of 90-110% for all analytes except mercury (80-120%) and cyanide (85-115%)?

**LEVEL IV ONLY:**  
 Y  N  NA Was a midrange cyanide standard distilled?  
 Y  N  NA Are all correlation coefficients  $\geq 0.995$ ?  
 Y  N  NA Were recalculated results acceptable? See Level IV Initial and Continuing Calibration Recalculation Worksheet for recalculations.

#	Date	Calibration ID	Analyte	%R	Associated Samples	Qualification of Data
1	6/5/08	CCV (2230)	TEL	113.4	A11 A2	It hit P
			U	115.8	↓	↓

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





**VALIDATION FINDINGS WORKSHEET**  
Field Blanks

METHOD: Trace Metals (EPA SW846 6010B/6020/7000)

N/A Were field blanks identified in this SDG?  
 N/A Were target analytes detected in the field blanks?

Blank units: ug/L Associated sample units: mg/Kg

Sampling date: 6/12/08 Soil factor applied: [Blank]

Field blank type: (circle one) Field Blank / Rinsate / Other: R Associated Samples: All Soil

Analyte	Blank ID	Action Level	Sample Identification												
	1	3													
Ca	48.2														
Fe	59.1	118.2													
Mg	6.1														
Na	11.0														
Sr	0.80														
Tl	1.5	0.40 / 0.44													

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:  
Samples with analyte concentrations within five times the associated field blank concentration are listed above, these sample results were qualified as not detected, "U".



LDC #: 19191AY  
SDG #: See work

Page: 1 of 1  
Reviewer: MH  
2nd Reviewer: CH

**VALIDATION FINDINGS WORKSHEET**  
ICP Interference Check Sample

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".  
(Y) (N) (N/A) Were ICP interference check samples performed as required?  
(Y) (N) (N/A) Were the AB solution percent recoveries (%R) within the control limits of 80-120%?

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

#	Date	ICS Identification	Analyte	Finding	Associated Samples	Qualifications
1	6/17/08	ICS4B	S	120.2	All AA	wore P (Residue, No Interferants)

Comments:

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

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 Was a matrix spike analyzed for each matrix in this SDG?  
 N N/A Were matrix spike percent recoveries (%R) within the control limits of 75-125? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.

N N/A Were all duplicate sample relative percent differences (RPD)  $\leq 20\%$  for water samples and  $\leq 35\%$  for soil samples?  
 N N/A Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations.

#	MS/MSD ID	Matrix	Analyte	MS %Recovery	MSD %Recovery	RPD (Limits)	Associated Samples	Qualifications
1	4/5	Soil	Sb	53.5	55.4		A1 Soil	J-WS/A
			Mg	64.6	161.1			J-WS/A
			Nb	42.1	46.5			J-WS/A
			Si	393.7	361.5			J-WS/A
			Ti	239.7	300.9			J-WS/A
			Hg	52.6	128.9			J-WS/A
			Kd					J-WS/A
			Sv		74.8			J-WS/A
			Zn <del>30K</del>		125.9			J-WS/A
			Mg			23.6		No qual (LCSIR)
			Mg			36.4		
			Hg			37.1		

Comments: Al, Co, Fe, Mn, 74X

LDC #: 19191A4  
 SDG #: see cover

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

Page: 1 of 1  
 Reviewer: MM  
 2nd Reviewer: C

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 Was a laboratory control sample (LCS) analyzed for each matrix in this SDG?

N N/A Were all aqueous LCS percent recoveries (%R) within the control limits of 80-120% and all soil LCS %R within laboratory established control limits. *Let's try it*

**LEVEL IV ONLY:**

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

#	LCS ID	Matrix	Analyte	%R (limits)	Associated Samples	Qualifications
1	LCS	As	Pd	87.0 (85-115)	A1 AA	J-107/P

Comments:

LDC #: 1919/AY  
 SDG #: See above

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

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

METHOD: Metals (EPA SW 846 Method 6020)

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

Y (N) N/A If the response to either of the above questions is no, were the samples reanalyzed as required ?

#	Internal Standard	Associated Metals	%R (Limits)	Associated Samples	Qualifications
1	S <sub>2</sub> 45	129.434 Si, Sr	129.434	3	J/MJ/A

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

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".  
 N N/A If analyte concentrations were > 50X the MDL (ICP), or >100X the MDL (ICP/MS), was a serial dilution analyzed?  
 Y N/A Were ICP serial dilution percent differences (%D) ≤10%?  
 Y N/A Is there evidence of negative interference? If yes, professional judgement will be used to qualify the data.

**LEVEL IV ONLY:**

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

#	Date	Diluted Sample ID	Matrix	Analyte	%D (Limits)	Associated Samples	Qualifications
1		T53-9J-08-101	Soil	Fe	10.4	A1 Soil	JLF/A

Comments: None

LDC #: 19191A4  
 SDG #: See cover

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

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

**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 =  $\frac{\text{Found} \times 100}{\text{True}}$  Where, Found = concentration (in ug/L) of each analyte measured in the analysis of the ICV or CCV solution  
 True = concentration (in ug/L) of each analyte in the ICV or CCV source

Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	Recalculated		Reported		Acceptable (Y/N)
					%R	%R	%R	%R	
ICV	ICP (Initial calibration)	Li	4067	4000	101.7	101.7	101.7	101.7	Y
	GFAA (Initial calibration)								
ICV	CVAA (Initial calibration)	Hg	2.33	2.50	93.2	93.2	93.2	93.2	Y
CCV	ICP (Continuing calibration)	S	52670	50000	105.3	105.3	105.3	105.3	↓
	GFAA (Continuing calibration)								
CCV	CVAA (Continuing calibration)	Hg	4.89	5.0	97.8	97.8	97.8	97.8	Y
ICV	ICP/MS (Initial calibration)	Mn	1018.9	1000	101.9	101.9	101.9	101.9	↓
CCV	ICP/MS (Continuing calibration)	Sn	1072	1000	107.2	107.2	107.2	107.2	↓

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 #: 1919/AN  
 SDG #: Self cover

Page: 1 of 1  
 Reviewer: AM  
 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		Acceptable (Y/N)
					%R / RPD / %D	%R / RPD / %D	
TESA3	ICP interference check	Ag	19.106	20	95.5	95.5	Y
LC3	Laboratory control sample	Ba	488	520	93.8	93.9	
4	Matrix spike	Li (SSR-SR)	105.1	104.3	100.8	100.8	
4/5	Duplicate	Cu	24.5	22.5	8.5	8.5	
T3B-6J-08-101	ICP serial dilution	Ca	67207	62288	9.9	9.9	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 #: 19191 A40  
 SDG #: see cover

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

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

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

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

- Y  N  N/A Have results been reported and calculated correctly?
- Y  N  N/A Are results within the calibrated range of the instruments and within the linear range of the ICP?
- Y  N  N/A Are all detection limits below the CRDL?

Detected analyte results for 3 were recalculated and verified using the following equation:

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

Recalculation:

$$B = \frac{18093 \mu\text{g/L} \times 0.1 \text{ L} \times 2}{0.58 \times 0.9132} = 79163 \mu\text{g/kg}$$

- 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 (µg/kg)	Calculated Concentration (µg/kg)	Acceptable (Y/N)
3	Al	9530	9530	Y
	As	10.6	10.6	
	Ba	91.6	91.6	
	Be	0.58	0.58	
	B	1.9	1.9	
	Cd	0.083	0.083	
	Ca	30100	30100	
	Cv	12.1	12.1	
	Co	6.7	6.7	
	Cu	13.8	13.8	
	Fe	13000	13000	
	Pb	8.3	8.3	
	Mg	9800	9800	
	Mn	312	312	
	Mo	0.45	0.45	
	Ni	14.1	14.1	
	Pd	0.57	0.57	
	P	740	740	
K	1770	1770		
Si	523	523		
Ag	0.15	0.15		
Na	1820	1820	✓	



LDC #: 19191A4  
SDG #: cer cover

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

Page: 2 of 9  
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".

- Y  N  N/A Have results been reported and calculated correctly?
- Y  N  N/A Are results within the calibrated range of the instruments and within the linear range of the ICP?
- Y  N  N/A Are all detection limits below the CRDL?

Detected analyte results for 3 were recalculated and verified using the following equation:

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

Recalculation:

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

$$Zr = \frac{59.735 \times 0.12 \times 2}{0.59 \times 0.9132} = 25.29 \text{ } \mu\text{g}/\mu\text{g}$$

Sample ID	Analyte	Reported Concentration ( $\mu\text{g}/\mu\text{g}$ )	Calculated Concentration ( $\mu\text{g}/\mu\text{g}$ )	Acceptable (Y/N)
<u>3</u>	<u>SV</u>	<u>291</u>	<u>291</u>	<u>Y</u>
	<u>TR</u>	<u>0.40</u>	<u>0.40</u>	<u>↑</u>
	<u>SN</u>	<u>0.55</u>	<u>0.55</u>	
	<u>Ti</u>	<u>593</u>	<u>593</u>	
	<u>W</u>	<u>1.1</u>	<u>1.1</u>	
	<u>Y</u>	<u>2.1</u>	<u>2.1</u>	
	<u>✓</u>	<u>40.2</u>	<u>40.2</u>	
	<u>Zn</u>	<u>330</u>	<u>330</u>	
	<u>Zr</u>	<u>25.3</u>	<u>25.3</u>	
	<u>Hg (<math>\mu\text{g}/\mu\text{g}</math>)</u>	<u>21.2</u>	<u>21.2</u>	<u>✓</u>

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

**Project/Site Name:** BRC Tronox Parcel C  
**Collection Date:** June 12, 2008  
**LDC Report Date:** August 7, 2008  
**Matrix:** Soil/Water  
**Parameters:** Wet Chemistry  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8F130140

**Sample Identification**

RINSATE-2  
TSB-CJ-09-0  
TSB-CJ-09-10\*\*  
RINSATE-2MS  
RINSATE-2DUP  
TSB-CJ-09-0MS  
TSB-CJ-09-0MSD  
TSB-CJ-09-0DUP

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 5 soil samples and 3 water 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 Method 1664A 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
CCB1	Orthophosphate as P	0.102 mg/L	TSB-CJ-09-0
CCB2	Orthophosphate as P	0.126 mg/L	TSB-CJ-09-10**

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

Sample RINSATE-2 was identified as a rinsate. No contaminant concentrations were found in this blank with the following exceptions:

Rinsate ID	Sampling Date	Analyte	Concentration	Associated Samples
RINSATE-2	6/12/08	Sulfate	0.11 mg/L	All soil samples in SDG F8F130140

Sample concentrations were compared to concentrations detected in the field blanks as required by the QAPP. No sample data was qualified.

#### 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:

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
TSB-CJ-09-0MS/MSD (All soil samples in SDG F8F130140)	Oil & 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) and relative percent differences (RPD) 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 C  
Wet Chemistry - Data Qualification Summary - SDG F8F130140**

<b>SDG</b>	<b>Sample</b>	<b>Analyte</b>	<b>Flag</b>	<b>A or P</b>	<b>Reason</b>
F8F130140	TSB-CJ-09-0 TSB-CJ-09-10**	Oil & grease	J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)

**BRC Tronox Parcel C  
Wet Chemistry - Laboratory Blank Data Qualification Summary - SDG F8F130140**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel C  
Wet Chemistry - Field Blank Data Qualification Summary - SDG F8F130140**

No Sample Data Qualified in this SDG

LDC #: 19191A6  
 SDG #: F8F130140  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

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

**METHOD: (Analyte)** Bromide, Bromine, Chlorate, Chloride, Chlorine, Fluoride, Nitrate, Nitrite, Orthophosphate-P, Sulfate (EPA Method 300.0), O & G (EPA SW846 Method 9071B/EPA 1664A)

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: 6/12/08
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/LCSD
VII.	Sample result verification	A	Not reviewed for Level III validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N	
X	Field blanks	SW	R=1

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	RINSATE-2	A2	11		21		31	
2	TSB-CJ-09-0	Soil	12		22		32	
3	TSB-CJ-09-10**	↓	13		23		33	
4	RINSATE-2MS	A2	14		24		34	
5	RINSATE-2DUP	↓	15		25		35	
6	TSB-CJ-09-0MS	Soil	16		26		36	
7	TSB-CJ-09-0MSD	↓	17		27		37	
8	TSB-CJ-09-0DUP	↓	18		28		38	
9	MS		19		29		39	
10			20		30		40	

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



LDC #: 19191A6  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2  
 Reviewer: WEM  
 2nd Reviewer: ✓

Method: Inorganics (EPA Method See cover)

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical Holding Times</b>				
All technical holding times were met.	✓			
Cooler temperature criteria was met.	✓			
<b>II. Calibration</b>				
Were all instruments calibrated daily, each set-up time?	✓			
Were the proper number of standards used?	✓			
Were all initial calibration correlation coefficients > 0.995?	✓			
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?	✓			
Were titrant checks performed as required? (Level IV only)			✓	
Were balance checks performed as required? (Level IV only)	✓			
<b>III. Blanks</b>				
Was a method blank associated with every sample in this SDG?	✓			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	✓			
<b>IV. Matrix Spike 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 #: 19191A6  
 SDG #: *See cover*

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: *MM*  
 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?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were detection limits < RL?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>Overall assessment of data was found to be acceptable.</b>				
Field duplicate pairs were identified in this SDG.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Target analytes were detected in the field duplicates.	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>Field blanks were identified in this SDG.</b>				
Target analytes were detected in the field blanks.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

LDC #: 1919/A6  
 SDG #: See cover

**VALIDATION FINDINGS WORKSHEET**  
**Sample Specific Analysis Reference**

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

All circled methods are applicable to each sample.

Sample ID	Matrix	Parameter
1-3	Soil/Aa	Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
m 4.5	Aa	Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
1 6.7	Soil	Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
4 6-8	↓	Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH

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

VALIDATION FINDINGS WORKSHEET  
Blanks

LDC #: 1996A6  
SDG #: See com  
METHOD: Inorganics, Method: See com

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

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

Conc. units: mg/kg Associated Samples: CB1 = 2, CB2 = 3 (MB)

Analyte	Blank ID	Maximum ICB/QCB mg/L	Blank Action Limit	Sample Identification																	
<del>CB1</del>		0.102																			
0-Pb4-P																					
0-Pb4-P		0.176																			

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

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

LDC #: 1919/A6  
SDG #: See cover

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

METHOD: Inorganics, EPA Method See cover  
 N/A Were field blanks identified in this SDG?  
 N/A Were target analytes detected in the field blanks?  
 Blank units: mg/L Associated sample units: mg/kg  
 Sampling date: 6/12/08 Soil factor applied  
 Field blank type: (circle one) Field Blank / Rinse / Other: R Associated Samples: Art soils (>5x)

Analyte	Blank ID	Blank Action Limit	Sample Identification
504	1		
	0.11		

Blank units: Associated sample units:  
 Sampling date: Soil factor applied  
 Field blank type: (circle one) Field Blank / Rinse / Other: Associated Samples:

Analyte	Blank ID	Blank Action Limit	Sample Identification

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:  
 Samples with analyte concentrations within five times the associated field blank concentration are listed above, these sample results were qualified as not detected, "U".

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

METHOD: Inorganics, EPA Method See cover

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

- Y N N/A Was a matrix spike analyzed for each matrix in this SDG?
- Y N N/A Were matrix spike percent recoveries (%R) within the control limits of 75-125? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.
- Y N N/A Were all duplicate sample relative percent differences (RPD)  $\leq$  20% for water samples and  $\leq$  35% for soil samples?
- Y N N/A Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations.

**LEVEL IV ONLY:**

#	MS/MSD ID	Matrix	Analyte	MS %Recovery	MSD %Recovery	RPD (Limits)	Associated Samples	Qualifications
1	619	Soil	OTG	63	63		All soil	J-UG/A

Comments:

LDC #: 1919166

SDG #: See cover

Validatin Findings Worksheet  
Initial and Continuing Calibration Calculation Verification

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

Method: Inorganics, Method See cover

The correlation coefficient (r) for the calibration of  $ClO_3^-$  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	r <sup>2</sup>	r	r <sup>2</sup>	
Initial calibration	ClO3	s1	500	0.034	0.99996	0.99991			Y
		s2	2000	0.151					
		s3	4000	0.317					
		s4	10000	0.777					
		s5	20000	1.586					
Calibration verification	ClO3	4	2,851		96.3		NR		Y
Calibration verification	0-1000	8	8,002		100.02		100.03		Y
Calibration verification	100-11	0.160	0,1634		102.13		102.14		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.

**VALIDATION FINDINGS WORKSHEET**  
Level IV Recalculation Worksheet

METHOD: Inorganics, Method Cecl cover

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

$\%R = \frac{\text{Found}}{\text{True}} \times 100$  Where, Found = concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation, 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	OTG	1250	1330	94	94	Y
6	Matrix spike sample	BY	(SSR-SFI) 9.98 <del>20.9</del>	20.9	96	96	Y
8	Duplicate sample	ce	51.755	51.5722	0.35	0.35	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 #: 1919/186  
SDG #: see cover

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

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

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?
- X N N/A Are all detection limits below the CRQL?

Compound (analyte) results for 3 reported with a positive detect were recalculated and verified using the following equation:

Concentration =

$$F = \frac{\text{Area}}{0.287}$$

Recalculation:

$$F = \frac{0.215 \times 40 \text{ mL}}{0.287 \times 4g \times 0.913} = 8.2 \text{ mg/kg}$$

#	Sample ID	Analyte	Reported Concentration (mg/kg)	Calculated Concentration (mg/kg)	Acceptable (Y/N)
1	3	ClO <sub>3</sub>	16.4	16.4	Y
		Cl	900	900	↓
		Cl <sub>2</sub>	1800	1800	↓
		F	8.2	8.2	↓
		NO <sub>3</sub> -N	1.9	1.9	↓
		SO <sub>4</sub>	170	170	↓

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

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

**Project/Site Name:** BRC Tronox Parcel C  
**Collection Date:** June 12, 2008  
**LDC Report Date:** August 6, 2008  
**Matrix:** Soil/Water  
**Parameters:** Gasoline Range Organics  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8F130140

**Sample Identification**

RINSATE-2  
TSB-CJ-09-0  
TSB-CJ-09-10\*\*  
RINSATE-2MS  
RINSATE-2MSD  
TSB-CJ-09-0MS  
TSB-CJ-09-0MSD

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 4 soil samples and 3 water 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 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.
- 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) 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.

Sample RINSATE-2 was identified as a rinsate. No gasoline range organic contaminants were found in this blank.

## **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 an 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 an 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 an 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 C  
Gasoline Range Organics - Data Qualification Summary - SDG F8F130140**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel C  
Gasoline Range Organics - Laboratory Blank Data Qualification Summary - SDG  
F8F130140**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel C  
Gasoline Range Organics - Field Blank Data Qualification Summary - SDG  
F8F130140**

No Sample Data Qualified in this SDG

LDC #: 19191A7  
 SDG #: F8F130140  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 8/15/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: <u>6/12/08</u>
IIa.	Initial calibration	A	
IIb.	Calibration verification/ICV	A	<u>ICV = 15%</u>
III.	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	A	
IVc.	Laboratory control samples	A	<u>LCS 7</u>
V.	Target compound identification	A	Not reviewed for Level III validation.
VI.	Compound Quantitation and CRQLs	A	Not reviewed for Level III validation.
VII.	System Performance	A	Not reviewed for Level III validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N	
X.	Field blanks	ND	<u>E = 1</u>

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	RINSATE-2	W	11	<u>8168739MB</u>	21		31
2	TSB-CJ-09-0	S	12	<u>8168741MB</u>	22	<u>S</u>	32
3	TSB-CJ-09-10**	↓	13		23		33
4	RINSATE-2MS	W	14		24		34
5	RINSATE-2MSD	↓	15		25		35
6	TSB-CJ-09-0MS	S	16		26		36
7	TSB-CJ-09-0MSD	↓	17		27		37
8			18		28		38
9			19		29		39
10			20		30		40

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



LDC #: 19191A7  
 SDG #: SecDown

VALIDATION FINDINGS CHECKLIST

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

Method:  GC  HPLC

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	/			
Cooler temperature criteria was met.	/			
<b>II. Initial calibration</b>				
Did the laboratory perform a 5 point calibration prior to sample analysis?	/			
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?	/			
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?			/	
Did the initial calibration meet the curve fit acceptance criteria?	/			
Were the RT windows properly established?	/			
<b>III. Continuing calibration</b>				
What type of continuing calibration calculation was performed? <u>  </u> %D or %R	/			
Was a continuing calibration analyzed daily?	/			
Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?	/			
Were all the retention times within the acceptance windows?	/			
<b>IV. Blanks</b>				
Was a method blank associated with every sample in this SDG?	/			
Was a method blank analyzed for each matrix and concentration?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.			/	
<b>V. Surrogate spikes</b>				
Were all surrogate %R within the QC limits?	/			
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?			/	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?			/	
<b>VI. Matrix spike/Matrix spike duplicates</b>				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			
Was a MS/MSD analyzed every 20 samples of each matrix?	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	/			
<b>VII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	/			
Was an LCS analyzed per extraction batch?	/			

LDC #: 19191AT  
 SDG #: 2000WY

VALIDATION FINDINGS CHECKLIST

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

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 checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Were target compounds detected in the field blanks?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	

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

LDC #: 1919A7  
 SDG #: See below

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

METHOD: GC  HPLC \_\_\_\_\_

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

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

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

#	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	<u>10A2</u>	<u>5/27/08</u>	<u>FRD</u>	<u>18352700</u>	<u>18352700</u>	<u>17182732</u>	<u>17182732</u>	<u>3.915</u>	<u>3.915</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.

**VALIDATION FINDINGS WORKSHEET**  
Continuing Calibration Results Verification

METHOD: GC  HPLC \_\_\_\_\_

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

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

#	Standard ID	Calibration Date	Compound	Average CF (Ical)/ CCV Conc.	Reported		Recalculated	
					CF/Conc. CCV	%D	CF/Conc. CCV	%D
1	<del>LCAL-1201B</del> SPD	6/16/18		1.0	1.0360	3.6	1.0360	3.6
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 #: 1191A7  
 SDG #: 200000

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

Page: 6 of 7  
 Reviewer: [Signature]  
 2nd reviewer: [Signature]

METHOD: VGC 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: 3

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
JFT	AS	0.04	0.03592	90	90	0

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

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

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

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

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

LDC #: 899A7  
 SDG #: 22.com

METHOD:  GC  HPLC

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

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

RPD =  $\frac{((SSCMS - SSCMSD) * 2) / (SSCMS + SSCMSD)}{100} * 100$  MSD = Matrix spike duplicate

MS/MSD samples: 6/7

Compound	Spike Added (MS/MSD)		Sample Conc. (MS/MSD)	Spike Sample Concentration (MS/MSD)		Matrix spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		MS/MSD RPD	
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)	1.04	1.04	ND	0.95	0.958	90	90	90	90	0.30	0.30
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 Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

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

LCS/LCSD samples: 8168141

Compound	Spike Added (mg/L)		Spiked Sample Concentration (mg/L)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)	1.0	1.0	0.957	0.918	95	95	92	92	3.9	3.9
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.

LDC #: 199A  
 SDG #: Sec 201

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

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

METHOD:  GC  HPLC

Y/N N/A  
 Y/N N/A

Were all reported results recalculated and verified for all level IV samples?  
 Were all recalculated results for detected target compounds agree within 10% of the reported results?

Concentration =  $\frac{(A)(Fv)(Df)}{(RF)(Vs \text{ or } Ws)(\%S/100)}$   
 Example: Sample ID B Compound Name Nb 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 C  
**Collection Date:** June 12, 2008  
**LDC Report Date:** August 6, 2008  
**Matrix:** Soil/Water  
**Parameters:** Diesel Range Organics  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8F130140

**Sample Identification**

RINSATE-2  
TSB-CJ-09-0  
TSB-CJ-09-10\*\*  
RINSATE-2MS  
RINSATE-2MSD  
TSB-CJ-09-0MS  
TSB-CJ-09-0MSD

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 4 soil samples and 3 water 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 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.
- 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) 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.

Sample RINSATE-2 was identified as a rinsate. No diesel range organic contaminants were found in this blank.

## **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 an 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 an 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 an 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 C  
Diesel Range Organics - Data Qualification Summary - SDG F8F130140**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel C  
Diesel Range Organics - Laboratory Blank Data Qualification Summary - SDG  
F8F130140**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel C  
Diesel Range Organics - Field Blank Data Qualification Summary - SDG  
F8F130140**

No Sample Data Qualified in this SDG

LDC #: 19191A8  
 SDG #: F8F130140  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**

Level III/IV

Date: 6/15/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/12/08
IIa.	Initial calibration	A	
IIb.	Calibration verification/ICV	A	ICV = 1570
III.	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	A	
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	ND	R=1 -

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	RINSATE-2	W	11	8169241NB	21		31	
2	TSB-CJ-09-0	S	12	8170312MB	22		32	
3	TSB-CJ-09-10**	W	13		23		33	
4	RINSATE-2MS	W	14		24		34	
5	RINSATE-2MSD	W	15		25		35	
6	TSB-CJ-09-0MS	S	16		26		36	
7	TSB-CJ-09-0MSD	W	17		27		37	
8			18		28		38	
9			19		29		39	
10			20		30		40	

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

LDC #: 19A18  
 SDG #: SA-COW

**VALIDATION FINDINGS CHECKLIST**

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

Method:  GC  HPLC

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>II. Initial calibration</b>				
Did the laboratory perform a 5 point calibration prior to sample analysis?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?	<input type="checkbox"/>	<input 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? <input checked="" type="checkbox"/> %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 #: 19191A8  
 SDG #: See down

**VALIDATION FINDINGS CHECKLIST**

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

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 #: 1991A8  
 SDG #: Beckon

**VALIDATION FINDINGS WORKSHEET**  
Initial Calibration Calculation Verification

Page: 6 of 8  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

METHOD: GC  HPLC \_\_\_\_\_

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

CF = A/C  
 average CF = sum of the CF/number of standards  
 %RSD =  $100 * (S/X)$   
 A = Area of compound,  
 C = Concentration of compound,  
 S = Standard deviation of the CF  
 X = Mean of the CFs

#	Standard ID	Calibration Date	Compound	Reported		Recalculated		Reported		Recalculated	
				CF (100 std)	CF (102 std)	Average CF (initial)	Average CF (initial)	%RSD	%RSD		
1	10AC	5/16/08	DR 0	15394	15394	16023	16023	3.456	3.456	3.456	3.456
2											
3											
4											

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

LDC #: 1919188  
 SDG #: See COM

**VALIDATION FINDINGS WORKSHEET**  
Continuing Calibration Results Verification

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

METHOD: GC  HPLC \_\_\_\_\_

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

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

#	Standard ID	Calibration Date	Compound	Average CF (Ical)/ CCV Conc.	Reported		Recalculated	
					CF/Conc. CCV	%D	CF/Conc. CCV	%D
1	<u>20A2575</u>	<u>6/19/08</u>	<u>DRD</u>	<u>1000</u>	<u>979.873</u>	<u>2.0</u>	<u>979.80</u>	<u>2.0</u>
2	<u>20A2586</u>	<u>6/19/08</u>	<u>DRD</u>	<u>1000</u>	<u>1005.376</u>	<u>0.5</u>	<u>1005.32</u>	<u>0.5</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 #: 199AB3  
 SDG #: 200000

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

Page: 6 of 7  
 Reviewer: [Signature]  
 2nd reviewer: [Signature]

METHOD: ✓ GC     HPLC

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

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

Sample ID: 3

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery		Percent Difference
				Reported	Recalculated	
TPH	NS	0.50	21.8080	87	87	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 differences (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation:

%Recovery =  $100 * ((SC - SA) / SA)$  Where SSC = Spiked sample concentration, SA = Spike added, MS = Matrix spike, SC = Sample concentration, MSD = Matrix spike duplicate

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

MS/MSD samples: 6/7

Compound	Spike Added (MS/MSD)		Sample Conc. (MS/MSD)	Spike Sample Concentration (MS/MSD)		Matrix spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		MS/MSD RPD	
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)											
Diesel (8015)	85.8	86.6	ND	76.1	71.0	89	89	92	82	69	69
Benzene (8021B)											
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)											
Anthracene (8310)											
HMX (8330)											
2,4,6-Trinitrotoluene (8330)											

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

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: 8170312

Compound	Spike Added (1158)		Spiked Sample Concentration (1158)		LCS		LCSD		Percent Recovery		LCS/LCSD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)												
Diesel (8015)	83.3	NA	73.6	NA	88	88						
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.

LDC #: 1977AS  
 SDG #: 2e con

**VALIDATION FINDINGS WORKSHEET**  
Sample Calculation Verification

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

METHOD:  GC  HPLC

Y/N N/A  
 Y/N N/A

Were all reported results recalculated and verified for all level IV samples?  
 Were all recalculated results for detected target compounds agree within 10% of the reported results?

Concentration =  $\frac{(A)(Ev)(Df)}{(RF)(Vs \text{ or } Ws)(\%S/100)}$  Example:  
 Sample ID: 3 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 C  
**Collection Date:** June 12, 2008  
**LDC Report Date:** August 6, 2008  
**Matrix:** Soil/Water  
**Parameters:** Polynuclear Aromatic Hydrocarbons  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8F130140

**Sample Identification**

RINSATE-2  
TSB-CJ-09-0  
TSB-CJ-09-10\*\*  
RINSATE-2MS  
RINSATE-2MSD  
TSB-CJ-09-0MS  
TSB-CJ-09-0MSD

\*\*Indicates sample underwent EPA Level IV review



## Introduction

This data review covers 4 soil samples and 3 water 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 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.
- 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 an 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.

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	Column ID	Compound	%D	Associated Samples	Flag	A or P
6/4/08 (QICV768)	Not specified	Benzo(k)fluoranthene	16.6	All samples in SDG F8F130140	J+ (all detects)	A

Retention time windows were evaluated and considered technically acceptable for samples on which an 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.

Sample RINSATE-2 was identified as a rinsate. No polynuclear aromatic hydrocarbon contaminants were found in this blank.

## **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 an 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 an 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 an 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 C  
 Polynuclear Aromatic Hydrocarbons - Data Qualification Summary - SDG F8F130140**

SDG	Sample	Compound	Flag	A or P	Reason
F8F130140	RINSATE-2 TSB-CJ-09-0 TSB-CJ-09-10**	Benzo(k)fluoranthene	J+ (all detects)	A	Continuing calibration (ICV %D)

**BRC Tronox Parcel C  
 Polynuclear Aromatic Hydrocarbons - Laboratory Blank Data Qualification Summary  
 - SDG F8F130140**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel C  
 Polynuclear Aromatic Hydrocarbons - Field Blank Data Qualification Summary -  
 SDG F8F130140**

No Sample Data Qualified in this SDG

LDC #: 19191A9  
 SDG #: F8F130140  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 6/5/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/12/08
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	
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	ND	R=1

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	RINSATE-2	W	11	81687/87 MB	21		31	
2	TSB-CJ-09-0	W	12	8170313 MB	22		32	
3	TSB-CJ-09-10**	W	13		23		33	
4	RINSATE-2MS	W	14		24		34	
5	RINSATE-2MSD	W	15		25		35	
6	TSB-CJ-09-0MS	W	16		26		36	
7	TSB-CJ-09-0MSD	W	17		27		37	
8			18		28		38	
9			19		29		39	
10			20		30		40	

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

VALIDATION FINDINGS WORKSHEET

METHOD: \_\_\_ GC \_\_\_ HPLC

8310	8330	8151	8141	8141(Con't)	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 #: 19191A9  
 SDG #: See cover

**VALIDATION FINDINGS CHECKLIST**

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

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"/>	<input checked="" type="checkbox"/>	
Did the initial calibration meet the curve fit acceptance criteria?	<input checked="" type="checkbox"/>			
Were the RT windows properly established?	<input checked="" type="checkbox"/>			
<b>III. Continuing calibration</b>				
What type of continuing calibration calculation was performed? <u>&lt;</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>IV. 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>V. 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>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"/>			
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>VII. 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 #: 19191A9  
 SDG #: See COMV

VALIDATION FINDINGS CHECKLIST

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

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 checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Were target compounds detected in the field blanks?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	



LDC #: 199189  
 SDG #: 2020W

**VALIDATION FINDINGS WORKSHEET**  
Initial Calibration Calculation Verification

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

METHOD: GC  HPLC

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

- CF = A/C
- average CF = sum of the CF/number of standards
- %RSD =  $100 * (S/X)$
- A = Area of compound,
- C = Concentration of compound,
- S = Standard deviation of the CF
- X = Mean of the CFs

#	Standard ID	Calibration Date	Compound	Reported		Recalculated		Reported		Recalculated	
				CF ( / std)	CF ( / std)	Average CF (Initial)	Average CF (Initial)	%RSD	%RSD	%RSD	%RSD
1	<u>10A/C</u>	<u>6/4/08</u>	<u>C</u>	<u>807269</u>	<u>807269</u>	<u>806710</u>	<u>806710</u>	<u>1.821</u>	<u>1.821</u>	<u>17.820</u>	<u>17.820</u>
			<u>#</u>	<u>67485</u>	<u>67485</u>	<u>6260.8</u>	<u>6260.8</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 #: 19191A9  
 SDG #: See down

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

Page: 6 of 9  
 Reviewer: \_\_\_\_\_  
 2nd Reviewer: g

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	<u>8CA2B1</u>	<u>6/19/08</u>	<u>C</u>	<u>0.50</u>	<u>0.4790</u>	<u>4.2</u>	<u>0.4790</u>	<u>4.2</u>
			<u>P</u>	<u>✓</u>	<u>0.4289</u>	<u>14.2</u>	<u>0.4289</u>	<u>14.2</u>
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 #: 191A9  
 SDG #: SecCom

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

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

METHOD: GC  HPLC

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

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

Sample ID: 3

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
TPH	NS	25	21.7512	87	87	0

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

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

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

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

METHOD: GC  HPLC

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

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

Where

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

SC = Sample concentration  
MSD = Matrix spike duplicate

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

MS/MSD samples: 6/7

Compound	Spike Added		Sample Conc. (MS)	Spike Sample Concentration (MS, MSD)		Matrix spike		Matrix Spike Duplicate		MS/MSD	
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)											
Diesel (8015)											
Benzene (8021B)											
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310) P	68.2	68.2	ND	55.1	56.0	81	81	82	82	1.7	1.6
Anthracene (8310)	↓	↓	↓	49.1	50.9	72	72	75	75	3.6	3.6
HMX (8330)											
2,4,6-Trinitrotoluene (8330)											

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

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

LCS/LCSD samples: 8170313

Compound	Spike Added		Spiked Sample Concentration		LCS		LCSD		Percent Recovery		Percent Recovery		RPD	
	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) <u>P</u>	<u>66.7</u>	<u>NA</u>	<u>56.6</u>	<u>NA</u>	<u>85</u>	<u>85</u>								
Anthracene (8310)	<u>↓</u>	<u>↓</u>	<u>53.9</u>	<u>↓</u>	<u>81</u>	<u>81</u>								
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 #: 19191A9  
 SDG #: Be con

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

METHOD: GC  HPLC

Y  N  N/A  
 Y  N  N/A

Were all reported results recalculated and verified for all level IV samples?  
 Were all recalculated results for detected target compounds agree within 10% of the reported results?

Concentration =  $\frac{(A)(Fv)(Df)}{(RF)(Vs \text{ or } Ws)(\%S/100)}$   
 Example: Sample ID: S Compound Name: NP

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 C  
**Collection Date:** June 12, 2008  
**LDC Report Date:** August 7, 2008  
**Matrix:** Soil/Water  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8F130140

**Sample Identification**

RINSATE-2  
TSB-CJ-09-0  
TSB-CJ-09-10\*\*  
TSB-CJ-09-0MS  
TSB-CJ-09-0MSD

\*\*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 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.
- 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. 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	Affected Compound	Flag	A or P
7/3/08 (02JL08B1D2_20)	<sup>13</sup> C-2,3,7,8-TCDF	57.3	TSB-CJ-09-0 TSB-CJ-09-0MS TSB-CJ-09-0MSD	2,3,7,8-TCDF	J+ (all detects)	P

Date	Compound	%D	Associated Samples	Affected Compound	Flag	A or P
7/3/08 (02JL08B1D2_32)	<sup>13</sup> C-2,3,7,8-TCDF	71.8	TSB-CJ-09-0 TSB-CJ-09-0MS TSB-CJ-09-0MSD	2,3,7,8-TCDF	J+ (all detects)	P
6/26/08	<sup>13</sup> C-OCDD	34.6	8175566MB	OCDD	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.

Sample RINSATE-2 was identified as a rinsate. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank.

## VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Although the MS/MSD percent recoveries (%R) and relative percent differences (RPD) were not within QC limits for some compounds, the LCS percent recovery (%R) was within QC limits and no data were qualified.

## 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
8172352LCS	1,2,3,7,8,9-HxCDD	129 (74-126)	All water samples in SDG F8F130140	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
RINSATE-2	<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	26 (40-135) 37 (40-135) 33 (40-135)	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,7,8-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	J (all detects) UJ (all non-detects)	P
TSB-CJ-09-10**	<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	25 (40-135) 32 (40-135) 14 (40-135) 16 (40-135) 13 (40-135)	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 1,2,3,4,7,8-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 with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P
TSB-CJ-09-0	2,3,7,8-TCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	P

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 this report if data has been qualified.

### **XIV. Field Duplicates**

No field duplicates were identified in this SDG.

**BRC Tronox Parcel C  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG F8F130140**

SDG	Sample	Compound	Flag	A or P	Reason
F8F130140	TSB-CJ-09-0	2,3,7,8-TCDF	J+ (all detects)	P	Routine calibration (%D)
F8F130140	RINSATE-2	1,2,3,7,8,9-HxCDD	J+ (all detects)	P	Laboratory control samples (%R)
F8F130140	RINSATE-2	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,7,8-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	J (all detects) UJ (all non-detects)	P	Internal standards (%R)
F8F130140	TSB-CJ-09-10**	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 1,2,3,4,7,8-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)
F8F130140	TSB-CJ-09-0	2,3,7,8-TCDF OCDF	J (all detects) J (all detects)	P	Compound quantitation and CRQLs

**BRC Tronox Parcel C  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG F8F130140**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel C  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG F8F130140**

No Sample Data Qualified in this SDG



LDC #: 19191A21  
 SDG #: F8F130140  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

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

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/12/08
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	W	
V.	Blanks	A	
VI.	Matrix spike/Matrix spike duplicates	W	
VII.	Laboratory control samples	W	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	W	
X.	Target compound identifications	A	Not reviewed for Level III validation.
XI.	Compound quantitation and CRQLs	W	Not reviewed for Level III validation.
XII.	System performance	A	Not reviewed for Level III validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	ND	R=1

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	RINSATE-2	W	11	8172252MB	21	31
2	TSB-CJ-09-0	S	12	8175566MB	22	32
3	TSB-CJ-09-10**		13		23	33
4	TSB-CJ-09-0MS		14		24	34
5	TSB-CJ-09-0MSD		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 #: 19191A21  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 3  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	/			
Cooler temperature criteria was met.	/			
<b>II. GC/MS Instrument performance check</b>				
Was PFK exact mass 380.9760 verified?	/			
Were the retention time windows established for all homologues?	/			
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers $\leq 25\%$ ?	/			
Is the static resolving power at least 10,000 (10% valley definition)?	/			
Was the mass resolution adequately check with PFK?	/			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	/			
<b>III. Initial calibration</b>				
Was the initial calibration performed at 5 concentration levels?	/			
Were all percent relative standard deviations (%RSD) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	/			
Did all calibration standards meet the Ion Abundance Ratio criteria?	/			
Was the signal to noise ratio for each target compound $\geq 2.5$ and for each recovery and internal standard $\geq 10$ ?	/			
<b>IV. Continuing calibration</b>				
Was a routine calibration performed at the beginning and end of each 12 hour period?	/			
Were all percent differences (%D) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?		/		
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	/			
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	/			
Was a method blank performed for each matrix and concentration?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?		/		
<b>VI. Matrix spike/Matrix spike duplicates</b>				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?		/		
<b>VII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	/			

LDC #: 19191A21  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 3  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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 type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>IX. Internal standards</b>				
Were internal standard recoveries within the 40-135% criteria?	<input checked="" type="checkbox"/>	<input 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 checked="" type="checkbox"/>	<input type="checkbox"/>	<input 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 checked="" type="checkbox"/>	<input type="checkbox"/>	<input 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 checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did compound spectra contain all characteristic ions listed in the table attached?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the signal to noise ratio for each target compound and labeled standard $\geq 2.5$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Does the maximum intensity of each specified characteristic ion coincide within $\pm 2$ seconds (includes labeled standards)?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
For PCDF identification, was any signal ( $S/N \geq 2.5$ , at $\pm$ seconds RT) detected in the corresponding PCDPE channel?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Was an acceptable lock mass recorded and monitored?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input 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"/>	

LDC #: 19191A21  
SDG #: See cover

VALIDATION FINDINGS CHECKLIST

Page: 3 of 3  
Reviewer: [Signature]  
2nd Reviewer: [Signature]

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:

**VALIDATION FINDINGS WORKSHEET**  
**Routine Calibration**

LDC #: 19191A2  
SDG #: Seecow

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

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

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

- N N/A Was a routine calibration performed at the beginning and end of each 12 hour period?  
 N N/A Were all percent differences (%D) of RRFs  $\leq$  20% for unlabeled compounds and  $\leq$  30% for labeled?  
 N N/A Did all routine calibration standards meet the Ion Abundance Ratio criteria?

#	Date	Standard ID	Compound	Finding %D (Limit: $\leq$ 30.0%)	Finding Ion Abundance Ratio	Associated Samples	Qualifications
	7/2/08	021408B1022	13C-H	57.3		2.4-5	+ detects
		↓	13C-H	71.8		↓	↓
	6/2/08	26W08D5-2	13C-OCDD	34.6		8175586 MB	+ detects

PCDDs		Selected ions (m/z)		Ion Abundance Ratio		PCDFs		Selected ions (m/z)		Ion Abundance Ratio	
Tetra-		M/M+2		0.65-0.89		Tetra-		M/M+2		0.65-0.89	
Penta-		M+2/M+4		1.32-1.78		Penta-		M+2/M+4		1.32-1.78	
Hexa-		M+2/M+4		1.05-1.43		Hexa-		M+2/M+4		1.05-1.43	
Hexa- <sup>13</sup> C-HxCDF (IS) only		M/M+2		0.43-0.59		Hexa- <sup>13</sup> C-HxCDF (IS) only		M/M+2		0.43-0.59	
Hepta- <sup>13</sup> C-HpCDF (IS) only		M/M+2		0.37-0.51		Hepta- <sup>13</sup> C-HpCDF (IS) only		M/M+2		0.37-0.51	
Hepta-		M+2/M+4		0.88-1.20		Hepta-		M+2/M+4		0.88-1.20	
Octa-		M+2/M+4		0.76-1.02		Octa-		M+2/M+4		0.76-1.02	

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

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

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".  
 **N** **N/A** Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.  
 **N** **N/A** Was a MS/MSD analyzed every 20 samples of each matrix?  
 **Y** **(N) N/A** Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		4/5	All	%R and	RPD out	( ) ( )	2	No data (CAS in)
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		
						( ) ( )		

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

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

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

N  N/A Was a LCS required?

Y  N/A Was a LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed?

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

#	Date	Lab ID/Reference	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		<u>8172352CS</u>	<u>E</u>	<u>139(74-126)</u>	( )	( )	<u>M7203</u>	
				( )	( )	( )	<u>8172352MS</u>	<u>1 + 2 1/2 p</u>
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		



**VALIDATION FINDINGS WORKSHEET**  
**Internal Standards**

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

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

X N N/A Are all internal standard recoveries were within the 40-135% criteria?

Y N N/A Was the S/N ratio all internal standard peaks  $\geq 10$ ?

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		1	E	26 (40-135)	Y/N/P (G-E, K-P)
			F	37	↓
			G	33	
		3	E	25	Y/N/P (G-F, K-Q)
			F	32	↓
			G	14	
			H	16	↓
			I	13	
		4 (NS)	D	36 (40-135)	No Qual
			F	36	
			H	32	↓
			I	24	
			C	39	↓
			G	33	↓

Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used
<sup>13</sup> C-2,3,7,8-TCDF			
<sup>13</sup> C-2,3,7,8-TCDD		K. <sup>13</sup> C-1,2,3,4-TCDD	
<sup>13</sup> C-1,2,3,7,8-PeCDF		L. <sup>13</sup> C-1,2,3,7,8,9-HxCDD	
<sup>13</sup> C-1,2,3,7,8-PeCDD		M.	
<sup>13</sup> C-1,2,3,6,7,8-HxCDF		N.	
<sup>13</sup> C-1,2,3,6,7,8-HxCDD		O.	
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF		P.	
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD		Q.	
<sup>13</sup> C-OCDD		R.	
		T.	

LDC #: 1919A-2  
 SDG #: See cover

**VALIDATION FINDINGS WORKSHEET**  
Compound Quantitation and Reported CRQLs

Page: 101  
 Reviewer: R  
 2nd Reviewer: R

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

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

Y  N  N/A Were the correct internal standard (IS), quantitation ions and relative response factors (RRF) used to quantitate the compound?  
 Y  N  N/A Compound quantitation and CRQLs were adjusted to reflect all sample dilutions and dry weight factors (if necessary).

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		2	H.A > calcs range	2	blots / P

Comments: See sample calculation verification worksheet for recalculations

LDC #: 19191A2  
 SDG #: See cover

**VALIDATION FINDINGS WORKSHEET**  
Initial Calibration Calculation Verification

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

**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:

$RRF = (A_x)(C_w)/(A_w)(C_x)$   
 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,  
 $A_w$  = Area of associated internal standard  
 $C_w$  = Concentration of internal standard  
 $X$  = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (initial)	(%RSD)	Average RRF (initial)	(%RSD)	RRF (CS3 std)	(%RSD)	RRF (CS3 std)	(%RSD)
1	18A2	6/17/08	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.798	12.5	0.798	12.5	0.82	12.5	0.82	12.70
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.913	10.3	0.913	10.3	0.93	10.3	0.93	10.3
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	0.821	13.9	0.821	13.9	0.87	13.9	0.87	14.1
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.844	12.7	0.844	12.7	0.88	12.7	0.88	12.7
			OCDF ( <sup>13</sup> C-OCDD)	1.721	16.2	1.721	16.2	1.86	16.2	1.86	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)								
3			OCDF ( <sup>13</sup> C-OCDD)								
			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)								

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

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

LDC #: 19191 A21  
 SDG #: See cover

Page: 1 of 1  
 Reviewer: 9  
 2nd Reviewer: 8

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

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

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	<u>71628405</u>	<u>6/28/08</u>	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	<u>0.798</u>	<u>0.83</u>	<u>4.0</u>	<u>0.83</u>	<u>4.1</u>
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	<u>0.913</u>	<u>0.81</u>	<u>11.7</u>	<u>0.81</u>	<u>11.7</u>
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	<u>0.821</u>	<u>0.87</u>	<u>6.5</u>	<u>0.87</u>	<u>6.4</u>
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	<u>0.844</u>	<u>0.83</u>	<u>1.5</u>	<u>0.83</u>	<u>1.5</u>
			OCDF ( <sup>13</sup> C-OCDD)	<u>1.721</u>	<u>1.58</u>	<u>8.3</u>	<u>1.58</u>	<u>8.3</u>
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.

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

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

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

% Recovery =  $100 * (SSR - SR) / SA$       Where: SSR = Spiked sample result, SR = Sample result  
 SA = Spike added

MSR = Matrix spike percent recovery      MSDR = Matrix spike duplicate percent recovery

RPD =  $|MSR - MSDR| * 2 / (MSR + MSDR)$

MS/MSD samples: 4/5

Compound	Spike Added (PS/SA)		Sample Concentration (PSS)	Spiked Sample Concentration (PSS/SA)		Matrix Spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		Reported RPD	Recalculated RPD
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.		
2,3,7,8-TCDD	20.9	20.9	78	183	95.9	50.2	50.2	85	86	62	62
1,2,3,7,8-PeCDD	10.4	10.4	330	824	457	47.2	47.5	120	122	57	57
1,2,3,4,7,8-HxCDD	✓	✓	250	697	426	43.1	43.0	172	169	48	48
1,2,3,4,7,8,9-HpCDF	✓	✓	850	1910	1020	10.00	10.192	1650	1635	60	61
OCDF	20.9	20.9	4900	98200	50600	23.00	23.541	618	766	64	64

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

LDC #: 1991A21  
 SDG #: 2er colle

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

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

**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 =  $100 * SSC/SA$

Where: SSC = Spiked sample concentration  
 SA = Spike added

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

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 2175566

Compound	Spike Added (PS/G)		Spiked Sample Concentration (PS/G)		LCS		LCSD		Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc
2,3,7,8-TCDD	20.0	NA	21.2	NA	106	106	106	106						
1,2,3,7,8-PeCDD	100	↓	105	↓	105	105	105	105						
1,2,3,4,7,8-HxCDD	↓	↓	84.9	↓	85	85	85	85						
1,2,3,4,7,8,9-HpCDF	↓	↓	106	↓	106	106	106	106						
OCDF	200	↓	220	↓	110	110	110	110						

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>(a)</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>3</sub> <sup>37</sup> ClO	HpCDF		
	305.8987	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub>	TCDF		409.7788	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>2</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>3</sub> O	HpCDF (S)		
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> 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>2</sub> <sup>37</sup> ClO	HpCDF		
	319.8965	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD		423.7767	M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	321.8936	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> 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>2</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>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	333.9338	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> 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>2</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>3</sub> <sup>37</sup> ClO	HxCDFPE		479.7165	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>2</sub> <sup>37</sup> ClO	NCDFPE		
	[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>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO	OCDF
		341.8567	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		443.7399	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
		351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF (S)		457.7377	M+2	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
353.8970		M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDF (S)	459.7348	M+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDD		
355.8546		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	469.7780	M+2		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
357.8516		M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	471.7750	M+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDD (S)		
367.8949		M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	513.6775	M+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDFPE		
369.8919		M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDD (S)	[422.9278]	LOCK		C <sub>10</sub> F <sub>17</sub>	PFK		
408.7974		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDFPE							
[354.9792]		LOCK	C <sub>9</sub> F <sub>13</sub>	PFK							
3		373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	HxCDF						
		375.8178	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</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>4</sub> <sup>37</sup> ClO	HxCDF (S)						
	385.8610	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> 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>4</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>3</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD							
	401.8559	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</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>3</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>3</sub> <sup>37</sup> ClO	OCDFPE							
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK							

(e) 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 = 36.966903

S = internal/recovery standard

LDC #: 19191A21  
 SDG #: See cover

## VALIDATION FINDINGS WORKSHEET

### Sample Calculation Verification

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

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

(Y) N N/A . Were all reported results recalculated and verified for all level IV samples?  
(Y) N N/A . Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

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

- $A_x$  = 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)
- $V_s$  = Volume or weight of sample extract in milliliters (ml) or grams (g).
- RRF = Relative Response Factor (average) from the initial calibration
- Df = Dilution Factor.
- %S = Percent solids, applicable to soil and solid matrices only.

Example:

Sample I.D. 3, 2:

$$\text{Conc.} = \frac{(365016)(4000)}{(1342580)(1.72)(1004)(0.913)}$$

= 6.90  $\mu$ g/g

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