

LABORATORY DATA CONSULTANTS, INC.

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August 15, 2008

ERM 2525 Natomas Park Drive, Suite 350

Sacramento, CA 95833

ATTN: Ms. Maria Barajas-Albalawi

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:**

SDG#	<u>Fraction</u>
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** 

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## 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

<sup>\*\*</sup>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 (≥0.05)	All soil samples in SDG F8F130140	J (all detects) UJ (all non-detects)	Α

## 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	lodomethane	67.71684	All water samples in SDG F8F130140	J+ (all detects)	А

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	lodomethane	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)	Α

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

## 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)	А	Initial calibration (RRF)
F8F130140	RINSATE-2 TB-2 TB-1 6/12/08 TB-2 6/12/08	lodomethane	J+ (all detects)	A	Continuing calibration (%D)
F8F130140	RINSATE-2 TB-2 TB-1 6/12/08 TB-2 6/12/08	lodomethane	J+ (all detects)	А	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)	А	Continuing calibration (ICV %D)
F8F130140	TSB-CJ-09-0 TSB-CJ-09-10**	Ethanol	J (all detects) UJ (all non-detects)	А	Continuing calibration (RRF)
F8F130140	RINSATE-2	Nonanal Dimethyl disulfide	J+ (all detects) J+ (all detects)	А	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	Α

LDC #: 19191A1	VALIDATION COMPLETENESS WORKSHEET
SDG #: F8F130140	Level III/IV
Laboratory: Test America	<del></del>

2nd Reviewer:

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

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

	Validation Area		Comments
l.	Technical holding times	4	Sampling dates: 6/13/08
II.	GC/MS Instrument performance check	A	/ '
III.	Initial calibration	m/	PSD.12
IV.	Continuing calibration/ICV	W	1ev=2570
V.	Blanks	#	'
VI.	Surrogate spikes	W	
VII.	Matrix spike/Matrix spike duplicates	W	
VIII.	Laboratory control samples	W	205 B
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	1	Not reviewed for Level III validation.
XII.	Compound quantitation/CRQLs	A	Not reviewed for Level III validation.
XIII.	Tentatively identified compounds (TICs)	2	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	M	F=1. TB=2.5.6

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

1/3	RINSATE-2	W	11	8170291MB	21	3	31	
21/2	TB-2	1	12 /	8172125MB	22	W	32	
3	TSB-CJ-09-0	5	کر 13	817-36/MB	23	W(N)	33	
4	TSB-CJ-09-10**		14	8/72539MB	24	Y (N.D)	34	
5/2	-TB-1 6/12/08	N	15	, /	25		35	
6/2	-TB-2 6/12/08	1	16		26		36	
7	RINSATE-2MS		17		27		37	
8	RINSATE-2MSD	V	18		28		38	
9	TSB-CJ-09-0MS	9	19		29		39	
10	TSB-CJ-09-0MSD	V	20		30		40	



## **VALIDATION FINDINGS CHECKLIST**

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

Method: Volatiles (EPA SW 846 Method 8260B)

Metriod: Volatiles (LFA 34V 040 Metriod 02002)				
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times	<del></del>	T		
All technical holding times were met.				
Cooler temperature criteria was met.				
III. ec/Ms. instrument performance check	T T			
Were the BFB performance results reviewed and found to be within the specified criteria?				
Were all samples analyzed within the 12 hour clock criteria?				
III Initial calibration		1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1		
Did the laboratory perform a 5 point calibration prior to sample analysis?				
Were all percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?				
Was a curve fit used for evaluation?				
Did the initial calibration meet the curve fit acceptance criteria of ≥ 0.990?				
Were all percent relative standard deviations (%RSD) $\leq$ 30% and relative response factors (RRF) $\geq$ 0.05?				
IV. Continuing calibration				
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?	1			
Were all percent differences (%D) $\leq$ 25% and relative response factors (RRF) $\geq$ 0.05?				
V Blanks		- Jack		
Was a method blank associated with every sample in this SDG?				
Was a method blank analyzed at least once every 12 hours for each matrix and concentration?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
Vi. Surrogate spikes				
Were all surrogate %R within QC limits?		/		
If the percent recovery (%R) for one or more surrogates was out of QC limits, was a reanalysis performed to confirm samples with %R outside of criteria?				
Mil⇒Matrix splke/Matrix spike/duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.				
Was a MS/MSD analyzed every 20 samples of each matrix?				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	10/15/09/09/15			
VIII. Laboratory control samples 14. 11. 11.				
Was an LCS analyzed for this SDG2				



## **VALIDATION FINDINGS CHECKLIST**

Page: \_\_of\_\_ Reviewer: \_\_\_\_\_ 2nd Reviewer: \_\_\_\_\_\_

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per analytical batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX: Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?	***			
X Internal standards	ī	T T	ı	, μ τριματικό το πορού (π. 1990). 1
Were internal standard area counts within -50% or +100% of the associated calibration standard?	/			
Were retention times within ± 30 seconds of the associated calibration standard?	/	<u> </u>		
XI: Target compound identification		T	T T	T
Were relative retention times (RRT's) within ± 0.06 RRT units of the standard?	/	<u>                                     </u>	$\bigsqcup$	
Did compound spectra meet specified EPA "Functional Guidelines" criteria?		<u>                                     </u>	$\bigsqcup$	
Were chromatogram peaks verified and accounted for?				
XII.: Compound quantitation/CRQLs)	T T	T T		T
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?		<u> </u>		
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XIII. Tentatively dentified compounds (TICs)		net y		
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?				
Were relative intensities of the major ions within $\pm$ 20% between the sample and the reference spectra?				
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?				
XIV. Systemipariometrice				
System performance was found to be acceptable.	/			
XV (overall assessment of data s				
Overall assessment of data was found to be acceptable.				
XVI. Flaid dugilleates.				
Field duplicate pairs were identified in this SDG.				panta viii verrita
Target compounds were detected in the field duplicates.	_	[ _ <u> </u>		
XVII. i Ejelckolanks				Market Commence of the Commenc
Field blanks were identified in this SDG.				
Target compounds were detected in the field blanks.				

## TARGET COMPOUND WORKSHEET

METHOD: VOA (EPA SW 846 Method 8260B)

A. Chloromethane*	U. 1,1,2-Trichloroethane	OO. 2,2-Dichloropropane	III. n-Butylbenzene	CCCC 1-Chlorohexane
B. Bromomethane	V. Benzene	PP. Bromochloromethane	JJJ. 1,2-Dichlorobenzene	DDDD, Isopropyl alcohol
C. Vinyl choride**	W. trans-1,3-Dichloropropene	QQ. 1,1-Dichloropropene	KKK. 1,2,4-Trichlorobenzene	EEEE Acetonitrile
D. Chloroethane	X. Bromoform*	RR. Dibromomethane	LLL. Hexachlorobutadiene	FFF Acrolein
E. Methylene chloride	Y. 4-Methyl-2-pentanone	SS. 1,3-Dichloropropane	MMM. Naphthalene	GGGG. Acrdonitrile
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*	W. 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. Chlorobenzene⁴	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 2:2-2-Watting
M. 2-Butanone	GG. Xylenes, total	AAA. 1,3,5-Trimethylbenzene	UUU. 1,2-Dichlorotetrafluoroethane	2 LT 1.7 M TO 10000
N. 1,1,1-Trichloroethane	HH. Vinyl acetate	BBB. 4-Chlorotoluene	VVV. 4-Ethyltoluene	21 12 22
O. Carbon tetrachloride	II. 2-Chloroethylvinyl ether	CCC. tert-Butylbenzene	WWW. Ethanol	2000.
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	8888
R. cis-1,3-Dichloropropene	LL. Methyl-tert-butyl ether	FFF. 1,3-Dichlorobenzene	ZZZ. tert-Butyl alcohol	
S. Trichloroethene	MM. 1,2-Dibromo-3-chloropropane	GGG. p-Isopropyltoluene	AAAA. Ethyl tert-butyl ether	חחחח.
T. Dibromochloromethane	NN. Methyl ethyl ketone	HHH. 1,4-Dichlorobenzene	BBBB. tert-Amyl methyl ether	

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

SDG #: 2000/14

## **VALIDATION FINDINGS WORKSHEET** Initial Calibration

Reviewer: 2nd Reviewer:

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

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

Did the laboratory perform a 5 point calibration prior to sample analysis?

Were percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCC's and SPCC's? Was a curve fit used for evaluation? If yes, what was the acceptance criteria used for evaluation?\_

Did the initial calibration meet the acceptance criteria?

N N/A

N/A

More all % BSDs and BBEs within the velidation existing of 100 % DSD

LDC #: 1919 SDG #: 226

## VALIDATION FINDINGS WORKSHEET Continuing Calibration

2nd Reviewer: Reviewer:

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".

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

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

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

*	Date	Standard ID	Compound	Finding %D (Limit: <25.0%)	Finding RRF (Limit: <u>&gt;</u> 0.05)	Associated Samples	Qualifications
	80/8012	18861017	Durot Homo	8/5/9/8		A11420S	1+1+A
			7	25-04476		8172125418	4/2/7
	8/14/10	F0441777	MMM		60,000.0	41/501/5	JMJ/B
					•		
							7
	6/9/08	40410317	lado methana	67.71684		A11 H20 S	1+4cts/10
	/, /					8172125HB	

SDG #: 20c (19/4)

# VALIDATION FINDINGS WORKSHEET Field Blanks

Reviewer:

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

Were field blanks identified in this SDG?  Were target compounds detected in the field blank Blank units: Sampling date: Field blank type: (circle one) Field Blank / Rinsate Trip Blank	/ere field blanks identified in this SDG? /gre target compounds detected in the  Associated sample units: ////////////////////////////////////	d in this SDG detected in the sple units:	/ere field blanks identified in this SDG? /ere target compounds detected in the field blanks?  Associated sample units:  ()	er:	Assoc	Associated Samples:	N:S:
Compound	Blank ID				Š	Sample Identification	lon
	5	X					
Methylene chloride							
Acetone					ı		
Chloroform	11.0						
Didlinumethano 0.4	0.41						
	,						

Associated Samples: Field blank type: (circle one) Field Blank / Rinsate / Trip Blank / Other:

Associated sample units:

Blank units: Sampling date:

CRQL

Compound	Blank ID			Sa	Sample Identification	ion		
Methylene chloride								
Acetone								
Chloraform								
							,	
CROL		,	,					

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 also qualified as not detected, "U". Other contaminants within five times the field blank concentration were also qualified as not detected, "U".

: 19191A

## VALIDATION FINDINGS WORKSHEET Field Blanks

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

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

Were field blanks identified in this SDG?

N/N/A

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". Other contaminants within five times the field blank concentration were also qualified as not detected, "U".

LDC #: 991.4 SDG #: 50 COL

## VALIDATION FINDINGS WORKSHEET Surrogate Spikes

2nd Reviewer: Page: Reviewer:

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

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

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

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

Y N/A 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

Qualifications	1+2648	1+ L+ 15/4 (Novanal, 0000																
%Recovery (Limits)	(311-115)	(1)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
Surrogate	BFB	BFB																
Sample ID	SWSEK/18	/																
Date																		
*																		

QC Limits (Water)

QC Limits (Soil)

81-117

88-110

86-115 80-120 86-118

74-121 80-120 80-120

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

## Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Reviewer

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

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

| N/A | 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 YIN NA

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?

Qualifications RPD (Water) M SON Associated Samples QC Limits (Water) 61-145% W 00 RPD (Limits) RPD (Soil) < 22% 7 MSD %R (Limits) 17 (8) QC Limits (Soil) 59-172% 1/1 MS %R (Limits) 3 (8) Comethans 1 Compound Compound 1,1-Dichloroethene MS/MSD ID 00 0 Date İ #

Chlorobenzene

11% × 14%

71-120% 76-127%

< 21%

66-142%

59-139% 60-133%

62-137%

Trichloroethene

Benzene Toluene

>

တ်

S. 9

< 24%

< 21%

< 21%

< 13% < 13%

76-125% 75-130%

VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

2nd Reviewer: Reviewer:

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

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

Y IN NA

Was a LCS required? Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?

																							_	
Qualifications	No Gual		(MESN/SM)																					
Associated Samples	M +=0>	8172/25 MZ																						
RPD (Limits)	(12(520)	( )	( )	( )	( )	( )	( )	)	( )	( )	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
LCSD %R (Limits)	( )	(AH5+181	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
LCS %R (Limits)	293 42+40)	(apt-54) 991 phothampo)	( )	( )	( )	( )	( )	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
Compound	NN	Codomethan																						
TCS/TCSD ID	1521.56/12/18	<i>a</i> /																						
Date																								
#									H						1									

LDC# SDG # W

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer: Reviewer:

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

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

RRF =  $(A_{+})(C_{+})/(A_{+})(C_{+})$ average RRF = sum of the RRFs/number of standards %RSD = 100 ° (S/X)

A<sub>x</sub> = Area of compound,
C<sub>x</sub> = Concentration of compound,
S = Standard deviation of the RRFs
X = Mean of the RRFs

A<sub>a</sub> = Area of associated internal standard C<sub>b</sub> = Concentration of internal standard

					Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (	Compound (Reference Internal Standard)	RRF (SS std)	RRF (\$2\std)	Average RRF (initial)	Average RRF (initial)	%RSD	%RSD
-	'Ne'	1/0/08		(1st internal standard)	126050	17605.0	1585,0 1585,0 1795,0 1795,0	0.52831	160402	70407 190405
			44	(2nd internal standard)	× 5280	1.35512	40x20 40x62.051285.0 58x60	pathe	l`	(27450)
	(+)		DD7	(3rd internal standard)	3.57047	3.57047 3.57047	3.42599	3.42599	8225 8 6225 8	8.2556
2		30/0//		NNN (1st internal standard)	P=1250	P = 47.0	0741-9 0.741-9 0.73871 0.73871 256782567	0.73871	8/25.5	7355
	1	0/4/00		(2nd internal standard)	805850	0.59203	0.59203 0.59203 0.5536 0.55366 13.4744 13.4718	0.55366	1347144	134718
	(下)			(3rd internal standard)	1.11568	89511-1	1.11150	1.11150	2.41699	24169
က				(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

SDG #: Secoury LDC #: 19414

## Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

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

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 \* (ave. RRF - RRF)/ave. RRF RRF =  $(A_x)(C_x)/(A_x)(C_x)$ 

Where: ave. RRF = initial calibration average RRF RRF = continuing calibration RRF  $A_x$  = Area of compound,  $A_x$  = Area of  $C_x$  = Concentration of compound,  $C_x$  = Concentration of compound,

 $A_{is}$  = Area of associated internal standard  $C_{is}$  = Concentration of internal standard

					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	<b>Q</b> %	Ω%
-	FCAUTTO 6/16/08	80/91/9	(1st internal standard)	158831	88=12.0	885/50 885/50	0.89949 0.899	0.8991
		,	(2nd internal standard)	pox620	0.3/2/6	8/2/8.0 Stell		5/9/2
			$\mathcal{ODP}$ (3rd internal standard)		36203	3.65203 6.59782 6.5978	6.59782	82659
2	F44-1777	80/91/9	FALTT 6/6/08 NNNN (1st internal standard)	0.73871	45/5/0 75/0/0	1.721set	445 = 3468 E	2,3244
		\ \	000 0 (2nd internal standard)	10	0.57358	0.57358 0.57358 3.5975 3.5.985	3.5975	3.5.985
			OCO (3rd internal standard)	1.11150	1.10470	1-10470	0.61152	8115-0-5118
က			(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 #: <u>See CO</u>UN

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

Page:_	
Reviewer:	<u> </u>
2nd reviewer:	
	, <b>/</b> ~

Percent

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	34	1
1.2-Dichloroethane-d4	1/	48.4101	97	97	
Dibromofluoromethane		43.2435	86	86	

Sample ID: Percent Percent Surrogate Spiked Recovery Recovery Percent Surrogate Found Reported Recalculated Difference Toluene-d8 Bromofluorobenzene 1,2-Dichloroethane-d4 Dibromofluoromethane

Sample ID: Percent Percent Recovery Surrogate Recovery Surrogate Reported Recalculated Spiked Found

Difference Toluene-d8 Bromofluorobenzene 1,2-Dichloroethane-d4 Dibromofluoromethane

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8					
Bromofluorobenzene					
1,2-Dichloroethane-d4					
Dibromofluoromethane					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8					
Bromofluorobenzene					
1,2-Dichloroethane-d4					
Dibromofluoromethane					

LDC#: 19/15

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

2nd Reviewer. Page: Reviewer

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 SA = Spike added

SC = Sample concentration

RPD = I MSC - MSC I \* 2/(MSC + MSDC)

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD sample: \_\_

	Š	oike	Sample	Spiked Sample	ample	Matrix Spike	Spike	Matrix Spike Duplicate	Duplicate	SM	MS/MSD
Compound	<i>₹</i> \	Addød Aggs	Concentration	Concentration	tration (O)	Percent Recovery	ecovery	Percent Recovery	ecovery	Ľ	RPD
	MS	MSD		MS	MSD	Reported	Recalc	Renorted	Recalc	Renorted	Recalculated
1,1-Dichloroethene	52.4	52.4 59.0	QN	21.6	42.8	64	29	d	90	0.8	8.80
Trichloroethene			/	1.6#	49.9	76	the	96	26	151	1.6
Benzene				46.9	47.6	90	08	16	É	4,	7.5
Toluene	,		670	470	47.8	89	88	16	6	8.1	1.7
Chlorobenzene	/	<b>\</b>	@ N	48.4	470	68	89	90	90	7.	4.

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

LDC #: 19/1 SDG#

## Laboratory Control Sample Results Verification VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Reviewer:

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

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

% Recovery = 100 \* SSC/SA

SSC = Spiked sample concentration SA = Spike added Where:

RPD = I LCSC - LCSDC I \* 2/(LCSC + LCSDC)

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

LCS ID:

	Ş	sike	Spiked S	ample	SOI	S	LCSD		I CS/I	CS/I CSD
Compound	PA )	Added (A)	Concentration	tration	Percent Recovery	ecovery	Percent Recovery	scovery	R	RPD
	SOT	l csp	SDI	I CSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
1,1-Dichloroethene	25	ν¥	47.8	X-V	38	96				
Trichloroethene	1	,	79.5		99.	Ó				
			49.6		B	80				
	,		2.05		100	201				
Chlorobenzene	$\nearrow$	<b>\</b>	49.9		201	(0)				

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. LDC #: 19191A SDG #: <u>See COV</u>EN

Y/N N/A

Df

%S

## VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

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

Dilution factor.

Were all reported results recalculated and verified for all level IV samples?

Percent solids, applicable to soils and solid matrices

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

# .	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

<sup>\*\*</sup>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.
- 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 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 (≥0.05)	All samples in SDG F8F130140	J (all detects) UJ (all non-detects)	A
	N-(Hydroxymethyl)phthalimide	0.04408 (≥0.05)		J (all detects) UJ (all non-detects)	

## IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

Percent differences (%D) between the initial calibration RRF and the continuing calibration RRF were within the method criteria of less than or equal to 20.0% for calibration check compounds (CCCs).

For the purposes of technical evaluation, all compounds were evaluated against the 25.0% (%D) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
6/19/08	Phthalic acid	25.06878		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 N-(Hydroxymethyl)phthalimide	0.01066 (≥0.05) 0.04523 (≥0.05)	All samples in SDG F8F130140	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	А

### 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)	А	Initial calibration (RRF)
F8F130140	RINSATE-2 TSB-CJ-09-0 TSB-CJ-09-10**	Phthalic acid	J- (all detects) UJ (all non-detects)	А	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	VALIDATION COMPLETENESS WORKSHEET
SDG #: F8F130140	Level III/IV
Laboratory: Test America	

2nd Reviewer

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 6/1=/08
11.	GC/MS Instrument performance check	A	, ,
III.	Initial calibration	<i>3</i> W	
IV.	Continuing calibration/ICV	W	101 = 2570
V.	Blanks	A	/
VI.	Surrogate spikes	$\bigstar$	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	W	109
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	$\Diamond$	Not reviewed for Level III validation.
XII.	Compound quantitation/CRQLs	$\phi$	Not reviewed for Level III validation.
XIII.	Tentatively identified compounds (TICs)	N	Not reviewed for Level III validation.
XIV.	System performance	4	Not reviewed for Level III validation.
XV.	Overall assessment of data	A	
XVI.	Field duplicates	N	
XVII.	Field blanks	ND	R=1. \$

Note:

Validated Samples:

A = Acceptable

N = Not provided/applicable SW = See worksheet

ND = No compounds detected

R = Rinsate

D = Duplicate

TB = Trip blank
EB = Equipment blank FB = Field blank

\*\* Indicates sample underwent Level IV validation

1	RINSATE-2	W	11	8168351MB	21	W	31	
2	TSB-CJ-09-0	M	12	8168351MB 8170309MB	22 -	S	32	
3	TSB-CJ-09-10**	V	13	, , , , , , , , , , , , , , , , , , ,	23		33	
4	RINSATE-2MS	h1	14		24		34	
5	RINSATE-2MSD	V	15		25		35	
6	TSB-CJ-09-0MS	9	16		26		36	
7	TSB-CJ-09-0MSD	V	17		27		37	
8	PARATE-L-	h	18		28		38	
9		•	19		29		39	
10			20		30		40	

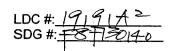
## LDC #: 191918 > SDG #: 787130140

## **VALIDATION FINDINGS CHECKLIST**

Page: of Reviewer: 2nd Reviewer:

Method: Semivolatiles (EPA SW 846 Method 8270C)

Method: Semivolatiles (EPA SW 846 Method 8270C)	<del></del>		<del></del>	
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times	100			
All technical holding times were met.	1/	1_		
Cooler temperature criteria was met.	$\bot$			
III GGMS Instrument performance check in				
Were the DFTPP performance results reviewed and found to be within the specified criteria?				
Were all samples analyzed within the 12 hour clock criteria?	1			
III: Initial calibration	T		T	
Did the laboratory perform a 5 point calibration prior to sample analysis?	1/	1		
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?	1 C	1/		
Did the initial calibration meet the curve fit acceptance criteria of ≥ 0.990?	<u></u>	ļ	/	
Were all percent relative standard deviations (%RSD) $\leq$ 30% and relative response factors (RRF) $\geq$ 0.05?		/		
IV. Gentifuling calibration (a)				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?				
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?				
Were all percent differences (%D) ≤ 25% and relative response factors (RRF) ≥ 0.05?		/		
V Blanks				
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.				
VI/Surrogate spikes				
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?				
VII. Matox spike/Matox spike duplicates vs. Lak (ii) (1. 16. 1) (1. 16. 16. 16. 16. 16. 16. 16. 16. 16. 1				and the second s
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?	7			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	7			
VIII. Laboratory control samples				
Was an LCS analyzed for this SDG?				
		_		



### **VALIDATION FINDINGS CHECKLIST**

Page: of Reviewer: 2nd Reviewer:

	T	T	T	1
Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?	-	-		
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
D. Regional Quality Assurance are quality Egrinol				en e
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
X. Internal standards				
Were internal standard area counts within -50% or +100% of the associated calibration standard?				
Were retention times within ± 30 seconds from the associated calibration standard?	7			
XI Target compound deministration with the last transfer and the second				States and the second second
Were relative retention times (RRT's) within ± 0.06 RRT units of the standard?			-	
Did compound spectra meet specified EPA "Functional Guidelines" criteria?				
Were chromatogram peaks verified and accounted for?				
XII. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XIII—rentatively identified compounds (FICs) (Exp. 1922)				en de la companya de
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?				
Were relative intensities of the major ions within $\pm$ 20% between the sample and the reference spectra?				
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?				
XIV, System, performance (1), 18, 18, 18, 18, 18, 18, 18, 18, 18, 18				alder and great and the second
System performance was found to be acceptable.	7	e e e e e e e e e e e e e e e e e e e		
V-Sveral assessment of Galas				
Overall assessment of data was found to be acceptable.	$\overrightarrow{A}$			Design Charles (Bra. 143)
KV/FREG pupilicates see the second				
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.			1	
XVII Fried danks 1965 - 2004 1968 1966 1966 1966 1966				Property of the second
Field blanks were identified in this SDG.		A		
Target compounds were detected in the field blanks.		1		

## **VALIDATION FINDINGS WORKSHEET**

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

A. Friendi	P. Bis(2-chioroethoxy)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-Dinitrophenoi*	WW. Carbazole	LLL. Benzo(g,h,l)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. Benzolc 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	m. Acetabhenono
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	uuu.
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	ww.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	WWW. 4-Ch Inobouzou + (h.j.o.)
XXX. A-f4) shoxmeth AAAA. 4-ch(nothery)	ly)>+h+halimide	XXX. Phenyl sultide	. 222. phany	disulfide

SDG #: The County METHOD: GC/MS BNA (EPA SW 846 Method 8270) LDC #: 19191A

VALIDATION FINDINGS WORKSHEET Initial Calibration

2nd Reviewer:

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

Y/N N/Y

Did the laboratory conduct an acceptable 5 point calibration prior to sample analysis?

Were percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCC's and SPCC's? Was a curve fit used for evaluation? If yes, what was the acceptance criteria used for evaluation?

Did the initial calibration meet the acceptance criteria? 

Were all %RSDs and RRFs within the validation criteria of ≤30 %RSD and ≥0.05 RRF?

Qualifications Associated Samples 0.04408 Finding RRF (Limit: >0.05) 20.0 Finding %RSD (Limit: <30.0%) thatic acid Compound XXX Standard ID 187 B 18/18 Date \*

VALIDATION FINDINGS WORKSHEET

Continuing Calibration

Reviewer: 2nd Reviewer:

> Jease see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". METHOD: GC/MS BNA (EPA SW 846 Method 8270)

SDG #: Se Com LDC #: 1919/A

Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's ? Were all %D and RRFs within the validation criteria of ≤25 %D and ≥0.05 RRF ?

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

Qualifications Associated Samples Finding RRF (Limit: >0.05) 33 0/0 25,06818 Finding %D (Limit: <25.0%) Hothalic aco Compound 9552740V Standard ID 9 Date 6

LDC #: 1919 SDG #: 200

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

Page:

2nd Reviewer: Reviewer:

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

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

Was a LCS required? Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?

r=====		_																						
Qualifications	No lenal	(MS/NSDW)																						
Associated Samples	M50,15.	817030 9NB																						
RPD (Limits)	( )	( )	( )	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(
LCSD %R (Limits)		( )	( )	( )	( )	( )	( )	( )	( )			( )	( )	( )		( )	( )	( )	( )	( )			( )	
LCS %R (Limits)	36 (54-90)		( )	( )	( )	( )	( )	(	( )	( )	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
Compound	HH																							
TCS/TCSD ID	8/10309405																							
Date																								
*																								

LDC #: 19191.4>

## VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: of Reviewer: 2nd Reviewer:

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

 $\label{eq:RRF} $$RF = (A_J(C_L)/(A_L)(C_J)$$ average RRF = sum of the RRFs/number of standards $$RSD = 100 * (S/X)$$$ 

A<sub>k</sub> = Area of compound,
C<sub>k</sub> = Concentration of compound,
S = Standard deviation of the RRFs, X =

 $A_k = Area of associated internal standard <math>C_k = Concentration of internal standard s, X = Mean of the RRFs$ 

		Calibration		Reported	Recalculated	Reported	Recalculated	Reported	
*	Standard ID	Date	Compound (Reference Internal Standard)	RRF		Average RRF	Average RRF	%RSD	Recalculated
	10/2	1,1,8	Phenol (1st internal standard)	11 (34.)	1 97 8td)	(initial)	(Inttial)		dens,
		0. 1.6	Naphthalene (2nd internal standard)	100438	1000	1.853/	1.8883/	1.070	1.070
			Fluorene (3rd internal standard)	1 4 177 B	Пξ	10/0/1	1000	1.328	1.32B
		~	Pentachlorophenol (4th internal standard)	07000	-1	1.422	1.41229	0.573	5720
			Bis(2-ethylhexyl)phithalate (5th internal standard)	090762	00000	4700.0	0.19634	10.265	10.356
			Benzo(a)pyrene (6th internal standard)	1000	20010	0.00045	0.86343	455.6	4500
N	CAR!	, ,		20051	13800	1-11/82	100 III	7878	4304
	11/2	6/18/08		10.57976	15-10 1-15-1	12517		200	6.4×6
		· /	Naphthalene (2nd internal standard) 1/1///	┵	٠١.	4.2/4	0.512/4	0.715111	115770
			Fluorene (3rd internal standard)		1.70/1	1.18205	1.18333	0.93562	0.9364
			Pentachlorophenol (4th internal standard)				\		
			Bis(2-ethylhexyl)phthalate (5th internal standard)						
	1		Benzo(a)pyrene (6th internal standard)						
e	1941	6/2/0	Phenof (1st internal standard) ///	1.60116	100	1 FT-43	==  =	H	4
		00/21/0	3	033010	00000	1256	ام	2 64252	26427
				1 32639	20000	1.27002	0.53002	1,4990	449CHD
			Pentachiorophenol (4th Internal standard) 222	+	N.	525-11	1.83325	1.22 192	1202)
			Ble(2 ethylhexyl)phthalate (5th internal standard) AAA		1000	0.5003	寸		87548
			Benzo(a)pyrene (6th internal standard)	1 1 1	2	1.50- 50	1.39-65	2,583	14851
					,	_			000/

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

SDG #:266

### Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Reviewer: 2nd Reviewer:

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 \* (ave. RRF - RRF)/ave. RRF RRF =  $(A_{\nu})(C_{\nu})/(A_{\nu})(C_{\nu})$ 

ave. RRF = initial calibration average RRF RRF = continuing calibration RRF Where:

 $A_{\rm b}$  = Area of associated internal standard  $C_{\rm b}$  = Concentration of internal standard

 $A_x$  = Area of compound,  $C_x$  = Concentration of compound,

					Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	RRF (CC)	RRF	Q%	<b>0%</b>
-	1545	6/9/19	Phenol (1st internal standard)	1.85537	180163	1.80165	1-600	000
		,	Naphthalene (2nd internal standard)	1.10001	1.08712	100100	1.0101	12000
			Fluorene (3rd internal standard)	14180	1 AURTR	コご	145/2016	1.0 (42
			Pentachlorophenol (4th internal standard)	0.19634	0.20730	02700	1.17832	0.428
			Bis(2-ethylhexyl)phthalate (5th internal standard)	08343	8×1×0	JAN X	220210	
			Benzo(a)pyrene (6th Internal standard)	~X		1. 12/00	9:4:0T	0.2 (84
7	104522B	80/6/08	Phrenol (1st internal standard)	0.51274	0.51326	DANT O	5 1001 0	90051
	,	, ,	Naphthalene (2nd internal standard) [/////	1.18223	- SOTOC	1	7	0-1010
			Fluorene (3rd internal standard)		1 2		acopor-	2,0469
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
6	10/529	6/19/08		1.575.1	1.59157	15001	1/4000	1:00
		/	Nลอาปาลอก (2nd internal standard) $M M_{ m N}$	0.33002	033954	100000	201102	0.974
			Fluorene (3rd internal standard)	1.022an	XX/0-	10101	I ·	2,885.8
			Pentachlerophenol (4th internal standard)	03/237	17/18/10	020000	0.585/9	0.58=9
			Bie(2-ethythexyt)phthalate (5th interrial standard)	0 2920	STORY!	0.70042	1.04A70	3.843/
			Benzo(a) ovrene (6th internal standard)	1	01/1/2	1.277.30	1024	1.639

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

LDC #: 191911= SDG #: 500 COW

### VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page:	
Reviewer:_	9
2nd reviewer:_	N
	Y

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	1/	36.6753	73	73	
Terphenyl-d14		33.4531	67	67	
Phenol-d5	75	53.9595	70	72	
2-Fluorophenol	1	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					

LDC #: 1919/17> SDG #: 500 COWN

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

Reviewer: 2nd Reviewer:

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:

SC = Sample concentation

RPD = I MSC - MSC I \* 2/(MSC + MSDC)

SSC = Spiked sample concentration SA = Spike added

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples: \_

	S.	ike	Sample	Spiked Sample	Sample	Matrix Spike	pike	Matrix Spike Duplicate	- Duplicate	US///SM	SD
Compound		Added	Conceptration	Concentration (MA)	tration ASA	Percent Recovery	ecovery	Percent Recovery	Recovery	RPD	0
	NS.	USM.		MS	MSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenol	\$	4	NB	340	2400		76	70	06	4.6	56
N-Nitroso-di-n-propylamine	1			2840	2858	00	20	52	56	76	9.6
4-Chloro-3-methylphenol				2/8/2	2670	83	83	86	22	12./	7.7
Acenaphthene				2780	2590	82	38	52	75	1.0	7.7
Pentachlorophenol				2590	240	52	75	06	72	7.4	7.2
Pyrene		<b>\</b>		2490	2340	73	1/2	89	88	0.0	4.0
		-		,	•						
											wa.

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 #:/9/9/X

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

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

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

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

% Recovery = 100 \* (SC/SA

SSC = Spike concentration SA = Spike added Where:

RPD = ILCSC - LCSDC I \* 2/(LCSC + LCSDC)

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

LCS/LCSD samples: \_

	as	ike	dS	ke	01	CS	TCSD	n.	I CS/	CS/I CSD
Compound	Added Added	<b>166</b> <b>165</b> )	Concentration	(fation	Percent Recovery	ecovery	Percent Recovery	ecovery	R	RPD
	SOL	I CSD	1.08	1 CSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenol	3330	λ¥Ν	2370	NA	12	71				
N-Nitroso-di-n-propylamine	V		2570		12	77				
4-Chloro-3-methylphenol			3600		28	78				
Acenaphthene			2520		757	76				
Pentachlorophenol			2270		89	68				
Pyrene		>	0955	<b>/</b>	88	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 #: 1919/A2
SDG #: <u>See COW</u>

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	
Reviewer:	9
2nd reviewer:	0.
,	<b>y</b> /

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

Ñ	M	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?

Conce	entratio	$n = \frac{(A_{*})(I_{*})(V_{*})(DF)(2.0)}{(A_{*})(RRF)(V_{*})(V_{*})(\%S)}$	Example:	=	110				
<b>,</b>	=	Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D	<u>₹</u>	-/V -:				
<u>.</u>	=	Area of the characteristic ion (EICP) for the specific internal standard							
<b>.</b>	=	Amount of internal standard added in nanograms (ng)	Conc. = ((	)(	)(	)(	)(	_)(	)
<b>'</b> 。	=	Volume or weight of sample extract in milliliters (ml) or grams (g).							
<b>'</b> i	=	Volume of extract injected in microliters (ul)	=						
<b>/</b> ,	=	Volume of the concentrated extract in microliters (ul)							
)f	==	Dilution Factor.							
<b>6</b> S	=	Percent solids, applicable to soil and solid matrices only.							
.0	==	Factor of 2 to account for GPC cleanup							

2.0	= Factor of 2 to accou	int for GPC cleanup				r
#	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

<sup>\*\*</sup>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.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/ECD Instrument Performance Check

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

### III. Initial Calibration

Initial calibration of single 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-09-0MS TSB-CJ-09-0MSD 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)	А
8169189MB	Not specified	Tetrachloro-m-xylene	58 (72-135)	All TCL compounds	J- (all detects) UJ (all non-detects)	Р

### 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)	Α

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)	А	

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)	Α	Continuing calibration (%D)
F8F130140	TSB-CJ-09-0	All TCL compounds	J+ (all detects)	А	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)	А	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	VALIDATION COMPLETENESS WORKSHEET	Date: <u>8/</u> 5
SDG #:	F8F130140	Level III/IV	Page:of_
Laborato	ory: Test America		Reviewer: Q
			2nd Reviewer:
METHO	D: GC Chlorinated Pa	esticides (EPA SW 846 Method 8081A)	<del>-y</del>

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
l.	Technical holding times	A	Sampling dates: 6/12/08
11.	GC/ECD Instrument Performance Check	A	/
III.	Initial calibration	A	+SD.12
IV.	Continuing calibration/ICV	M	ex=1570
V.	Blanks	A	
VI.	Surrogate spikes	W	
VII.	Matrix spike/Matrix spike duplicates	M	
VIII.	Laboratory control samples	-4	109
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	
Xb.	GPC Calibration	N	
XI.	Target compound identification	4	Not reviewed for Level III validation.
XII.	Compound quantitation and reported CRQLs	W	Not reviewed for Level III validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	ND	R=)

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

			<b></b>				
1	RINSATE-2	11	8169189MB	21	W	31	
2	TSB-CJ-09-0 \$	12	8169189MB 8170319MB	22	<u>s</u>	32	
3	TSB-CJ-09-0DL	13	,	23		33	
4	TSB-CJ-09-10**	14		24		34	
5	TSB-CJ-09-0MS	15		25		35	
6	TSB-CJ-09-0MSD	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	l. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	.00
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	Ŧ.
C. della-BHC	K. Endrin	S. aipha-Chlordane	AA. Aroclor-1254	-
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	.tr
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. DB 608	ÆK.
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. DB 1701	LL.
G. Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	E.E.	MM.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	FF.	NN.

Notes:

LDC #: 19191 A34 SDG #: <u>Seccoun</u>

### VALIDATION FINDINGS CHECKLIST

Page: /of / Reviewer: / 2nd Reviewer: /

Method: GC \_\_\_\_\_HPLC\_

Method: GC HPLC	==-			ı — J
Validation Area	Yes	No	NA	Findings/Comments
Georgia Booking unes			Г	
All technical holding times were met.			ļ	
Cooler temperature criteria was met.	200		HAVE S	
rapulat calciales of the control of				
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?	$\frac{1}{2}$			
Were the RT windows properly established?		8/25/25	e e e e e e e e e e e e e e e e e e e	
nV-@inflooring-calibration		17 T	TO T	T
What type of continuing calibration calculation was performed?%D or %R	/		<u> </u>	
Was a continuing calibration analyzed daily?		1	ļ	
Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?			1_	
Were all the retention times within the acceptance windows?		1	(esa)	
V-Blanks -		7	T S	
Was a method blank associated with every sample in this SDG?	4	_	┼	
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.	304.305			
Visioni gales alles			T	
Were all surrogate %R within the QC limits?		<del>                                     </del>	1	
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?		<u> </u>	1_	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?		SPEKEERE		
VII. Mariji sejike Mariji sejike dopikales i u su alam da kara kara kara kara kara kara kara k				T T
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?	7			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII Laboratory controls amples 200				Total
Was an LCS analyzed for this SDG?	1	4	+	
Was an LCS analyzed per extraction batch?	1		_1_	

LDC#:	19191 B3a
SDG #:	Lee cow

### **VALIDATION FINDINGS CHECKLIST**

Page: \_\_\_of\_\_\_ Reviewer: \_\_\_\_\_ 2nd Reviewer: \_\_\_\_\_

Validation Area	Yes	No	NA	Findings/Comments
Validation Area	1.63			· ····airigo commonto
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
XSP copioners as a company Assistance and Adventury Control (2005)				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
As paragraphics (the authorities to the second seco				
Were the retention times of reported detects within the RT windows?				
XI Eximposino quantitation/OROES				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions				
and dry weight factors applicable to lever 17 Validation				
System performance was found to be acceptable.				
Overall assessment of data was found to be acceptable.	1			
XIV.FEGOROBIES				
Were field duplicate pairs identified in this SDG?		/		
Were target compounds idetected in the field duplicates?				3,000,000
V Fad Danis				
Were field blanks identified in this SDG?	1	_/	1	
Were target compounds detected in the field blanks?			<u></u>	

LDC#: 19,9,1,39 SDG#: 22,000

METHOD: \_\_\_GC\_\_ HPLC

### VALIDATION FINDINGS WORKSHEET Continuing Calibration

Reviewer:

2nd Reviewer:\_\_

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

evel IV Only Y N N/A

Were the retention times for all calibrated compounds within their respective acceptance windows?

Qualifications	THACK		× ×														
Associated Samples	8 4-6.	1 1															
RT (limit)	(	(	(	)					(	(		(		7	(	(	
%D / RPD (Limit ≤ 15.0)	4:51	-									·						
Compound	A	,			-	-											
Detector/ Column	Chit																
Standard ID	XX42153																
# Date	2016119	, ,															

SDG #: See COUN LDC #. 19191839

### VALIDATION FINDINDS WORKSHEET Surrogate Recovery

Reviewer:

Page:\_

or No METHOD: VGC HPLC
Are surrogates required by the method? Yes\_

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

Did all surrogate recoveries (%R) meet the QC limits? Were surrogates spiked into all samples and blanks? N N/A

LDC#: 19,9,43a SDG #: See CON

VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

2nd Reviewer: Reviewer: Page:

**METHOD:** <u>/</u> GC \_\_HPLC Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

	Se Qualifications	\ \ 	(405 m)																							
SDG? on was performed? C limits?	Associated Samples	7																								
(MSD) analyzed for each matrix in this SDG? h matrix or whenever a sample extraction was tive percent differences (RPD) within QC limit	RPD (Limits)	£7 (530)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	)	
cate (MSD) analyzed to each matrix or whene relative percent differ	MSD %R (Limits)	161 (69-130)			( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(	( )	( )	(	( )	
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? Was an MS/MSD analyzed every 20 samples for each matrix or whenever a sample extraction was performed? Were the MS/MSD percent recoveries (%R) and relative percent differences (RPD) within QC limits?	MS %R (Limits)	288 (69-130)	( )	( )		( )		( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	
matrix spike (MS MS/MSD analyz e MS/MSD perce	Compound	<i>1. Y</i>																								
W N N/A Were a r	CI CUNIVA																									

SDG #: 5000 NV

# VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

Page: of A Reviewer: 2nd Reviewer:

METHOD: VGC HPLC

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

Level W/D Only
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?

Qualifications	John X								
Associated Samples	8								
Finding	exceeded calibrars								
Compound Name	Ö								
*									

Comments: See sample calculation verification worksheet for recalculations

LDC #: 1919/439 SDG #: Secon

### Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer:

> GC HPLC METHOD:

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

evel IV/D Only

N N N

N AVA

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 <40%?

If no, please see findings bellow.

#	Compound Name	Sample ID	/ %D Between Two Columns/Detectors Limit (≤ 40%)	Qualifications
	٨	7	12/8.5	Heta/+
Com	Comments:			

SDG # Jee CONV LDC#: 19191 A39

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

701	4	9
Page:	Reviewer	2nd Reviewer:

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

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

				Reported	Recalculated	Reported	Recalculated Reported	Renorted	Recalculated
*	Standard ID	Calibration Date	Compound	CF CF CF CF	C. Sstd)	ш	Average CF (initial)	%RSD	%RSD
	70	6/2//	2,4-006 (Ch.A)	3/366840	3/366840	22	3/3/287262	1.30/08	1.3011
	1	00/2/0	V (ch B)	21121/200	port relic	21121700 7121 700 20380278 20380278 2.78044 2.7804	20380279	2.78044	2.780K
1			F (ch. A)	64543204	655453200	81332132	£81332137	2.76/88	2.7619
7		0/,	1 1 0	28233800	2823386	28233860 28233800 30126 9612 30126 965 285	30/26962	Strell'	42///
	10/2	8/19/19	F (ORB)	3635 9100	36359/100	2635 flow 36359 loso 380/8 2886 3/8/8286 2.83696 2.837	3818282	2.83696	2.8370
			7	0095/800	12681560	20815604 12681400 13320 2078 133202078 8.79750 8.7925	13320208	8.79250	8.7925
т									
4									

Comments: Referto 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 #: 19191 \$39 SDG #: 500 COW

### Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Page:

> HPLC METHOD: GC\_

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

% Difference = 100 \* (ave. CF - CF)/ave. CF CF = A/C

Where:

ave. CF = initial calibration average CF
CF = continuing calibration CF
A = Area of compound
C = Concentration of compound

					Reported	Recalculated	Reported	Recalculated
Calibration Standard ID Date	ion		Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc. CCV	۵%	Q%
XCAU33 6/9/18 2.4-2	18 2.4'2		2.4'2026 (Ch.A)	0.025	Spc0.0	-0.0242	W	w. W
		- 1	(chB)		8700	8400	8.0	0.0
H	H	11	(chr A)		35000		الا. من	ر. س
0	0	ł	1		0.0283	0.0283	/: 8/	/3./
H	H	Ì	(chr B)		1500	1.500	4.0	4.0
2	6		1	<i>\</i>	0.0269	0.0269	7.4	7.4
					/			

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#: 1919, A3a. SDG#: See COUN

### VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page: Reviewer: 2nd reviewer:

METHOD: //GC HPLC

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Surrogate Colu	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
TOUX	chB	0.020	0.020 0.01992	001	001	0
<i>1043</i>	1	1	50110.0	85	l X	0
			,			

sample ID:						
Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	

Sample ID:

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

LDC #: 19,9,439 SDG #: Secoun

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

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

> Z GC METHOD:

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

SC = Sample concentration

RPD =(((SSCMS - SSCMSD) \* 2) / (SSCMS + SSCMSD))\*100

þ

Ŋ

MS/MSD samples:\_\_

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

MSD = Matrix spike duplicate

	Spi	ke	Sample	Spike Sample	ample	Matrix spike	spike	Matrix Spike Duplicate	Duplicate	GSM/SM	SD
Compound	Added	) Sed	Come.	Concentration (	tration (2)	Percent Recovery	tecovery	Percent Recovery	ecovery	RPD	0
	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)											
4	727	721	NB	16.1	15-6	8	9	90	30	) / /	<i>w</i>
0	1	7	99	23.2	4:00	96	95	10	à	3.00	3.5
		:			24 4-11-24 4-	and the contract of the second	Ofciococ bas	the column of	Total ac	receipt of a no	* 00250

LDC#: 1919, 439 SDG#: 500 COW

# VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicate Results Verification

2nd Reviewer: Reviewer

> GC HPLC METHOD:

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

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

Where:

SC = Concentration

RPD = I SSCLCS - SSCLCSD I \* 2/(SSCLCS + SSCLCSD)

SSC = Spiked sample concentration
SA = Spike added
LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples: 8/703/

	ຜູ້	Spike	Spiked	Sample	רכ	rcs	rcsd	SD	/SOT	rcs/rcsp
Compound		2000	Sonoo	Concentration	Percent !	Percent Recovery	Percent Recovery	Recovery	~	RPD
	SOT	LCSD	rcs	rcsD	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)										
4	16.7	Ν¥	(5.	NX	90	90				
Q.	1	//	0.21	7	<u> </u>	201				

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 #: 19196/439 SDG #500 COVON

## VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

WETHOD: ∠GC\_\_HPLC

ا ور ا	Were all
AE I HOD:	A/N N N N
	ZZ
=	

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

Concentration= (A)(Fv)(Df) (RF)(Vs or Ws)(%S/100)

A= Area or height of the compound to be measured Fv= Final Volume of extract Df= Dilution Factor

RF= Average response factor of the compound In the initial calibration Vs= Initial volume of the sample Ws= Initial weight of the sample %S= Percent Solid

Sample ID.

Example:

Compound Name

Concentration =  $\frac{(8297556)}{(14470464)}(10000)$  (1)

= 5.32 M/rx

098,588595)

Qualifications				
Recalculated Results Concentrations				
Reported Concentrations				
Compound				
Sample ID				
#				

1	

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

<sup>\*\*</sup>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.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.

None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/ECD Instrument Performance Check

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

### III. Initial Calibration

Initial calibration of 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)	А

### 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

SDG	Sample	Compound	Flag	A or P	Reason
F8F130140	TSB-CJ-09-0	All TCL compounds	J+ (all detects)	А	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

	19191A3b	VA	LIDATIO				SS WORKSHE	ET	Date:
	:F8F130140			Le	evel III/	IV			Page: /of/
abora	tory: Test America								Reviewer: 9
NETH(	OD: GC Polychlorina	ted Biph	enyls (EPA	SW 846 N	Method 8	308	2)		Zila Noviowali.
			wood for oo	ah af tha f	ollowing	vo	idation areas Valid	dation fir	adings are noted in attache
	mpies listed below w on findings workshee		wed for ea	Cii Oi tiie i	ollowing	va	iuation areas. Vaii	Janon III	ndings are noted in attache
	Validati	on Area					Co	mments	<u> </u>
l.	Technical holding times			A	Sampling	j da	tes: 6/12/0	18	
II.	GC/ECD Instrument Per	formance	Check	$\sim$					
III.	Initial calibration			1					
IV.	Continuing calibration/IC	CV		A	10	V :	< 1570		
V.	Blanks			A					
VI.	Surrogate spikes			M					
VII.	Matrix spike/Matrix spike	e duplicate	S	A					
VIII.	Laboratory control samp	les		$\triangleleft$	10	_	>		
IX.	Regional quality assura	nce and qu	ality control	N					
Xa.	Florisil cartridge check			N					
Xb.	GPC Calibration			N					
XI.	Target compound identi	fication		A-	Not revie	ewe	d for Level III validation	1.	
XII.	Compound quantitation	and report	ed CRQLs	1	Not revie	ewe	d for Level III validation	1.	
XIII.	Overall assessment of o	lata		A					
XIV.	Field duplicates			$\mathcal{N}$					
XV.	Field blanks			ND	<b>R</b> =				
ote:	A = Acceptable N = Not provided/applic SW = See worksheet	able	R = Rir	lo compound nsate ield blank	ds detected	þ	D = Duplicate TB = Trip blank EB = Equipmen	t blank	
alidate	ed Samples: **	Indicates s	ample underv	vent Level IV	/ validation	)			
T		[1]	8168	2371	N3 1 24	T		31	·
	RINSATE-2	W 11	15100	2-11	/65 21	_		31	

RINSATE-2MS RINSATE-2MSD TSB-CJ-09-0MS TSB-CJ-09-0MSD 

TSB-CJ-09-0

TSB-CJ-09-10\*\*

LDC #: 19191 A3b SDG #: <u>See COU</u>

### VALIDATION FINDINGS CHECKLIST

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

Method:	/_ (	3C	HPLC

Method:/_ GC HPLC				
Validation Area	Yes	No	NA	Findings/Comments
technicalholding times				
Il technical holding times were met.				
Cooler temperature criteria was met.				
Annual callus ation.				
Did the laboratory perform a 5 point calibration prior to sample analysis?		1		
Vas a linear fit used for evaluation? If yes, were all percent relative standard leviations (%RSD) < 20%?	/	_	-	
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?	ļ		-	
Did the initial calibration meet the curve fit acceptance criteria?	<del> </del>	ļ	<del>                                      </del>	
Nere the RT windows properly established?	] _		***	
y. Continuing calibration				
What type of continuing calibration calculation was performed?%D or				
%R				
Was a continuing calibration analyzed daily?  Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?	1			
	1/	1		
Were all the retention times within the acceptance windows?				AND THE RESERVE OF THE PERSON
V. Blanks		1		
Was a method blank associated with every sample in this SDG?	1/	+	1	
Was a method blank analyzed for each matrix and concentration?	<del> </del>	+	+	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
VII Serrogate spikes			7	
Were all surrogate %R within the QC limits?	-	+	-	
If the percent recovery (%R) of one or more surrogates was outside QC limits, wa a reanalysis performed to confirm %R?	s /	1_		
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?				
VII. Mairix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each				
were a matrix spike (MS) and matrix spike duplicate (MSS) analyzed to matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/	1		
Was a MS/MSD analyzed every 20 samples of each matrix?	/	1	_	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?			annia ak	
VIII*Laboratory/control/samples ************************************				Same and the same of the same
Was an LCS analyzed for this SDG?	4	4		
Was an LCS analyzed per extraction batch?	$\bot\!$		$\perp$	



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### **VALIDATION FINDINGS CHECKLIST**

Page: of 2 Reviewer: 2nd Reviewer:

	ГТ			
Validation Area	Yes	No	NA	Findings/Comments
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?			~ 0.000 0.000	
IX: Regional chalify Assurance and ordality Control 4.5.				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
X spanget compound identification				
Were the retention times of reported detects within the RT windows?		Sour Assessor	15770.55504	
XI: Composind quantilation/CRQLs			ı	
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII System performance				
System performance was found to be acceptable.				
XIII rojerallessessmentol dala (ad es 11 an 12				
Overall assessment of data was found to be acceptable.				
XIV. Electrophicales 1788 17 1987 17 17 17 18 18 18 18 18 18 18 18 18 18 18 18 18				
Were field duplicate pairs identified in this SDG?				
Were target compounds idetected in the field duplicates?				
XV. Field blanks				
Were field blanks identified in this SDG?				
Were target compounds detected in the field blanks?				

# **VALIDATION FINDINGS WORKSHEET**

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

A. alpha-BHC	I. Dieldrin	Q. Endrin ketone	Y, Aroclor-1242	GG.
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH.
C. delta-BHC	K. Endrin	S. aipha-Chlordane	AA. Aroclor-1254	H.
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.

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Notes:		

100 #:18/8/43P SDG #: 226

### VALIDATION FINDINDS WORKSHEET Surrogate Recovery

Page:

2nd Reviewer:\_ Reviewer:

METHOD: ✓ GC HPLC
Are surrogates required by the method? Yes ✓

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? M N/A

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

_	Sample	Detector	,,,,,,	ć							
#	Q)	Column	È E	Surrogate							
	N	4 /	(	C	$\parallel$	%K (Limits)				Qualifications	
		1/1	<u> </u>	850	-	533	57	(25)	1+1	4/4	
								1			
					-						
					-			^			
					+						
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					+			(			
					$\parallel$	)		(			
T					+			ſ			
					$\dashv$			(			T
					-	)		(			
	Surrogate Compound		Surrogal	Surrogate Compound		Surrogate Compound		Contraction			
∢	Chlorobenzene (CBZ)	ß	Oct	Octacosane	2	Renzo(e)Durono		Suitogate compound	ponua		
В	4-Bromofluorobenzene (BFB)	I	Ortho	Ortho-Terphenyl	z	alian (Control	,	1-Chloro-3-Nitrobenzene	enzene Y	Tetrachloro-m- xylene	
٥	a,a,a-Trifluorotoluene	-	Fluorob	Fluorobenzene (FBZ)	c	Occupientyi-U14		3,4-Dinitrotoluene	ene		
۵	Bromochlorobenene	 	n-Tr	n-Triacontane	٥	Decachioroppeny (DCB)	3	Tripentyltin			
Ш	1,4-Dichlorobutane	×	Ţ	Hexacosane	6	I-methylnaohthalene	>	Tri-n-propyttin			
	1.4-Difluorobenzene (DFB)	-	Brom	Bromobenzene	,	Distilled by Ideas Acid (DCAA)	≥	Tributyl Phosphate	nate		

LDC #:/9/9/ SDG #: 200

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

of	6	Y
Page:	Reviewer:	2nd Reviewer:

HPLC METHOD: GC\_

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

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

Recalculated Reported Recalculated	%RSD	(3.0	39164 9582 9.582								
Reported	Average CF (initial)	<b> </b>	39164								
Recalculated	CF (572 std)	75188	45676								
Reported	CF ( <i>SO</i> std)		45676								
	Compound	CAX-CLPOST	(H ) H)								
	Calibration Date	80/12/5									
······································	Standard ID	1946				•					
	#	-		,	1			က		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.

SDG #: See Coul LDC #: 19/9/1826

## Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Page: /of/	Reviewer:	Ond Reviewer.
	_	2nd

HPLC METHOD: GC

using the following calculation:

% Difference = 100 \* (ave. CF - CF)/ave. CF .CF = A/C

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

Where: ave. CF = initial calibration average CF CF = continuing calibration CF A = Area of compound C = Concentration of compound

-			T						Ī
Recalculated	Q%	2.5		8.0	2:2				
Reported	Q%	Z. &		9.	2.5				
Recalculated	CF/Conc. CCV	1027,438 1027,40		99/8768 992.0	09:5/01				
Reported	CF/Conc. CCV	884 (507)		8918166	1075.6662				
	Average CF(Ical)/ CCV Conc.	0001		020/	1000				
	Compound	BB (dut)		BB (Ch. A)	V (Ch.B)				
	Calibration Date	84/81/9		8//8//7					
	Standard ID	Editas		Pet 428					
	#	-		2		ო		4	L

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#:1919/36 SDG#26\_COM

# VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Reviewer: 2nd reviewer: 2

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

	Percent Difference				
	Percent Recovery	Recalculated	20		
	Percent Recovery	Reported	18	/	
	Surrogate Found		798561		
	Surrogate Spiked		30.08		
	Column/Detector		ch A		
Sample ID: 🎅	Surrogate		DOB		

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ample ID:						
Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	

Sample ID:

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

LDC #: 19191.A30 SDG #: 5ee agm

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

2nd Reviewer:

METHOD:

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

SC = Sample concentration

RPD =(((SSCMS - SSCMSD) \* 2) / (SSCMS + SSCMSD))\*100

MS/MSD samples:

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

MSD = Matrix spike duplicate

	- C	Cond.	Concent/ation	/ation						
ne (8015) ne (8021B)		Trans	4/	2	Percent Recovery	ecovery	Percent Recovery	covery	RPD	
9 9 9	205		MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
) ) )   O										
ואופווומוזם (הסהיונט)										
2,4-D (8151)										
Dinoseb (8151)										
Naphthalene (8310)										
Anthracene (8310)										
HMX (8330)										
2,4,6-Trinitrotoluene (8330)										
728 (73	174	P N	193	178	114	7	102	102	X:4	<i>\( \rangle \)</i>

Comments: Refer to Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualificatio 10.0% of the recalculated results.

MSDCLCNew.wpd

LDC #: 191836 SDG #: 500 COWN

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

2nd Reviewer: Reviewer:

> CGC HPLC METHOD:

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

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

SC = Concentration

LCSD = Laboratory control sample duplicate percent recovery

RPD = 1 SSCLCS - SSCLCSD 1 \* 2/(SSCLCS + SSCLCSD) LCS/LCSD samples: 8/703/5

SSC = Spiked sample concentration SA = Spike added LCS = Laboratory control sample percent recovery Where:

Recalc. CS/CSD RPD Reported Recalc. Percent Recovery CSD Reported Recalc. Percent Recovery CS Reported CSD Spiked Sample Conceptration (ACT) 00 CCS CSD 4 Adda င္ပ 2,4,6-Trinitrotoluene (8330) Naphthalene (8310) Methane (RSK-175) Anthracene (8310) Benzene (8021B) Gasoline (8015) Dinoseb (8151) Diesel (8015) 2,4-D (8151) HMX (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.

SDG #: 200 Com LDC #: 1919/Jah

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page: 2nd Reviewer: Reviewer:

> GC HPLC METHOD:

A/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?

(A)(Fv)(Df)	(RF)(Vs or Ws)(%S/100)	A sea or height of the company to be measured
Concentration=	(R)	Area or height
ဝ		Į

Compound Name\_

 $\emptyset$ 

Sample ID.

Example:

RF= Average response factor of the compound In the initial calibration A= Area or height of the compound to be I Fv= Final Volume of extract Df= Dilution Factor

Concentration =\_

Vs= Initial volume of the sample Ws= Initial weight of the sample %S= Percent Solid

Qualifications				
Recalculated Results Concentrations (				
Reported Concentrations (				
Compound				
Sample ID				
#				

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

<sup>\*\*</sup>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.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. Calibration

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)	Р

### 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	Р

### 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)	А
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)	А
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)	А

### 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)	Р

### 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:

Internal Standard	%R (Limits)	Analyte	Flag	A or P
Scandium-45	129.434 (30-120)	Silicon	J (all detects)	А
		Strontium	J (all detects) UJ (all non-detects)	
			Scandium-45 129.434 (30-120) Silicon	Scandium-45 129.434 (30-120) Silicon J (all detects) UJ (all non-detects) J (all detects)

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

### IX. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

### X. ICP Serial Dilution

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

Diluted Sample	Analyte	%D (Limits)	Associated Samples	Flag	A or P
TSB-GJ-08-10'L	Iron	10.4 (≤10)	All soil samples in SDG F8F130140	J (all detects)	А

### 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

				4 5	
SDG	Sample	Analyte	Flag	A or P	Reason
F8F130140	RINSATE-2	Thallium Uranium	J+ (all detects) J+ (all detects)	Р	Calibration (CCV %R)
F8F130140	RINSATE-2	Sulfur	None	Р	ICP interference check sample analysis (%R)
F8F130140	TSB-CJ-09-0 TSB-CJ-09-10**	Silicon Titanium Potassium Zinc	J+ (all detects)	А	Matrix spike/Matrix spike duplicates (%R)
F8F130140	TSB-CJ-09-0 TSB-CJ-09-10**	Magnesium	J (all detects) UJ (all non-detects)	А	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)	А	Matrix spike/Matrix spike duplicates (%R)
F8F130140	RINSATE-2	Palladium	J- (all detects) UJ (all non-detects)	Р	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)	А	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	Α
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

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

SDG	#: 19191A4 #: F8F130140 ratory: Test America	_ <b>VALII</b> 	OITAC		LETENI evel III/IV	ESS WORKS	SHEET	Date: 8/5/ Page: 1 of / Reviewer: Wy 2nd Reviewer: 9
MET	HOD: Metals (EPA SW 8	346 Metho	d 6020/6	8010B/700	00)			
	samples listed below wer ation findings worksheets		d for eac	ch of the fo	ollowing va	alidation areas.	Validation findin	gs are noted in attached
	Validation	Area					Comments	
I.	Technical holding times			A	Sampling d	ates: 6/12/4	8	
II.	Calibration			5N		/		
III.	Blanks			5W				
IV.	ICP Interference Check Sa	mple (ICS)	Analysis	3W				
V.	Matrix Spike Analysis			5W	2 M S	/ MSD		
VI.	Duplicate Sample Analysis			N	)	1		
VII.	Laboratory Control Sample	s (LCS)		3W	Les			
VIII.	Internal Standard (ICP-MS)	)		SW	N.T	ben'ewal!	~ \eu 3	>
IX.	Furnace Atomic Absorption	QC		N	N.+ 1	Milicel		
Χ.	ICP Serial Dilution			SW		4		
XI.	Sample Result Verification			A	Not review	ed for Level III valid	dation.	
XII.	Overall Assessment of Dat	a		A				
XIII.	Field Duplicates		· ·	N,				
XIV	Field Blanks			γW	R=)			
Note: /alida	A = Acceptable N = Not provided/applicable SW = See worksheet ted Samples: ** Indicates sam		R = Rins FB = Fie	ld blank	detected	D = Duplica TB = Trip b EB = Equip		
1	RINSATE-2	11	pr		21		31	
2	TSB-CJ-09-0 40-1	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	
Votes	S:							

### VALIDATION FINDINGS CHECKLIST

Page: of A Reviewer: uu 2nd Reviewer:

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

Method:Metals (EPA SW 846 Method 6010/7000/6020)				
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times		ki ili		
All technical holding times were met.	1	<u> </u>		
Cooler temperature criteria was met.			100.00	
II.:Calibration:				
Were all instruments calibrated daily, each set-up time?	/			
Were the proper number of standards used?	1		<b> </b>	
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)				
III UBlanks				
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.	/		and the same	
IV IGP lighterference Check Sample				
Were ICP interference check samples performed daily?	/			
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?	12-64-76-76		0.000	
IV: Matrix spike/Matrix spike duplicates				
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.		/		
V-Laboratory control samples:				
Was an LCS anaylzed for this SDG?	$\checkmark$			
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?		$\checkmark$		
limits for soils?  V) Furnace Atomic Absorption QC				
If MSA was performed, was the correlation coefficients > 0.995?			_	
Do all applicable analysies 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# 9 Af SDG#: Lee wow

### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 2
Reviewer: WM
2nd Reviewer: 9

Validation Area	Yes	No	NA	Findings/Comments
VII. ICR Serial Dilution				The state of the s
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the IDL?	Z			> 100 x mor for 20%
Were all percent differences (%Ds) < 10%?		/		<b>,</b>
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.		/		
Vill Internat Standards (EPA/SW/846/Method B02b)				
Were all the percent recoveries (%R) within the 30-120% of the intensity of the internal standard in the associated initial calibration?	ж	V		
If the %Rs were outside the criteria, was a reanalysis performed?		<b>V</b>	X	
IX Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?			V	
Were the performance evaluation (PE) samples within the acceptance limits?			/	
X, Sample Result Verification - to East, Surgary				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	1			
XI Overall assessment of geta				
Overall assessment of data was found to be acceptable.				
XII. Field duplicates				P. 6 (1.10)
Field duplicate pairs were identified in this SDG.		$\sqrt{}$	4000	
Target analytes were detected in the field duplicates.				
XIII.Fied.blanks				
Field blanks were identified in this SDG.			ocatica (f.)	
Target analytes were detected in the field blanks.	1			

LDC #: 1919 Ay SDG #: <u>( u we</u>

### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page:_	of	
Reviewer:	hun	
2nd reviewer:	q	

All circled elements are applicable to each sample.

Sample ID	Matrix	Target Analyte List (TAL)
1-3	Mafsoil	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
n45	soil	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
1-3	102/50:	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
m 4-5	1.مو	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,
	10	Analysis Method
ICP		(Li,S)
CP-MS		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
CP-MS		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Zt.
GEAA		Al, Sh, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Ph, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN

ICP-MS

Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Zi.

GFAA

Al, Sh, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Ph, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,

Comments: Mercury by CVAA if performed

Nb: Niobium, Pd: Palladium, P: Phosphorus, Pt: Platinum, S: Sulfur, W: Tungsten, U: Uranium, Zr: Zirconium

LDC #: (9 (9 | 16

### VALIDATION FINDINGS WORKSHEET Calibration

Reviewer:\_\_\_\_ Page:

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

Were all instruments calibrated daily, each set-up time, and were the proper number of standards used?

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 KON Was

Man N/A Are

N N/A Wes

Was a midrange cyanide standard distilled?

Are all correlation coefficients >0.995?
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	
<u> </u>	8-12-19 1	(0124) /22		7:31	F1 A	なけん	
L			7	1145	7	7	
			_				
<u></u>							
<u></u>							
L							
L							
Ш							Π
<u> </u>							
ı ŏ	Comments						

SDG #: See Cover LDC #: 19191A4

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000) Sample Concentration units, unless otherwise noted: ug/L

VALIDATION FINDINGS WORKSHEET PB/ICB/CCB QUALIFIED SAMPLES

Page: of Y

Reviewer: MAZ 2nd Reviewer: Q

Sample Identification Blank Action Maximum ICB/CCB<sup>a</sup> (1011) 5. 0.1 2.7 Maximum PB<sup>a</sup> 0.89 21.5 Maximum PB<sup>a</sup> mq/Ka) Analyte S Cd Fe

Samples with analyte concentrations within five times the associated ICB, CCB or PB concentration are listed above with the identifications from the Validation Completeness Worksheet. These sample results were qualified as not detected, "U".

Note: a - The listed analyte concentration is the highest ICB, CCB, or PB detected in the analysis of each element.

LDC #: 19191A4 SDG #: See Cover

Sample Concentration units, unless otherwise noted: \_mg/Kg, except Hg ug/Kg\_ METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

VALIDATION FINDINGS WORKSHEET PB/ICB/CCB QUALIFIED SAMPLES

Soil preparation factor applied:

Associated Samples: All Soil

Page: 2 of 7 Reviewer: 2nd Reviewer:

Sample Identification																	
Sample															-		
			.44				16.5										
	2 3		0.40 / 0.44	71.0 1.1/1.1			21.2 / 36.5										
	Blank Action 2		0.22	0.54 / 1		**************************************											
	Maximum EICB/CCB <sup>a</sup> A (ug/l)		1.1	1.5	2.7	8.0	0.1										
	Maximum PB <sup>a</sup> (ug/l)																
	Maximum PB <sup>a</sup> (mg/Kg)																
	Analyte	Sb	П	W	۸	Į.	Hg (ug/Kg)										

Samples with analyte concentrations within five times the associated ICB, CCB or PB concentration are listed above with t qualified as not detected, "U".

Note: a - The listed analyte concentration is the highest ICB, CCB, or PB detected in the analysis of each element.

19191A4.wpd

SDG #: See Cover LDC #: 19191A4

# **VALIDATION FINDINGS WORKSHEET**

Field Blanks

Reviewer: MH 2nd Reviewer:

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

Were field blanks identified in this SDG?

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

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

ď Field blank type: (circle one) Field Blank / Rinsate / Other: Soil factor applied Sampling date: 6/12/08

Associated Samples: All Soil

Sample Identification 0.40 / 0.44 ന Action Level 118.2 Blank ID 48.2 11.0 0.80 59.1 6.1 Analyte င္မ æ δ Na ര് F

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".

SDG #:

# VALIDATION FINDINGS WORKSHEET ICP Interference Check Sample

Reviewer: МИ Вeviewer: Page: of 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 ICP interference check samples performed as required?
| Y | N | N | Were the AB solution percent recoveries (%R) within the control limits of 80-120%?

| LEVEL IV ONLY:
| Y | N | N | Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations.

	4	T	7	ī	_					1			7	 7		1	i	I
Qualifications	wore/ ( Riverta No interpera	2																
Associated Samples	411 AA																	
Finding	75.4																	
Analyte	δ,																	
ICS Identification																		
# Date	8-16/9 1	-															Offinitering.	

(9.6. SDG #: LDC #:

### Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

Page: Reviewer:\_ 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".

NA N/A Was a matrix spike analyzed for each matrix in this SDG?

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/A

Were all duplicate sample relative percent differences (RPD)  $\leq$  20% for water samples and  $\leq$ 35% for soil samples? Y N N/A W

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

Y) N/A

				MS	OSW	428		
	MS/MSD ID	Matrix	Analyte	%Recovery	%Recovery	RPD (Limits)	Associated Samples	Qualifications
	4/2	\$3;	5b	53.5	55.4		1,05 H	I-/45/4
		\	Ма	64,6	1 (6   1			V/ 13/15
1			09/	42.1	465			W/ H1/-15
			5,	393.1	361-5			ンナイナ
			í- F	731.7	900.9			<b>N</b>
			146	52.6				7-1434
			K 0		128.9			
			ر ح		8.40			7- /ux/n
			2n 3x		(250)			1 2 9 1
1			Mark			23.6		A 4 P
			H.g			₹ 7° ¢		( ) ( ) ( ) ( ) ( ) ( ) ( ) ( )
			à			3.7		
	,		P.					
	-							
								*
1								
1								
Com	Comments:	Ca, Fe	Mn 7 4X	×				
				-				

1919/49 LDC #: SDG #:\_

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

Page: Reviewer: 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".  $\frac{\sqrt{N_0}}{\sqrt{N_0}}$  Was a laboratory control sample (LCS) analyzed for each matrix in this SDG?

YNNA Was a laboratory control sample (LCS) analyzed for each matrix in this SDG?
YNNA Were all aqueous LCS percent recoveries (%R) within the control limits of 80-120% and all soil LCS %R within laboratory established control limits. LEVEL IV ONLY:

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

*	CS ID	Matrix	Analyte	%R (limits)	Associated Samples	Qyalffcations
_	103	1 dg	Pa	( SU-112) 07/8	A1 A4	d /2n/-∑
			-			
L						
Comments:	ents:					

# VALIDATION FINDINGS WORKSHEET Internal Standards (ICP-MS)

Page: of Beviewer:

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 internal standard in the initial calibration standard?

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

II THE TESPOISE TO BITTED OF THE ADOVE THESTON IS TO, WELD THE SATISTICAL AS TOTALLED .	l Samples	76434												
		1-14	Sγ											
T N/N/A II (THE LESPOINSE TO E	*	3,45												

LDC #: (9191A

# VALIDATION FINDINGS WORKSHEET **ICP Serial Dilution**

Page: 2nd Reviewer: Reviewer:

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? N N/A N/N/A N/A

Were ICP serial dilution percent differences (%D) <10%?

Is there evidence of negative interference? If yes, professional judgement will be used to qualify the data.

(A) N/A W

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

			is acceptable	200 - 200	Necessary VVO		
#	t Date	Diluted Sample ID	Matrix	Analyte	%D (Limits)	Associated Samples	Qualifications
		TSB-9J-08-101	501)	τe.		A. S	4/4
							1
<u> </u>							
Ш							
Ш							
<u>_</u>							
ပိ	Comments	14 A 2001 A 2001	\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \				
)							

SDG #: (9191A4

# VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

Page: of Reviewer: Mtg

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 = <u>Found</u> × 100 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

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
771	ICP (initial calibration)	[]	4.67	000	(1)	6101	Y
	GFAA (Initial calibration)					,	
75	CVAA (Initial calibration)	149	2,33	2, 50	£%b	43,2	>
cer	ICP (Continuing calibration)	2	52610	00005	5'501	(عد }	$\rightarrow$
	GFAA (Continuing calibration)						
cu	CVAA (Continuing calibration)	(45	4.89	0°S	816	816	}
M	ICP/MS (Initial calibration)	) <u>7</u>	10/8-9	(000)	6-10)	10/9	
41	ICP/MS (Continuing callbation)	>N	2601	( 00 J	10)	7 %	<u> </u>

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.

SDG #: Sel cont LDC #. 1919/AN

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

Page: \_\_of\_ 2nd Reviewer: Reviewer:\_\_\_

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 = Found x 100 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:

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

RPD = IS-DL × 100 (S+D)/2

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

%D = 1-SDR x 100

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

					Recalculated	Renorted	
Sample ID	Type of Analysis	Element	Found / S / I (units)	True / D / SDR (units)	%R/RPD/%D	%R/RPD/%D	Acceptable (Y/N)
TUSARS	ICP interference check	Ag	(9. pb)	Z	gr.J	92-5	<b>&gt;</b>
421	Laboratory control sample	Ba	887	95	8.8	93.9	
f	Matrix spike	L)	(SSR-SR)	£'\$0)	800)	8.00)	
5/7	Dupilcate	3	ンさ	2225	8.5	J, à	
TSB-45-100 serial dilution	ICP serial dilution	رود	Laly	ffing	9.9	2.9	Jo

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	ALP ,
SDG #:	Ce	cover

# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	1012
Reviewer:	my
2nd reviewer:	$\mathcal{I}$

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

		•			
Please Y N Y N Y N	see qua N/A N/A N/A	alifications below for all question Have results been reported an Are results within the calibrate Are all detection limits below t	d calculated correctly?  d range of the instruments	•	
	ed analy	rte results for	3	were recalculate	d and verified using the
Concent	•	(RD)(FV)(Dil) (in. Vol.)(%S)	Recalculation:	1	
RD FV In. Vol. Dil	=======================================	Raw data concentration Final volume (ml) Initial volume (ml) or weight (G) Dilution factor	B= 18013 4	12 X 0, 12 X 2 0-59 X 0.9132	= 7.91 63 mg/mg
%S	=	Decimal percent solids			

Sample ID	Analyte	Reported Concentration ( Wy / kg )	Calculated Concentration ( Mg 上な)	Acceptable (Y/N)
ζ	Al	9530	9530	4
	Az	10-6	10.6	1
	. Ba	91.6	91%	
	Be	0.58	0.78	
	B	2,9	1.9	
	<u> </u>	0,083	0,083	
	Ca	30/00	30100	
	4	12.1	[21]	
	Co	6.7	6.7	
	Gu	13.8	13.8	
	l-e	13000	13000	
	Pb	8.3	8.3	
	<u>Mg</u>	9800	9800	
	Mn	312	312	
	Mo	0.45	0,45	
	N.	(4.1	14.	
	þa	0-59	0-57	
	P	149	743	
	<u> </u>	1770	1770	
	31	£>3	523	
	Aq	0.15	0,15	
	Va	1870	1820	.//

LDC #:	19191A	$\frac{\varphi}{}$
SDG #:_	Cu	wen

# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	101 1
Reviewer:_	my
2nd reviewer:	V

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

Please Y N M N	see qua N/A N/A N/A	alifications below for all questi Have results been reported Are results within the calibra Are all detection limits below	ions answered "N". Not applicable of and calculated correctly? ated range of the instruments and w with the CRDL?	questions are identified as vithin the linear range of the	"N/A". e ICP?
	ed analy ng equal	rte results fortion:	3	were recalculated ar	nd verified using the
Concent	ration =	(RD)(FV)(Dil) (In. Vol.)(%S)	Recalculation:		
RD FV	=	Raw data concentration Final volume (ml)	7r= ±7.735	X 0.18 X2	_ = 25, 29 mg/mg/mg
In. Vol. Dil %S	= =	Initial volume (ml) or weight (G) Dilution factor Decimal percent solids	Č	-3 0× -1113	

Sample ID	Απαίγτο	Reported Concentration ( Wy Lu )	Calculated Concentration ( \Mg \rightarrow \Cappa )	Acceptable (Y/N)
3	Sr	291	2910	Y
	Tl	0,40	6,40	
	SN	0.57	0.55	
	Tì	193	593	
	W	1-1		
	y	2./	40,2	
	<u> </u>	40.2		
	₹h	<u> </u>	33-0	
	<del></del>	75.3	72'3	
	Hg 1 mg/mg	7 7,2	ン、ン	/
	10	1		7

# 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

<sup>\*\*</sup>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, Chorine, 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.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

# I. Technical Holding Times

All technical holding time requirements were met.

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

### II. Calibration

## a. Initial Calibration

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)	Α

# 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

SDG	Sample	Analyte	Flag	A or P	Reason
F8F130140	TSB-CJ-09-0 TSB-CJ-09-10**	Oil & grease	J- (all detects) UJ (all non-detects)	А	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

Method 300.0), O 8	nerica  b) Bromide, G (EPA S) below were	Bromi W846	ne, Chlorate Method 90	Le e, Chloride 718 <b>アモ</b> 収)	evel III/IV e, Chorine, A (664/	Fluoride, Nitrate, N	litrite Orthoph	Page:
	Validation	Area				С	omments	
Technical hold				A	Sampling da	//		
Ila. Initial calibrati				A		1		
Ilb. Calibration ve				A			\	
III. Blanks				SW				
IV Matrix Spike/I	//atrix Spike D	uplicate	es	5W	) h	5/M50/bup		
V Duplicates				Α	7			
VI. Laboratory co	ntrol samples			A	Les/Le	5D		
VII. Sample result				<b>A</b> -	Not review	ed for Level III validation	on.	
VIII. Overall asses				A				
IX. Field duplicat				Ŵ				
X Field blanks				5~	R=			
Note: A = Acceptal N = Not provi SW = See wo Validated Samples:	ded/applicable orksheet		R = Rin	eld blank		D = Duplicate TB = Trip blant EB = Equipme		
1 RINSATE-2	As	11			21		31	
2 TSB-CJ-09-0	500)	12			22		32	
3 TSB-CJ-09-10*	1.	13			23		33	
4 RINSATE-2MS	ps	14			24		34	
5 RINSATE-2DUI	L	15			25		35	
6 TSB-CJ-09-0M	Sail	16			26		36	
7 TSB-CJ-09-0M	SD J	17			27		37	
8 TSB-CJ-09-0DL		18			28		38	
9 MB		19			29		39	
10		20			30		40	
Notes:								

LDC #:	19191	\$6
SDG #:	Ses	cover

Method: Inorganics (EPA Method Sel Cylin

### **VALIDATION FINDINGS CHECKLIST**

Page: 1 of 1 Reviewer: 1 pm 2nd Reviewer: 1

Findings/Comments Validation Area ( Technical holding brues All technical holding times were met. Cooler temperature criteria was met. 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) 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. CONTROL OF THE PROPERTY OF THE 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

Was an LCS anaylzed for this SDG?

Was an LCS analyzed per extraction batch?

within the 80-120% (85-115% for Method 300.0) QC limits?

VI. Resonated and Quality Control

Were performance evaluation (PE) samples performed?

Were the LCS percent recoveries (%R) and relative percent difference (RPD)

Were the performance evaluation (PE) samples within the acceptance limits?

LDC #:	19/91	46
SDG #:	(u	ww/

## VALIDATION FINDINGS CHECKLIST

Page: Vof Y Reviewer: W4/ 2nd Reviewer: V

	T	<del></del>	T	
Validation Area	Yes	No	NA	Findings/Comments
VII. Sample Basiuti Verflicaton			îŽi:	(*************************************
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	^			
Were detection limits < RL?				
<b>建设地区地区地区地区地区</b>		關		
Overall assessment of data was found to be acceptable.	1			
Field duplicate pairs were identified in this SDG.				
Target analytes were detected in the field duplicates.				
Field blanks were identified in this SDG.				
Target analytes were detected in the field blanks.	/		T	

LDC #:_	91917	16
SDG #:_	Su	cove-

# VALIDATION FINDINGS WORKSHEET Sample Specific Analysis Reference

Page: \_\_\_of \_\_\_ Reviewer: \_\_\_\_\_\_ 2nd reviewer: \_\_\_\_\_\_

All circled methods are applicable to each sample.

Sample ID	Matrix	Parameter
1-3	501/102	Br Bromine Cl Chlorine F NO, NO, SO, O-PO, Chlorate CIO, Q+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
a 4-5	AD.	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
16.1	ζο;	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
26.8	<u> </u>	Br) Bromine C) Chlorine 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:		 ·	 
*			
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# VALIDATION FINDINGS WORKSHEET

Blanks

Page: of

Reviewer: 2nd Reviewer:

METHOD: Inorganics, Method

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

| N | N/A | Were all samples associated with a given method blank?
| N | N/A | Were any inorganic contaminants detected above the reporting limit in the method blanks? If yes, please see qualifications below.

3

					 		******	 		 			 	
											,	,		
ECB1:3 ( NO)														
ec B 2 2	Sample Identification													
CB 1: 2	Sample			-										
.														
Associated Samples:														
Associated														
	Blank	Action LIM		•										
ž	Maximum	ICB/CCB W C/V	20/0		0,126	•								
1	Blank ID													
Conc. units: W.	Analyte		X	d-had-o	0-124-P									
			-3		1482	5								

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

LDC #: 1919 A6 SDG #: See core

# VALIDATION FINDINGS WORKSHEET FIELD FIELD BIANKS

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Page:	Reviewer:	Reviewer:
		2nd

u	Sample Identification		Blank	Blank ID	Analyte
A1 501 6 (>TX)	Associated Samples:	Blank / Rinsate / Other: 段	one) Field	eld blank type: (circle one) Field Bl	eld blank
		Soil factor applied	80/2-	ate: 6	ampling date:
		d sample units: Phylogen	My Associated		lank units:
		s detected in the field blanks?	get analyte:		N/A
		Were field blanks identified in this SDG?	d blanks id		N N/A
2nd Reviewer:		fre Course	A Method	ETHOD: Inorganics, EPA Method	ЕТНОВ: 1

Analyte	DIANK ID						Sample Identification	ntification		
		Action Limit								
504	11.0									
-										
							,			
							,			
Blank units:		Associate	d sample un	ils.						
Sampling da	ate:		Soil fact	or applied	ı.					
Field blank type: (circle one) Field Blank / Rinsate / Other:	type: (circle	one) Field	Blank / Rins	ate / Other:		Asso	Associated Samples:	les:		

Analyte	Blank ID	Blank					Sample Id	Sample Identification		
		Action								
CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE CITATIFIED BY THE FOLLOW	CHOLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE DIJATIFIED BY THE FOIL OWING STATEMENT	TOUATIFIED	ALL RESULT	S NOT CIRCLE	D WERE OUT	IEIEN BV 112E	TOT CAMBRIS ES			

blank concentration are listed above, these sample results were qualified as not detected, "U".

1919 46 LDC #:

# Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Page: Reviewer:\_

METHOD: Inorganics, EPA Method\_

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

Y) N/A

Was a matrix spike analyzed for each matrix in this SDG?

Were matrix spike percent recoveries (%R) within the control limits of 75-125? If the sample concentration exceeded the spike concentration by a factor Y NA NA

Were all duplicate sample relative percent differences (RPD)  $\leq$  20% for water samples and  $\leq$ 35% for soil samples? of 4 or more, no action was taken. (Y) N N/A WA

Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations. AN NA

` [								
*	ai asw/sw	Matrix	Analyte	MS %Recovery	MSD %Recovery	RPD (Limits)	Associated Samples	Qualifications
=	6/9	501-	1 5+0	<u> </u>	<u> </u>		1 /2:05 118/	J-/nJ/4
			,					
					-			
ا ا	Comments:					-		
			***************************************	The second secon	**************************************			

9/1/6/6/ LDC#:

# Initial and Continuing Calibration Calculation Verification Validatin Findings Worksheet

Reviewer: Mul

Method: Inorganics, Method

he wen

was recalculated.Calibration date:\_ The correlation coefficient (r) for the calibration of \_\_

An initial or continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula: Where, %R = Found X 100

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

					Recalculated	Reported	Acceptable
Type of analysis	Analyte	Standard	Conc. (ug/L)	Area	r or r²	r or r²	(Y/N)
Initial calibration		s1	500	0.034			
	CIO3	s2	2000	0.151	96666.0	0.99991	>
		83	4000	0.317			,
		84	10000	0.777			
		s5	20000	1.586			
$\mathcal{L}$ Calibration verification	Les?	4	128%		5.95	MR	>
$\mathcal{L}_{\mathcal{N}}$ Calibration verification	dtago	&	8,302		۲0°0°)	(00,0)	
$c_{\mathcal{L}}$ Calibration verification	M-201	09170	p 6910		(02,13	j 1, ≤0)	->

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.

(9/9/A6 SDG #: # CO

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

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

METHOD: Inorganics, Method

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

Where %R = Found x 100

Found =

concentration of each analyte <u>measured</u> in the analysis of the sample. For the matrix spike calculation, Found = SSR (spiked sample result) - SR (sample result), concentration of each analyte in the source. True ==

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

 $RPD = \underbrace{1S \cdot D!}_{(S+D)/2} \times 100 \text{ Where,}$ 

(S) (D)

Original sample concentration Duplicate sample concentration

	-				Receipulated	Reported	
Sample ID	Type of Analysis	Element	Found / S (units)	True / D (unita)	%R / RPD	%R/RPD	Acceptable (Y/N)
	Laboratory control sample						•
Lcs		0+14	251	(330	44	94	<b>&gt;</b>
Q.	Natrix spike sample	1	89.99 (AS-ASS)	6.6%	76	96	
8	Dupilcate sample	ન	81,785	5/2/2	0.3)	a.35	

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

LDC #:_	1919/16
	ie com
	,

# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	1 01
Reviewer:	Ми
nd reviewer:	¥

<del></del>		2nd reviewer:
METHOD: Inorganics, Method	Su con	
Have results been reported Are results within the calibration limits below	d and calculated corrected range of the insow the CRQL?	Not applicable questions are identified as "N/A". ectly? struments? reported with a positive detect were
Compound (analyte) results for	g equation:	
F = Area  O.287	Recalculation:	0.215 X 4 9 X 0.913 = 8.2 mg/m

#	Sample ID	Analyte	Reported Concentration (W~\/~(\)	Calculated Concentration ( W. M., )	Acceptable (Y/N)
	3	cl03	16.4	16rg	<u> </u>
<b></b>			900		
<b> </b>		d>	1800	[800	
ļ		F ,	1.9	8.2	
		603-7V 504	170	170	
<b> </b>		, , , ,	1,0	1/0	
<u> </u>			5		
-					
<b>_</b>					-
<u> </u>			<u></u>		-
-			<u> </u>		<del>                                     </del>
-					1
	-				

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

<sup>\*\*</sup>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.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.

None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

# I. Technical Holding Times

All technical holding time requirements were met.

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

## II. Calibration

## a. Initial Calibration

Initial calibration of compounds was performed as required by the method.

The percent relative standard deviations (%RSD) of calibration factors for compounds were less than 20.0%.

## b. Calibration Verification

Calibration verification was performed at required frequencies. The percent differences (%D) of amounts in continuing standard mixtures were within the 15.0% QC limits.

The percent differences (%D) of the second source calibration standard were less than or equal to 15.0% for all compounds.

### III. Blanks

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

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

SDG#	: 19191A7 : F8F130140 atory: Test America	\	/ALIDATIO		PLETEN evel III/		SS WORKSHEET		Date: <u>8/5/0</u> Page: <u>/of /</u> Reviewer:
	<b>OD:</b> GC Gasoline R	ange C	Organics (EPA	\ SW846 N	Method 8	01	5B)		2nd Reviewer:
	amples listed below vion findings workshe		viewed for ea	ch of the f	ollowing	val	lidation areas. Validatio	n findi	ngs are noted in attached
	Validat	ion Ar	ea				Comm	ents	
١.	Technical holding times	S		<del> </del> <del> </del>	Sampling	da da	tes: 6/12/00	35	
IIa.	Initial calibration			À			/ '		
IIb.	Calibration verification/	ICV		\$	lev	/ =	= 15%		
111.	Blanks			A					
IVa.	Surrogate recovery			1					
IVb.	Matrix spike/Matrix spik	e duplic	ates	<b>*</b>					
IVc.	Laboratory control sam	ples		A	100	<u> </u>	\$		
V.	Target compound ident	tification		<b></b>	Not revie	we	d for Level III validation.		
VI.	Compound Quantitation	n and CF	RQLs	1	Not revie	ewe	d for Level III validation.		
VII.	System Performance			<b>1</b>	Not revie	ewe	d for Level III validation.		
VIII.	Overall assessment of	data		1					
IX.	Field duplicates			N					
X	Field blanks			ND	2=	L			
Note:	A = Acceptable N = Not provided/appl SW = See worksheet	icable I	R = Rinsate	o compound			D = Duplicate = Trip blank EB = Equipment blani	k	
Validate	d Samples: **		s sample underw	vent Level IV	validation			<del></del>	· · · · · · · · · · · · · · · · · · ·
1 !	RINSATE-2	W 1		739M	<u>3 21</u>	4		31	
2	TSB-CJ-09-0	9 12	81681	41 MB	22	+	5	32	
3	TSB-CJ-09-10**	1/ 13	3		23	$\perp$		33	
4 1	RINSATE-2MS	W 14			24			34	
5 1	RINSATE-2MSD	15	<u> </u>		25	_		35	
6	TSB-CJ-09-0MS	9 10	8		26	4		36	
7 -	TSB-CJ-09-0MSD	V 17	,		27	$\perp$		37	
8		18	3		28	$\downarrow$		38	
9		19	)		29	$\perp$		39	
10		20	)		30	$\perp$		40	
Votes:									

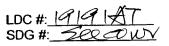
LDC #: 19191A7 SDG #: <u>Sector</u>

# VALIDATION FINDINGS CHECKLIST

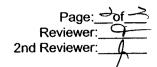
Page: of Reviewer: 1

Method:	V	GC	 HPLC	

Validation Area  Yes No NA Findings/Comments  I secanical holding times  All technical holding times were met.  Cooler temperature criteria was met.  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	5
All technical holding times were met.  Cooler temperature criteria was met.  It initial calibration  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	
All technical holding times were met.  Cooler temperature criteria was met.  It is fault calibration.  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	
Cooler temperature criteria was met.  # fanial calis class:  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	
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	
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	
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?	(***)
1V-Continuing calibration	
What type of continuing calibration calculation was performed?%D or %R	
Was a continuing calibration analyzed daily?	
Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?	
Were all the retention times within the acceptance windows?	
V:Blanks	
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.	
Vi Sprogate spikes	
Were all surrogate %R within the QC limits?	
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?	
VII. Manus spike Manus spike duplicates 1988 1988 1988 1988 1988 1988 1988 198	
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?	
VIII*Laboratory control samples: ***	
Was an LCS analyzed for this SDG?	
Was an LCS analyzed per extraction batch?	



# **VALIDATION FINDINGS CHECKLIST**



Validation Area	Yes	No	NA	Findings/Comments
Were the LCS percent recoveries (%R) and relative percent difference (RPD)	163	110	•••	1 manga communic
within the QC limits?				
IX: Regional Quality Assurance and additive country.				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
A garget compound identification ( ).				
Were the retention times of reported detects within the RT windows?				
XI. composind quantitation/CRQLS				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XI/ASYstemperformance				
System performance was found to be acceptable.				
XIII Overallarssessment of data (2) 45 45 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5				
Overall assessment of data was found to be acceptable.				
Xiv. Field upplicates				
Were field duplicate pairs identified in this SDG?				
Were target compounds idetected in the field duplicates?				
XV. Field Hanks	tegi.			
Were field blanks identified in this SDG?				
Were target compounds detected in the field blanks?		/		

LDC#:191947 SDG#:2ec.Cown

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

2nd Reviewer:\_\_ Reviewer:

> HPLC METHOD: GC\_

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

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

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
Calibration Standard ID Date	Calibration Date		Compound	CF (0.7 std)	CF (0. std)	Average CF (initial)	Average CF (initial)	%RSD	%RSD
0 pt 12/2/2 1/2/	0 XX	A80		00/ESE 81	18352,20	21852 1835 17182/32 17182/33 3.915 3.915	17182732	3.915	3,915
10 2/0/s	80/10/5						,		
								·	

Comments: Referto 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 #: 1919147 SDG #: 5ee COWN

# Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer:\_ Reviewer:

> HPLC METHOD: GC\_

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

% Difference = 100 \* (ave. CF - CF)/ave. CF CF = A/C

Where: ave. CF ≈ initial calibration average CF CF = continuing calibration CF

punoduoo	tration of compound
Area of	Concen
۱	Ü

Recalculated	Q%	8							
Reca	_	3.0							
Reported	<b>0</b> %	8.8							
Recalculated	CF/Conc. CCV	1.0350							
Renorted	CF/Conc. CCV	1.0360							
	Average CF(Ical)/ CCV Conc.	0./							
	Compound	00 p							
	Calibration Date	3 . / . / .	21/91/9						
	Standard ID	SMEH-7407				-			
	#:	-		2		က		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 #: 1919/47 SDG #: 200 COULV

# VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Reviewer: 2

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:

Percent Difference Recalculated Percent Recovery 0 Reported Percent Recovery 0 0.03592 Surrogate Found Surrogate Spiked 0.04 Column/Detector V Surrogate

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

# Sample ID:

Spiked

SDG #22 CON LDC #: 1919-14

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

2nd Reviewer: Page:

> ပ္ပ METHOD:

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

SC = Sample concentration

RPD =(((SSCMS - SSCMSD) \* 2) / (SSCMS + SSCMSD))\*100

MS/MSD samples:

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

MSD = Matrix spike duplicate

NO Recalc. 0 MS/MSD RPD  $_{o}^{\omega}$ Reported Recalc. Matrix Spike Duplicate Percent Recovery Reported 9 Ü Recalc. Percent Recovery Matrix spike 4 Reported 856 MSD Concentration Spike Sample 0 356 SΕ Sample Conc MSD Ø 40 WS 2,4,6-Trinitrotoluene (8330) (RSK-175) (8021B) (8015) (8310) (8015) (8151)(8330)(8151) (8310) Compound Naphthalene Anthracene Gasoline Benzene Methane Dinoseb Diesel 2,4-D Χ×Ε

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 #: 00 97.4 SDG #W

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

2nd Reviewer: Reviewer:\_\_

METHOD: V 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:

SC ≈ Concentration

RPD = I SSCLCS - SSCLCSD I \* 2/(SSCLCS + SSCLCSD)

SSC = Spiked sample concentration
SA = Spike added
LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples: 8/68/4

	้	pike	Spiked	Sample	TCS	S	TC	rcsp	/SOT	TCS/TCSD
Compound	( ) A	MS/B	Conce MS	Concentration MF/FS	Percent Recovery	Recovery	Percent f	Percent Recovery	æ	RPD
	SOT	TCSD	, SOT	CSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)	1.0	1.0	4560	0.918	95	95	93	93	S. 6	3.9
Diesel (8015)										
Benzene (8021B)										
Methane (RSK-175)										
2,4-D (8151)										
Dinoseb (8151)										
Naphthalene (8310)										
Anthracene (8310)										
HMX (8330)								5		
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 #: 779747 SDG #: 24 COUN

# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

METHOD: / GC HPLC

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= (A)(Fv)(Df)	Example:		
IF.	(K)	Compound Name	NB
A= Area or height of the compound to be measured Fv= Final Volume of extract Of= Dilution Earthr			

Concentration =\_

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
<u> </u>					
Somments:	ents:				

### 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

<sup>\*\*</sup>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.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.

None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. Calibration

### a. Initial Calibration

Initial calibration of compounds was performed as required by the method.

The percent relative standard deviations (%RSD) of calibration factors for compounds were less than 20.0%.

### b. Calibration Verification

Calibration verification was performed at required frequencies. The percent differences (%D) of amounts in continuing standard mixtures were within the 15.0% QC limits.

The percent differences (%D) of the second source calibration standard were less than or equal to 15.0% for all compounds.

### III. Blanks

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

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

SDG # _abora	t: <u>19191A8</u> #: <u>F8F130140</u> atory: <u>Test America</u> <b>IOD:</b> GC Diesel Range C	- -		Le	evel	III/I\	′	ORKSHEE	Т	Date: \$\frac{5}{0}\$  Page: of /  Reviewer: 2nd Reviewer:
	tion findings worksheets.			ch of the fo	ollow	ing va	alidation	areas. Valida	tion findi	ngs are noted in attached
	Validation	<u>Area</u>		н				Com	ments	
1.	Technical holding times	····		A-	Sam	pling d	ates:	6/12/0	8	
IIa.	Initial calibration			<del>4</del>						
IIb.	Calibration verification/ICV			<u> </u>	K	2V =	3/5/	0		
III.	Blanks			4					<u>,</u>	
IVa.	Surrogate recovery			4						
IVb.	Matrix spike/Matrix spike du	olicate	s	<b>★</b>	ļ					
IVc.	Laboratory control samples			<b>₫</b>		10	<u> </u>			
V.	Target compound identificati	on		A	Not	review	ed for Lev	el III validation.		
VI.	Compound Quantitation and	CRQ	_s	$\triangleleft$	Not reviewed for Level III validation.					
VII.	System Performance	System Performance		Not reviewed for Level III validation.						
VIII.	Overall assessment of data									
IX.	Field duplicates									
Х.	Field blanks			ND	2	=	-			
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	11	81692	4/11	3	21			31	
	TSB-CJ-09-0	12	817031			22			32	
	TSB-CJ-09-10**	13				23			33	
	- V	14				24			34	
	RINSATE-2MSD	15				25			35	
	TSB-CJ-09-0MS	16				26			36	
7	TSB-CJ-09-0MSD	17				27			37	
8	<b>V</b>	18				28			38	
Ħ						<u> </u>				

Notes:\_

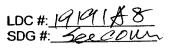
20



### VALIDATION FINDINGS CHECKLIST

Method: GC HPLC

Method: GC HPLC			,	
Validation Area	Yes	No	NA	Findings/Comments
f Tastuicalmoiding tures				
All technical holding times were met.				
Cooler temperature criteria was met.	7			
		11.00		
(Philia calibration)				
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%?	/		<u> </u>	
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?		e e e e e e e e e	NEW MARS (NO	
1V. Continuing calibration				
What type of continuing calibration calculation was performed?%D or%R				
Was a continuing calibration analyzed daily?	//	1		
Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?	<u> </u>	<b> </b>		
Were all the retention times within the acceptance windows?				
V/Blacks				
Was a method blank associated with every sample in this SDG?	/	1		
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.		/		
V. Surrogale spikes				
Were all surrogate %R within the QC limits?				
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?			/	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?			/	
VII. Marrix spike/Martx spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	1	1		
Was a MS/MSD analyzed every 20 samples of each matrix?	1			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII?Laboratory.comretsamples 70%				
Was an LCS analyzed for this SDG?	(			
Was an LCS analyzed per extraction batch?	1			
I VI do dil LOO dilaiyeed poi ova dodoi oddoi.				



### **VALIDATION FINDINGS CHECKLIST**

Page: of 2
Reviewer: 2nd Reviewer:

Validation Area	Yes	No	NA	Findings/Comments
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX: Regionals evallity Assurance and dealify (control v				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
X stanger compound identification				
Were the retention times of reported detects within the RT windows?				
XI: Compound quantifation/CRQLS				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII System ceromance — — — — — — — — — — — — — — — — — — —				
System performance was found to be acceptable.				
XIII Toverall assessment of data (w. 1773). The control of the con				
Overall assessment of data was found to be acceptable.				•
XIV: Field applicates				
Were field duplicate pairs identified in this SDG?			<u> </u>	
Were target compounds idetected in the field duplicates?	İ	,	/	
XV. Fieldblanks				
Were field blanks identified in this SDG?				
Were target compounds detected in the field blanks?				

SDG #: LECON LDC #: 19/9/ 48

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

100		م
Page:	Reviewer:	and Reviewer.

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

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 Date Compound    CALL   Standard ID Date					Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
10AC 5/16/08	#	Standard ID	Calibration Date	Compound	CF (/∂Dstd)	CF (ODstd)	Average CF (initial)	Average CF (initial)	%RSD	%RSD
74 60 4	-		80/91/5		76851	76851	16023	16023	3.486	3.456
3 8 4 4	П		,							
6 4	2									
3										
6 4	П									
4	3									
4										
4										
	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.

SDG #: See Con LDC #: 19/9/48

## Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Reviewer: 2nd Reviewer:

METHOD: GC\_

HPLC

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

% Difference = 100 \* (ave. CF - CF)/ave. CF CF = A/C

ave. CF = initial calibration average CF
CF = continuing calibration CF
A = Area of compound
C = Concentration of compound Where:

_					Reported	Recalculated	Reported	Recalculated
St	Standard ID	Calibration Date	Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc. CCV	<b>0%</b>	Q%
The M	\		DR0	0.46/	82/8/626	V`	0.8	0,8
LV	3425K	29/28/1/3/21/2	2RO	1001	PKCE:2001	1005.32	2.0	5.0
		80/6//0						
L.								

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

Page: Reviewer: 2nd reviewer:

METHOD: VGC \_\_ HPLC

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

Where: SF = Surrogate Found SS = Surrogate Spiked

% Recovery: SF/SS \* 100

Sample ID: 🍼						
Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
HAL	$\sim$	0.50	28.85	287	/ 80	O
					/	

흗	Į
0	
E	
-	

ample ID:						
Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	

### Sample ID:

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

SDG #: See COM LDC #: 1919.48

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

2nd Reviewer: Reviewer:

> ) | | METHOD:

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

MS/MSD samples:

SC = Sample concentration

SSC = Spiked sample concentration SA = Spike added MS = Matrix spike RPD =(((SSCMS - SSCMSD) \* 2) / (SSCMS + SSCMSD))\*100

MSD = Matrix spike duplicate

100 Recalc. MS/MSD RPD 1 Reported By Recalc. Matrix Spike Duplicate Percent Recovery DO Reported 8 Recalc. Percent Recovery Matrix spike a Reported Ø 7/10 MSD Concentration (MS/13) Spike Sample MS Sample S S Conc. 0 MSD 8 Spike Added 858 2,4,6-Trinitrotoluene (8330) (RSK-175) (8021B) (8015)(8015) (8151) (8310) (8330) (8310) (8151) Compound Naphthalene Anthracene Gasoline Benzene Methane Dinoseb Diesel 2,4-D XMH

LDC #:/9/9/43 SDG#50

# <u>Laboratory Control Sample/Laboratory Control Sample Duplicate Results Verification</u> VALIDATION FINDINGS WORKSHEET

2nd Reviewer:

LGC\_HPLC METHOD:

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:

SC = Concentration

RPD = I SSCLCS - SSCLCSD I \* 2/(SSCLCS + SSCLCSD)

SSC = Spiked sample concentration
SA = Spike added
LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples:\_\_

	ds	ike	Spiked	Sample	SOT	Ş	CSD	SD	I/SOI	TCS/TCSD
Compound	Ad (M)	Added/ (M/S/S	Conce	Concentration	Percent Recovery	Recovery	Percent Recovery	Recovery	Ŗ	RPD
	, rcs	rcsp	LCS	rcsp	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)										
Diesel (8015)	83,3	NA	9:86	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 #: MINES SDG #: V

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

2nd Reviewer: \_\_ Reviewer:

ပ္

GC HPI	Were all repo
METHOD:	Y N N/A

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

Example:	Sample ID.
Concentration= (A)(Fv)(Df)	(RF)(Vs or Ws)(%S/100)

Compound Name\_

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

Concentration =\_

Vs= Initial volume of the sample Ws= Initial weight of the sample %S= Percent Solid

Qualifications				
Recalculated Results Concentrations (				
Reported Concentrations (				
Compound				
Sample ID				
#				

	#	Sample ID	Compound	Reported Concentrations (	Recalcuated Results Concentrations (	Qualifications
ې -	Omments.	onte:				
,	ָ -	2		The second secon		

### 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-2MSD TSB-CJ-09-0MS

TSB-CJ-09-0MSD

<sup>\*\*</sup>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.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.

None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. Calibration

### a. Initial Calibration

Initial calibration of compounds was performed as required by the method.

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

Retention time windows were evaluated and considered technically acceptable for samples on which 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)	А

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

	19191A9	V	ALIDATIC				S WORKSHEET	•	Date 3/5/1
	: F8F130140			Le	evel III.	/IV			Page: // of /
_abora	tory: Test America								Reviewer: 2nd Reviewer:
METH	OD: GC Polynuclea	ar Aroma	atic Hydrocar	bons (EPA	SW 84	6 Me	thod 8310)		ZIId Neviewer.
	•		-					e ,	
	mples listed below on findings worksh		viewed for ea	ach of the fo	ollowing	valic	lation areas. Validation	on find	ings are noted in attache
- andati	on mange worker								
	Valida	tion Ar	ea				Comn	nents	
l.	Technical holding time	es	-	1	Samplin	g date	s: 6/12/05	3	
lla.	Initial calibration			1			/ /		
IIb.	Calibration verification	/ICV		I m/	10	/≤	15/0		
111.	Blanks			4					
IVa.	Surrogate recovery		· · · · · · · · · · · · · · · · · · ·	4					
IVb.	Matrix spike/Matrix spi	ike duplica	ates	<del> </del>					
IVc.	Laboratory control sar	nples		<u> </u>	10	<u> </u>			
V.	Target compound ider	ntification		<u> </u>	Not rev	iewed	for Level III validation.		
VI.	Compound Quantitation	on and CF	QLs	A	Not rev	iewed	for Level III validation.		
VII.	System Performance			<b>A</b>	Not rev	iewed	for Level III validation.		
VIII.	Overall assessment o	f data		<u> </u>					
IX.	Field duplicates			N					
X	Field blanks			ND	R =	1			
Note:	A = Acceptable N = Not provided/app SW = See worksheet		R = Ri	No compound nsate Field blank	ls detecte	d	D = Duplicate TB = Trip blank EB = Equipment blan	nk	
√alidate	d Samples: ** Indicates	s sample	underwent Leve	I IV validation	1				
1 F	RINSATE-2	W 11	8168.	487NL	<b>3</b> 2	1		31	
	rsb-cj-09-0	S 12	81703	13 MB	22	2		32	
3 7	ГSB-CJ-09-10**	13			23	3		33	
4 F	RINSATE-2MS	W 14			24	4		34	
5 F	RINSATE-2MSD	√ 15			2	5		35	
6 1	rsb-cj-09-0Ms	5/ 16			26	3		36	
7 7	rsb-cj-09-0Msd	<b>V</b> 17			27	7		37	
8		18			28	3		38	
9		19			29	<u> </u>		39	
10		20			3(	٠ I		140	

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-Dinitrotolune	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. Suiprofos	
O. Phenanthrene	О.		O. Chlorpyrifos		
P. Pyrene	P.		P. Fenthion		
Ö	O		Q. Parathion-ethyl		
œ			R. Trichloronate		
Š.			S. Merphos		
			T. Stirofos		
			U. Tokuthion		

cmpd\_list.wpd

LDC #: 1919149 SDG #: <u>See cow</u>

### **VALIDATION FINDINGS CHECKLIST**

Page: / of Z Reviewer: 2nd Reviewer: 4

Method: GC HPLC

Method: GC / HPLC		_		
Validation Area	Yes	No	NA	Findings/Comments
f Festinical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.				
(Espuial calibration				
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?		****	77.519	
IV. Continuing calibration	) T	T	T IV	T
What type of continuing calibration calculation was performed?%D or%R				
Was a continuing calibration analyzed daily?	1/		_	
Were all percent differences (%D) ≤ 15%.0 or percent recoveries 85-115%?	<del>                                     </del>	1	ļ	
Were all the retention times within the acceptance windows?	ESTAGE STATE	200 A Military J	Creation 1	
V/Blacks		5 1	1 8	
Was a method blank associated with every sample in this SDG?	1	1	<u> </u>	
Was a method blank analyzed for each matrix and concentration?	1/	1		
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.			1_	
V) Surgete spices				
Were all surrogate %R within the QC limits?		1		
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?	3		K	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?	No. of the last of	en processo din		
VII. Marrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.				
Was a MS/MSD analyzed every 20 samples of each matrix?	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	/	1_		
VIII1Laboratory control samples 30%				T
Was an LCS analyzed for this SDG?	16	4	+	
Was an LCS analyzed per extraction batch?	1	<u> </u>		1

LDC#:	1919	18	9	
SDG #:				N

### **VALIDATION FINDINGS CHECKLIST**

Validation Area	Yes	No	NA	Findings/Comments
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
X: Regional Phality Assurance and Otality Control	1			
Were performance evaluation (PE) samples performed?		/		
Were the performance evaluation (PE) samples within the acceptance limits?				
X Target compound identification				
Were the retention times of reported detects within the RT windows?  XI: compound quantifation/CRQLs				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII SVStemperformance				
System performance was found to be acceptable.				
XIII overallessessment of data in the second				
Overall assessment of data was found to be acceptable.				
XIV Field dublicates 19				
Were field duplicate pairs identified in this SDG?				
Were target compounds idetected in the field duplicates?				
XV. Field blanks				
Were field blanks identified in this SDG?			<u> </u>	
Were target compounds detected in the field blanks?		/		

LDC #:1919/A SDG # 700 C GC / HPLC

METHOD:

VALIDATION FINDINGS WORKSHEET **Continuing Calibration** 

Page: Reviewer:

2nd Reviewer:

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

Y M N/A

Were the retention times for all calibrated compounds within their respective acceptance windows?

		Qualifications	1244 B		,																		
	Accordated Committee	Associated Samples	00040 14415	A11+1845																			
	RT (limit)			(	( )	( )	)	7		( )	(	,		)	,	(	(	( )		7		7	•
	%D / RPD (Limit < 15.0)	771	()																				
	Compound	7																					
Detector/	Column	NS																					
	Standard ID	- 1	(/6/)																				
<u> </u>		6/4/0 8	,																		- 5:		

LDC #:19/9/14 9 SDG #: 20

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Page: Reviewer:\_

> HPLC METHOD: GC

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

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

A = Area of compound,

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

Recalculated	%RSD	/581	17.820 17.820	,						
Reported	%RSD	/=& ·1	17.820							
Recalculated	Average CF (initial)	806T10	12608							
Reported	Average CF (initial)	0 11908	67485 67485 6260 8 6260 8							
Recalculated	CF ( / std)	807269	5485							
Reported	CF ( ( std)	807269	67485							
	Compound	V	A							
	Calibration Date	/ / ~	2/4/08							
	Standard ID		JAN JAN							
	#	-			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 #: (21, 91, 4, 9, 80G #: 50e com)

## Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Lot	7	۵
Page:_	Reviewer:_	2nd Reviewer:

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

using the following calculation:

% Difference = 100 \* (ave. CF - CF)/ave. CF CF = A/C

Where:

ave. CF = initial calibration average CF
CF = continuing calibration CF
A = Area of compound
C = Concentration of compound

					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc. CCV	Q%	۵%
<b> </b>		2/01/	0		0	Ø.	4.2	Z'F
		0////	Q		0.4289	0.4289	- 7º1	74.
							,	
~								
6						·		
Γ								
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 #: 19149 SDG #: 100000

# VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Sample ID: 3		oo - ourogaie opined				
Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
HAL	NS	25	2151/2	87	87	Ø
					/	

Sample ID:						
Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	

Sample ID:						
Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	

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

2nd Reviewer: Reviewer:

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

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

SC = Sample concentration

RPD =(((SSCMS - SSCMSD) \* 2) / (SSCMS + SSCMSD))\*100

MSD = Matrix spike duplicate

MS/MSD samples:

	Spike	e (	Sample	Spike	Spike Sample	Matrix	Matrix spike	Matrix Spike Duplicate	Duplicate	MS/MSD	ISD
Compound	Added )	() SE	Cond ( //43)	Concentration ( )	Hation (	Percent	Percent Recovery	Percent Recovery	ecovery	RPD	a
	/ SW	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) ₹	682	589	an	1:55	55.0	18	18/	82	82	1.7	9.1
Anthracene (8310)	1	1	7	49.1	50.9	73	72	75	75	.d.	3
HMX (8330)											
2,4,6-Trinitrotoluene (8330)											

10.0% of the recalculated results.

LDC #191914 SDG# 7

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

2nd Reviewer:

GC / HPLC METHOD:

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

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

Where:

SC = Concentration

RPD = I SSCLCS - SSCLCSD I \* 2/(SSCLCS + SSCLCSD)

SSC = Spiked sample concentration SA = Spike added LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples: 87703/

	ίς	pike	Spiked	Sample	רכ	rcs	27	TCSD	/SOT	TCS/FCSD
Compound	A Ž	Added LAGS	Souce	Concentration	Percent Recovery	Recovery	Percent	Percent Recovery	æ	RPD
	SOT	TCSD	, SOT	CCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)										
Diesel (8015)										
Benzene (8021B)										
Methane (RSK-175)										
2,4-D (8151)										
Dinoseb (8151)										
Naphthatene (8310)	T.79	VN	266	NA	5.8	85				
Anthracene (8310)	1	1	53.9	1	/8	18				
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.

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# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

OD: GC / HPLC

GC  ☐ HPLC	Were all reported results	Were all recalculated res
METHOD:	A/N	A/N N/A
AET	(Z	Z
	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	ilculated and verified for all for detected target compounds.	Vere all reported results recalculated and verified for all level IV samples?  Vere all recalculated results for detected target compounds agree within 10% of the reported results?	rted results?
oncentration∍'	(RE)(Vs or Ws)(%S/100)	Example.		e
-		Sample ID	Compound Name	5

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

Qualifications				
Recalculated Results Concentrations (				
Reported Concentrations	-			
Compound				
Sample ID				
#				

Somments:

### 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

<sup>\*\*</sup>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.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25%.

The exact mass of 380.9760 of PFK was verified. The static resolving power was at least 10,000 (10% valley definition) for samples on which EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by EPA Level III criteria.

### III. Initial Calibration

A five point initial calibration was performed as required by the method.

Percent relative standard deviations (%RSD) were less than or equal to 20.0% for unlabeled compounds and less than or equal to 30.0% for labeled compounds.

The ion abundance ratios for all PCDDs and PCDFs were within validation criteria.

The minimum S/N ratio for each target compound was greater than or equal to 2.5 and and greater than or equal to 10 for each recovery and internal standard compound for samples on which EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by EPA Level III criteria.

### IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

All of the routine calibration percent differences (%D) between the initial calibration RRF and the routine calibration RRF were less than or equal to 20.0% for unlabeled compounds and less than or equal to 30.0% for labeled compounds 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)	Р

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)	Р
6/26/08	<sup>13</sup> C-OCDD	34.6	8175566MB	OCDD	J+ (all detects)	Р

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)	Р

## 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 1,2,3,4,6,7,8-HpCDF	J (all detects) UJ (all non-detects)	Р
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 1,2,3,4,6,7,8-HpCDF	J (all detects) UJ (all non-detects)	Р

## 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)	Р

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)	Р	Routine calibration (%D)
F8F130140	RINSATE-2	1,2,3,7,8,9-HxCDD	J+ (all detects)	Р	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)	Р	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 0CDF	J (all detects) UJ (all non-detects)	Р	Internal standards (%R)
F8F130140	TSB-CJ-09-0	2,3,7,8-TCDF OCDF	J (all detects) J (all detects)	Р	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

### **VALIDATION COMPLETENESS WORKSHEET** LDC #: 19191A21 Level III/IV SDG #: F8F130140 Reviewer: Laboratory: Test America 2nd Reviewer: METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290) The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets. Validation Area Comments Technical holding times Sampling dates: 11. GC/MS Instrument performance check III. Initial calibration Routine calibration/I IV. V. Blanks VI. Matrix spike/Matrix spike duplicates 1C5 VII. Laboratory control samples VIII. Regional quality assurance and quality control Ν IX. Internal standards Target compound identifications Not reviewed for Level III validation. X. Not reviewed for Level III validation. Compound quantitation and CRQLs XI. Not reviewed for Level III validation. XII. System performance XIII. Overall assessment of data XIV. Field duplicates Field blanks XV. A = Acceptable ND = No compounds detected D = Duplicate Note: TB = Trip blank N = Not provided/applicable R = Rinsate FB = Field blank EB = Equipment blank SW = See worksheet Validated Samples: \*\* Indicates sample underwent Level IV validation RINSATE-2 31 22 32 TSB-CJ-09-0 13 23 33 3 TSB-CJ-09-10\*\* 34 24 TSB-CJ-09-0MS 14 25 35 TSB-CJ-09-0MSD 15 5 26 36 6 16 27 37 17 28 38 8 18 29 39 19

30

40

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Notes:

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LDC #: 19191A21 SDG #: <u>Becover</u>

## **VALIDATION FINDINGS CHECKLIST**

Page: / of Page: / Of

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times		1		
All technical holding times were met.	7			
Cooler temperature criteria was met.				
II. GC/MS Instrument performance check				
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% ?				
ls the static resolving power at least 10,000 (10% valley definition)?	Ĺ			
Was the mass resolution adequately check with PFK?	1			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?				
III. Initial calibration	,	<b></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?	/			
IV. Continuing calibration	,	,		
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?				
V. Blanks				
Was a method blank associated with every sample in this SDG?				
Was a method blank performed for each matrix and concentration?		ļ	/	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?				
VI. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	_			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VII. Laboratory control samples				
Was an LCS analyzed for this SDG?	<u> </u>			

LDC #: 1919/A>1 SDG #: <u>See cover</u>

## **VALIDATION FINDINGS CHECKLIST**

Page: of 3
Reviewer: 9
2nd Reviewer: 4

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
VIII. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
IX; Internal standards		,		r
Were internal standard recoveries within the 40-135% criteria?		/		
Was the minimum S/N ratio of all internal standard peaks $\geq$ 10?				
X. Target compound identification				
For 2,3,7,8 substituted congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard?	/	r		
For 2,3,7,8 substituted congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration?	-			
For non-2,3,7,8 substituted congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution?				
Did compound spectra contain all characteristic ions listed in the table attached?	/			
Was the Ion Abundance Ratio for the two quantitation ions within criteria?				
Was the signal to noise ratio for each target compound and labeled standard $\geq$ 2.5?				
Does the maximum intensity of each specified characteristic ion coincide within $\pm$ 2 seconds (includes labeled standards)?				
For PCDF identification, was any signal (S/N $\geq$ 2.5, at $\pm$ seconds RT) detected in the corresponding PCDPE channel?			/	
Was an acceptable lock mass recorded and monitored?	/			
XI. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII. System performance				·
System performance was found to be acceptable.				
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.				
XIV. Field duplicates				I
Field duplicate pairs were identified in this SDG.				

LDC #: 19191A2/ SDG #: Zee conv

## **VALIDATION FINDINGS CHECKLIST**

Page: 3 of 3
Reviewer: 9
2nd Reviewer: 9

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	a. 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:

SDG #: 1919/4=/

## **VALIDATION FINDINGS WORKSHEET** Routine Calibration

2nd Reviewer: Reviewer:

> Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290) Y N N/A

Was a routine calibration was performed at the beginning and end of each 12 hour period?

Were all percent differences (%D) of RRFs  $\leq$  20% for unlabeled compounds and  $\leq$  30% for labeled? Did all routine calibration standards meet the Ion Abundance Ratio criteria?

N N/A

*	Date	Standard ID	Compound	Finding %D (Limit: <30.0%)	(%)	Finding Ion Abundance Ratio	Assoc	Associated Samples	Qualifications	ions
	20/6/2	021108B1DZX	13C-H	67.3			2.4-5	-5	1 toleto	1
	`			-			/			
		1 -32	2 13C-H	71.80					1	
	811999	26 WOS/D5- 2	13 C-0000	34.6			81750	56 MB	1+ Jo 74	1
	/ /						,			
			~							
1										
1										
- 1										
ΙÍ		PCDDs S	Selected ions (m/z)	lon Abundance Ratio		PCDFs		Selected ions (m/z)	Ion Abund	Ion Abundance Ratio
	Tetra-		M/M+2	0.65-0.89		Tetra-		M/M+2	0.65-	0.65-0.89
	Penta-		M+2/M+4	1.32-1.78	. L	Penta-		M+2/M+4	1.32-	1.32-1.78
	Неха-		M+2/M+4	1.05-1.43	-1-	Неха-		M+2/M+4	1.05-	1.05-1.43
	Hexa- <sup>13</sup> C-Hx(	Hexa-13C-HxCDF (IS) only	M/M+2	0.43-0.59		Hexa-13C-HxCDF (IS) only	>	M/M+2	0.43-0.59	0.59
	Hepta-13C-Hp	Hepta-13C-HpCDF (IS) only	M/M+2	0.37-0.51		Hepta-13C-HpCDF (IS) only	γlr	M/M+2	0.37-0.51	0.51
	Hepta-		M+2/M+4	0.88-1.20		Hepta-		M+2/M+4	0.88-1.20	1.20
	Octa-		M+2/M+4	0.76-1.02		Octa-		M+2/M+4	0.76-1.02	1.02

## Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Reviewer:

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/A | N/A | Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this

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?

Y IN NA

Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

(DC #: 14/4/47 SDG #: See

## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Page:

2nd Reviewer:\_ Reviewer:

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 (Y)N N/A

Was a LCS required? Was a LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed?

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

<u></u>			T	T	<del>_</del> _	7	7/	<del>-</del> T-	<del></del>	7	1	7		_	_	<del>- T-</del>		 		_	-	7/	<del></del>		<b></b> -		
	Qualifications	1 total																									
	Associated Samples	MHZOS	8172352NR																								
	RPD (Limits)	( )	( )	( )	( )	( )	( )	(		)	)	)	)						-	( )	( )	( )	( )			(	
CSD	%H (LIMITS)	)	(	( )	( )	( )	( )	)	( )	)	· ·	( )	· ·	(	-		)			^   -	( )	( )	· ·	<u> </u>	( )		
LCS %D (Timite)	(culling)	(32/14/18)	( )	`	( )	( )	( )	( )	( )	( )	( )	( )	( )	(	( )	( )	( )			^	( )	( )	( )	( )	<u> </u>	( )	
Compound	. 11																										
Lab ID/Reference	200000000000000000000000000000000000000	21/225045																									
Date																	i										
*																				1	$\parallel$	$\top$	$\top$				

LDC #: 19191431 SDG #: 200 COWN

## VALIDATION FINDINGS WORKSHEET Internal Standards

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

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". Are all internal standard recoveries were within the 40-135% criteria?  $\frac{Y(N)A}{Y(N)A}$  Was the S/N ratio all internal standard peaks  $\geq$  10?

*	Date	Lab ID/Reference	Internal Standard	•	% Recovery (Limit: 40-135%)	Qualifications	suc
		_	2	()	26 (40-125	4/ M//	(G-E, K-P
			1	W	_		
			#	33	)		
					)	•	
		8	-	255	)	1 -VAJA P 10-4	F. K-X)
			1	W W	)	/ / / (	
			V	14	)		
			1	1	)		
			I	(3)	<b>A</b> )	<b>\</b>	
					)		
		4(NS)	0	38	140-136	5) No and	Z
			1	38	)		
			<i>H</i> ,	a U	Ò	(	
	,	**	7	40	)		
			0	88			
			Ų	H	W )		
					<b>)</b>	(	
					)		
					)	(	
					)		
		Internal Standards	Check Standard Used		Recovery Standards		Check Standard Used
Ą.	<sup>13</sup> C-2,3,7,8-TCDF	JE		K.	¹³C-1,2,3,4-TCDD		
В.	13C-2,3,7,8-TCDD	Q		نـ	<sup>13</sup> C-1,2,3,7,8,9-HxCDD		
Ö	<sup>13</sup> C-1,2,3,7,8-PeCDF	CDF		Œ.			
<u>.</u>	<sup>13</sup> C-1,2,3,7,8-Pe	CDD		ź			
ші	13C-1,2,3,\$,7,8-HxCDF	HXCDF		o			
u.	<sup>13</sup> C-1,2,3,6,7,8-HxCDD	Чхсрр		a.			
Ö	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	3-HpCDF		Ö			
ΤÌ	13C-1,2,3,4,6,7,8-HpCDD	з-НрСDD		œ			
	13C-OCDD			<b>I</b> -			

LDC #: 1919, AN SDG #: Secover

# VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

Page: of A Reviewer: 2nd Reviewer:

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? Compound quantitation and CRQLs were adjusted to reflect all sample dilutions and dry weight factors (if necessary).

Qualifications	Jets/F								
Associated Samples	Z								
Finding	H. & > call sange								
Sample ID	γ								
Date									
#									

Comments: See sample calculation verification worksheet for recalculations

LDC #: 1919/ A2 SDG #: Sec COV

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Page: Reviewer:

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 =  $(A_{\lambda})(C_{\alpha})/(A_{\alpha})(C_{\lambda})$ average RRF = sum of the RRFs/number of standards %RSD = 100 \* (S/X)

 $\begin{array}{l} A_x = Area \ of \ compound, \\ C_x = Concentration \ of \ compound, \\ S = Standard \ deviation \ of \ the \ RRFs, \end{array}$ 

 $A_{\rm s}=A{\rm rea}$  of associated internal standard  $C_{\rm s}=Concentration$  of internal standard X=Mean of the RRFs

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF ( @\$\$ std)	RRF ( CS \$\inf\$std)	%RSD	%RSD
-	1str	0/-//	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.798	0.798	0.82	0.87	25.61	1270
		00/11/0	6/1//00 2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	5.83	0.9/3	0.93	003	102	10 3
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1580	188.0	187	0.87	681	17/
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	7×128.0	D. 8444	0.88	XXX	800	10/
			OCDF (%c-OCDD)	156.1	1.721	1.86	18	16.4	76/
7			2,3,7,8-TCDF (1°C-2,3,7,8-TCDF)						
			2,3,7,8-TCDD (¹³C-2,3,7,8-TCDD)						
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)						
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)						
			OCDF (3c-OCDD)						
က			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)						
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)						
			1,2,3,6,7,8-HxCDD (13C-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 (19C-OCDD)						

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

LDC #: 9/9/ 42/

## VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

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

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

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF =  $(A_u)(C_u)/(A_u)(C_v)$ 

Where: ave. RRF = initial calibration average RRF RRF = continuing calibration RRF

 $A_x = Area of compound,$  $C_x = Concentration of compound,$ 

 $A_{is}$  = Area of associated internal standard  $C_{is}$  = Concentration of internal standard

					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	<b>0%</b>	d%
	2 W68/05	89897 541891	2,3,7,8-TCDF (1°C-2,3,7,8-TCDF)	0.798	0.83	0.83	70	4
	•	/	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	5/60	180	0.88	1.7.	K 1 1
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	0.82/	187	78.0	15.0	4.0
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	0.844	0.83	1830	1.5	1.51
			OCDF (3C-OCDD)	1-7-1	1.58	85:	W.	( ) &
7			2,3,7,8-TCDF (13C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)					
			OCDF (13C-OCDD)					
က			2,3,7,8-TCDF (°C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)					
			OCDF ("c-OCDD)					

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

LDC #:/9/9/

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

2nd Reviewer:\_\_ Reviewer:

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

SSR = Spiked sample result, SR = Sample result SA = Spike added Where:

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

N MS/MSD samples:

RPD = I MSR - MSDR I \* 2/(MSR + MSDR)

	dS	ike	Sample	Spiked Sample	Sample	Matrix Spike	Spike	Matrix Spike Duplicate	- Duplicate	Reported	Recalculated
Compound	PAG.	Added ( )	Concentration (P5K)	Concentration (PS/9)	tration (9)	Percent Recovery	Recovery	Percent Recovery	ecovery	RPD	RPD
	MS	MSD	, , , , , , , , , , , , , , , , , , ,	MS	MSD	Reported	Recalc	Reported	Recalc	32 8 8	
2,3,7,8-TCDD	20.9	20.9	78	183	95.9		502	58	36	62	62
1,2,3,7,8-PeCDD	701	701	330	824	457	472	214	120	/22	25	57
1,2,3,4,7,8-HxCDD			250	269	426	157	430	172	691	87	48
1,2,3,4,7,8,9-HpCDF		1	000	19100	19100 10200 10100 10192	00/01	10192	2591	1635	09	19
OCDF	209	808	00067	98.200	1758C 0018C 00925 00086	23/00	1/2560	819	766	79	79
	\	,	/		:	,					

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 #: 19.91421 SDG #: 20.00/e1

## Laboratory Control Sample Results Verification VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Reviewer: Page:

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

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratoy 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 = ILCS - LCSD I\* 2/(LCS + LCSD)

LCS ID: 8/75546

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

	ďS	ike	Spiked S	ample	ני	CS	LCSD	n	I CS/I CSD	CSD
Compound	A ()	Added (+75/9)	Concentration (FS/Q)	tration	Percent Recovery	tecovery	Percent Recovery	ecovery	RPD	۵
	SOI	I CSD	SUI	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	0.02	λX	2,2	¥	501	901				
1,2,3,7,8-PeCDD	100		105		105	105				
1,2,3,4,7,8-HxCDD			849		385	85				
1,2,3,4,7,8,9-HpCDF	<i>/</i>		901		901	901			-	
OCDF	200		220	->	011	011				

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.

# lons Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Analyte	HPCDF HPCDF HPCDF (S) HPCDD HPCDD HPCDD (S) NCDPE PFK	OCDF OCDF OCDD OCDD (S) OCDD (S) DCDPE PFK	
Elemental Composition	C12H <sup>26</sup> C1,37C1O C12H <sup>26</sup> C1,37C1O 13C12H <sup>26</sup> C1,0 13C12H <sup>26</sup> C1,0 13C12H <sup>26</sup> C1,0 13C12H <sup>26</sup> C1,37C1 <sub>2</sub> O <sub>2</sub> 13C12H <sup>26</sup> C1,37C1 <sub>2</sub> O <sub>2</sub> 13C12H <sup>26</sup> C1,37C1 <sub>2</sub> O <sub>2</sub> C12H <sup>26</sup> C1,37C1 <sub>2</sub> O <sub>2</sub> C5F7,	C <sub>12</sub> **Cl <sub>3</sub> **ClO C <sub>12</sub> **Cl <sub>3</sub> **ClO <sub>2</sub> C <sub>12</sub> **Cl <sub>3</sub> **ClO <sub>2</sub> C <sub>12</sub> **Cl <sub>3</sub> **ClO <sub>2</sub> 1*C <sub>12</sub> **Cl <sub>3</sub> **Cl <sub>2</sub> O <sub>2</sub> C <sub>12</sub> **Cl <sub>3</sub> **Cl <sub>2</sub> O <sub>2</sub> C <sub>12</sub> **Cl <sub>3</sub> **Cl <sub>2</sub> O <sub>2</sub>	
Ion ID	M M M M M M M M M M M M M M M M M M M	M M M M M M M M M M M M M M M M M M M	
Accurate Mass <sup>(a)</sup>	407.7818 409.7788 417.8250 419.8220 423.7767 425.7737 435.8169 437.8140 479.7165 [430.9728]	441.7428 443.7399 457.7377 459.7348 469.7780 471.7750 513.6775	
Descriptor	4	rv	
Analyte	TCDF TCDF (8) TCDP (8) TCDD TCDD (8) TCDD (8) TCDD (8)	Pecde Pecde (S) Pecde (S) Pecde Pecde Pecde (S) Pecde (S) Pecde (S) Pecde (S)	HKCDF HKCDF (S) HKCDD (S) HKCDD HKCDD (S) HKCDD (S) HKCDD (S)
Elemental Composition	C <sub>12</sub> H <sub>3</sub> *Cl <sub>1</sub> O C <sub>12</sub> H <sub>3</sub> *Cl <sub>1</sub> O '1 <sub>C<sub>12</sub>H<sub>3</sub>*Cl<sub>2</sub>O '1<sub>C<sub>12</sub>H<sub>3</sub>*Cl<sub>2</sub>O C<sub>12</sub>H<sub>3</sub>*Cl<sub>2</sub>O<sub>2</sub> C<sub>12</sub>H<sub>3</sub>*Cl<sub>2</sub>O<sub>2</sub> '1<sub>C<sub>12</sub>H<sub>3</sub>*Cl<sub>2</sub>O<sub>2</sub> '1<sub>C<sub>12</sub>H<sub>3</sub>*Cl<sub>3</sub>*Cl<sub>2</sub>O<sub>2</sub> '1<sub>C<sub>12</sub>H<sub>3</sub>*Cl<sub>3</sub>*Cl<sub>2</sub>O<sub>2</sub> C<sub>12</sub>H<sub>3</sub>*Cl<sub>3</sub>*Cl<sub>2</sub>O<sub>2</sub> C<sub>12</sub>H<sub>3</sub>*Cl<sub>3</sub>*Cl<sub>2</sub>O<sub>2</sub> C<sub>12</sub>H<sub>3</sub>*Cl<sub>3</sub>*Cl<sub>2</sub>O<sub>2</sub></sub></sub></sub></sub></sub>	C <sub>12</sub> H <sub>3</sub> 2C <sub>1</sub> 37ClO C <sub>12</sub> H <sub>3</sub> 2C <sub>1</sub> 37ClO C <sub>12</sub> H <sub>3</sub> 2C <sub>1</sub> 37C <sub>1</sub> O C <sub>12</sub> H <sub>3</sub> 2C <sub>1</sub> 37C <sub>1</sub> O C <sub>12</sub> H <sub>3</sub> 2C <sub>1</sub> 37Cl <sub>2</sub> O C <sub>12</sub> H <sub>3</sub> 2C <sub>1</sub> 37ClO <sub>2</sub> C <sub>12</sub> H <sub>3</sub> 2C <sub>1</sub> 37ClO <sub>2</sub> C <sub>12</sub> H <sub>3</sub> 2C <sub>1</sub> 37C <sub>1</sub> O <sub>2</sub> C <sub>12</sub> H <sub>3</sub> 2C <sub>1</sub> 37C <sub>1</sub> O <sub>2</sub> C <sub>12</sub> H <sub>3</sub> 2C <sub>1</sub> 37C <sub>1</sub> O <sub>2</sub> C <sub>12</sub> H <sub>3</sub> 2C <sub>1</sub> 37ClO	C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> 7ClO C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> 7ClO C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> 7Cl <sub>2</sub> O C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> 7ClO C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> 7ClO <sub>2</sub> C <sub>12</sub> H <sub>2</sub> *Cl <sub>4</sub> 7Cl <sub>2</sub> O <sub>2</sub> C <sub>12</sub> H <sub>2</sub> *Cl <sub>4</sub> 7Cl <sub>2</sub> O <sub>2</sub> C <sub>12</sub> H <sub>2</sub> *Cl <sub>4</sub> 7Cl <sub>2</sub> O <sub>2</sub> C <sub>12</sub> H <sub>2</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> O <sub>2</sub> C <sub>12</sub> H <sub>2</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> O <sub>2</sub> C <sub>12</sub> H <sub>2</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> O <sub>2</sub>
Ol nol	M M M M M M M M M M M M M M M M M M M	M + 4 A M + 4	M + 4 M + 4 M + 2 M + 4 M + 4 M + 4 M + 4
Accurate mass <sup>(s)</sup>	303.9016 305.8987 315.9419 317.9389 319.8965 321.8936 331.9368 333.9338 375.8364 [354.9792]	339.8597 341.8567 351.9000 353.8970 355.8546 357.8516 367.8949 369.8919 409.7974 [354.9792]	373.8208 375.8178 383.8639 385.8610 389.8156 391.8127 401.8559 403.8529 445.7555 [430.9728]
Descriptor	-	a	ю

(a) The following nuclidic masses were used:

H = 1.007825 C = 12.000000 <sup>13</sup>C = 13.003355 F = 18.9984

O = 15.994915 $^{36}CI = 34.968853$  $^{37}CI = 36.965903$ 

S = internal/recovery standard

LDC #: 1919/A>1 SDG #: See COVEN

## **VALIDATION FINDINGS WORKSHEET**

Sample Calculation Verification

Page:_	
Reviewer:	9-
2nd reviewer:	Q.
_	

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

R	N	N/A	
/ 🔽	Ν	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?

Concent	ration	$= \frac{(A_{\circ})(I_{\circ})(DF)}{(A_{\circ})(RRF)(V_{\circ})(\%S)}$
$A_{x}$	=	Area of the characteristic ion (EICP) for the compound to be measured
$A_{is}$	=	Area of the characteristic ion (EICP) for the specific internal standard
l <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).
RRF	=	Relative Response Factor (average) from the initial calibration
Df	=	Dilution Factor.
0/ 0		Percent colide applicable to soil and solid matrices

Example:			
Sample I.D.	3	, <u> </u>	

Conc. = (3650/6) (4000	<del>)</del> )()
Conc. = (3650/6) (4000 (134258b) 1.72	(1004)(0913
ŕ	
= 6.90 pg/g	
( / )	

#	Sample ID	Compound .	Reported Concentration ( )	Calculated Concentration ( )	Qualification
<u> </u>					
<b></b>					
ļ					