

### 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 G, Data Validation

Dear Ms. Barajas-Albalawi

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

### LDC Project # 19188:

- SDG # Fraction
- F8F120180 Volatiles, Semivolatiles, Chlorinated Pesticides, Polychlorinated Biphenyls, Metals, Wet Chemistry, Gasoline Range Organics, Diesel Range Organics, Polynuclear Aromatic Hydrocarbons, Dioxins/Dibenzofurans

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

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

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

Sincerely,

Erlinda T. Rauto Operations Manager/Senior Chemist

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

Project/Site Name: BRC Tronox Parcel G

Collection Date: June 11, 2008

LDC Report Date: August 7, 2008

Matrix: Soil/Water

Parameters: Volatiles

Validation Level: EPA Level III & IV

Laboratory:

TestAmerica, Inc.

### Sample Delivery Group (SDG): F8F120180

TSB-GJ-09-10' TSB-GJ-09-20'\*\* TSB-GJ-09-30' TSB-GJ-09-40' TB-2 6/11/08

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 4 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8260B for Volatiles.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- 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 F8F120180	J (all detects) UJ (all non-detects)	A

### IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

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

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

Date	Compound	%D	Associated Samples	Flag	A or P
6/19/08 (LCAL0317)	lodomethane	67.71684	All water samples in SDG F8F120180	J+ (all detects)	A

The percent differences (%D) of the second source calibration standard were less than or equal to 25.0% for all compounds with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
5/28/08 (LICV9881)	lodomethane	31.67513	All water samples in SDG F8F120180	J+ (all detects)	Α
5/28/08 (LICV9881)	2-Hexanone	25.04476	All water samples in SDG F8F120180	J- (all detects) UJ (all non-detects)	A

All of the continuing calibration RRF values were within method and validation criteria with the following exceptions:

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
6/16/08 (FCAL1777)	Ethanol	0.00209 (≥0.05)	All soil samples in SDG F8F120180	J (all detects) UJ (all non-detects)	A

### V. Blanks

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

Sample TB-2 6/11/08 was identified as a trip blank. No volatile contaminants were found in this blank with the following exceptions:

Trip Blank ID	Sampling Date	Compound	Concentration	Associated Samples
TB-2 6/11/08	6/11/08	Dichloromethane	0.47 ug/L	All soil samples in SDG F8F120180

Sample concentrations were compared to concentrations detected in the field blanks. The sample concentrations were either not detected or were significantly greater (>10X for common contaminants, >5X for other contaminants) than the concentrations found in the associated field blanks.

### VI. Surrogate Spikes

Surrogates were added to all samples and blanks as required by the method. All surrogate recoveries (%R) were within QC limits with the following exceptions:

Sample	Surrogate	%R (Limits)	Compound	Flag	A or P
8172125MB	Bromofluorobenzene	117 (79-115)	All TCL compounds	J+ (all detects)	Р

### VII. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Percent recoveries (%R) and relative percent differences (RPD) were not within QC limits. Since there were no associated samples, no data were qualified.

### VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Although the percent recoveries (%R) and relative percent difference (RPD) for some compounds in the LCS/LCSD were not within QC limits, the MS/MSD percent recoveries (%R) were within QC limits and no data were qualified.

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

All internal standard areas and retention times were within QC limits.

### XI. Target Compound Identifications

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

### XII. Compound Quantitation and CRQLs

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

### XIII. Tentatively Identified Compounds (TICs)

Tentatively identified compounds were not reported by the laboratory.

### XIV. System Performance

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The system performance was acceptable for samples on which an EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

### XV. Overall Assessment of Data

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

### **XVI. Field Duplicates**

No field duplicates were identified in this SDG.

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

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

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

No Sample Data Qualified in this SDG

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

No Sample Data Qualified in this SDG

### VALIDATION COMPLETENESS WORKSHEET

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

### Level III/IV

	11
Date:	<u> 8 5/08</u> -
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Reviewer:	· 4
Reviewer:	

2nd

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
١.	Technical holding times	A	Sampling dates: 6/11/08
11.	GC/MS Instrument performance check	Å	· · ·
111.	Initial calibration	W	75D.Y2
IV.	Continuing calibration/ICV	m/	10/0 2570
V.	Blanks	A	
VI.	Surrogate spikes	Ŵ	
VII.	Matrix spike/Matrix spike duplicates	m	TSB-GJ-08-10'- No Splass'd Notenal
VIII.	Laboratory control samples	ŚW	LCS D
IX.	Regional Quality Assurance and Quality Control	N	
Х.	Internal standards	$\mathbf{A}$	
XI.	Target compound identification	A	Not reviewed for Level III validation.
XII.	Compound quantitation/CRQLs	$\mathbf{A}$	Not reviewed for Level III validation.
XIII.	Tentatively identified compounds (TICs)	N	Not reviewed for Level III validation.
XIV.	System performance	A	Not reviewed for Level III validation.
XV.	Overall assessment of data	Å	/
XVI.	Field duplicates	N	l
XVII.	Field blanks	$\sim$	TB=5

Note:

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

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

D = Duplicate TB = Trip blank EB = Equipment blank

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

1	TSB-GJ-09-10'	٦ ا	1 4	8170291MB	21	(5)	31	
2	TSB-GJ-09-20'**	1	2 4	8172125MB	22	N	32	
3	TSB-GJ-09-30'	1	3	8172361MB	23	(N)	33	
4	TSB-GJ-09-40'	1	4	/	24		34	
5	TB-2 6/11/08	1	5		25		35	
6		1	6		26		36	
7		1	7		27		37	
8		1	8		28		38	
9		1	9		29		39	
10		2	0		30		40	

LDC #: <u>/91884</u>/ SDG #: <u>See CO W</u>

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				Ref. 1
All technical holding times were met.				
Cooler temperature criteria was met.			CHEMISTRA	
II/GC/MS Instrument performance check				
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		2010 1		
Did the laboratory perform a 5 point calibration prior to sample analysis?	$\square$			
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?	$\square$		-	
Did the initial calibration meet the curve fit acceptance criteria of $\geq 0.990?$				
Were all percent relative standard deviations (%RSD) $\leq$ 30% and relative response factors (RRF) $\geq$ 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?	/	~		
Were all percent differences (%D) $\leq$ 25% and relative response factors (RRF) $\geq$ 0.05?				
V Blanks				
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.				
M Surrogate spikes				
Were all surrogate %R within QC limits?	Ľ			
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?				
VIII 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.	/	7		
Was a MS/MSD analyzed every 20 samples of each matrix?	$\square$	-		
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?	1			

LDC #: <u>19188</u> A / SDG #: <u>See CO W 14</u>

### VALIDATION FINDINGS CHECKLIST

Page:	<u>2of</u>
Reviewer:	<u></u>
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?	е	$\langle$		
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			tu L	
Were internal standard area counts within -50% or +100% of the associated calibration standard?	$\langle \rangle$			
Were retention times within <u>+</u> 30 seconds of the associated calibration standard?				
XI Target compound identification				
Were relative retention times (RRT's) within <u>+ 0.06 RRT units of the standard?</u>	1			
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	<b>/</b>			
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 lever to validation:				
XIII - Tentatively identified compounds (TICs)				
XIII Tentatively identified compounds (TICs) Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?				
XIII. Tentatively identified compounds (TICs) Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum? Were relative intensities of the major ions within <u>+</u> 20% between the sample and the reference spectra?				
XIII: Tentatively identified compounds (TICs)   Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?   Were relative intensities of the major ions within ± 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)?			/	
All y weight factors applicable to rever v validation?   X[I]. Tentatively identified compounds (TICs)   Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?   Were relative intensities of the major ions within ± 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.				
All y weight factors applicable to rever v validation?   XIII. Tentatively identified compounds (TICs)   Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?   Were relative intensities of the major ions within ± 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. Systemiperformance   System performance				
All y weight factors applicable to rever v validation?   XIII. Tentatively identified compounds (TICs)   Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?   Were relative intensities of the major ions within ± 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   System performance was found to be acceptable.				
All y weight factors applicable to rever v validation?   XIII. Tentatively identified compounds (TICs)   Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?   Were relative intensities of the major ions within ± 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   System performance was found to be acceptable.   XV. Overall assessment of data.				
All y weight factors applicable to rever v validation?   XIII. Tentatively identified compounds (TICs)   Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?   Were relative intensities of the major ions within ± 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   System performance was found to be acceptable.   XV. Overall assessment of data was found to be acceptable.				
ally weight factors applicable to rever it validation?   XIII. Tentatively identified compounds (TICs)   Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?   Were relative intensities of the major ions within ± 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   System performance was found to be acceptable.   XV. Overall assessment of data   Overall assessment of data was found to be acceptable.   XM. Field duplicates				
dify weight factors applicable to rever in validation?   XIII. Tentatively identified compounds (TICs)   Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?   Were relative intensities of the major ions within ± 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. Systemiperformance   System performance was found to be acceptable.   XV. Overall assessment of data was found to be acceptable.   XVI. Field duplicates   Field duplicate pairs were identified in this SDG.				
Ally weight factors applicable to level it validation?   XIII. Tentatively identified compounds (TICs)   Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?   Were relative intensities of the major ions within ± 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]   System performance was found to be acceptable.   XV. Overall assessment of data   Overall assessment of data was found to be acceptable.   XVI. Field duplicates   Field duplicate pairs were identified in this SDG.   Target compounds were detected in the field duplicates.				
Ally weight factors applicable to level it valuation?   XIII. Tentatively identified compounds (TICs)   Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?   Were relative intensities of the major ions within ± 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)?   XW_System performance   System performance was found to be acceptable.   XV_Overall assessment of data   Overall assessment of data was found to be acceptable.   XVI Fleid duplicates   Field duplicate pairs were identified in this SDG.   Target compounds were detected in the field duplicates.   XVII Eleid blanks;				
All y weight factors applicable to reven to validation:   XIIIrentatively.identified compounds (TICs)   Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?   Were relative intensities of the major ions within ± 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   System performance was found to be acceptable.   XV_Overall assessment of data was found to be acceptable.   XV_System performance between the identified in this SDG.   Target compounds were detected in the field duplicates.   XVI_Eleid blanks   Field blanks were identified in this SDG.				

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-Dichlaropropene	KKK. 1,2,4-Trichlorobenzene	EEEE. Acetonitrile
D. Chloroethane	X. Bromoform*	RR. Dibromomethane	LLL. Hexachlorobutadiene	FFFF. Acrolein
E. Methylene chloride	Y. 4-Methyl-2-pentanone	SS. 1,3-Dichloropropane	MMM. Naphthalene	GGGG. Acrylonitrile
F. Acetone	Z. 2-Hexanone	TT. 1,2-Dibromoethane	NNN. 1,2,3-Trichlorobenzene	HHHH. 1,4-Dioxane
G. Carbon disulfide	AA. Tetrachloroethene	UU. 1,1,1,2-Tetrachloroethane	000. 1,3,5-Trichlorobenzene	IIII. Isobutyl alcohol
H. 1,1-Dichloroethene**	BB. 1,1,2,2-Tetrachloroethane*	VV. Isopropylbenzene	PPP. trans-1,2-Dichloroethene	JJJJ. Methacrylonitrile
I. 1,1-Dichloroethane*	CC. Toluene**	WW. Bromobenzene	QQQ. cis-1,2-Dichloroethene	KKKK. Propionitrile
J. 1, 2-Dichloroethene, total	DD. Chlorobenzene*	XX. 1,2,3-Trichloropropane	RRR. m,p-Xylenes	LLLL. Ethyl ether
K. Chloroform**	EE. Ethylbenzene**	YY. n-Propylbenzene	SSS. o-Xylene	MMMM. Benzyl chloride
L. 1,2-Dichloroethane	FF. Styrene	ZZ. 2-Chlorotoluene	TTT. 1,1,2-Trichloro-1,2,2-trifluoroethane	NNNN. Z, Z - D'MAHU Prutoul
M. 2-Butanone	GG. Xylenes, total	AAA: 1,3,5-Trimethylbenzene	UUU. 1,2-Dichlorotetrafluoroethane	0000. Dimetly disultide
N. 1, 1, 1-Trichloroethane	HH. Vinyl acetate	BBB. 4-Chlorotoluene	VVV. 4-Ethyltoluene	dddd
O. Carbon tetrachloride	II. 2-Chloroethylvinyl ether	CCC. tert-Butylbenzene	WWW. Ethanol	<u>aaaa.</u>
P. Bromodichloromethane	JJ. Dichlorodifluoromethane	DDD. 1,2,4-Trimethylbenzene	XXX. Di-isopropyl ether	RRR.
Q. 1,2-Dichloropropane**	KK. Trichlorofluoromethane	EEE. sec-Butytbenzene	YYY. tert-Butanol	SSSS.
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	טטטט.
T. Dibromochloromethane	NN. Methyl ethyl ketone	HHH. 1,4-Dichlorobenzene	BBBB. tert-Amyl methyl ether	vvvv.

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

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## VALIDATION FINDINGS WORKSHEET **Initial Calibration**

đ Page: 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".

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

Did the initial calibration meet the acceptance criteria?

	Qualifications	A MV														
\$	Associated Samples	W/50% 5.	SIMIPCOTIS													
RSD and ≥0.05 RRF	Finding RRF (Limit: ≥0.05)	10000														
ion criteria of ≤30 %	Finding %RSD (Limit: ≤30.0%)															
RFs within the validat	Compound	MNN														
Vere all %RSDs and R	Standard ID	1012								-						
<u>V N/A</u> V	Date	6/9/08	/ /													
긝	#															:

LDC #:<u>/9/884/</u> SDG #:*222 COW* 

## VALIDATION FINDINGS WORKSHEET **Continuing Calibration**

ਰਿੱ Page:\_\_ 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". M N/A Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?

s ?	Qualifications	Vt/et=/A	X/MY/X			1/W/B		1+ 1+ 1+ 1A										
ia for all CCC's and SPCC'	Associated Samples	5.81721×5MB				M Sils .	8176291NB	5.817×7514B	/									
) within method criter d ≥0.05 RRF ?	Finding RRF (Limit: ≥0.05)					1 00207		•										
sponse factors (RRF iteria of ≤25 %D and	Finding %D (Limit: <u>≤</u> 25.0%)	3/.67573	22+70-5C					67.71684										
(%D) and relative re vithin the validation cr	Compound	10 domethand	N			N N N		10 domethour										
re percent differences re all %D and RRFs w	Standard ID	11219881	(1ey)		4	FCALITT	/	20440317										
<u>Y (N) N/A</u> We	# Date	543/08				6/16/0 8	\ \ \	 6/19/08	/ /									

LDC #: /9/28//		VAL	IDATION F	INDINGS	s WORKS ks	ЭНЕЕТ			Pa Reviev	ge: /of/ ver:
METHOD: GC/MS VOA (EPA	SW 846 Metho	d 8260)							zna heviev	ver.
<u>Y N N/A</u> Were field bla <u>Y N N/A</u> Were target c Blank units: Ass	anks identified i compounds det ociated sampl	n this SDG? ected in the field e units:	blanks?							
Sampling date: <u>\$////0</u> Field blank type: (circle one)	Field Blank $H$	Rinsate / Trip Blar	hk) Other:		Associat	ted Sample	35: AL	( soils		
Compound	Blank ID				Samp	ole Identificat	tion			
	5									
Methylene chloride										
Acetone				: 						
Chloroform										
Dichloromethaus	0.47									
	-									
CROL										
Blank units:Ass Sampling date:	sociated samp	e units:	- - - -							
Field blank type: (circle one)	) Field Blank /	Rinsate / Trip Bla	nk / Other:		Associa	ted Sample	ss:			
Compound	Blank ID				Sami	ple Identifica	tion			
Methylene chloride										
Acetone										
Chloraform										
CRQL										
CIRCLED RESULTS WERE NOT QL Common contaminants such as Mer	JALIFIED. ALL RE thviene chloride. A	SULTS NOT CIRCLEE	D WERE QUALIF nd Carbon disult	IED BY THE F	OLLOWING S	STATEMENT: moles within	ten times the	essociated field	blank concentrati	on were qualified a

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

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ГРС	SDG

## VALIDATION FINDINGS WORKSHEET Surrogate Spikes

Page: Reviewer: 2nd Reviewer:

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

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

Y/N N/A 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 out of outside of criteria? IN/N/A

Qualifications	1+ late to																		
ry (Limits)	(57 + 6 - 2)		-	(	(	( )	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(	
%Recover	1.7																		C Limits (Water)
Surrogate	2-7-5																		ĕ
Sample ID	SUZSIS																		<u>OC Limits (Soil)</u>
# Date																			

SUR.1SB

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

81-117



## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

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

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) N/A Was a LCS require

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

F=====		`		<u> </u>																				
Qualifications	No leva L	$\sim$	M QSN/SNI																					
Associated Samples	5. BITHYNNB																							
RPD (Limits)	( 02×) C/	(	( )	( )	( )	(	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(	( )	( )	( )
LCSD %R (Limits)	( )	181 (45+40)	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
LCS %R (Limits)	293 (42-140)	ne 16645-140)	( )	(	( )	( )	( )	( )	( )	( )	()	( )	( )	( )	()	( )	(	( )	( )	()	( )	( )		(
Compound	NN	lodometha																						
rcs/rcsd ID	8172/25/25/	\$ ,	/											-										
Date																								
#																								

LCSLCSD.1SB



VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

ď J Reviewer:\_\_ 2nd Reviewer: Page:\_\_\_

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:

 $\label{eq:RF} RF = (A_x)(C_x)(A_x)(C_x) \\ average \ RFF = sum \ of the \ RFFs/number \ of standards \\ \ \%RSD = 100 \ \bullet (S/X) \\ \end{cases}$ 

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

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

					Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound	Reference Internal Standard)	RRF ( 🌮 std)	RRF (Sold)	Average RRF (initial)	Average RRF (initial)	%RSD	%RSD
-	1241	6/9/08	¥	(1st internal standard)	12605.0	D.SOGTI	0.5831	0.52831	7.04091	7.0405
	É H	/ , / _	44	(2nd internal standard)	0 2592	1.35512	Q.29404	1=40+	074.01	127450
			AAA	(3rd internal standard)	3.57047	3.57047	3.42599	3.42599	8255-8	8.250
7	Yell	1/2/18	NNNN	(1st internal standard)	6=1720	0.74120	0.73871	0.73871	5,5079	7355
		m/L/a	0000	(2nd internal standard)	0.59203	0.59203	992550	0.55366	13.47144	134718
	( <del>]</del> )		000	(3rd internal standard)	1.11568	89511-1	1.11.50	R25111.1	2.41699	2,4169
ო				(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 gualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

SDG #: Second LDC # 19/384

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

້ວ້ Reviewer: 2nd Reviewer:\_ Page:

# 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<sub>x</sub>)(C<sub>s</sub>)/(A<sub>s</sub>)(C<sub>x</sub>)

Where: ave. RRF = initial calibration average RRF RRF = continuing calibration RRF A<sub>x</sub> = Area of compound, C<sub>x</sub> = Concentration of compound,

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

20 Recalculated *d*o 899 3.5-985 2244 10 °° Ò 3 324/2 59782 N 89949 2974 1010 Reported 4 **0**% ð M 55203 5)358 57288 **Recalculated** 31216 20/02 040 RRF (CC) ï η 0 0 0.57288 858/2.0 0470 0.72154 2 Reported 3 6520 RRF (CC) ģ 1.31 3.42599 5 Average RRF 029404 0.7387, 65 30 0.583 (initial) Compound (Reference internal Standard) (2nd internal standard) (1st internal standard) (3rd internal standard) (2nd internal standard) (3rd internal standard) (2nd internal standard) (2nd internal standard) (1st internal standard) (3rd internal standard) (1st internal standard) (3rd internal standard) (1st internal standard) NNNN 0000 440  $\checkmark$ 6/08 Calibration 116/08 Date FCA21778 Standard ID 124 \* 2 ო 4

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



### VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page:_	
Reviewer:	9-
2nd reviewer:	

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

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

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8	50	45.0289	90,	90	0
Bromofluorobenzene	,	42.1691	84	84	1
1,2-Dichloroethane-d4		45.1855	90	90	
Dibromofluoromethane		4.4.0752	88	38	

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

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

### Sample ID:

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

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

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

### Sample ID:\_

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

:#:19133341	3#:500 COW
#	#
ГРС	SDG

## VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Results Verification

ð Page: Reviewer: 2nd 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 Where: SSC = Spike

Where: SSC = Spiked sample concentration . SA = Spike added

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

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

LCS ID: 8170291

	ŝ	pike	Spiked :	Sample		Ş	10	SD C	1 CS/I	csn
Compound	Ac Ac	idea 1915)	Concer	tration (U)	Percent F	Recovery	Percent F	tecovery	RF	Q
	I CS	1 CSD		1 CSD	Renorted	Recalc	Renorted	Recalc	Renorted	Recalculated
1,1-Dichloroethene	25	Υ¥	47.8	¥-N	9¢	96				
Trichloroethene	-	,	79.5-		99,	99				
Benzene			49.6		J	99				
Toluene			2.05		100	201				
Chlorobenzene	$\bigwedge$	1	49.94		201	10)				
Comments: Refer to Laboratory	v Control Sa	mole findings	s worksheet foi	r list of aualific	cations and a	scoriated car	mnlas whan re	anorted results	do not agree wi	hin 10 0% of the

recalculated results.

LDC #:<u>/9/88</u> SDG #:<u>2000</u>

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	
Reviewer:	4
2nd reviewer:	

)

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

YN N/A YN 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?

Concer	ntratio	$n = \frac{(A_{x})(I_{y})(DF)}{(A_{xs})(RRF)(V_{y})(\%S)}$
A <sub>x</sub>	=	Area of the characteristic ion (EICP) for the compound to be measured
A <sub>is</sub>	=	Area of the characteristic ion (EICP) for the specific internal standard
l <sub>s</sub>	=	Amount of internal standard added in nanograms (ng)
RRF	=	Relative response factor of the calibration standard.
V <sub>o</sub>	=	Volume or weight of sample pruged in milliliters (ml) or grams (g).
Df	=	Dilution factor.
%S	=	Percent solids, applicable to soils and solid matrices only.

Example:				
Sample I.D	2	ND		
Conc. = (	) (	)(		)
(	)(	)(	)(	
_				

	0niy.				
#.	Sample ID	Compound	Reported Concentration ()	Calculated Concentration ()	Qualification
				-	
		<u> </u>			

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

Project/Site Name: BRC Ironox Parce	l G
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Soil

Collection Date: June 11, 2008

LDC Report Date: August 6, 2008

Matrix:

Parameters: Semivolatiles

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

### Sample Identification

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

\*\*Indicates sample underwent EPA Level IV review

### Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### **II. GC/MS Instrument Performance Check**

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

### III. Initial Calibration

Initial calibration was performed using required standard concentrations.

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

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

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

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
6/18/08	Phthalic acid	0.01422 (≥0.05)	All samples in SDG F8F120180	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	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	J- (all detects) UJ (all non-detects)	A

The percent difference (%D) of the second source calibration standard were less than or equal to 25.0% for all compounds.

All of the continuing calibration RRF values were within method and validation criteria with the following exceptions:

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
6/18/08	Phthalic acid N-(Hydroxymethyl)phthalimide	0.01330 (≥0.05) 0.04331 (≥0.05)	8168439MB	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A
6/19/08	Phthalic acid N-(Hydroxymethyl)phthalimide	0.01066 (≥0.05) 0.04523 (≥0.05)	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A

### V. Blanks

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

No field blanks were identified in this SDG.

### VI. Surrogate Spikes

Surrogates were added to all samples and blanks as required by the method. All surrogate recoveries (%R) were within QC limits.

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

All internal standard areas and retention times were within QC limits with the following exceptions:

Sample	Internal Standards	Area (Limits)	Compound	Flag	A or P
TSB-GJ-09-10'	Perylene-d12	198321 (281395-1125580)	3321 (281395-1125580) Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene		A
TSB-GJ-09-20'**	Perylene-d12	191974 (281395-1125580)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) UJ (all non-detects)	A
TSB-GJ-09-30'	Perylene-d12	206248 (281395-1125580)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) UJ (all non-detects)	A
TSB-GJ-09-40'	Perylene-d12	212988 (281395-1125580)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) UJ (all non-detects)	A

### **XI. Target Compound Identifications**

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

### XII. Compound Quantitation and CRQLs

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

### XIII. Tentatively Identified Compounds (TICs)

Tentatively identified compounds were not reported by the laboratory.

### XIV. System Performance

The system performance was acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

### XV. Overall Assessment of Data

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

### XVI. Field Duplicates

No field duplicates were identified in this SDG.

### BRC Tronox Parcel G Semivolatiles - Data Qualification Summary - SDG F8F120180

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

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

No Sample Data Qualified in this SDG

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

No Sample Data Qualified in this SDG

### VALIDATION COMPLETENESS WORKSHEET

LDC #: 19188A2 SDG #: F8F120180

### Level III/IV



Laboratory: Test America

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	$\mathbf{A}$	Sampling dates: 6/11/08
11.	GC/MS Instrument performance check	$\overline{4}$	· · · ·
III.	Initial calibration	m	
IV.	Continuing calibration/ICV	TW	KY======70
V.	Blanks	A	
VI.	Surrogate spikes	Æ	
VII.	Matrix spike/Matrix spike duplicates	A	TSB-&1-08-101
VIII.	Laboratory control samples	W	409
IX.	Regional Quality Assurance and Quality Control	N	
Х.	Internal standards	w	
XI.	Target compound identification	$\mathbf{A}$	Not reviewed for Level III validation.
XII.	Compound quantitation/CRQLs	Ä	Not reviewed for Level III validation.
XIII.	Tentatively identified compounds (TICs)	N	Not reviewed for Level III validation.
XIV.	System performance	Ą	Not reviewed for Level III validation.
XV.	Overall assessment of data	A	
XVI.	Field duplicates		
XVII.	Field blanks	L Ň_	

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

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

D = Duplicate TB = Trip blank EB = Equipment blank

Validated Samples:

\*\* Indicates sample underwent Level IV validation

1	TSB-GJ-09-10' 5	11	81687439MB	21	31	
2	TSB-GJ-09-20'**	12		22	32	
3	TSB-GJ-09-30'	13		23	33	
4	TSB-GJ-09-40'	14		24	34	
5	11. Januari, 11. J	15		25	35	
6		16		26	36	
7		17		27	37	
8		18		28	38	
9	· · · · · · · · · · · · · · · · · · ·	19		29	39	
10		20		30	40	

LDC #: 19188A2 SDG #: <u>See coun</u>

### **VALIDATION FINDINGS CHECKLIST**



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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times		1		
All technical holding times were met.	1			
Cooler temperature criteria was met.				
II. GC/MS Instrument performance check	1		1	
Were the DFTPP performance results reviewed and found to be within the specified criteria?	$\square$	/		
Were all samples analyzed within the 12 hour clock criteria?	<b> </b>			
10. Initial calibration				ang banang sa
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?	$\mathcal{V}$			<
Did the initial calibration meet the curve fit acceptance criteria of $\geq 0.990$ ?				
Were all percent relative standard deviations (%RSD) $\leq$ 30% and relative response factors (RRF) $\geq$ 0.05?	ŀ	/		
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?				
Were all percent differences (%D) $\leq$ 25% and relative response factors (RRF) $\geq$ 0.05?		(		
V. Blanks				
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.		/		
VI. Surrogate spikes				
Were all surrogate %R within QC limits?	$\square$			
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?			$\langle$	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?				1
VII. 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.				
Was a MS/MSD analyzed every 20 samples of each matrix?	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences				
			, iP	
Was an LCS analyzed for this SDG?	$\left[ \right]$			

LDC #: 1918842 SDG #: <u>See COWN</u>

### VALIDATION FINDINGS CHECKLIST

Page:	<u>≥of</u>
Reviewer:	9
2nd Reviewer:	

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		/		
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		(* 1. j. j.		
Were internal standard area counts within -50% or +100% of the associated calibration standard?	Ø			
Were retention times within + 30 seconds from the associated calibration standard?				
Were relative retention times (RRT's) within ± 0.06 RRT units of the standard?	4			
Did compound spectra meet specified EPA "Functional Guidelines" criteria?				
Were chromatogram peaks verified and accounted for? XII. Compound quantitation/CRQLs		- 11 I		
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?	7			
XIII. Tentatively identified compounds (TICs)		4		
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?			$\land$	
Were relative intensities of the major ions within <u>+</u> 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)?		1		
XIV, System performance			1.55	and the second
System performance was found to be acceptable.				
XV Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			
XVI. Fleid duplicates				
Field duplicate pairs were identified in this SDG.		$\square$		
Target compounds were detected in the field duplicates.			7	
XV/II. Field blanks				
Field blanks were identified in this SDG.				/
Target compounds were detected in the field blanks.			7	

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

UUU. 1.2.4.5-Tetradolorobenzeul www. 4-chlinobenzen+hiol MMM. Bis(2-Chloroisopropyl)ether 000. N-Nitrosodimethylamine Acetaphenone KKK. Dibenz(a,h)anthracene JJJ. Indeno(1,2,3-cd)pyrene LLL. Benzo(g,h,i)perylene Benzenethio 1 disulfide lli. Benzo(a)pyrene\*\* QQQ. Benzyl alcohol PPP. Benzoic Acid SSS. Benzidine RRR. Pyridine NNN. Aniline Ë ZZZ: Phenyl EEE. Bis(2-ethylhexyl)phthalate BBB. 3,3'-Dichlorobenzidine GGG. Benzo(b)fluoranthene HHH. Benzo(k)fluoranthene AAA. Butylbenzylphthalate CCC. Benzo(a)anthracene FFF. Di-n-octylphthalate\*\* TT. Pentachlorophenol\*\* XX. Di-n-butylphthalate YY. Fluoranthene\*\* UU. Phenanthrene W. Anthracene DDD. Chrysene WW. Carbazole ZZ. Pyrene XXX Pheny I sulfide. QQ. N-Nitrosodiphenylamine (1)\*\* MM. 4-Chlorophenyl-phenyl ether RR. 4-Bromophenyl-phenylether PP. 4,6-Dinitro-2-methylphenol SS. Hexachlorobenzene KK. 2,4-Dinitrotoluene HH. 2,4-Dinitrophenol\* EE. 2,6-Dinitrotoluene GG. Acenaphthene\*\* LL. Diethylphthalate 00. 4-Nitroaniline FF. 3-Nitroaniline JJ. Dibenzofuran II. 4-Nitrophenol\* NN. Fluorene H-H+Johoxymethy) >+ halimide P. Bis(2-chioroethoxy)methane X. Hexachlorocyclopentadiene\* V. 4-Chloro-3-methylphenol\*\* R. 1,2,4-Trichlorobenzene U. Hexachlorobutadiene\*\* Y. 2,4,6-Trichlorophenol\*\* AA. 2-Chloronaphthalene Q. 2,4-Dichlorophenol\*\* W. 2-Methylnaphthalene Z. 2,4,5-Trichlorophenol CC. Dimethylphthalate 2-ch(répheny) >u (toue DD. Acenaphthylene T. 4-Chloroaniline BB. 2-Nitroaniline S. Naphthalene H. 2,2'-Oxybis(1-chloropropane) J. N-Nitroso-di-n-propylamine\* B. Bis (2-chloroethyf) ether E. 1,4-Dichlorobenzene\*\* D. 1,3-Dichlorobenzene F. 1,2-Dichlorobenzene O. 2,4-Dimethylphenol K. Hexachloroethane C. 2-Chlorophenol N. 2-Nitrophenol\*\* G. 2-Methylphenol I. 4-Methylphenol L. Nitrobenzene M. Isophorone A. Phenol\*\* AAAA . XXX.

VALIDATION FINDINGS WORKSHEET

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# VALIDATION FINDINGS WORKSHEET

Lot L	4	
Page:	Reviewer:	2nd Reviewer.

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

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

Did the laboratory conduct an acceptable 5 point calibration prior to sample analysis? N N/A <u>Y / N N/A</u>

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? Y N N/A Y N N/A

nd ≥0.05 RRF ?	Qualifications															
	Associated Samples	M+B+C														
	Finding RRF (Limit: <u>&gt;</u> 0.05)	-22410.0	0.04408													
criteria of ≤30 %RSD ₅	Finding %RSD (Limit: <u>&lt;</u> 30.0%)															
s within the validation	Compound	thethalic acid	XXX													
/ere all %RSDs and RR	Standard ID	Ich.														
N N/A	≄ Date'	6/3/3	、 、 、						 							
≻∥Ľ					<u> </u>										1	

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Page: /of / Reviewer: 2nd Reviewer:	Qualifications	The the			A-MU A	XINXX	```										
for each instrument? CCC's and SPCC's ?	Associated Samples	RAK			MTNB												
<b>NINGS WORKSHEET</b> <b><u>3</u> <u>Calibration</u> tre identified as "N/A". tours of sample analysis to in method criteria for all 0 5 RRF ?</b>	Finding RRF (Limit: <u>≥</u> 0.05)	0 8810.0	0.04331			0.0/066	0.04523										
VALIDATION FIND <u>Continuing</u> applicable questions a t least once every 12 h nse factors (RRF) withi a of ≤25 %D and ≥0.0	Finding %D (Limit: <u>≤</u> 25.0%)				25:06818												
od 8270) ons answered "N". Not ons anavderd analyzed a %D) and relative respo hin the validation criteri	Compound	thethalize acid	$\chi \chi \chi$		HAHVAI'S acid		XXX										
NA (EPA SW 846 Metho INA (EPA SW 846 Metho ions below for all questi is a continuing calibratic ire percent differences ( ire all %D and RRFs with	Standard ID	1CA15197			PCS22ADV												
LDC #: 19/2412 SDG #: 200 METHOD: GC/MS B Please see qualificat V N N/A We Y N N/A We	# Date	6/12/08			6/9/08	//											

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## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Page: <u>\_\_\_\_\_\_\_</u>of\_\_\_\_ Reviewer: 2nd Reviewer: \_

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

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

				_	_										-		_								
Qualifications	- Fult	No leual		(masn/sn)																					
Associated Samples	WH Det																								
RPD (Limits)	( )	(	(			( )	( )	( )		( )	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(
LCSD %R (Limits)	( )	( )	(	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(	( )	( )	( )	( )	( )	(	( )	( )
LCS %R (Limits)	19 (12/20)		( )	( )		( )	( )	( )		( )	(	( )	( )	(	(	( )	(	( )	( )	( )	( )	( )	( )	( )	( )
Compound	++++																								
rcs/rcsb iD	216843918																								
Date																									
*																									

LDC	#: 19182 #: 200 A	Anto A		ALIDATION FINDINGS WORK Internal Standards	SHEET	Page: of A
N Plea	THOD: GC/N Se see quali <u>V N/A</u> V	IS BNA (EPA SW 846 Meth fications below for all ques Were all internal standard ε Were the retention times of	ood 8270) tions answered "N' area counts within f the internal s <u>t</u> and	". Not applicable questions are identif -50 to +100 of the associated calibra ards within +/- 30 seconds of the ret	ified as "N/A". ation standard? tention times of the associated calib	2nd Reviewer:
#	Date	Sample ID	Internal Standard	Area (Limits)	RT (Limits)	Qualifications
		/	PRY	198321 (281395-1123	¢580)	K TM K
		Ν	XX	19,974 C	(	
		u)	PRY	2062485	(	
		4	TRY	2/2988C	C	
						(FF - 222)
				A.		
C *	limite ara advie					

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

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

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SDG #: 200 - 2010

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

ō Page: 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

 $\label{eq:RFF} RFF = (A_{\rm J})(C_{\rm h})(C_{\rm J})$  average RFF = 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,

A<sub>k</sub> = Area of associated internal standard C<sub>k</sub> = Concentration of internal standard X = Mean of the RRFs

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

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рс	SDG

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



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 =  $(A_{\nu})(C_{\kappa})/(A_{\kappa})(C_{\nu})$ 

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

 $A_{\mathbf{k}} = Area$  of associated internal standard  $C_{\mathbf{k}} = Concentration of internal standard$ 

Recalculated	۵%	2 8971	0770	AR TEO	1, 5, 8, 2, 3	a root	N a Za V	7-1910	, 10 KO	1-21-2-	_			7 492 1	X V XX C	2 1010	10000	262.0	1 1
Reported	۵%	2.89721	1.97399	1.24855-0	02585.5	0.97842	1.3606 2	0.10052	2 1246	- natar				n adds	2 887 10	018810	2 SALIC	176921	
Recalculated	RRF (CC)	1.80162	1.08712	1.4087X	0.20730	0.87 8x	1.15094	0.9326	1.20707					1.59157	1.339021	1.01788	0.38045	0.29958	
Reported	RRF (CC)	1-30162	1.08712	1.40878	0.20730	88178.0	1.12694	0.51326	20702.1					1.59157	033954	×8710.1	0.750 dc	0.39958	
	Average RRF (initial)	TE228.1	1.10001	1.41229	0.19634	0.86343	1-11182	0.5474	1.18223					1.575-90	0.33002	1.02385	0.36637	24265.0	
	Compound (Reference Internal Standard)	Phenol (1st internal standard)	Naphthalene (2nd internal standard)	Fluorene (3rd internal standard)	Pentachtorophenol (4th internal standard)	Bis(2-ethylhexyl)phthalate (5th internal standard)	Benzo(a)pyrene (6th internal standard)	Phenol (1st internal standard)	Naphthalene (2nd internal standard) $\mathcal{M}\mathcal{M}\mathcal{M}$	Fluorene (3rd internal standard)	Pentachlorophenol (4th internal standard)	Bis(2-ethylhexyl)phthalate (5th internal standard)	<u>Benzo(a)pyrene (6th internal standard)</u>	Phenol (1st internal standard) $VVV$	Naphthalene (2nd internal standard) $WWW$	Fluorene (3rd internal standard)	Pentachlorophenel (4th internal standard) 222	Bie(2 ethythexyl)phthalate (5th internal standard)	Renzo(a) nurene /8th internal standard)
	Calibration Date	0/0/19	~					6/0/08	· · ·					6/19/08					
	Standard ID	Tees W-Y						InAc228	-					104529					
	#	-			+			N						0					

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 #:1918842 REDW SDG #: -

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

Page:\_ 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 =  $(A_{a})(C_{a})/(A_{a})(C_{a})$ 

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

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

					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q%	0%
-	VCANS195	6/3/08	Phenol (1st internal standard)	1.85537	1.8774	1.87174	ULEXX 0	1000
	/	/ /	Naphthalene (2nd internal standard)	1.1090	1.10135	1.10135	0 KANTN	n hand
			Fluorene (3rd internal standard)	1.41229	1.398.01	1.39501	820101	20101
	-		Pentachlorophenol (4th internal standard)	0.19634	0.2020	0.203/0	12072 2	27472
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.86343	0.87788	0.870 88	1. 86222	0.460
			Benzo(a)pyrene (6th internal standard)	-1118,2	1.11507	76311.1	08-62.0	0.2924
~	NCA15196	e/18/08	Phienol (1st internal standard)	6.512T4	23185.0	1 52185	42914-	1.7767
		/ /	Maphihalene (2nd internal standard) $\mathcal{W}\mathcal{U}\mathcal{U}$	82-21.1	1.17316	1.17316	076745	N-724 0
			Fluorene (3rd internal standard)				C T	<u> </u>
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
		· · ·	Benzo(a) pyrene (6th internal standard)					
e	Jettel M	6/8/18	Phenol (1 st internal standard) $VVV$	1.67590	1.60400	1.60400	1.78300	- 78.2.2
	-		Maphthelene (2nd internal standard) $MM_{ m N}$	0.33002	0.33744	0.33744	2 20747	1 2 B C
	`		Fluorana (3rd internal standard)	1.02385	1.03316	1.03316	0 9092	00000
			Pentachlorophenol (4th internal standard)	0.3637	0.38=74	1 3824	2.468m >	6111 P
			Bis(2-ethylhexyl)phthalate (5th internal)दियोगिती	0.39265	039671	0.39671	1.03291	1 0 332
			Benzo(a)pyrene (6th internal standard)					
Com	ments: <u>Refer t</u>	o Continuing C	alibration findings worksheet for list of	qualifications and	1 associated samp	oles when reported	results do not agree	e within 10.0% of the

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recalculated results

LDC #: 19 SDG #: See C

### VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page:	<u>/of /</u>
Reviewer:	Q
2nd reviewer:	

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

### Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5		33.9134	65		
2-Fluorobiphenyl		35.9420	68		
Terphenyl-d14		39.8482	78		
Phenol-d5		52.0537	66		
2-Fluorophenol		50,9295	65		
2,4,6-Tribromophenol		52.8840	69		
2-Chiorophenol-d4					
1,2-Dichlorobenzene-d4					

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

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	50	32.5367	65	65	0
2-Fluorobiphenyl	1	33.9843	68	68	
Terphenyl-d14	V	38.7625	78	78	
Phenol-d5	75	49.6403	66	66	
2-Fluorophenol		49.04-21	65	65	
2,4,6-Tribromophenol		52.0744	69	69	$\checkmark$
2-Chiorophenol-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 # 018842	SDG #: See COW
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# Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification VALIDATION FINDINGS WORKSHEET

Page: <u>lof</u> Reviewer: <u>9</u> 2nd Reviewer:

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

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

% Recovery = 100 \* (SC/SA

Where: SSC = Spike concentration SA = Spike added

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

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

LCS/LCSD samples: 25168437

csn	ď	Recalculated									
I CS/I	Υ.	Renorted									
sn	Recovery	Recalc									
	Percent I	Renorted									
Ş	Recovery	Recalc	ト	77	77	121	67	01			
Г	Percent F	Renorted	14	77	トト	75	67	70			
ike	Mration パン)	1 CSD	AM				/	$\bigwedge$			
Sp	Concer ( July	I CS	3350	2570	2560	2152	2240	2350			
ike	ted ES)	I CSD	NĂ					Ņ			
Sp	Add	LCS	3370					V			
	Compound		Phenol	N-Nitroso-di-n-propylamine	4-Chloro-3-methylphenol	Acenaphthene	Pentachlorophenoi	Pyrene			

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 #: 1918842 SDG #: Sec court

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	(of
Reviewer:	9
2nd reviewer:	

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

YN N/A YN 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	entratic	$n = (A_{,})(I_{,})(V_{,})(DF)(2.0) - (A_{,})(RRF)(V_{,})(V_{,})(%S)$	Example:		1-					
A,	=	Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D	2.	NZ	<u> </u>				
A <sub>is</sub>	=	Area of the characteristic ion (EICP) for the specific internal standard								
l,	=	Amount of internal standard added in nanograms (ng)	Conc. = ((	)()(	<u>)(</u>	()(	_)(	)(	_)(	)
V <sub>°</sub>	=	Volume or weight of sample extract in milliliters (ml) or grams (g).								
V,	=	Volume of extract injected in microliters (ul)	=							
V,	=	Volume of the concentrated extract in microliters (ul)								
Df	-	Dilution Factor.								
%S	=	Percent solids, applicable to soil and solid matrices only.								
2.0	=	Factor of 2 to account for GPC cleanup								
[ <b></b>	Î									

			Reported	Calculated	
#	Sample ID	Compound	()	()	Qualification
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	<u></u>				
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					<u></u>
		-		·	
1	1	4	1 .		

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

Project/Site Name: BRC Tronox, Parcel G

Collection Date: June 11, 2008

LDC Report Date: August 6, 2008

Matrix:

Parameters: Chlorinated Pesticides

Soil

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

### Sample Identification

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

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 4 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8081A for Chlorinated Pesticides.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### **II. GC/ECD Instrument Performance Check**

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

### III. Initial Calibration

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

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

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

Retention time windows were evaluated and considered technically acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples on which a Level III review was performed.

### **IV. Continuing Calibration**

Continuing calibration was performed at required frequencies.

The percent differences (%D) of calibration factors in continuing standard mixtures were within the 15.0% QC limits with the following exceptions:

Date	Standard	Channel	Compound	%D	Associated Samples	Flag	A or P
6/18/08	KCAL092	A	Toxaphene	15.2	TSB-GJ-09-30' TSB-GJ-09-40'	J+ (all detects)	A
6/18/08	KCAL095	A	2,4'-DDD	22.6	TSB-GJ-09-30' TSB-GJ-09-40'	J+ (all detects)	Ρ

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

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

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

### V. Blanks

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

No field blanks were identified in this SDG.

### VI. Surrogate Spikes

Surrogates were added to all samples and blanks as required by the method. All surrogate recoveries (%R) were within QC limits.

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Pesticide Cleanup Checks

### a. Florisil Cartridge Check

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

### b. GPC Calibration

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

### XI. Target Compound Identification

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

### XII. Compound Quantitation and Reported CRQLs

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

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

No field duplicates were identified in this SDG.

### BRC Tronox, Parcel G Chlorinated Pesticides - Data Qualification Summary - SDG F8F120180

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

### BRC Tronox, Parcel G

Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG F8F120180

No Sample Data Qualified in this SDG

BRC Tronox, Parcel G

Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG F8F120180

No Sample Data Qualified in this SDG

### VALIDATION COMPLETENESS WORKSHEET

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

### Level III/IV

	111
Date:	8/4/08-
Page:_	<u>/of /</u>
Reviewer:	<u>Q</u>
2nd Reviewer:	

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 6/11/08
II.	GC/ECD Instrument Performance Check	A	
111.	Initial calibration	Å	KSD.12
IV.	Continuing calibration/ICV	in	1ex \$ 1570
V.	Blanks	Ă	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	Å	75B-G1-08-10
VIII.	Laboratory control samples	A	10-3
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	
Xb.	GPC Calibration	N	
XI.	Target compound identification	A	Not reviewed for Level III validation.
XII.	Compound quantitation and reported CRQLs	$\mathbf{A}$	Not reviewed for Level III validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks		

Note:

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

ND = No compounds detected R = Rinsate

FB = Field blank

D = Duplicate TB = Trip blank EB = Equipment blank

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

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

LDC #: 19188A39 SDG #: <u>Sec. Cour</u>1

### VALIDATION FINDINGS CHECKLIST

Method: GC HPLC	<u> </u>			
Validation Area	Yes	No	NA	Findings/Comments
f Teconicalitolding tunes				
All technical holding times were met.	/			
Cooler temperature criteria was met.				
1 millionation				
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?	$\square$		ļ	
Were the RT windows properly established?		e de la contra	SHALL DE	
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%?		/	1	
Were all the retention times within the acceptance windows?		-	1966298	
V-Blacks			1996) T	
Was a method blank associated with every sample in this SDG?			<b> </b>	
Was a method blank analyzed for each matrix and concentration?	1			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.			1	and the particular second state and the second state of the second resonance and the second
VI. Songette splikes				
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?			<	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?			/	
VII Marix spike/Marix 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?	/	1	<u> </u>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII*Laboratory control samples				1 1
Was an LCS analyzed for this SDG?	$\downarrow /$		<u> </u>	
Was an LCS analyzed per extraction batch?				1

LDC #: 19188 A39 SDG #: <u>Seccour</u>

### 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?	1	-		
IX: Regional Quality Assurance and Ordality Control				
Were performance evaluation (PE) samples performed?		$\square$		/
Were the performance evaluation (PE) samples within the acceptance limits?	C C a Tangata	New Joseph Control of C		
X. Target compound identification				
Were the retention times of reported detects within the RT windows?			101.0. 3 Said	
XI. Composind quantitation/CRQLs				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XIIIS)Slempedorpance				
System performance was found to be acceptable.	$\left( \right)$			
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.				
XIV. Field dublicates				
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?			7	

## VALIDATION FINDINGS WORKSHEET

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

A. alpha-BHC	I. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	CG.
B. beta-BHC	J. 4,4"-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH.
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	H,
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	.rr
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. DB 608	Ϋ́Υ.
F. Aldrin	N. Endosulfan sulfate	V. Arocior-1016	DD. DB 1701	LL.
G. Heptachlor epoxide	0. 4,4'-DDT	W. Aroclor-1221	EE	MM.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	EF.	ŻŻ

Notes:

LDC #:∠ SDG #: 1

METHOD: \_\_\_\_GC \_\_\_HPLC

## VALIDATION FINDINGS WORKSHEET <u>Continuing Calibration</u>

\_of Reviewer: Q Page:

2nd Reviewer:

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". What type of continuing calibration calculation was performed? %D or RPD W N/A Were continuing calibration standards analyzed at the required frequencies? Y M N/A

Did the continuing calibration standards meet the %D / RPD validation criteria of ≤15.0%? evel IV Only Y/N N/A

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

	-	Qualifications	1+dets/1	U+ letert												
	Accordiated Cameral		3-4													
	RT (limit)					(	(								)	( )
%D/RPD	(Limit < 15.0)	15 21	2.2	N 1 1												
	Compound	Z	246-40	Apr 1.5												
Detector/	Column	Chr A	1													
	Standard ID	KCAZ092	KCALOGS													
		Sul 1/2		• •												

LDC #: 1918 89/39 SDG #: Zee COW

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

6 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

Indard ID Calibratio						Kecalculateo	Keported	Recalculated
12/2)	Loi	Compound	CF (D S std)	CF Sstd)	Average CF (initial)	Average CF (initial)	%RSD	%RSD
20/2/10/	9 2	2.4- 00 E (Ch. A)	37366 840	37366844	37378556	3/3/25269	1. 30/08	1.3011
	× x	V (ChB)	211217200	bac relic	20380278	20380279	2.78044	2.78ax
	. ,	F (ch.A)	645423200	655453200	\$1332132 a	681332137	a.76/88	2.7619
	2	0 ( V )	28233800	2823386	30/26/96/2	30/26962	547-117	11124
te 6/16/11.	2	F (ch B)	0a0/15892	363591000	38018-38085	28878/all	2.83696	2.8370
		0	128/5600	12681560	133202078	133202078	8.79750	8.7925
	L							
	1							
	<u> </u>							
	L							

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 #:260 

**Continuing Calibration Results Verification** VALIDATION FINDINGS WORKSHEET

5 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

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

Where:

Recalculated  $\mathcal{L}$ 0% ſ N Ŋ ()Reported %۵ d' Ŋ N 2220.0 5 Recalculated CF/Conc. CCV SYC O X 220 0 0 0.0255 CF/Conc. CCV 500 320 2 2 2 5 0 Reported Ó 0 Average CF(lcal)/ 200.0 X CCV Conc. 220. 0 AN N Compound 4 もりと 00 4.0 0 4 H  $\mathcal{E}_{\mathcal{U}}$ 6/18/08 Calibration Date Ŕ 0 Standard ID *b* K/A ¥ 2 ო 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 #:*191881339* SDG #:*5ec.01*4.11

## VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

of 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

Sample ID: 2-

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
TCNX	Ch A	0.020	0.01360	50	e E	0
D013	$\checkmark$	1	0.01958	98	98	0
			~			

### Sample ID:

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

### Sample ID:

~	>
R	N
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#	#
LDC	SDG

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

័ច Page: Reviewer: 2nd Reviewer:

> CC 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

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

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

LCSD = Laboratory control sample duplicate percent recovery SC = Concentration

LCS/LCSD samples: 276 37

	S .	pike	Spiked	Sample		cs	ГС	SD	TCS/L	CSD
Compound	NY)	doed W	Conce	pration \$15	Percent	Recovery	Percent	Recovery	RP	Q
	rcs		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)										
H	16.7	NA	15.0	NÅ	06	90				
$\mathcal{O}$	1	//	16.8	N	101	101				
Comments: <u>Refer to Labor</u>	atory Control	Sample/Lab	oratory Control	Sample Dupli	icate findings wo	orksheet for lis	st of qualifications	s and associate	d samples wh	nen reporte

V:\Validation Worksheets\GC\LCSDCLC\_GC.wpd

results do not agree within 10.0% of the recalculated results.

00 #17188439	06 #: <i>See cowN</i>
ĽDC	SDG

## VALIDATION FINDINGS WORKSHEET Sample Calculation Verification



Concentration= (A)(Fv)(Df)	Example:	
(001/202) Visit in sal/ ini)		
<ul> <li>A= Area or height of the compound to be measured</li> <li>Fv= Final Volume of extract</li> <li>Df= Dilution Factor</li> </ul>		
RF= Average response factor of the compound	Concentration =	
Vs= Initial volume of the sample Ws= Initial weight of the sample %S= Percent Solid		

#	Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications
Comme	ints:				

SAMPCALew.wpd

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

Project/Site Name:	BRC Tronox Parcel G
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Collection Date: June 11, 2008

LDC Report Date: August 6, 2008

Matrix:

Parameters: Polychlorinated Biphenyls

Soil

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

### Sample Identification

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

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 4 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8082 for Polychlorinated Biphenyls.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- 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 was performed for the primary (quantitation) column as required by the method.

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

Retention time windows were evaluated and considered technically acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples on which a Level III review was performed.

### IV. Continuing Calibration

Continuing calibration was performed at required frequencies.

The percent differences (%D) of calibration factors in continuing standard mixtures were within the 15.0% QC limits.

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

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

### V. Blanks

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

No field blanks were identified in this SDG.

### VI. Surrogate Spikes

Surrogates were added to all samples and blanks as required by the method. All surrogate recoveries (%R) were within QC limits.

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Pesticide Cleanup Checks

### a. Florisil Cartridge Check

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

### **b. GPC Calibration**

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

### XI. Target Compound Identification

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

### XII. Compound Quantitation and Reported CRQLs

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

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

No field duplicates were identified in this SDG.

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化化合物 化结核化 经保证 化乙基 输出 机运行的 医结节 医外侧的 医黄色的 经营销 医神经神经 化过度 化化合体的 医外外的 网络小麦瓜 医外外外的 化乙基苯基乙基

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

No Sample Data Qualified in this SDG

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

No Sample Data Qualified in this SDG

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

No Sample Data Qualified in this SDG

LDC #: <u>19188A3b</u> SDG #: <u>F8F120180</u> Laboratory: <u>Test America</u>

### Level III/IV

Date: <u>8/4/08</u>
Page:/of
Reviewer:
2nd Reviewer:

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/11/0-8
II.	GC/ECD Instrument Performance Check	N	/ /
111.	Initial calibration	A	
IV.	Continuing calibration/ICV	A	1CV = 1570
V.	Blanks	$\mathbf{A}$	
VI.	Surrogate spikes	$\mathbf{A}$	
VII.	Matrix spike/Matrix spike duplicates	A	TSB-GJ-08-10
VIII.	Laboratory control samples	A	105
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	
Xb.	GPC Calibration	N	
XI.	Target compound identification	A	Not reviewed for Level III validation.
XII.	Compound quantitation and reported CRQLs	A	Not reviewed for Level III validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	N	

Note:

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

Validated Samples:

\*\* Indicates sample underwent Level IV validation

1	TSB-GJ-09-10' 5	11	8168762MB	21	31	
2	TSB-GJ-09-20'**	12		22	32	
3	TSB-GJ-09-30'	/13		23	33	
4	TSB-GJ-09-40'	14		24	34	
5		15		25	35	
6		16		26	36	
7		17		27	37	
8		18		28	38	
9		19		29	39	
10		20		30	40	

LDC #:<u>19188435</u> SDG #:<u>3@\_COW</u>

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### VALIDATION FINDINGS CHECKLIST

1

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

Method: GC HPLC				]
Validation Area	Yes	No	NA	Findings/Comments
Crectorical bolding tunes				AND STATES
All technical holding times were met.	$\square$			
Cooler temperature criteria was met.				
1: multi-calorabon				
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%?	$\square$			
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?		S. 1995	C 70743 013	
1V Continuing calibration			CARCO T	
What type of continuing calibration calculation was performed?%D or %R				
Was a continuing calibration analyzed daily?	Ζ,		<b> </b>	
Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?			<u> </u>	
Were all the retention times within the acceptance windows?				
V-Blanks		n F	`≪ T	n an
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.				
V). Somogate spikes		i i i i		
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?	2.20 M 12.00	Let speak		
VII Matrix spike/Matrix spike duplicates			<u>.</u>	
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?	$\Box$	ł		
Was an LCS analyzed per extraction batch?	1/			

LDC #: 19188A SDG #: <u>Sec CO</u>

### VALIDATION FINDINGS CHECKLIST

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Reviewer: C	
2nd Reviewer:	

Validation Area	Yes	No	NA	Findings/Comments
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
X.Regional Quality Assurance and Weality/Control				
Were performance evaluation (PE) samples performed?		$\square$		
Were the performance evaluation (PE) samples within the acceptance limits?			(	
X-Target compound (dentification				
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?				
XIISVslemperformance				
System performance was found to be acceptable.	[			
XIII Noveralitassessment of data				
Overall assessment of data was found to be acceptable.	17			
XIV_FIeld Toppleares				
Were field duplicate pairs identified in this SDG?		[	^	
Were target compounds idetected in the field duplicates?			/	
XV. Fieldblanks				
Were field blanks identified in this SDG?		1		
Were target compounds detected in the field blanks?			1	

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	Ŧ
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	ï
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. Arocior-1016	DD. DB 1701	LL
G. Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	Ë	MM.
H. Endosultan I	P. Methoxychlor	X. Aroclor-1232	FF.	Ż

Notes:

1.1
LDC #: 18/82431 SDG #: 20

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

	f	
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/Caverage 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
#	Standard ID	Calibration Date	Compound	CF ( <i>ら</i> つ std)	CF ( <i>ら</i> で std) /	Average CF (initial)	Average CF (initial)	%RSD	%RSD
-	2	_/ /_	( FER (RTX-CAPONT)	33154	33154	27972	27972	(3.0	(2.0
		Raliels	TAS ( 1 H)	45676	45676	39164	39164	~ &ib	9.582
$\square$								-	
7									
e									
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 #: 19185 ABA

VALIDATION FINDINGS WORKSHEET Continuing Calibration Results Verification

đ Page: 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 \_ W ·CF = A/C

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

A = Area of compound C = Concentration of compound

Recalculated 160 N 0% J 5 Ď M Reported ۵° η 4 Recalculated CF/Conc. CCV 952 Ν 33 1902 937,3342 CF/Conc. CCV Reported 952.1 Average CF(Ical)/ 0001 CCV Conc. 020 tool H MY-CLASS Compound ، ۲ BB ( BB R 10/18/03 18/18/ Calibration Date 2802 20. Standard ID Alt Hed. **#**£ 0 ო 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 #: /9/88/35/ SDG #:500 COWN

## VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

/ of / 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

Sample ID:

		_		_
Percent Difference		0		
Percent Recovery	Recalculated	102	_	
Percent Recovery	Reported	102		
Surrogate Found		20.4758		
Surrogate Spiked		Q Q		
Column/Detector		ch A		
Surrogate		000		

## Sample ID:

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

## Sample ID:

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

1918813b	See COWN
LDC #:/	SDG # M

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

, o Page: -/ Reviewer: 2nd Reviewer:

METHOD: CC 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

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

LCS/LCSD samples: 3/8276 2

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

SC = Concentration

LCSD = Laboratory control sample duplicate percent recovery

	IS .	oike	Spiked	l Sample	Ľ	cs	ГС	sD	LCS/	csD
Compound	X Y Y	Idea	Conce	P / O	Percent	Recovery	Percent I	Recovery	R	0
	LCS	LCSD	rcs	<b>LCSD</b>	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)										
BB	167	4NA	121	$N\mathcal{A}$	601	102				
Comments: Refer to Labora	atory Control	Sample/Labo	oratory Control	l Sample Dupli	icate findings w	orksheet for lis	t of qualifications	s and associate	d samples w	hen reported

V:\Validation Worksheets\GC\LCSDCLC\_GC.wpd

results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET **Sample Calculation Verification** 



METHOD: 4 GC HPLC

۲	A
Z	Z
Z	Z
X	K

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:			
(NF)(VS 01 WS)(VOV 100)	Sample ID.	Compound Name	C N	
<ul> <li>A= Area or height of the compound to be measured</li> <li>-v= Final Volume of extract</li> <li>Dilution Factor</li> </ul>		-		
KF= Average response factor of the compound In the initial calibration	Concentration =			
/s= Initial volume of the sample Vs= Initial weight of the sample				
SE Percent Solid				

#	Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications
Jomme	ants:				

SAMPCALew.wpd

## LDC Report# 19188A4

وربينا ويعتقوه فرارم فارتد فالتراميني

## Laboratory Data Consultants, Inc. Data Validation Report

G

Collection Date: June 11, 2008

LDC Report Date: August 8, 2008

Matrix: Soil

Parameters: Metals

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

## Sample Identification

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

NAMES AND AND A STREET AND A

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 4 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Methods 6010B, 6020, and 7000 for Metals. The metals analyzed were Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Lithium, Magnesium, Manganese, Molybdenum, Mercury, Nickel, Niobium, Palladium, Phosphorus, Platinum, Potassium, Selenium, Silicon, Silver, Sodium, Strontium, Sulfur, Thallium, Tin, Titanium, Tungsten, Uranium, Vanadium, Zinc, and Zirconium.

This review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004) as there are no current guidelines for the methods stated above.

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

Blanks are summarized in Section III.

Field duplicates are summarized in Section XIII.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- 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.

## III. Blanks

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

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
ICB/CCB	Antimony Thallium Tungsten Vanadium Lithium Mercury	1.3 ug/L 1.1 ug/L 1.4 ug/L 2.7 ug/L 8.0 ug/L 0.1 ug/Kg	All samples in SDG F8F120180

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

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

No field blanks were identified in this SDG.

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

The frequency of analysis was met.

The criteria for analysis were met.

## V. Matrix Spike Analysis

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

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

## VI. Duplicate Sample Analysis

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

## VII. Laboratory Control Samples (LCS)

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

## VIII. Internal Standards (ICP-MS)

All internal standard percent recoveries (%R) were within QC limits for samples on which a Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

## IX. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

## X. ICP Serial Dilution

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

Diluted Sample	Analyte	%D (Limits)	Associated Samples Flag		A or P
TSB-GJ-08-10'L	lron	10.4 (≤10)	All samples in SDG F8F120180	J (all detects)	A

## XI. Sample Result Verification

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

## XII. Overall Assessment of Data

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

## XIII. Field Duplicates

No field duplicates were identified in this SDG.

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

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

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

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

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

No Sample Data Qualified in this SDG

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

## Level III/IV

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 6/11) 08
11.	Calibration	A	7 1
111.	Blanks	- Sw	
IV.	ICP Interference Check Sample (ICS) Analysis	A	
V.	Matrix Spike Analysis	SW	2hs/1430
VI.	Duplicate Sample Analysis	N	
VII.	Laboratory Control Samples (LCS)	A	Luy
VIII.	Internal Standard (ICP-MS)	A	pit veriewed for lend 3
IX.	Furnace Atomic Absorption QC	N	hit utrazes
Х.	ICP Serial Dilution	4W	
XI.	Sample Result Verification	A	Not reviewed for Level III validation.
XII.	Overall Assessment of Data	Â	
XIII.	Field Duplicates	N,	
XIV.	Field Blanks	μ	

Note: A = Acceptable N = Not provided/applicable 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  $4 \times 3$ 

SW = See worksheet

	-7011		 			
1	TSB-GJ-09-10'	11	21		31	
2	TSB-GJ-09-20'**	12	22		32	
3	TSB-GJ-09-30'	13	23		33	
4	TSB-GJ-09-40'	14	24	······································	34	
5	bB	15	25		35	
6		16	26		36	
7		17	27		37	
8		18	28		38	
9		19	29		39	
10		20	30		40	

Notes:

LDC #: 19188Auf SDG #: <u>Sue cover</u>

### VALIDATION FINDINGS CHECKLIST



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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical fiolding times			1965	
All technical holding times were met.	1			
Cooler temperature criteria was met.				
II. Calibration				
Were all instruments calibrated daily, each set-up time?	1		ļ	
Were the proper number of standards used?	/		<u> </u>	
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)	1		1442.201744	
III Blanks				
Was a method blank associated with every sample in this SDG?	1			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	/			
M-ICP interference Check Sample				
Were ICP interference check samples performed daily?	1			
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?			elector de	
IV-Matric 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) $\leq$ 20% for waters and $\leq$ 35% for soil samples? A control limit of +/- RL(+/-2X RL for soil) was used for samples that were $\leq$ 5X the RL, including when only one of the duplicate sample values were $\leq$ 5X the RL.		/		
V. Liaboratory control samples		902).		
Was an LCS anaylzed for this SDG?	1			
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?	/			
VI, 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% OC limits?		]		

LDC #\_\_\_\_\_9188/14 SDG #\_\_\_\_\_SU cover

### VALIDATION FINDINGS CHECKLIST

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4

Validation Area	Yes	No	NA	Findings/Comments
MILICE Senal Dilution	9月7日 全下的		2199) 21 (14)	
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the IDL?			ļ	
Were all percent differences (%Ds) < 10%?			ļ	
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.		1		
Vill Internal Standards (EPA SW 846 Method 6020)				
Were all the percent recoveries (%R) within the 30-120% of the intensity of the internal standard in the associated initial calibration?	/			
If the %Rs were outside the criteria, was a reanalysis performed?		and the all states		
IX: Regional Quality Assurance and Quality Control :				
Were performance evaluation (PE) samples performed?			_	
Were the performance evaluation (PE) samples within the acceptance limits?			-	
X Sample Result Vertication				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XI. Overall assessment of dela				
Overall assessment of data was found to be acceptable.	/			
XII Field duplicates				
Field duplicate pairs were identified in this SDG.		$\checkmark$		· · · · · · · · · · · · · · · · · · ·
Target analytes were detected in the field duplicates.				
XIII:Field blanks				
Field blanks were identified in this SDG.		/		
Target analytes were detected in the field blanks.			7	

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## VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference Sample Specific Element Reference

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All circled elements are applicable to each sample.

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Sample ID	Matrix	Target Analyte List (TAL)
1-4	50,-	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
	,	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
1-4	50:1	Nb, Pd, P, Pt, <u>Sn, Sr,</u> Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
	]	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
	H	Analysis Method
ICP		Li, 8,
ICP-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
ICP-MS		Nb, Pd, P. Pt, Sn, Sr, Ti, W, U, Zr, 7
GFAA		Al Sh As Ba Be Cd Ca Cr Co Cu Fe Pb Mg Mn Hg Ni K Se Ag Na TL V Zn Mo B Si CN

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

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## VALIDATION FINDINGS WORKSHEET PB/ICB/CCB QUALIFIED SAMPLES



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11       0.23       11       0.23       11       0.21       11       11         W       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14       14	Sb			1.3							
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	μ			1.1	0.22						
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	Hg (ug/Kg)			0.1			22.0 / 52.4				

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LDC #: {	SDG #:

## Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

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

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". <u>N N/A</u> 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. A'N N'A

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

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

Qualifications	4/+T +L	J-1 47/1	$\mathcal{T}$	J-/R/A	V/+T.+L	V/In/-L				/	J		No great ( LCG h )					A		V			
Associated Samples	A1/	-																		Ţ	-		
ろうろう RPD (Limits)												1	6'a(	1.8 + Her	20.3	29.4	2600	20.9	0.87	9.25			
MSD %Recovery	1345.4	39.4	60.9	29.7		44.6	5610	69.8	1,10	60.6	62.2											アオヘ	
MS %Recovery	140.1	53.2	7246	40.6	8 4 61	65.4	1.89															X 5v . T.	
Analyte	ري	56	Cr	4Nr	ф	~ ``		Lì,	ίN	M	Zn		۲۶	510	8 E	Le C	Ce.	1.1	Ŵ	J		Ma Mu	f
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## VALIDATION FINDINGS WORKSHEET **ICP Serial Dilution**

2nd Reviewer: Page: 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". $\overrightarrow{Y}$  N N/AIf analyte concentrations were > 50X the MDL (ICP), or >100X the MDL (ICP/MS), was a serial dilution analyzed? $\overrightarrow{Y}$  N/AWere ICP serial dilution percent differences (%D)  $\leq 10\%$ ?Y N/AIs there evidence of negative interference? If yes, professional judgement will be used to qualify the data.

**JEVEL IV ONLY:** 

Qualifications	J/4		and the second																						
	F																								
Associated Samples	41																								
%D (l imits)	(o,4																								
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Diluted Sample ID	758-47-08-10																								Ne, 4 2 100X
# Date	1																								comments:
	# Date Diluted Sample ID Matrix Analyte %D (1 inits) Associated Samples Oualifications	# Date Diluted Sample ID Matrix Analyte %D (Limits) Associated Samples Qualifications 1 T5B-G了-0名イロ Soil Fe (ロ・チ Ani J1)	#     Date     Diluted Sample ID     Matrix     Analyte     */D (1 inits)     Associated Samples     Oualifications       1 $75B - (77 - 0540^{1})$ $5_{3}$ ? $7e$ $1e_{1}$ $71$ $31$ $7e$ $1e_{1}$ $71$	#     Date     Diluted Sample ID     Matrix     Analyte     *An (1 inits)     Associated Samples     Qualifications       1 $75B - (r_3 - \delta 5 + 0)$ 50;1 $Fe$ $(e, \psi)$ $A1$ $2J + A$	#     Date     Diluted Sample ID     Matrix     Analyte     *AD (1 inits)     Associated Samples     Oualifications       1     75B-GJ-05A0     Soil     Fe     10.4     A1 $3Jf/A$	#     Date     Diluted Sample ID     Matrix     Analyte     *AD (1 inits)     Associated Samples     Oualifications       1     75B-(47-0640)     50:1     Fe     10.4     A1 $3D$ $3D$	#     Date     Diluted Sample ID     Matrix     Analyte     */n (1 inits)     Associated Samples     Qualifications       1 $T \leq B - (T_T - \delta f + 0)$ $S_0$ ; 1 $T_c$ $(e, \psi)$ $A_{11}$ $\Im L + A_1$ 1 $T \leq B - (T_T - \delta f + 0)$ $S_0$ ; 1 $T_c$ $(e, \psi)$ $A_{11}$ $\Im L + A_1$	#     Date     Diluted Sample ID     Matrix     Analyte     %D (1 inits)     Associated Samples     Qualifications       1 $T \leq B - (r_1 I o \delta T o)^2 < 5_3 $	#     Date     Diluted Sample ID     Matrix     Analyte     %D (1 imits)     Associated Samples     Qualifications       1 $75B - G_T - \delta E_T o^2 S_0$ ;     Fe $(e, \psi)$ $A_1$ $32T + A_1$ 1 $75B - G_T - \delta E_T o^2 S_0$ ;     Fe $(e, \psi)$ $A_1$ $32T + A_1$	#     Date     Diluted Sample ID     Matrix     Analyte     "Analyte     Sample ID       1 $T \leq B - (T_1 - \delta f - d_1)$ $F \leq 10$ , $\psi$ $A_1$ $\sqrt{J} \int A_1$ 1 $T \leq B - (T_1 - \delta f - d_1)$ $F \leq 10$ , $\psi$ $A_1$ $\sqrt{J} \int A_1$ 1 $T \leq B - (T_1 - \delta f - d_1)$ $T = 0$ $T = 0$	#     Date     Diluted Sample ID     Matrix     Analyte     "Analyte     "Analyte Samples     Outsilitrations       1     T5B-GT_0510     Soil     Fe $10, \psi$ A1 $\Im L_T^A$ 1     T5B-GT_0510     Soil     Fe $10, \psi$ A1 $\Im L_T^A$	#     Date     Diluted Sample ID     Matrix     Analytic Amoles     Samples       1 $75B-43-0\delta_10^2$ $S_3$ :1 $Fc$ $(e,\psi)$ $A_1$ $\sqrt{3}Lf/4$ 1 $75B-43-0\delta_10^2$ $S_3$ :1 $Fc$ $(e,\psi)$ $A_1$ $\sqrt{3}Lf/4$ 1 $75B-43-0\delta_10^2$ $S_3$ :1 $Fc$ $(e,\psi)$ $A_1$ $\sqrt{3}Lf/4$	#     Date     Dimed Sample ID     Matrix     Analyte     SAD (Limits)     Associated Samples       1 $TSB-GTo^2 STo^2 So^2 To^2 So^2 To^2 So^2 To^2 So^2 To^2 To^2 To^2 To^2 To^2 To^2 To^2 T$	#     Date     Diluted Sample ID     Matrix     Analyte     "Diluted Sample ID       1 $7 \leq P - (\tau_T - \delta_T - \delta_T)^2$ $5 \circ i$ $\mathcal{F} \in$ $(\circ, \psi$ $\mathcal{A}_1$ $\mathcal{N}_1 \mathcal{N}_1 \mathcal{A}_1$ 1 $7 \leq P - (\tau_T - \delta_T - \delta_T)^2$ $5 \circ i$ $\mathcal{F} \in$ $(\circ, \psi$ $\mathcal{A}_1$ $\mathcal{N}_1 \mathcal{N}_1 \mathcal{A}_1$ 1 $7 \leq P - (\tau_T - \delta_T - \delta_T)^2$ $5 \circ i$ $\mathcal{F} \in$ $(\circ, \psi$ $\mathcal{A}_1$ $\mathcal{N}_1 \mathcal{N}_1 \mathcal{A}_2$ 1 $7 \leq P - (\tau_T - \delta_T)^2$ $5 \circ i$ $\mathcal{F} \in$ $(\circ, \psi$ $\mathcal{A}_1$ $\mathcal{N}_1 \mathcal{N}_2 \mathcal{A}_2$	#     Tate     Diluted Sample ID     Matrix     Analytic with inits     Associated Samples       1 $759$ - $G_{1}T_{-}0\xi_{1}d^{2}$ $\zeta_{0}$ , $U$ $H_{1}$ $\mathcal{M}_{1}$ 1 $759$ - $G_{1}T_{-}0\xi_{1}d^{2}$ $\zeta_{0}$ , $U$ $H_{1}$ $\mathcal{M}_{1}$ 1 $759$ - $G_{1}T_{-}0\xi_{1}d^{2}$ $\zeta_{0}$ , $U$ $H_{1}$ $\mathcal{M}_{1}$ 1 $10$ , $U$ $10$ , $U$ $M_{1}$ $\mathcal{M}_{1}$	#     Inter Analytic     Marity     Analytic     And timits;       1 $7 \le B - \zeta_1 7 - \delta_5 1 \delta_5$ $5_3 \cdot 1$ $F \in (e, \psi)$ $A_1 1$ $\Delta_1 1 + \Delta_2$ 1 $7 \le B - \zeta_1 2 - \delta_5 1 \delta_5$ $5_3 \cdot 1$ $F \in (e, \psi)$ $A_1 1$ $\Delta_1 1 + \Delta_2$ 1 $7 \le B - \zeta_1 2 - \delta_5 1 \delta_5$ $5_3 \cdot 1$ $F \in (e, \psi)$ $A_1 1$ $\Delta_1 1 + \Delta_2$ 1 $P = 0$ 1 $P = 0$ $P = 0$ $P = 0$ $P = 0$ 1 $P = 0$ $P = 0$ $P = 0$ $P = 0$ 1 $P = 0$ $P = 0$ $P = 0$ $P = 0$ 1 $P = 0$ $P = 0$ $P = 0$ $P = 0$ 1 $P = 0$ $P = 0$ $P = 0$ $P = 0$ 1 $P = 0$ $P = 0$ $P = 0$ $P = 0$ 1 $P = 0$ $P = 0$ $P = 0$ $P = 0$ 1 $P = 0$ $P = 0$ $P = 0$ $P = 0$ 1 $P = 0$ $P = 0$ $P = 0$ $P = 0$ 1 $P = 0$ $P = 0$ $P = 0$ $P = 0$ 1 $P = 0$ $P = 0$ $P = 0$ 1 $P = 0$	and the line of sample in the line of the line of sample in the line of the line line of the lin	#     Date     Titled Sample ID     Matrix     Analytic Sample     Mail Hinting       1 $T_2B-G_1^2-\delta_5^2 1^\circ$ $S_3$ /1 $F_2$ $(e, \psi)$ $A_1$ $\Im_1 \mathcal{M}_2$ 1 $T_2B-G_1^2-\delta_5^2 1^\circ$ $S_3$ /1 $F_2$ $(e, \psi)$ $A_1$ $\Im_1 \mathcal{M}_2$ 1 $T_2B-G_1^2-\delta_5^2 1^\circ$ $S_3$ /1 $F_2$ $(e, \psi)$ $A_1$ $\Im_1 \mathcal{M}_2$ 1 $T_2B-G_1^2-\delta_5^2 1^\circ$ $S_3$ /1 $F_2$ $(e, \psi)$ $A_1$ $\Im_1 \mathcal{M}_2$ 1 $T_2B-G_1^2-\delta_5^2 1^\circ$ $S_3$ /1 $F_2$ $(e, \psi)$ $A_1$ $\Im_1 \mathcal{M}_2$ 1 $T_2B-G_1^2-\delta_5^2 1^\circ$ $S_3$ /1 $F_2$ $(e, \psi)$ $A_1$ $\Im_1 \mathcal{M}_2$ 1 $T_3$ $T_3$ $T_3$ $T_3$ $T_3$ $T_3$ 1 $T_3$ $T_3$ $T_3$ $T_3$ $T_3$ 1 $T_3$ $T_3$	a     Date     Date     Martic     Analytic     Analytic     Analytic       1 $75B-4c_T-0\xi-10^{1}$ $5_3$ :1 $F_c$ $(o, \psi)$ $A_1$ $31F_A$ 1 $75B-4c_T-0\xi-10^{1}$ $5_3$ :1 $F_c$ $(o, \psi)$ $A_1$ $31F_A$ 1 $15B-4c_T-0\xi-10^{1}$ $5_3$ :1 $F_c$ $(o, \psi)$ $A_1$ $31F_A$ 1 $10-10^{1}$ $10-10^{1}$ $10-10^{1}$ $10-10^{1}$ $10-10^{1}$ 1 $10-10^{1}$ $10-10^{1}$ $10-10^{1}$ $10-10^{1}$ 1 $10-10^{1}$ $10-10^{1}$ $10-10^{1}$ 1 $10-10^{1}$ $10-10^{1}$ $10-10^{1}$ 1 $10-10^{1}$ $10-10^{1}$ $10-10^{1}$ 1 $10-10^{1}$ $10-10^{1}$ $10-10^{1}$ 1 $10-10^{1}$ $10-10^{1}$ $10-10^{1}$ 1 $10-10^{1}$ $10-10^{1}$ $10-10^{1}$ 1 $10-10^{1}$ $10-10^{1}$ $10-10^{1}$ 1 $10-10^{1}$ $10-10^{1}$ $10-10^{1}$ 1 $10-10^{1}$ $10-10^{1}$ $10-10^{1}$ 1 $10-10^{1}$ $10-10^{1}$ $10-10^{1}$ 1 $10-10^{1}$ $10$	and the interval stands     Matrix     Matrix     Matrix     Matrix     Matrix       1 $75B-G_{1-0}\delta_{10}$ $5_{0}1$ $F_{0}$ $4_{1}$ $3_{1}M_{1-1}$ 1 $75B-G_{1-0}\delta_{10}$ $5_{0}1$ $F_{0}$ $4_{1}$ $3_{1}M_{1-1}$ 1 $15B-G_{1-0}\delta_{10}$ $5_{0}1$ $F_{0}$ $4_{1}$ $3_{1}M_{1-1}$ 1 $10-M_{1-1}$ $10-M_{1-1}$ $3_{1}M_{1-1}$ $3_{1}M_{1-1}$ 1 $10-M_{1-1}$ $10-M_{1-1}$ $10-M_{1-1}$ 1 $10-M_{1-1}$ $10-M_{1-1}$ $10-M_$	$\mu$ Date     Introdet Sample     Matrix     Accordinat Sample     Matrix     Accordinat Sample       1     TSB-GT_o\$T_o\$T_o\$T_     S_31     Fe     (a, $\psi$ $A_1$ $\Delta D f A_1$ 1     TSB-GT_o\$T_o\$T_o\$T_o\$T_o\$T_o\$T_o\$T_o\$T_o\$T_o\$	I     Diamet Smatial     Matrix     Matrix     Anolde     Contract Samples       I     T5B-GGT-0E10     S21     Fe     (e.f.)     Au     Difference       I     T5B-GGT-0E10     S21     Fe     (e.f.)     Au     Difference       I     I     S21     Fe     (e.f.)     Au     Difference       I     I     I     I     I     Difference       I     I     I     I     I     I       I     I     I     I     I     I       I     I     I     I     I     I       I     I     I     I     I     I       I     I     I     I     I     I       I     I     I     I     I     I       I     I     I     I     I     I       I     I     I     I     I     I       I     I     I     I     I     I       I     I     I     I     I     I       I     I     I     I     I     I       I     I     I     I     I     I       I <tdi< td=""> <tdi< td="">     I     <tdi< td=""> <td< td=""><td><math>\mu</math>     Date     Date     Matrix     Matrix     Matrix       1     T5B-GT-0510     Soll     Fc     (e.f.     All       1     T5B-GT-0510     Soll     Fc     (e.f.     All       1     T     T     T     T     T       1     T     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T       1     <tdt< td=""><td>Allow         Diluted Sample II         Matrix         Andrix         Sectional Sample II         Dunited Sample II           1         T_2_BG_1^2</td><td><math>\mu</math>     Dirical Samole ID     Marrix     Anotical Samole ID     Marrix     Anotical Samole ID     Marrix     Conditications       1     T <math>\mathcal{L}</math>       1     T <math>\mathcal{L}</math>       1     T <math>\mathcal{L}</math>       1     T <math>\mathcal{L}</math>       1     T <math>\mathcal{L}</math>       1     T <math>\mathcal{L}</math> <math>L</math></td></tdt<></td></td<></tdi<></tdi<></tdi<>	$\mu$ Date     Date     Matrix     Matrix     Matrix       1     T5B-GT-0510     Soll     Fc     (e.f.     All       1     T5B-GT-0510     Soll     Fc     (e.f.     All       1     T     T     T     T     T       1     T     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T     T       1     T     T     T       1 <tdt< td=""><td>Allow         Diluted Sample II         Matrix         Andrix         Sectional Sample II         Dunited Sample II           1         T_2_BG_1^2</td><td><math>\mu</math>     Dirical Samole ID     Marrix     Anotical Samole ID     Marrix     Anotical Samole ID     Marrix     Conditications       1     T <math>\mathcal{L}</math>       1     T <math>\mathcal{L}</math>       1     T <math>\mathcal{L}</math>       1     T <math>\mathcal{L}</math>       1     T <math>\mathcal{L}</math>       1     T <math>\mathcal{L}</math> <math>L</math></td></tdt<>	Allow         Diluted Sample II         Matrix         Andrix         Sectional Sample II         Dunited Sample II           1         T_2_BG_1^2	$\mu$ Dirical Samole ID     Marrix     Anotical Samole ID     Marrix     Anotical Samole ID     Marrix     Conditications       1     T $\mathcal{L}$ 1     T $\mathcal{L}$ 1     T $\mathcal{L}$ 1     T $\mathcal{L}$ 1     T $\mathcal{L}$ 1     T $\mathcal{L}$ $L$

LDC #: (9186AV

VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

Page: <u>fof</u> Reviewer: <u>W1</u>1 2nd Reviewer: <u>C</u>

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:

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	
%R = <u>Found</u> × 100 True	

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
Ter	ICP (Initial calibration)	S	42700	4000	8,301	3.90)	7
	GFAA (Initial calibration)						
ICV	CVAA (Initial calibration)	Hq	2,33	as.'z	43.2	93.2	7
cu	ICP (Continuing calibration)	ر ا_ا	4754	<i>دە</i> مر	95-1	95-1	7
	GFAA (Continuing calibration)	;					
cw	CVAA (Continuing calibration)	1HE	4.98	5-0	9.13	946	7
ANI	ICP/MS (Initial calibration)	° Y	1.800)	( 00 )	1.2.8	8-001	
cev	ICP/MS (Continuing calibation)	A5	908.3	لحدم	90,8	90.5	7
			and the second	and a second design of the	ومحتمد والواحدة والمستعد والمستعد والمستعد والمستعم والمستعم والمستعم والمستعم والمستعم والمستعم والمستعد والم		

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.

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		SDG #:

## VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

Page: <u>Lof</u> Reviewer: <u>her</u> 2nd Reviewer: <u>C</u>

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:

Where, 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). True = Concentration of each analyte in the source. %R = Found\_x 100 True

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

RPD = <u>[S-D]</u> × 100 Where, S = Original sam (S+D)/2 D = Duplicate sar

S = Original sample concentration D = Duplicate sample concentration An ICP serial dilution percent difference (%D) was recalculated using the following formula:

%D = <u>||-SDR|</u> × 100

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

					Recalculated	Reported	
Sample ID	Type of Analysis	Element	Found / S / I (units)	True / D / SDR (units)	%R / RPD / %D	%R / RPD / %D	Acceptable (Y/N)
SUSAL	ICP interference check	the state	(0, 1, 0)	as)	1 - 4	104	7
1cs	Laboratory control sample	6J	9 <i>P S</i>	[x]	46.2	76.2	
1-8~[1-8-1-	/ Matrix spike	20	(ssr-sr) (b 4, 2	6 • ~ ( 0 )	91.3	99.3	
	Duplicate	3	70.05	1/ w	(~)	1,0	
	ICP serial dilution	ð	7(19)	(6568	2 . 8	2.8	X

Comments: Refer to appropriate worksheet for list of gualifications 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

Page:	<u>(or</u>
Reviewer:	hu
2nd reviewer:	

METHOD: Trace Metais (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/AHave results been reported and calculated correctly?(Y) N N/AAre results within the calibrated range of the instruments and within the linear range of the ICP?(Y) N N/AAre all detection limits below the CRDL?

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

Concentration = <u>(RD)(FV)(Dil)</u> (In. Vol.)(%S) RD = Raw data concentration FV = Final volume (ml) In. Vol. = Initial volume (ml) or weight (G)

Recalculation: 

Dil = Dilution factor

%S = Decimal percent solids

Sample ID	Analyte	Reported Concentration ( Mg/Rg )	Calculated Concentration ( W.G / KL )	Acceptable (Y/N)
7	S	J3300	5)200	Y
	AR	(0100	(0100	/I
	As	27-6	27-6	
	Ba	64.6	64-6	
	Be	0.64	0.6 Y	
	Ca	75800	15800	
	C_	22.2	22,2	
	Co	5.7	5.6	
	Cu	13-5	13-5	
	Fe	13200	13200	
	pь	1.1	7.)	
	Mg	18200	18200	
	My O	<u>ino</u>	170	
	NI	14.7	14.6	
	p J	<u> </u> _/	1.1	
	<u> </u>	528	528	
	K	210	2/10	
	51	549	549	
	Ag	o(14)	0,14	
	Na	944	943	
	4x	505	505	
	Tì	518	557	

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### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	Tor 2
Reviewer:	My
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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". $\widehat{(N)}$  N N/AHave results been reported and calculated correctly? $\widehat{(N)}$  N N/AAre results within the calibrated range of the instruments and within the linear range of the ICP? $\widehat{(N)}$  N N/AAre all detection limits below the CRDL?

2010/2016/2016

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

Recalculation:

 In. Vol.)(%S)

 RD
 =

 FV
 =

 Final volume (ml)

 In. Vol.
 =

 Initial volume (ml) or weight (G)

 Dil
 =

 Dilution factor

 %S
 =

 Decimal percent solids

Concentration =

(RD)(FV)(Dil)

V= 45.91 %/2×0.1e×5 = 57.67 mg/mg

Sample ID	Analyte	Reported Concentration ( Mg/Kg )	Calculated Concentration ( W. 6, / 12; )	Acceptable (Y/N)
2	И	<u>ー りし</u> ふり	3.7	Ý
	V	51-4	57.7	1
	Zh	91.5	91,2	
	27	31.7	31.6	
		·		
				·
		·····		

## LDC Report# 19188A6

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	BRC Tronox Parcel G
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Collection Date: June 11, 2008

LDC Report Date: August 7, 2008

Matrix: Soil

Parameters: Wet Chemistry

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

## Sample Identification

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

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 4 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 300.0 for Bromide, Bromine, Chlorate, Chloride, Chorine, Fluoride, Nitrate as Nitrogen, Nitrite as Nitrogen, Orthophosphate as Phosphorus, and Sulfate and EPA SW 846 Method 9071B for Oil & Grease.

The review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- 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
ICB/CCB	Orthophosphate as P	0.102 mg/L	All samples in SDG F8F120180

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

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

No field blanks were identified in this SDG.

## IV. Matrix Spike/Matrix Spike Duplicates

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

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

## V. Duplicates

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable. Results were within QC limits.

## VI. Laboratory Control Samples

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

## **VII. Sample Result Verification**

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

## VIII. Overall Assessment of Data

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

## **IX. Field Duplicates**

No field duplicates were identified in this SDG.

## BRC Tronox Parcel G Wet Chemistry - Data Qualification Summary - SDG F8F120180

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

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

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

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

No Sample Data Qualified in this SDG

ALIDATION C	COMPLETENESS	WORKSHEET
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Level III/IV

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LDC #: <u>19188A6</u> SDG #: <u>F8F120180</u> Laboratory: <u>Test America</u> V

## METHOD: (Analyte) Bromide, Bromine, Chlorate, Chloride, Chorine, Fluoride, Nitrate-N, Nitrite-N, Orthophosphate-P, Sulfate (EPA Method 300.0), O & G (EPA SW846 Method 9071B)

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

	Validation Area		Comments
	Technical holding times	A	Sampling dates: 6/11/-8
lla.	Initial calibration	A	
IIb.	Calibration verification	A	
	Blanks	GW	
١٧	Matrix Spike/Matrix Spike Duplicates	SW	> My/MSP / Dup
v	Duplicates	A	
VI.	Laboratory control samples	A	Les
VII.	Sample result verification	Á	Not reviewed for Level III validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N	
L x	Field blanks	N	

Note:

A = Acceptable N = Not provided/applicable SW = See worksheet ND = No compounds detected R = Rinsate

FB = Field blank

D = Duplicate TB = Trip blank EB = Equipment blank

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

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

Notes:\_

1918816 LDC #: SDG #:

### VALIDATION FINDINGS CHECKLIST

## Method:Inorganics (EPA Method Su Wir

Validation Area	Yes	No	NA	Findings/Comments
C. Technical holding times		24	117	(A
All technical holding times were met.	/			
Coolor tomporaturo critoria was met.				
Lize Star 2001		和过		
Were all instruments calibrated daily, each set-up time?				
Were the proper number of standards used?				
Were all initial calibration correlation coefficients > 0.995?	1			
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)	V	a wide d	K	
Was a method blank associated with every sample in this SDG?	$\angle$			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	$\checkmark$			
NAMALIK SIKON ADUSAKA UDAK RESAMITADIK DESETISATI KATA TALAH				
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) $\leq$ 20% for waters and $\leq$ 35% for soil samples? A control limit of $\leq$ CRDL( $\leq$ 2X CRDL for soil) was used for samples that were $\leq$ 5X the CRDL, including when only one of the duplicate sample values were $\leq$ 5X the CRDL.	7			
Was an LCS anaylzed for this SDG?	2			
Was an LCS analyzed per extraction batch?	$\checkmark$			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% (85-115% for Method 300.0) QC limits?				
VI. Regional Granty Assistance and Quality Control Last Control Last Control				
Were performance evaluation (PE) samples performed?			-4	
Were the performance evaluation (PE) samples within the acceptance limits?			$\Delta$	

LDC # 19188A6 SDG #: 10 com

### VALIDATION FINDINGS CHECKLIST

Page: Vof V Reviewer: MM 2nd Reviewer: V

Validation Area	Yes	No	NA	Findings/Comments
VII. Sampe Result Verification		a itai	a.E.M	
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	~			
Were detection limits < RL?	/			
	44			
Overall assessment of data was found to be acceptable.	/			
Field duplicate pairs were identified in this SDG.		~		
Target analytes were detected in the field duplicates.			1	
Field blanks were identified in this SDG.	-	1		
Target analytes were detected in the field blanks.				

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## VALIDATION FINDINGS WORKSHEET Sample Specific Analysis Reference

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All circled methods are applicable to each sample.

Sample ID	Matrix	Parameter
1-4	507	Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>3</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> Ø+G/TPH
		Br Bromine CI 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 CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI 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 CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI 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 CI 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 CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI 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 CI 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 CI 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 CI 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 CI 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 CI 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 CI 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 CI 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 CI 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 CI 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 CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH

Comments:

VALIDATION FINDINGS WORKSHEET Blanks

ō Page: Reviewer:\_\_\_\_\_\_\_2nd Reviewer:\_\_\_\_\_\_

METHOD: Inorganics, Method

LDC #: 19188A6

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SDG #:

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

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					-								
	ų												
	ple Identificatic							•					
<u>A</u> )	Sam												
les:													
siated Samp													
Asso		2	1-5/6.3										
	Blank	Action Limit										-	
5 T	Maximum	ICB/CCB	10										
1/ Bru :	Blank ID												
Conc. units	Analyte		9-404-0										

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".

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## Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

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) くうち METHOD: Inorganics, EPA Method\_

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. Were all duplicate sample relative percent differences (RPD)  $\leq 20\%$  for water samples and  $\leq 35\%$  for soil samples? Phase see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". <u>V N N/A</u> Was a matrix spike analyzed for each matrix in this SDG? <u>Y N N/A</u> Were matrix spike percent recoveries (%R) within the control limits of 75-125? If the sample conc

Y/N N/A

EVEL IV ONLY:

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

			WS	DSM			
di dsw/sw #	Matrix	Analyte	%Recovery	%Recovery	RPD (Limits)	Associated Samples	Qualifications
-bo-17-451 1	0 501	019	63	<u></u> {9		A.	J-/ht/A
					-		
		•					
Comments:							

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LDC #: 19188 MG	) I I I	tial and Con	/alidatin Fin Itinuing Cali	ldings Worksl bration Calcu	heet lation Verifica	tion	Page: of Reviewer:t 2nd Reviewer:
Method: Inorganics, M	ethod	el cruer					
The correlation coefficient (	r) for the calibra	tion of $\frac{15Y}{}$	was recalcu	lated.Calibration	date: $b/l^{\mathcal{F}}$	/•لا	
An initial or continuing calit	oration verificati	on percent reco	very (%R) was r	recalculated for ea	ich type of analysis	s using the followi	ng formula:
%R ≡ <u>Found X 100</u>		Where,	Found = concen	ntration of each an	alyte <u>measured</u> in	the analysis of the	ICV or CCV solution
True			True = concen	ıtration of each an	alyte in the ICV or	CCV source	
					Recalculated	Reported	Acceptable
Type of analysis	Analyte	Standard	Conc. (ug/L)	Area	r or r²	r or r²	(V/N)
Initial calibration		s1	250	0.02			
	Б	s2	500	0.039	0.99997	0.99997	7
		s3	1000	0.076			
		S4	2500	0.196			
		s5	5000	0.396			
$\mathcal{LCV}$ Calibration verification	lloz	4000	7-95		98	MR	7
દ અ Calibration verification	μ	000	949.5		9495	9495	-
CCV Calibration verification	J-hud-o	Boon	1856 *		98.20	98,20	-)

Comments: Refer to Calibration Verification findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.
19188 A6	Lee corre
	SDG #:

VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

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

METHOD: Inorganics, Method \_\_\_\_\_\_

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

%R = Found x 100	Where,	Found ≂	concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation,
True			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:

Original sample concentration Duplicate sample concentration	-
۲ ۲ ۵	
RPD = <u>iS-Di</u> × 100 Where, (S+D)/2	

					Receiculated	Reported	
Sample (D	Type of Analysis	Element	Found / S (unite)	True / D (unlta)	<b>%</b> R / RPD	%R / RPD	Acceptable (Y/N)
	Laboratory control sample						
<b>L</b> CS		W2-N	(. <i>F</i> )	~ 9~	36	36	<u>}</u>
	Matrix spike sample		(SSR-SR)				-
T58-CJ-09	- 0	otd	880	0 / 2 / 0	<i>e</i> ~	63	
$\uparrow$	Duplicate sample	705	ممحا	0841	<i>c</i> _	~	>
					$\sim$	-	

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.

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LDC #: (9188776 SDG #:

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

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reported with a positive detect were

METHOD: Inorganics, Method \_\_\_\_

Sie inen

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". Have results been reported and calculated correctly? (Y) N N/A Are results within the calibrated range of the instruments? YN NA Are all detection limits below the CRQL? EN N/A

2

Compound (analyte) results for recalculated and verified using the following equation:

Concentration =

Recalculation:

eloz= Aven x 40ml 0.079 x 48x/05/12

			Reported Concentration	Calculated Concentration	Acceptable
#	Sample ID	Analyte	(mg/m)	(mg/y)	(Y/N)
1	2	0-p04-p	1.5	1-5	<u> </u>
		Chlority	3,7	3.7	
		cl	244	rep	
		Cl 2	488	488	
		F	0.58	0.59	
		LOZ-N	5.3	5.5	
		504	11600	11600	X
			<u>.</u>		
┣				+	
<b> </b>					
			L	<u> </u>	<u></u>

Note:

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

Project/Site Name: BRC Tr	onox Parcel G
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Collection Date: June 11, 2008

LDC Report Date: August 6, 2008

Matrix:

Parameters: Gasoline Range Organics

Soil

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

### Sample Identification

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

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 4 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8015B for Gasoline Range Organics.

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

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. Calibration

### a. Initial Calibration

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

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

### b. Calibration Verification

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

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

### III. Blanks

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

No field blanks were identified in this SDG.

### **IV. Accuracy and Precision Data**

### a. Surrogate Recovery

Surrogates were added to all samples and blanks as required by the method. All surrogate recoveries (%R) were within QC limits.

### b. Matrix Spike/Matrix Spike Duplicates

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

### c. Laboratory Control Samples

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

### V. Target Compound Identification

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

### **VI. Compound Quantitation and CRQLs**

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

### VII. System Performance

The system performance was acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

### **VIII. Overall Assessment of Data**

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

### IX. Field Duplicates

No field duplicates were identified in this SDG.

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

No Sample Data Qualified in this SDG

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

No Sample Data Qualified in this SDG

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

No Sample Data Qualified in this SDG

VALIDATION	COMPL	<b>ETENESS</b>	WORKSHEET
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LDC #: <u>19188A7</u> SDG #: <u>F8F120180</u> Laboratory: <u>Test America</u>

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### Level III/IV

1.1
Date: 8/4/18
Page:of
Reviewer:
Reviewer:

2nd

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/11/08
lla.	Initial calibration	A	/ /
IIb.	Calibration verification/ICV	$\mathbf{A}$	$ e  \leq  5/2$
- 111.	Blanks	A	
IVa.	Surrogate recovery	$\mathbf{A}$	
IVb.	Matrix spike/Matrix spike duplicates	NA	Fiert Diffed - TSB4 J-08-10'
IVc.	Laboratory control samples	$\mathbf{A}$	LCSD
V.	Target compound identification	A	Not reviewed for Level III validation.
VI.	Compound Quantitation and CRQLs	$\mathbf{A}$	Not reviewed for Level III validation.
VII.	System Performance	$\mathbf{A}$	Not reviewed for Level III validation.
VIII.	Overall assessment of data	Ah	
IX.	Field duplicates	N	
Х.	Field blanks		

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

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

1	TSB-GJ-09-10'	11	816526914B	21	31	
2	TSB-GJ-09-20'**	12		22	32	
3	TSB-GJ-09-30'	/13		23	33	
4	TSB-GJ-09-40'	14		24	34	
5		15		25	35	
6		16		26	36	
7		17		27	37	
8		18		28	38	
9		19		29	39	
10		20		30	40	

Notes:

Note:

LDC #: <u>/9188 Å7</u> SDG #: <u>200 CP WY</u>

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### VALIDATION FINDINGS CHECKLIST

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

		<u>مارد</u>	<b>NIA</b>	Eindinge/Commonte
Validation Area	Yes	<u>NO</u>		Findings/comments
f. Technical holding times			E Second	
All technical holding times were met.	$ \rightarrow $			
Cooler temperature criteria was met.	23393)			
(L'Initial calibration				Γ
Did the laboratory perform a 5 point calibration prior to sample analysis?	$\leq$			
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?	$\leq$			
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?		/	$\left  \right $	
Did the initial calibration meet the curve fit acceptance criteria?				
Were the RT windows properly established?				
tv Continuing calibration				
What type of continuing calibration calculation was performed?%D or %R				
Was a continuing calibration analyzed daily?			<u> </u>	
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.		/	1	
VI. Surrogate 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. Matrix spike/Matrix spike duplicates	1997. T		5 (2) 1	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?			1	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?			//	
VIII*Laboratory control samples	Ser			
Was an LCS analyzed for this SDG?		X		
Was an LCS analyzed per extraction batch?				

LDC #: 19188-AT SDG #: Ber COWN

### 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?				
IX: Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?		_		
Were the performance evaluation (PE) samples within the acceptance limits?	Sec. To start	1		
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 System performance				
System performance was found to be acceptable.				
XIII. Overall assessment of data			ps age	
Overall assessment of data was found to be acceptable.	/			
XIV. Field dublicates				
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?			/	

SDG #: See COM LDC #: /9/88

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

/ of đ 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	Recalculated
#	Standard ID	Calibration Date	Compound	CF (Ø./std)	CF ( Ø, / std)	Average CF (initial)	Average CF (initial)	%RSD	%RSD
-	. 101		GR0	00/c5881	183520	17,8-732	1718=732	3915	3915
	144	& olders							
2									
ო									
4									
				-					

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results.

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

SDG #:2er COWN LDC #:/9/8847

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

ď J Page: 2nd Reviewer: Reviewer:

HPLC METHOD: GC V 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

#     Standard ID     Calibration     Calibration     Calibration     Conc.     CFConc.       1 $\angle CAE \overline{AMB}$ $\angle I/3 \gamma B$ $\overrightarrow{AVE}$ $\angle I/2$ $\bigcirc GCV Conc.$ 2 $\angle ADE \overline{AMB}$ $\angle I/3 \gamma B$ $\overrightarrow{AVE}$ $\angle I/2$ $\bigcirc GCV Conc.$ 3 $\angle I/3 \gamma B$ $\overleftarrow{AVE}$ $\angle I/2$ $\bigcirc GV \overline{ABB}$ 4 $\boxed{AVE}$ $\overleftarrow{AVE}$ $\boxed{I/2}$ $\boxed{B} \overline{ABB}$ 4 $\boxed{AVE}$ $\overbrace{I/2}$ $\boxed{B} \overline{ABB}$ $\boxed{B} \overline{ABB}$					Reported	Recalculated	Reported	Recalculated
$\frac{1}{2} \frac{202326}{6} \frac{1}{6} \frac{3}{8} \frac{3}{5} \frac{5}{6} \frac{2}{10} \frac{3}{10} $	#	Calibration Standard ID Date	Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc. CCV	ΨD	Ω%
$2 \frac{1}{2} \sqrt{2} \sqrt{2} \sqrt{2} \sqrt{2} \sqrt{2} \sqrt{2} \sqrt{2} $	-	10423983 1.1.	GR0	1,0	0.9982	0.9982	۲.	ح. 0
$\frac{2}{2} \frac{\partial k_{2} h_{0} k_{0}}{\partial k_{0}} = \sqrt{14} \sqrt{6} \frac{7}{6} \frac{1}{2} \frac{1}{2}$		13/13/05						
2 W2 406 6/14/18 FRO 1.0 2.9824 3 19 19 19 19 19 19 19 19 19 19 19 19 19								
	2	2042 406 2 / 11/ 1.8	GRO	0.1	0. 9824	0.98 zy	d.	de ;
		a./hi/a						
	6							
	4							
	1							

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

ď Reviewer:\_\_\_\_\_2nd reviewer:\_\_\_\_ Page:

# 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

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S	L	

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
ZF7	NS	0.04	0.03383	85	28	Ú

### Sample ID:

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

### Sample ID:

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

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Ø	1
#	#
ГРС	SDG



METHOD: GC HPLC

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

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

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

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

SC = Concentration

LCSD = Laboratory control sample duplicate percent recovery

0	
B16526	
samples:	
CS/LCSD	

	S.	pike	Spiked	Sample	Ľ	S	ГО	sp	I/SO1	.csp
Compound	¥ )	dded /	Conce (M.	entration SIST	Percent I	Recovery	Percent F	Recovery	R	0
	rcs		LCS /	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)	1.0	0.1	1.0	0.944	100	201	44	gif	4.0	S.B
Diesel (8015)										
Benzene (8021B)										
Methane (RSK-175)										
2,4-D (8151)										
Dinoseb (8151)										
Naphthalene (8310)										
Anthracene (8310)										
HMX (8330)										
2,4,6-Trinitrotoluene (8330)										
						-				

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

:#:1912247	3#: <i>500 CO</i> WN
LDC #:	SDG #:

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page: /of / Reviewer: 2nd Reviewer:

METHOD: 🗸 GC \_\_ HPLC

N/A     Were all recalculated results for c       entration=     (A)(Fv)(Df)       (RF)(Vs or Ws)(%S/100)	letected target compounds agree within 10% of the reported results? Example:	
ea or height of the compound to be measured	Sample ID. 🕘 Compound Name 🛛 📈 Ď	
inal Volume of extract ilution Factor		
verage response ractor of the compound the initial calibration itial volume of the comple		
itial volume of the sample tital weight of the sample ercent Solid		

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

SAMPCALew.wpd

Comments: \_

### LDC Report# 19188A8

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

Project/Site Name: BRC 1	Fronox Parcel G
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Collection Date: June 11, 2008

LDC Report Date: August 6, 2008

Matrix:

Parameters: Diesel Range Organics

Soil

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

### Sample Identification

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

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 4 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8015B for Diesel Range Organics.

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

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. Calibration

### a. Initial Calibration

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

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

### b. Calibration Verification

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

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

### III. Blanks

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

No field blanks were identified in this SDG.

### **IV. Accuracy and Precision Data**

### a. Surrogate Recovery

Surrogates were added to all samples and blanks as required by the method. All surrogate recoveries (%R) were within QC limits.

### b. Matrix Spike/Matrix Spike Duplicates

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

### c. Laboratory Control Samples

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

### V. Target Compound Identification

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

### **VI. Compound Quantitation and CRQLs**

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

### VII. System Performance

The system performance was acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

### **VIII. Overall Assessment of Data**

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

### **IX. Field Duplicates**

No field duplicates were identified in this SDG.

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

No Sample Data Qualified in this SDG

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

No Sample Data Qualified in this SDG

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

No Sample Data Qualified in this SDG

### VALIDATION COMPLETENESS WORKSHEET

LDC #: <u>19188A8</u> SDG #: <u>F8F120180</u> Laboratory: <u>Test America</u>

### Level III/IV

1.1.5
Date: <u>8/4/08</u>
Page: //of /
Reviewer:
2nd Reviewer:

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

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

	Validation Area		Comments
Ι.	Technical holding times	A	Sampling dates: 6/11/08
lla.	Initial calibration	A	
llb.	Calibration verification/ICV	Å	a  = 1570
111.	Blanks	$\mathbf{A}$	
IVa.	Surrogate recovery	$ \mathbf{A} $	
IVb.	Matrix spike/Matrix spike duplicates	KA	aieut seited TSB 4 -08-10
IVc.	Laboratory control samples	A	LCS
V.	Target compound identification	A	Not reviewed for Level III validation.
VI.	Compound Quantitation and CRQLs	$\mathbf{A}$	Not reviewed for Level III validation.
VII.	System Performance	A	Not reviewed for Level III validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	Ń	
Х.	Field blanks		

 Note:
 A = Acceptable
 ND = No compounds detected
 D = Duplicate

 N = Not provided/applicable
 R = Rinsate
 TB = Trip blank

 SW = See worksheet
 FB = Field blank
 EB = Equipment blank

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

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

Notes:

LDC #: 19188/18 SDG #: <u>See Con</u>

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Method: GC HPLC				· · · · · · · · · · · · · · · · · · ·
Validation Area	Yes	No	NA	Findings/Comments
Trechnical holding times				
All technical holding times were met.	$\square$			
Cooler temperature criteria was met.			RANKERS	
II Tontial 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) $\leq 20\%$ ?				
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?		/		
Did the initial calibration meet the curve fit acceptance criteria?				
Were the RT windows properly established?		SPECIA		
IV: Continuing calibration	े <u>के लि</u> T	in de la comunicación I		
What type of continuing calibration calculation was performed?%D or%R				
Was a continuing calibration analyzed daily?				
Were all percent differences (%D) $\leq$ 15%.0 or percent recoveries 85-115%?	Ľ/			
Were all the retention times within the acceptance windows?	/		1263274	
V-Blanks		2 7		
Was a method blank associated with every sample in this SDG?	4	ļ		
Was a method blank analyzed for each matrix and concentration?	1/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
VI. Surrogate spikes		的 (1) 子		
Were all surrogate %R within the QC limits?	14		<b> </b>	
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?			4	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?		There are a set of the		
VII. Matrix spike/Matrix spike duplicates	<u></u>	СС Т	<u>а</u> Т	<u></u>
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	56			an a
Was an LCS analyzed for this SDG?		1	<u> </u>	
Was an LCS analyzed per extraction batch?				

LDC #: 1918878 SDG #: See CONIN

### 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?	/			
IX: Regional Quality Assidance and Quality Control	1.5			
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?		1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1		
X. Target compound identification				
Were the retention times of reported detects within the RT windows?		*#060000000		
XI. Compound quantitation/CRQLs	/			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XII. System performance	e (NASCESSION)		2.2005 	
System performance was found to be acceptable.				
XIII. Qverall assessment of data	<u>,</u>	ana National National		
Overall assessment of data was found to be acceptable.	/			
XIV Field duplicates				
Were field duplicate pairs identified in this SDG?		/		
Were target compounds idetected in the field duplicates?			/	
XV. Field blanks		9 		
Were field blanks identified in this SDG?		/		
Were target compounds detected in the field blanks?			/	



## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

بے ا Page: 2nd Reviewer: Reviewer:

HPLC METHOD: GC V 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
#	Standard ID	Calibration Date	Compound	CF (/ &Zstd)	CF ( /b&td)	Average CF (initial)	Average CF (initial)	%RSD	%RSD
-			DE C	15394	16394	16023	16023	3.456	3,456
	10the	Z/16/08							
		/							
2									
ო									
4									
				-					

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

INICLC.1SB

LDC #: 19108940 SDG #: See COM

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

ď 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

eported Recalculated	%D	0.3		15 °E (5'					<u> </u>	
	CF/Conc. CCV	, 996.53		557480/		· ·				
Reported	CF/Conc. CCV	465,966		695 7E "						
	Average CF(Ical)/ CCV Conc.	1000		0 a0 1						
	Compound	DRO		DEO						
	Calibration Date	21,-108	~ // /	6/17/08						
	Standard ID	Zether		604.537						
	#	-		7		Э		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.

CONCLC.1S

LDC #: 1912848 SDG #500 COMV

# VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

9 Page: / of / 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

٨ Sample ID:

	D1001	Recovery	Recovery	Difference	
		Reported	Recalculated		
25.0	1062.10	SS	20	Ś	
0.5°	10/2/2			ASS BSS BSS	ASS SS SS

### Sample ID:

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

### Sample ID:

							Г
Surrogate	Column/Detector	Spiked	surrogate Found	Percent Recovery	Percent Recovery	Percent Difference	
				Reported	Recalculated		1
							<u>17</u>
							1
							<u> </u>
							T

.DC #:/9/8848	SDG #: Sec COWN
ĕ	SD



METHOD: \_\_\_\_GC \_\_\_HPLC

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

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

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

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

SC = Concentration

LCSD = Laboratory control sample duplicate percent recovery

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n)
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N
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$\mathbb{N}$
q
ples:
am
S S
CSD s
CS/LCSD s

							y				T	i	 
LCSD	PD	Recalc.											
LCS/	æ	Reported											
so So	kecovery	Recalc.											
Ľ	Percent F	Reported											
S	Зесоvегу	Recalc.		e X									
ГС	Percent F	Reported		83									
Sample	ntration 5/13	rcsp		NA	*								
Spiked	Concel ( Me	LCS		68.9									
oike	dea 765	LCSD		NA									
S	Ad M	RCS		83.3									
	Compound		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)	

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

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification



Were all reported results recalcula	Were all recalculated results for de
Y N N/A	YN NA
	Y N N/A Were all reported results recalculate

ed and verified for all level IV samples? ected target compounds agree within 10% of the reported results?

Concentration= (A)(Fv)(Df)	Example:			
(KF)(VS OF VVS)(702/100)	Sample ID.	Compound Name	$\sim \varphi$	ł
A= Area or height of the compound to be measured FV= Final Volume of extract				
Di= bilution ractor RF= Average response factor of the compound	Concentration =			
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				
#	· · · · · ·			

Comments: \_\_\_\_

SAMPCALew.wpd

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

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

### Sample Identification

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

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 4 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8310 for Polynuclear Aromatic Hydrocarbons.

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

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. Calibration

### a. Initial Calibration

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

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

Retention time windows were evaluated and considered technically acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples on which a Level III review was performed.

### b. Calibration Verification

Calibration verification was performed at required frequencies.

The percent differences (%D) of calibration factors in continuing standard mixtures were within the 15.0% QC limits with the following exceptions:

Date	Detector	Compound	%D	Associated Samples	Flag	A or P
6/16/08	Not specified	Benzo(g,h,i)perylene	15.2	TSB-GJ-09-10' TSB-GJ-09-20'**	J+ (all detects)	A

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

Date	Detector	Compound	%D	Associated Samples	Flag	A or P
6/4/08	Not specified	Benzo(k)fluoranthene	16.6	All samples in SDG F8F120180	J+ (all detects)	A

Retention time windows were evaluated and considered technically acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples on which a Level III review was performed.

### III. Blanks

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

No field blanks were identified in this SDG.

### **IV. Accuracy and Precision Data**

### a. Surrogate Recovery

Surrogates were added to all samples and blanks as required by the method. All surrogate recoveries (%R) were within QC limits.

### b. Matrix Spike/Matrix Spike Duplicates

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

### c. Laboratory Control Samples

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

### V. Target Compound Identification

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

### **VI. Compound Quantitation and CRQLs**

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

### VII. System Performance

The system performance was acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

### VIII. Overall Assessment of Data

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

### IX. Field Duplicates

No field duplicates were identified in this SDG.

### BRC Tronox Parcel G Polynuclear Aromatic Hydrocarbons - Data Qualification Summary - SDG F8F120180

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

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

No Sample Data Qualified in this SDG

### **BRC Tronox Parcel G**

Polynuclear Aromatic Hydrocarbons - Field Blank Data Qualification Summary - SDG F8F120180

No Sample Data Qualified in this SDG

### VALIDATION COMPLETENESS WORKSHEET

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

### Level III/IV

Date:8/4/08
Page: / of /
Reviewer:
2nd Reviewer:

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

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

	Validation Area		Comments
١.	Technical holding times	$\mathbf{A}$	Sampling dates: 6/11/08
lla.	Initial calibration	A	
lib.	Calibration verification/ICV	W	$ a  \leq  570$
- 111.	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates		78B-GJ-08-10'
IVc.	Laboratory control samples	$\mathbf{A}$	209
V.	Target compound identification	A	Not reviewed for Level III validation.
VI.	Compound Quantitation and CRQLs	$\mathbf{A}$	Not reviewed for Level III validation.
VII.	System Performance	A	Not reviewed for Level III validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N/	
Х.	Field blanks	$\square$	

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

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

D = Duplicate TB = Trip blank EB = Equipment blank

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

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

Notes:

Note:
LDC #: 1918879 SDG #: <u>500 cow</u>

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Method: GC HPLC				
Validation Area	Yes	No	NA	Findings/Comments
T Technical holding tures				
All technical holding times were met.	$\leq$			
Cooler temperature criteria was met.		-	WEITHING	
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 used?		/		
Did the initial calibration meet the curve fit acceptance criteria?			/	
Were the RT windows properly established?				
IV: Continuing calibration			<u>1998)</u> 1	T
What type of continuing calibration calculation was performed?%D or%R	/			
Was a continuing calibration analyzed daily?		Ĺ	<u> </u>	
Were all percent differences (%D) $\leq$ 15%.0 or percent recoveries 85-115%?				
Were all the retention times within the acceptance windows?				
V Blanks	e I	80000 1	20534 T	1
Was a method blank associated with every sample in this SDG?		<u>[</u>	ļ	
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 Serrogate spikes		<u></u>	<u>.</u> T	1
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?		THE CAN BE OF		
VII. Matrix spike/Matrix spike duplicates	())) <b>(</b> ) T	1 1	т. Т.	1
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				an a
Was an LCS analyzed for this SDG?	$ \zeta $	4		
Was an LCS analyzed per extraction batch?				

### VALIDATION FINDINGS CHECKLIST

	Page:	<u> </u>	<u> </u>
	Reviewer:	9	
2nd	<b>Reviewer:</b>		

Validation Area	Yes	No	NA	Findings/Comments
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		Ł		
IX: Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?	<u> </u>			
Were the performance evaluation (PE) samples within the acceptance limits?				
X. Target compound identification	<u> </u>	<u>7 10 10</u> T	NUMBER OF T	
Were the retention times of reported detects within the RT windows?				
XI. Compound quantitation/CRQLs		T T	<u>se p</u> e	
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.	$\square$			
XIII. Overall assessment of data				
Overail assessment of data was found to be acceptable.	$\square$			
XIV: Field duplicates				
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?	T		$\square$	

METHOD: GC HPLC

VALIDATION FINDINGS WORKSHEET

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	С. 2,4,5-Т	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
Е. 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	ll. Sulprofos	
O. Phenanthrene	Ö.		O. Chlorpyrifos		
P. Pyrene	ď		P. Fenthion		
Ċ	a		Q. Parathion-ethyl		
œ			R. Trichloronate		
Ċ.			S. Merphos		
			T. Stirofos		
			U. Tokuthion		

Notes:

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SDG #: Ser CON LDC # 19188

- GC \_ HPLC

METHOD:

# VALIDATION FINDINGS WORKSHEET **Continuing Calibration**

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2nd Reviewer:

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". What type of continuing calibration calculation was performed? <u>%D</u> or <u>RPD</u> <u>What N/A</u> Were continuing calibration standards analyzed at the required frequencies?

Y N NIA

Did the continuing calibration standards meet the %D / RPD validation criteria of <15.0%? Level IV Only

Y N N/A

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

		Qualifications	Vtolets A			Ntdets / O											
		Associated Samples	MTATC			1-2											
	RT (limit)								( )	- -	(	(	(				(
	(Limit ≤ 15.0)	12 6			15.2												
	Compound				Ø												
Detector/	Column	<			NS												
	Standard ID	8 EV768	(12V)		QCA2873	/											
	Uate	6440	· · · · · · · · · · · · · · · · · · ·		6/16/08	~	 										
4	*																

SDG #: *Zee cew* LDC #: 19/887

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

6 6 Page: 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

Recalculated	%RSD	1-28.1	12820							
Reported	%RSD	1-8-1	17.820							
Recalculated	Average CF (initial)	806710	62608							
Reported	Average CF (initial)	806710	8-0924							
Recalculated	CF ( / std)	807269	67485	-						
Reported	CF ( / std)	805-69	67485							
	Compound		-4							
	Calibration Date	1/1/04	e the o	×						
	Standard ID	. <i>Per</i> l	122							
	#	-			ы		9		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.

INICLC.1SB

LDC #: 171 8849 SDG #: Second

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

đ 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

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

					Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc. CCV	Ω%	۵%
-	8042862	X1. 1. 18	J	0.50	0.5344	0.5344	6.9	6.9
		n./a//a	Ĥ		0.4834	0.4834	E.	e E
			-					
ы	804-873	6/16/08	IJ	0.50	0.5307	0.5307	6./	6./
	/		٩		0.4984	72867.0	С.	N
ю								
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.

CONCLC.1S

LDC #: <u>71874</u> 9 SDG #: <u>5ac CO W</u>V

# VALIDATION FINDINGS WORKSHEET **Surrogate Results Verification**

Page: Reviewer: <u>C</u> 2nd reviewer: \_\_\_\_

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

٨ Sample ID:

	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recoverv	Percent Recoverv	Percent Difference
				Reported	Recalculated	
NS   ~	r	5.0	18:35-8	20	73	0

### Sample ID:

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

### Sample ID:

( <del>,</del>			
Percent Difference			
Percent Recovery	Recalculated		
Percent Recoverv	Reported		
Surrogate Found			
Surrogate Spiked			
Column/Detector			
Surrogate			



METHOD: GC HPLC

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

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

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

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

SC = Concentration

LCSD = Laboratory control sample duplicate percent recovery

8158
N
5
2
Ø
samples:
CS/LCSD

	<u>v</u>	pike	Spiked	Sample	ΓC	S	ГС	sD	rcs/I	-csp
Compound	××)	ided AB	Concei	P C	Percent R	ecovery	Percent	Recovery	R	0
	rcs	LCSD	rcs	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)										
Diesel (8015)										
Benzene (8021B)										
Methane (RSK-175)										
2,4-D (8151)										
Dinoseb (8151)										
Naphthalene (8310)	61.7	4N	52.6	ΝĂ	79	79				
Anthracene (8310)	N/	$\mathcal{A}$	5/.2	7	77	77				
HMX (8330)	>					-				
2,4,6-Trinitrotoluene (8330)										

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

0C #: 1918849	DG#:2000
LDC	SDG

# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page: <u>/</u>of / Reviewer: <u></u> 2nd Reviewer:

METHOD: GC VHPLC

ts?	P		
ted resul	~	, ,	
V samples? ree within 10% of the repor	Compared Name		
ated and verified for all level l' letected target compounds ag	Example:		Concentration =
Were all reported results recalcul Were all recalculated results for d	= (A)(Fv)(Df) (RF)(Vs or Ws)(%S/100)	ght of the compound to be measured ne of extract ctor	sponse factor of the compound calibration re of the sample it of the sample lid
Y N NA Y N NA	Concentration=	A= Area or hei Fv= Final Volun Df= Dilution Fac	RF= Average res In the initial Vs= Initial volum Ws= Initial weigh %S= Percent Sol

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

SAMPCALew.wpd

Comments: \_

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

Project/Site Name:	BRC Tronox Parcel G
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Collection Date: June 11, 2008

LDC Report Date: August 8, 2008

Matrix:

Parameters: Dioxins/Dibenzofurans

Soil

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

### Sample Identification

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

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 4 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

This review follows USEPA Contract Laboratory Program National Functional Guidelines for Polychlorinated Dioxins/Dibenzofurans Data Review (September 2005) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

### III. Initial Calibration

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

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

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

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

### **IV. Routine Calibration (Continuing)**

Routine calibration was performed at the required frequencies.

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

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

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

### V. Blanks

Method blanks were reviewed for each matrix as applicable. No polychlorinated dioxin/dibenzofuran contaminants were found in the method blanks.

No field blanks were identified in this SDG.

### VI. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

### VII. Laboratory Control Samples (LCS)

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

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

### VIII. Regional Quality Assurance and Quality Control

Not applicable.

### IX. Internal Standards

All internal standard recoveries were within QC limits with the following exceptions:

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
TSB-GJ-09-30'	<ul> <li><sup>13</sup>C-2,3,7,8-TCDF</li> <li><sup>13</sup>C-1,2,3,7,8-PeCDF</li> <li><sup>13</sup>C-1,2,3,7,8-PeCDD</li> <li><sup>13</sup>C-1,2,3,4,7,8-HxCDF</li> <li><sup>13</sup>C-1,2,3,4,6,7,8-HxCDD</li> <li><sup>13</sup>C-1,2,3,4,6,7,8-HpCDD</li> <li><sup>13</sup>C-0CDD</li> </ul>	38 (40-135) 26 (40-135) 27 (40-135) 18 (40-135) 21 (40-135) 11 (40-135) 16 (40-135) 9.7 (40-135)	1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,7,8,9-HxCDD 0CDD 1,2,3,7,8-PeCDF 2,3,7,8-TCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-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)	Ρ

### X. Target Compound Identifications

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

### XI. Compound Quantitation and CRQLs

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

### XII. System Performance

The system performance was acceptable for samples on which EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by EPA Level III criteria.

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

No field duplicates were identified in this SDG.

### BRC Tronox Parcel G Dioxins/Dibenzofurans - Data Qualification Summary - SDG F8F120180

SDG	Sample	Compound	Flag	A or P	Reason
F8F120180	TSB-GJ-09-40'	2,3,7,8-TCDF	J+ (all detects)	Р	Routine calibration (%D)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-30' TSB-GJ-09-40'	1,2,3,7,8,9-HxCDD OCDD	J+ (all detects) J+ (all detects)	Р	Laboratory control samples (%R)
F8F120180	TSB-GJ-09-30'	1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,7,8-PeCDF 2,3,7,8-PeCDF 2,3,7,8-PeCDF 1,2,3,4,7,8-PeCDF 1,2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HxCDF 1,2,3,4,7,8,9-HxCDF 1,2,3,4,7,8,9-HxCDF 0,2,3,4,7,8,9-HxCDF	J (all detects) UJ (all non-detects)	Ρ	Internal standards (%R)

### BRC Tronox Parcel G

Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG F8F120180

No Sample Data Qualified in this SDG

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

No Sample Data Qualified in this SDG

### VALIDATION COMPLETENESS WORKSHEET

LDC #: 19188A21 SDG #: \_\_\_\_\_\_F8F120180

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Level III/IV

Date: Page: Reviewer: 2nd Reviewer

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Laboratory: Test America

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
١.	Technical holding times	A	Sampling dates: 4/11/08
11.	GC/MS Instrument performance check	A	
111.	Initial calibration		
IV.	Routine calibration/IO	Im	
V.	Blanks		
VI.	Matrix spike/Matrix spike duplicates	$ $ $\wedge$	client Defied
VII.	Laboratory control samples	-mr	229
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	Iw	
<b>X</b> .	Target compound identifications		Not reviewed for Level III validation.
XI.	Compound quantitation and CRQLs	Å	Not reviewed for Level III validation.
XII.	System performance	A	Not reviewed for Level III validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	N	

Note:

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

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

D = Duplicate TB = Trip blank EB = Equipment blank

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

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

Notes:

LDC #: <u>191887</u> SDG #:<u>5@</u>C

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

Validation Area	Yes	No	NA	Findings/Comments
1. Technical holding times				
All technical holding times were met.	/			
Cooler temperature criteria was met.	/			
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?	$\square$			
Were the retention time windows established for all homologues?				
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers $\leq 25\%$ ?				
Is the static resolving power at least 10,000 (10% valley definition)?				
Was the mass resolution adequately check with PFK?	$\mathbf{Z}$			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?				
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?	$\square$			
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?				
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	·	ř	r	
Was an LCS analyzed for this SDG?	$\mathbb{Z}$		l	

LDC #: 1918842 SDG #: <u>3a Cou</u>

### VALIDATION FINDINGS CHECKLIST

	Page: -> of ->	
	Reviewer: 9	
2nd	Reviewer:	

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		/		
VIII. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
IX. Internal standards				
Were internal standard recoveries within the 40-135% criteria?		<		
Was the minimum S/N ratio of all internal standard peaks $\geq$ 10?				
X. Target compound identification				
For 2,3,7,8 substituted congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard?			/	
For 2,3,7,8 substituted congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration?			/	
For non-2,3,7,8 substituted congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution?			/	
Did compound spectra contain all characteristic ions listed in the table attached?			$\checkmark$	
Was the Ion Abundance Ratio for the two quantitation ions within criteria?			$\angle$	
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?			$\langle \rangle$	
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		F		
System performance was found to be acceptable.				
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.				
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.		/		



### VALIDATION FINDINGS CHECKLIST



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Validation Area	Yes	No	NA	Findings/Comments
Target compounds were detected in the field duplicates.			/	
XV: Field blanks				
Field blanks were identified in this SDG.				/
Target compounds were detected in the field blanks.				

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:

LDC #: 1918842

# VALIDATION FINDINGS WORKSHEET **Routine Calibration**



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". KN N/A V/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?

			1	1		<u> </u>	 1	-	-	 -	1	1	 	_								
	Qualifications	It dats A													Ion Abundance Ratio	0,65-0,89	1.32-1.78	1.05-1.43	0.43-0.59	0.37-0.51	0.88-1.20	0.76-1.02
-	ssociated Samples														Selected ions (m/z)	M/M+2	M+2/M+4	M+2/M+4	M/M+2	M/M+2	M+2/M+4	M+2/M+4
o criteria?	Finding Ion Abundance Ratio A	4													PCDFs	Tetra-	Penta-	Неха-	Hexa- <sup>13</sup> C-HxCDF (IS) only	Hepta- <sup>13</sup> C-HpCDF (IS) only	Hepta-	Octa-
on Abundance Ratio	Finding %D (Limit: <u>≤</u> 30.0%)	37.2													Abundance Ratio	0.65-0.89	1.32-1.78	1.05-1.43	0.43-0.59	0.37-0.51	0.88-1.20	0.76-1.02
in standards meet the	Compound	13C-H													ected ions (m/z)   Ion /	M/M+2	M+2/M+4	M+2/M+4	M/M+2	M/M+2	M+2/M+4	M+2/M+4
id all routine calibratic	Standard ID	03/2021025													CDDs Sel				DF (IS) only	CDF (IS) anly		
N N/A D	Date	7/7/0 8	\ \ \												4	Tetra-	Penta-	Hexa-	Hexa- <sup>13</sup> C-HxC	Hepta-"C-Hp(	Hepta-	Octa-
AL	*																					

LDC #: 191 8542 SDG #:

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

5 Ø Page: 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". N N/A Was a LCS required? N N/A Was a LCS analyzed every 20 samples for each matrix or whenever a sample extraction was per V N/A Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the OC limit

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 /%B) and relative percent difference (PDD) with the CO matrix of

		7		- 17	1	1		-	_	-				_	_							_					_
	Qualifications	(1+1.4 J		>																							
limits?	Associated Samples	1 3-2	8170292NB	11/2 1/10																							
<b>RD</b> ) within the QC	RPD (Limits)	)	)			( )	( )	( )	( )	( )	( )			) (		)	· ·	)	· ·	( )	( )	( )	( )	()	()	)	
ercent difference (i	LCSD %R (Limits)	)	( )	()	( )	( )	( )	(	()	(	( )	( )	( )	( )	( )	( )	- ( )	( )	( )	(	( )	( )	( )	( )	( )	( )	( )
(76r) and relative p	LCS %R (Limits)	Ge1-12) 281	1521(74-144	( )	(	( )	()	(	( )	( )	( )	( )	(	( )	( )	^ ~	( )	( )	( )	( )	( )	( )	( )	(	<b>^</b>	( )	(
Il recoveries	Compound	Ŵ	$\approx 6$	/																							
Meie nie roo peice	Lab ID/Reference	817049315	/ .																								
1/1/1	# Date																										
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# VALIDATION FINDINGS WORKSHEET Internal Standards

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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". <u>Y/N N/A</u> Are all internal standard recoveries were within the 40-135% criteria? <u>N/N N/A</u>

۱ŀ							
	Date	Lab ID/Reference	Internal Standard		% Recovery (Limit: 40-135	(%	Qualifications
1 1		M	A	S S	- 07)	( -5 81	1 1 4 4 (B-&)
			C	26	)	(	
1			$\mathcal{D}$	27	)	(	
			Ź	81	)	(	
			Ŧ	7/2	)	(	
			Left -	///	)	(	
			$t^{\prime}$	12	~		
			,T	9.4	)	(	
						(	
						(	
1						(	
					)	(	
					)	(	
					)	(	
1					)	(	
					<b>`</b>	(	
					)	(	
					)	(	
					)	(	
					)	(	
		Internal Standards	Check Standard Used		Recovery S	tandards	Check Standard Used
	<sup>13</sup> C-2,3,7,8-TCD	ЪF		¥	<sup>13</sup> C-1,2,3,4-TCDD		
	<sup>13</sup> C-2,3,7,8-TCD	D		<u>نـ</u>	<sup>13</sup> C-1,2,3,7,8,9-HxCDD		
	<sup>13</sup> C-1,2,3,7,8-Pe	SCDF		ž			
	<sup>13</sup> C-1,2,3,7,8-Pe	CDD		ż			
1	<sup>13</sup> C-1,2,3, <b>6</b> ,7,8-1	HxCDF		o			
÷ F	<sup>13</sup> C-1,2,3,6,7,8-1	HxCDD		<u>م:</u>			
- 1	<sup>13</sup> C-1,2,3,4,6,7,E	8-HpCDF		Ö			
_ I	<sup>13</sup> C-1,2,3,4,6,7,5	8-HpCDD		œ			
	<sup>13</sup> C-OCDD						

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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:

 $\label{eq:RFF} RFF = (A_{\rm J})(C_{\rm s})/(A_{\rm s})(C_{\rm s})$  average RRF = sum of the RRFs/number of standards %RSD = 100 \* (S/X)

 $A_x = Area of compound,$  $C_x = Concentration of compound,$ S = Standard deviation of the RRFs,

 $\label{eq:associated} \begin{array}{l} A_{\mathbf{k}} = Area \mbox{ of associated internal standard} \\ C_{\mathbf{k}} = Concentration \mbox{ of internal standard} \\ X = Mean \mbox{ of the RRFs} \end{array}$ 

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF ( CS3 std)	RRF ( / 2 S = Stid)		
-	10/2-	0/-//	2.3.7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	8.798	0.798	1820			мчэ <b>л</b>
		6/17/00	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	560	0,00	10 C		10.5	N. C.
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1080	100	181	100	0.91	
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	2778.0	D RILL	82 0	00.0	4	
			OCDF ("C-OCDD)	1261	1241	281	000	2.0	
N			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)				90:		16-21
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)						
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)						
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)						
			OCDF (1°C-OCDD)						
e			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)						
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)						
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)						
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)						
		. v	OCDF ( <sup>1</sup> C-OCDD)						

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





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:

Where:	
ance = 100 * (ave. RRF - RRF)/ave. RRF	٨.)(٥.)/(٩.)(٥.)
% Differe	3RF = (

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

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

 $A_{\mathbf{s}} = Area$  of associated internal standard  $C_{\mathbf{s}} = Concentration of internal standard$ 

L								
					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	۵%	Q%
	29/10 0 Safes	1 200/08	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.798	28.0	ínx (	5	le L
	, ,	20/1/2	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	516.0	082	a a a		
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	128.0	0.94	660	11	
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	0.844	0.89	0.89	5.2	
			OCDF ("C-OCDD)	1.72/	1.60	1.62	8	
2			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
		·	1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)					
			OCDF ("c-OCDD)					
e			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)					
Ī			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
1			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
T			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDD)					
							1	

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.

	2
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БС	SDG

# VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Results Verification

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

The percent recoveries (%R) and Relative Percent Difference (RPD) of the 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 Where: SSC = Spiked sample concentration SA = Spike added

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

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

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	_							_	 		
I CS/I CSD RPD	þ	Recalculated									
	Reported										
	ecovery	Recalc									
50 T	Percent R	Renorted									
S.	Recovery	Recalc	96	1021	126	9/	6//	<pre></pre>			
	Percent F	Renorted	96	701	) A	6	611				
Sample	tration	L CSD	$\mathcal{N}$								
Spiked (	Spiked S Concen		19.2	to d/	901	30.8	238				
ike Jed	ded 5/3	I CSD	N/A	_			1				
Sp	Ţ.	I CS	20.0	001	1	$\bigwedge$	000				
	Compound		2,3,7,8-TCDD	1,2,3,7,8-PeCDD	1,2,3,4,7,8-HxCDD	1,2,3,4,7,8,9-HpCDF	OCDF				

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.

Descriptor	Accurate mass <sup>(a)</sup>	lon ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(a)</sup>	Di nol	Elemental Composition	Analvte
-	303.9016 305.8987 315.9419 317.9389	₩ <sup>₩</sup> ₩ + <sup>₩</sup> ₩	C <sub>12</sub> H, <sup>35</sup> Cl <sub>4</sub> O C <sub>12</sub> H, <sup>35</sup> Cl <sub>4</sub> O <sup>13</sup> C <sub>12</sub> H, <sup>46</sup> Cl <sub>4</sub> O <sup>13</sup> C <sub>12</sub> H, <sup>46</sup> Cl <sub>4</sub> O	TCDF TCDF TCDF (S) TCDF (S)	4	407.7818 409.7788 417.8250	M M M M M M M M M M M M M M M M M M M	C <sub>12</sub> H <sup>38</sup> Cl <sub>8</sub> 7ClO C <sub>12</sub> H <sup>38</sup> Cl <sub>8</sub> 7ClO 13C <sub>12</sub> H <sup>38</sup> Cl <sub>2</sub> 0 13C <sub>12</sub> H <sup>38</sup> Cl <sub>2</sub> O	HpCDF HpCDF HpCDF (S)
	319.8965 321.8936 331.9368 333.9338 333.9338 375.8364 375.8364	M 4 2 M 4 4 M 4 4 CCK 5 CCK 5	C <sub>17</sub> 2 <sup>4</sup> , C <sub>1</sub> C <sub>12</sub> H, SCI, O <sub>2</sub> C <sub>12</sub> H, SCI, O <sub>2</sub> 13C <sub>12</sub> H, SCI, O <sub>2</sub> 13C <sub>12</sub> H, SCI, SCIO <sub>2</sub> 13C <sub>12</sub> H, SCI, SCIO <sub>2</sub> C <sub>12</sub> H, SCI, SCIO <sub>2</sub> C <sub>17</sub> H, SCI, SCIO C <sub>6</sub> F <sub>13</sub>	TCDD (3) TCDD (3) TCDD (3) HXCDPE		419.8220 423.7767 425.7737 435.8169 437.8140 479.7165 [430.9728]	∩ M M M M M H + + 2 0 + + + 2 + 2 + 2 + 2 + 2 0 + + + 2 + 2 + 2 + 2 + 2 + 2 + 2 + 2 +	ັດ <sub>ເ</sub> 2H ແ cl ູ ກັດເວ ດ <sub>ເ2</sub> H ແ cl ູ ກັດເວ ດ <sub>ເ2</sub> H ແ cl ູ ກັດເວ ເ <sub>2</sub> H ແ cl ູ ກັດເວ ເຣ ເ 2 ດ ເ 2 H ແ cl ູ ກັດເວ ດ ເ 2 ດ ເ 2 ດ 5 F ,	HPCDF HPCDD HPCDD HPCDD (S) NCDPE (S) PFK
N	339.8597 331.8567 351.9000 353.8970 355.8546 355.8546 355.8546 355.8546 355.8546 355.8546 355.8949 360.8919 408.7974 [354.9792]	M M M M M M M M M M M M M M M M M M M	C <sub>1</sub> H <sub>3</sub> ±Cl <sub>3</sub> rCl0 C <sub>1</sub> H <sub>3</sub> ±Cl <sub>3</sub> rCl0	PeCDF PeCDF PeCDF (S) PeCDD PeCDD PeCDD PeCDD (S) PFK	۵	441.7428 443.7399 457.737 459.7348 469.7780 471.7750 513.6775 [422.9278]	M + 2 M + 4 M + 4 M + 4 M + 4 C C K A + 4 C C K	Cr_ascl, arClO Cr_ascl, arClO Cr_ascl, arCl_2O Cr_ascl, arCl_2O Cr_ascl, arCl_2O 13Cr_ascl, arCl_2O 13Cr_ascl, arCl_2O 13Cr_ascl, arCl_2O Cr_0F_17 Cr_0F_17	0CDF 0CDF 0CDD 0CDD (S) 0CDPE 9FK
m	373.8208 375.8178 383.8639 383.8639 385.8610 389.8156 391.8127 401.8559 403.8559 403.8559 403.8529 445.7555 [430.9728]	M M M H 2 M M M M H + 2 M M M M H + 2 M M + 4 2 M M + 4 2 CCK 4 4	C <sub>12</sub> H <sub>2</sub> sec1, srClO C <sub>12</sub> H <sub>2</sub> sec1, srClO C <sub>12</sub> H <sub>2</sub> sec1, srCl <sub>2</sub> O 13 C <sub>12</sub> H <sub>2</sub> sec1, srCl <sub>2</sub> O 13 C <sub>12</sub> H <sub>2</sub> sec1, srCl <sub>2</sub> O C <sub>12</sub> H <sub>2</sub> sec1, srCl <sub>2</sub> O C <sub>12</sub> H <sub>2</sub> sec1, srCl <sub>2</sub> O 13 C <sub>12</sub> H <sub>2</sub> sec1, srCl <sub>2</sub> O 13 C <sub>12</sub> H <sub>2</sub> sec1, srCl <sub>2</sub> O C <sub>12</sub> H <sub>2</sub> sec1, srCl <sub>2</sub> O	HXCDF HXCDF HXCDF HXCDD HXCDD HXCDD HXCDD S(S) S(S) S(S) S(S) S(S) S(S) S(S) S(					
a) The fc	I Ilowing nuclidic masse	s were used:							

The following nuclidic masses were used:

H = 1.007825C = 12.0000000  $^{13}$ C = 13.003355 F = 18.9984

O = 15.994915<sup>35</sup>Cl = 34.968853
<sup>37</sup>Cl = 36.965903

S = internal/recovery standard

C:\WPDOCS\WRK\DIOXIN90\TCI90.21

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



### LDC #: 191884-1 VALIDATION FINDINGS WORKSHEET SDG #: 2ac Cover Sample Calculation Verification

Page:	
Reviewer:	<u>q</u>
2nd reviewer:	-

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

N N/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 10.0% of the reported results?

Concer	ntration	$u = \frac{(A_{.})(I_{.})(DF)}{(A_{is})(RRF)(V_{o})(\%S)}$
A <sub>x</sub>	=	Area of the characteristic ion (EICP) for the compound to be measured
A <sub>is</sub>	=	Area of the characteristic ion (EICP) for the specific internal standard
l <sub>s</sub>	11	Amount of internal standard added in nanograms (ng)
V.	==	Volume or weight of sample extract in milliliters (ml) or grams (g).
RRF	=	Relative Response Factor (average) from the initial calibration
Df	=	Dilution Factor.
%S	=	Percent solids, applicable to soil and solid matrices only.





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