

### LABORATORY DATA CONSULTANTS, INC.

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

August 11, 2008

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. These SDGs were received on July 11, 2008. Attachment 1 is a summary of the samples that were reviewed for each analysis.

### LDC Project # 19097:

### SDG # Fraction

 F8F120137, Volatiles, Semivolatiles, Chlorinated Pesticides, Polychlorinated
 F8F120167 Biphenyls, Metals, Wet Chemistry, Gasoline Range Organics, Diesel Range Organics, Polynuclear Aromatic Hydrocarbons, Dioxins/Dibenzofurans

The data validation was performed under EPA Level III 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,

alle area for

Erlinda T. Rauto <sup>v</sup> Operations Manager/Senior Chemist

						_										_																		
		s																															73	
		×												_	_																		0	
		s			L				<u> </u>																								0	
		3																															0	pdw.
		s																															0	19097ST.wpd
		3																															0	190
	0&G (9071B 1664A)	s	0	2	2																												4	
	0&G (9071B 1664A)	N	1	0	0						Γ																						-	L.
	<b>,</b>	S	0	2	S																												4	
	s0, (300.0)	3	1	0	0																												Ţ	
		S	0	~	Q	5		1					$\uparrow$	$\square$																		+	4	
	NO <sup>2</sup> NO <sup>2</sup> NO <sup>2</sup>	≥	-	0							$\vdash$	$\uparrow$	1-	$\square$	$\square$																	+	-	
-		S	0	2			$\square$				1	$\vdash$	$\square$	<u> </u>	1						$\vdash$								-			+	4	
0	hlor luor	3	1	0			1	$\vdash$	+		-	┢	$\vdash$	┢	$\vdash$			<u> </u>						$\square$				 			-+	+		
Cel	ate C	- s	0	5	Q			┢	$\vdash$	┢	╞──	┢	$\vdash$	┢	┢─	╞				$\square$				$\vdash$	$\left  \right $						-+	+	4	
Parcel G	Bromide Chloride Bromine Chlorine Chlorate Fluoride	3	-	0	1000		$\vdash$		+	┢	┢	┢	+	$\left  \right $	-				-					$\left  - \right $							$\dashv$	+		Ś
1 Contraction 1	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	s S	0		S S		-	+	┢	$\vdash$	┢	┼──	┢	┢	┢				-									-			+	-+		DUP
Tronox,	Dioxins (8290)	3	1	0	0			┢	┢	$\vdash$		┢──													_	_		 					4	, and
l 1 1					thermost the			-	┝	$\vdash$		┢		-																		+		MSD
BRC	PAHs (8310)	s /	0	2	2	-		-	$\left  \right $	╞			-	-																		-	4	8 MS/
	3 <b></b>	3	-	0	0				┢	-	-																			_	_	+	ᅴ	Iclude
Attachment 1 -Sacramento /	DRO (8015)	S	0	2	2				_														~~								_		4	not ir
en le		≥	-	0	0				_	<u> </u>		ļ																 			-+		-	ts do
Atta	GRO (8015)	S	<u> </u>	3	Ś																												2	coru
acr	98	≥	-	0	0																												-	mple
		S	0	2	2																												4	se sa
(ERM	Met (SW1	≥	-	0	0																											T	-	The
	Bs 82)	S	0	2	2																										Τ		4	ation)
#19097	PCBs (8082)	≥	-	0	0																										1		-	valida
f19	J. t	S	0	2	ç																		-								+	1	4	el III
<b>5</b>	Pest. (8081A)	≥	-		0																							_		-	$\uparrow$			s Lev
LDC		S	-	2	<b>C</b> 1			<b> </b>	<u> </u>		$\vdash$	<u> </u>		-							╡			$\neg$	+						-		4	lls are
	SVOA (8270C)	≥	-	0	0					-		$\vdash$	$\vdash$									-+		$\neg$	-+	-+	+			-+	+			er ce
		- S	0		2					-	$\vdash$						-	-						$\dashv$	+	-	-	 _		-+	$\rightarrow$	+	4	all oth
	VOA (8260B)	3	~	-	0	$\vdash$		⊢		-		-		-	$\left  - \right $		$\dashv$	$\dashv$				-		-	+	-				-+		-	m m	ion (s
		-				$\vdash$			┢	┝		-					-	-				-+		-	-+		$\dashv$			-	+	_	Ψ	Shaded cells indicate Level IV validation (all other cells are Level III validation). These sample counts do not include MS/MSD, and DUPs
	(3) DATE DUE		08/01/08	08/01/08	08/01/08																													2 2
		-															-								_	_		 						Leve
	DATE REC'D		07/11/08	07/11/08	07/11/08																													icate
e e	28		1/20	120	1/20																													ls ind
EDD				~	~																					1		-		1		+		od Cel
Pac		ir/Soi	0137	0167	0167																												х	Shade
9,064 Pages-CD 80/20 EDD	SDG#	Water/Soil	F8F120137	F8F120167	F8F120167																											i		57
		Matrix: \	۳	щ	щ																													
10° °	Ä	,⊂ L	·····	_		_								-														 						

19097ST.wpd

### LDC Report# 19097B1

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

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

Collection Date: June 11, 2008

LDC Report Date: July 22, 2008

Matrix: Soil/Water

Parameters: Volatiles

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

### Sample Delivery Group (SDG): F8F120167

TSB-GJ-08-10 TSB-GJ-08-20\*\* TSB-GJ-08-30\*\* TSB-GJ-08-40 TB-1 6/11/08 TSB-GJ-08-10MS TSB-GJ-08-10MSD

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 6 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8260B for Volatiles.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- 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 F8F120167	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 F8F120167	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 F8F120167	J+ (all detects)	A
5/28/08 (LICV9881)	2-Hexanone	25.04476	All water samples in SDG F8F120167	J- (all detects) UJ (all non-detects)	А

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

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P	
6/16/08	Ethanol	0.00209 (≥0.05)	All soil samples in SDG F8F120167	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-1 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-1 6/11/08	6/11/08	Acetone	1.1 ug/L	All soil samples in SDG F8F120167

Sample "RINSATE 1" (from SDG F8F120137) was identified as a rinsate. No volatile contaminants were found in this blank with the following exceptions:

Rinsate Blank ID	Sampling Date	Compound	Concentration	Associated Samples
RINSATE 1	6/11/08	Dichloromethane	3.3 ug/L	All soil samples in SDG F8F120167

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

### VI. Surrogate Spikes

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

Sample	Surrogate	%R (Limits)	Compound	Flag	A or P	
F8F200000-125	Bromofluorobenzene	117 (79-115)	All TCL compounds	J+ (all detects)	Ρ	

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

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

### XI. Target Compound Identifications

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

### XII. Compound Quantitation and CRQLs

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

### XIII. Tentatively Identified Compounds (TICs)

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

### XIV. System Performance

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

### XV. Overall Assessment of Data

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

### **XVI. Field Duplicates**

No field duplicates were identified in this SDG.

### BRC Tronox Parcel G Volatiles - Data Qualification Summary - SDG F8F120167

SDG	Sample	Compound	Flag	A or P	Reason
F8F120167	TSB-GJ-08-10 TSB-GJ-08-20** TSB-GJ-08-30** TSB-GJ-08-40	Ethanol	J (all detects) UJ (all non-detects)	A	Initial calibration (RRF)
F8F120167	TB-1 6/11/08	lodomethane	J+ (all detects)	A	Continuing calibration (%D)
F8F120167	TB-1 6/11/08	lodomethane	J+ (all detects)	A	Continuing calibration (ICV %D)
F8F120167	TB-1 6/11/08	2-Hexanone	J- (all detects) UJ (all non-detects)	A	Continuing calibration (ICV %D)
F8F120167	TSB-GJ-08-10 TSB-GJ-08-20** TSB-GJ-08-30** TSB-GJ-08-40	Ethanol	J (all detects) UJ (all non-detects)	A	Continuing calibration (RRF)

### BRC Tronox Parcel G Volatiles - Laboratory Blank Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

### BRC Tronox Parcel G Volatiles - Field Blank Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

### VALIDATION COMPLETENESS WORKSHEET Level III/IV

LDC #: <u>19097B1</u> SDG #: <u>F8F120167</u> Laboratory: Test America

### Date: 7/21/08 Page: \_\_\_\_\_\_ Reviewer: \_\_\_\_\_\_ 2nd Reviewer: \_\_\_\_\_\_

A CARLES AND A CONTRACTOR OF AND

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
<b>I</b> .	Technical holding times	A	Sampling dates: 6/11/08
11.	GC/MS Instrument performance check	Å	, ,
111.	Initial calibration	SW	% psD, 12 20.990
IV.	Continuing calibration/ICV	.sw	ICVERS
V.	Blanks	A	
VI.	Surrogate spikes	4	
VII.	Matrix spike/Matrix spike duplicates	SW	Runsate 2 Les IP
VIII.	Laboratory control samples	SW	Les IP
IX.	Regional Quality Assurance and Quality Control	N	
<b>X</b> .	Internal standards	Δ	
XI.	Target compound identification	Δ	Not reviewed for Level III validation.
XII.	Compound quantitation/CRQLs	A	Not reviewed for Level III validation.
XIII.	Tentatively identified compounds (TICs)	A	Not reviewed for Level III validation.
XIV.	System performance	Δ	Not reviewed for Level III validation.
XV.	Overall assessment of data	۵	
XVI.	Field duplicates	N	
XVII.	Field blanks	ふら	TB=5 R= Rusate 1, \$4 # FSF/20/3

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:

Note:

\*\*Indicates sample underwent Level IV validation

	JUILT U	Jav	Υ <u></u>				
11	TSB-GJ-08-10	11 1	F8 F180000-291	21/	8 170a9 /	31	
21	TSB-GJ-08-20**	12	18 F2 00000-125		8172125	32	
3	TSB-GJ-08-30**	13	F8F200000-361	23 <b>3</b>	817236/	33	
<b>†</b> 4	TSB-GJ-08-40 ,	14		24		34	
452	B= 10000001 TB-1 6/11/08	15		25		35	
6	TSB-GJ-08-10MS	16		26		36	
7	TSB-GJ-08-10MSD	17		27		37	
8		18		28		38	
9		19		29	·····	39	
10		20		30		40	

19097B. LDC #:\_\_\_\_\_ SDG #:\_\_\_\_\_ con

### VALIDATION FINDINGS CHECKLIST

Page: / of <u>2</u> Reviewer: <u>7</u> 2nd Reviewer: <u></u>

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

Validation Area	Yes	No	NA	Findings/Comments
It recention holding times as a state of the second state of the second state of the second state of the second				
All technical holding times were met.				
Cooler temperature criteria was met.				
II. COMSUMMENDERCOMMENDER				
Were the BFB performance results reviewed and found to be within the specified criteria?	/			
Were all samples analyzed within the 12 hour clock criteria?				
Did the laboratory perform a 5 point calibration prior to sample analysis?	$\leq$		<b> </b>	
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?	$\leq$			
Did the initial calibration meet the curve fit acceptance criteria of $\geq 0.990?$	$\leq$			
Were all percent relative standard deviations (%RSD) $\leq$ 30% and relative response factors (RRF) $\geq$ 0.05?		_	-	
NV Continuingreationance				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?	$\square$			
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?		/	_	
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.		_	-	n
Were all surrogate %R within QC limits?	1			
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?			-	-
Min Matrix spike Matrix spite dubre less a constant de la constant de la constant de la constant de la constant				
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?	-	-		
Were the MS/MSD percent recoveries (%R) and the relative percent differences RPD) within the QC limits?		4	-	
////www.carenz.control_camplester.com/control_carenzester.com/				
Vas an LCS analyzed for this SDG?	1			

LDC #: 190978) SDG #: pu coner

### VALIDATION FINDINGS CHECKLIST

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

	T	T	T	
Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per analytical batch?		ļ		
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?			1	
IX Regional Coality Asstrance and Ouality Control				
Were performance evaluation (PE) samples performed?		ļ	<u> </u>	
Were the performance evaluation (PE) samples within the acceptance limits?				$\vdash$
Kinnemal standards		90. SP		
Were internal standard area counts within -50% or +100% of the associated calibration standard?	/	-		
Were retention times within + 30 seconds of the associated calibration standard?	/			
Xistangaredhiooundisemtications in the second state of the second s				<b>2443年1月1日,新聞会社会社会社会社</b>
Were relative retention times (RRT's) within ± 0.06 RRT units of the standard?				
Did compound spectra meet specified EPA "Functional Guidelines" criteria?			1	
Were chromatogram peaks verified and accounted for?		-		
All Compound Iquannian on/PROLS A Second				这个。这种的问题,如此的" <u>这个</u> "的
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?		_		
		200 C 200	977 NE 7 X 2	
XIIVEEDAIIVAVAUAUIIIAACOMECINOSI(IISS) variaties and serves serves serves as a serve serves and serv				
XIII tertalized value the documpoints (1953) constant a second se		-		
Were the major ions (> 10 percent relative intensity) in the reference spectrum				
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		-		
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? Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?				
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? Did the raw data indicate that the laboratory performed a library search for all				
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? Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?				
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? Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)? We system performance System performance was found to be acceptable.				
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? Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)? We system performance System performance was found to be acceptable.				
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? Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)? System performance was found to be acceptable. System performance was found to be acceptable.				
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)? System performance was found to be acceptable. Overall assessment of data was found to be acceptable.				
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? Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)? System performance was found to be acceptable. System performance was found to be acceptable.				
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)? System performance was found to be acceptable. Overall assessment of data was found to be acceptable.				
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)? System performance was found to be acceptable. System performance was found to be acceptable. System to f data was found to be acceptable. System performance was found to be acceptable. System performance was found to be acceptable.				

TARGET COMPOUND WORKSHEET

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

A. Chioromethane*	U. 1,1,2-Trichloroethane	00. 2,2-Dichloropropane	III. n-Butylbenzene	CCCC.1-Chlorohexane
B. Bromomethane	V. Benzene	PP. Bromochloromethane	JJJ. 1,2-Dichiorobenzene	DDDD. Isopropyl alcohol
C. Vinyl choride**	W. trans-1,3-Dichloropropene	QQ. 1,1-Dichloropropene	KKK. 1,2,4-Trichlorobenzene	EEEE. Acetonitrile
D. Chloroethane	X. Bromoform*	RR. Dibromomethane	LLL. Hexachiorobutadiene	FFF. 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. Tetrachioroethene	UU. 1,1,1,2-Tetrachioroethane	000. 1,3,5-Trichlorobenzene	IIII. Isobutyl alcohol
H. 1,1-Dichloroethene**	BB. 1,1,2,2-Tetrachioroethane*	VV. Isopropylbenzene	PPP. trans-1,2-Dichloroethene	JJJJ. Methacrylonitrile
I. 1,1-Dichloroethane*	CC. Toluene**	WW. Bromobenzene	QQQ. cis-1,2-Dichloroethene	KKKK. Propionitrile
J. 1,2-Dichloroethene, total	DD. Chlorobenzene*	XX. 1,2,3-Trichloropropane	RRR. m,p-Xylenes	LLLL. Ethyl ether
K. Chloroform**	EE. Ethylbenzene**	YY. n-Propylbenzene	SSS. o-Xylene	MMMM. Benzyl chloride
L. 1,2-Dichloroethane	FF. Styrene	ZZ. 2-Chlorotoluene	TTT. 1, 1, 2-Trichloro-1, 2, 2-trifluoroethane	NNNN.
M. 2-Butanone	GG. Xylenes, total	AAA. 1,3,5-Trimethylbenzene	UUU. 1,2-Dichlorotetrafluoroethane	0000.
N. 1,1,1-Trichloroethane	HH. Vinyl acetate	BBB. 4-Chlorotoluene	VVV. 4-Ethyltoluene	рерр.
O. Carbon tetrachloride	II. 2-Chloroethylvinyl ether	CCC. tert-Butylbenzene	WWW. Ethanol	<u>aaaa.</u>
P. Bromodichioromethane	JJ. Dichlorodifluoromethane	DDD. 1,2,4-Trimethylbenzene	XXX. Di-isopropyl ether	RRR.
Q. 1,2-Dichloropropane**	KK. Trichlorofluoromethane	EEE. sec-Butylbenzene	YYY. tert-Butanol	SSSS.
R. cis-1,3-Dichloropropene	Lt Methyl-tert-butyl ether	FFF. 1,3-Dichlorobenzene	ZZZ. tert-Butyl alcohol	TTTT.
S. Trichloroethene	MM. 1,2-Dibromo-3-chloropropane	GGG. p-lsopropyltoluene	AAAA. Ethyl tert-butyl ether	ບບບບ.
T. Dibromochloromethane	NN. Methyl ethyl ketone	HHH. 1,4-Dichlorobenzene	BBBB. tert-Amyl methyl ether	www.

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

COMPNDL. 1sb.wpd

	5
0110	cone
190	3
#	] ₩
LDC	SDG

### VALIDATION FINDINGS WORKSHEET **Initial Calibration**

Page: / of / 9 Ø Reviewer:

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

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

	Qualifications	J/11/1														
		FX F180000-29/	+ A/1 50:75													
RSD and ≥0.05 RRF ?	Finding RRF (Limit: <u>&gt;</u> 0.05)	0.0022/								-						
i criteria / ion criteria of ≤30 %f	Finding %RSD (Limit: <30.0%)															
I meet the acceptance RFs within the validat	Compound	mm														
UID the initial calibration meet the acceptance criteria / Were all %RSDs and RRFs within the validation criteria of ≤30 %RSD and ≥0.05 RRF ?	Standard ID	FKAL-BRC		-												
	Date	6/2/08				 						 				 
1	) #															

INICAL.1SB

Lee cones 1816061 LDC #: SDG #:

## VALIDATION FINDINGS WORKSHEET **Continuing Calibration**



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

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

Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's ? Were all %D and BBEs within the validation criteria of 735 %D and 50 05 BBE ? Was a continuing calibration standard analyzed at least once every 12 hours for each instrument? N/A N/A

	Qualifications	J+/A det	J-/4J/A		Jr/Adel		1/4J/A											
	Associated Samples	All water f	F8F20000-1x	,	1	 -	A// So: 17	F&F 180000-29	^									
עטט אארי ג	Finding RRF (Limit: <u>&gt;</u> 0.05)						0.00204											
	Finding %D (Limit: ≤25.0%)	5/529.18	25. 04176		67.71684													
	Compound	Icdo no thank	4	- 1/ /	Iodome thank		5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5											
	Standard ID	1836 1217			1CAL @317		FCAL 1777 BKC											
	ate	\$/24/08	,		6/19/0X	 	6/16/00	•								 		
-	2 #	4	1	!·	Ł													

LDC # <u>14071</u> 0/ SDG # <b>Lee Coren</b>		VALIDATION FINDINGS WORKSHEET	FINDINGS WC	<b>JRKSHEET</b>		Page: of <u></u> Reviewer: <b></b>
D: GC/	MS VOA (EPA SW846 Method 8260B) Were field blanks identified in this SDG?	62				2nd Reviewer:
<u>Y N N/A</u> Were target of Blank units: <u>n3/b</u> Assoc	Were target compounds detected in the field blanks? はんし、Associated sample units: <u>いまん</u> 子	the field blanks?			-	
Sampling date: <u>L////</u> 2 Field blank type: (circlé one) Field Blank / Rinsate / Trip Blank / Other:	) Field Blank / Rinsate /	Trip Blank / Other:	TB A:	Associated Samples:	All soils (ND)	(MD)
Compound	Blank ID			Sample Identification		
	ح					
Methylene chloride						
Acetone	1./					
C <del>hlorefor</del> m						
		-				
	-					
CRQL						
CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT: Common contaminants such as Methylana chlorida. Acatoma and Carbon disultida that ware detacted in samples within tan times the associated field have concentration were qualified as not	NALIFIED. ALL RESULTS N Mulana chinida Acatona 2-B	IOT CIRCLED WERE QUAL	FIED BY THE FOU	OWING STATEMENT:	and field field for the second second	4 F-911

3

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

~

SDG #: COURT		Field Blanks		Reviewer:
METHOD: GC/MS VOA (EPA SW 846 Method 8260B) <u>Y N/NA</u> Were field blanks identified in this SDG?	hod 8260B) I in this SDG?			
Y N N/A Were target compounds detected in the field blanks? Blank units: <u>vg /L</u> Associated sample units: <u>vg //</u> Field blank type: (circle one) Field Blank / Rinsate / Trip Blank / Other: P = R/nSG/P	stected in the field blanks? e units: <u>47</u> /7 Rinsate / Trip Blank / Other:	R= Rinsafe / Associate	/ A //	A/1 80:1/5 CND)
Compound Blank ID R	Blank ID		Sample Identification	
X0/11/9				
Pi'c h lorone than 3.3				
Acetone				
Chtoroform				
CROL				
Blank units: Associated sample units: Associated sample units: Associated Sample units: Eield blank type: (circle one) Field Blank / Rinsate / Trip Blank / Other	e units: ' Rinsate / Trip Blank / Other:	Associat	Associated Samples:	
Compound Blank ID	Blank ID		Sample identification	
Supervision of the supervision o				
Methylene chloride				
Acetone				
Chloroform				
CRAL				
CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:	RESULTS NOT CIRCLED WERE ON		G STATEMENT:	

2:54

Common contaminants such as Methylene chloride. Acetone, 2-Butanone and Carbon disulfide that were detected in samples within ten times the associated field blank concentration were qualified as not detected, "U". Other contaminants within five times the field blank concentration were also qualified as not detected, "U".

FBLKASC2.1SB

181	9.02
82606	22
·/-:#	;#
ğ	5DG

## VALIDATION FINDINGS WORKSHEET Surrogate Spikes

0 /of 2nd Reviewer: Page: Reviewer:

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

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". <u>Y NDN/A</u> 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? Z

Qualifications	J+/Patt																					
(Limits)	( 19-115)	( )	)	( )	(	)	( )	( )	( )	( )	( )	( )	(	( )	) (	( )	) (	] ( )	( )	( )	) (	
%Recovery (Limits)	117																					<u>QC Limits (Water)</u> 88-110
Surrogate	BFB																					SI
Sample ID	F8 F2 00000-12																					QC Limits (Soil) d8 81-117
Date																						SMC1 (TOL) = Toluene-d8
#																						SMC1 (

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

14126061	he cones
*	#
ğ	SDG

## VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

ر م Page: 2nd Reviewer: Reviewer:

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

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

A/A

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?

Date	DI DSM/SM	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
	647	<b>AA</b>	(	(D2/-33-15D)	())	# /	U idjew moto on
		HH	( )	( )	( 02 ) 82	1	u:d/sw t1
			(	( )	~		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )			
	Pinsai 2ms 10		(	( )	(01)	hre	no que
				( )	(		
			( )	( )			
			( )	)	(		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			(	(			
			)	( )	( )		-
			(	(	( )		
			).	)	( )		
			( )	( )	( )		
	Compound	Ind	ac tir	C Limits (Soil)	RPD (Soll)	QC Limits (Water)	RPD (Waler)
ŗ	1,1-Dichloroethene		95 	36-172%	< 22%	61-145%	34 <b>7</b>
Ś	Trichloroethene				<u>&lt;</u> 24%	71-120%	×. ₹
<u>&lt;.</u>	Benzene				× 21%	76-12.7%	
oc.	Toluene				10 + C		
c	Chiombanzana						

MSD.1SB

ŝ

1011101	Les could
	sDG #:

## VALIDATION FINDINGS WURNDREL Laboratory Control Samples (LCS)

rage: / ol / 2nd Reviewer: Reviewer:

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

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

AN A/A >

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

	2															,						_		
Qualifications	no quest carps	1/ ms/p:n				-																		
Associated Samples	Al walers	521-0000 - 182																						
RPD (Limits)	1/2 (20)	· ( )	( )	( )	)	( )	( )	( )	)	( )	( )	( )	( )	( )	())	(	( )	( )	( )	(			(	()
LCSD %R (Limits)	( )	( ahl-sh ) / 21		( )	(	( ) )	( )	( )	( )	( )	( )	( )	( )	()	(	( )	(	( )	( )	( )	( )	( )	( )	(
LCS %R (Limits)	893 ( 42-140)	Locknothan 166 (45-140)	( )	( )	(	(	(	(	(	( )	( )	( )	( )	( )	( )	( )	( )	(	(	(	( )	( )	(	(
Compound	Y	Loobus than									_													
rcs/rcsp iD	Class scielis																							
Date																								
*																								

LCSLCSD.1SB

cover 191909181 22 SDG #: LDC #:

# VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

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

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

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

 $\label{eq:RFF} RRF = (A_x)(C_x)/(A_x)(C_x) \\ average \ RRF = sum \ of the \ RRFs/number \ of standards \\ %RSD = 100^{-\epsilon} (S/X) \\ \end{cases}$ 

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

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

#         Standard ID         calibration Date         calibration compound (reference internal standard)         RRF ( $S^2$ etc)         RRF ( $S^2$ etc)         RRF ( $S^2$ etc)         Average RRF ( $S^2$ etc)         Average RRF (initial)         Average RRF (initial)         Average RRF ( $S^2$ ( $S^2$ etc)         A					Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
$   \begin{array}{ c c c c c c c c c c c c c c c c c c c$	*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	RRF (57 std)	RRF (ک) std)	Average RRF (initial)	Average RRF (initial)	%RSD	%RSD
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	-	ICAL-F	60/6/9	viny (ch for tolk (1st internal standard)		0. 38029	0.40478	0.40678	13.28749	13.27
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$					2.07630	2.036>		E01161	9.6757	
				_	1.42 JSB	1.42753		1/3011	5.1783	L
	7			(1st internal standard)						
				(2nd internal standard)						
				(3rd internal standard)						
	e			(1st internal standard)						
	Τ			(2nd internal standard)						
				(3rd internal standard)						
(2nd internal standard)	4			(1st internal standard)						
(3rd internal standard)				(2nd internal standard)						
				(3rd internal standard)						

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

19097B eore 2 LDC #: SDG #:

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

đ 2nd Reviewer: 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\_x)(C\_s)/(A\_s)(C\_x)

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

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

6.95693 6.66812 21428.8 Serer. Recalculated 0% 26926 5 6.668/3 1.22 825 2.32412 Reported ۵% 1.44262 **Recalculated** 9.1088b 451e2.0 0.38285 RR 5 5 1 0. 38 205 124242 9.801.6 Reported P2/27.0 RRF (CC) Average RRF (initial) PILSCAI 60776.1 0.40478 17861.0 Compound (Reference internal Standard) (2nd internal standard) (2nd internal standard) (3rd internal standard) (2nd internal standard) (3rd internal standard) (2nd internal standard) (1st internal standard) (3rd internal standard) (1st internal standard) (1st internal standard) (3rd internal standard) (1st internal standard) Dim = Hyy yent 222 יבא EF J 116/08 Calibration 6/16/08 Date FCAL1777 FCALITIS Standard ID BRC # 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 #: 19097B/ SDG #: <u>per coner</u>

### VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page:_	<u>_/_of_/</u>
Reviewer:	<u>*7</u>
2nd reviewer:_	́^
	Ý

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

### Sample ID: 2

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8	\$19.0	46.9579	94	94	0
Bromofluorobenzene	4	46.9877	94	94	1
1,2-Dichloroethane-d4		47.6177	95	95.	
Dibromofluoromethane		47.0909	94	94	

### Sample ID:

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

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

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

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

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

### Sample ID:

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

LDC #: 1909713/ SDG #: pu cover

VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates Results Verification

Ņ of a 2nd Reviewer: Page: Reviewer:

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

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

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

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

MSC = Matrix spike percent recovery

MSDC = Matrix spike duplicate percent recovery

SC = Sample concentration

MS/MSD sample:  $b \leftarrow 7$ 

	S	Spike	Sample	Spiked Sample	sample	<u>Matrix Spike</u>	Spike	Matrix Spike Duplicate	Duplicate	WS	MS/MSD
Compound	8 3 )	Added wy My	Concentration	Concentration	iration 152	Percent Recovery	ecovery	Percent Recovery	ecovery	-	RPD
	WS	MSD		SM	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
1,1-Dichloroethene	53 3	53.9		6.2%	47.3	₽\$¢	87	Z2	z	ð, Ó	2.0
Trichloroethene				5.5.5	58.7	104	104	601	601	2:0	5.6
Benzene				7.64	s:02	33	93	76	44	2 N	2.2
Toluene				e.ar	51.2	hb	76	56	56	1.7	1.7
Chlorobenzene				49.7	5/.3	33	66	95	95	2.5	3.2

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

1826061	conor
190	Z
LDC #:_	SDG #:

# VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Results Verification

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

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

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

% Recovery = 100 \* SSC/SA Where: S

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

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

CSID: 8/7029/ - 102

LCSD = Laboratory control sample duplicate percent recovery

	Sp	Spike	Spiked S	ample		S	I CSD		1 CS/I	CS/LCSD
Compound	Adde ( 1097 /	lleg	Concentration	tration	Percent Recovery	kecovery	Percent Recovery	ecovery	RPD	0
	1 CS	1 CSD	1 CS		Renorted	Recalc	Renorted	Recalc	Renorted	Recalculated
1,1-Dichloroethene	RD	ANOS	47. K	k A	76	46				
Trichloroethene	/		5.67		66	66				
Benzene			1 64		66	99				
Toluene			2.25		001	Cal				
Chlorobenzene	Ţ	1	49.9		100	001	NA NA			
-										
		1								
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	y Control Sa	mple findings	worksheet for	list of qualifi	cations and a	ssociated sa	mples when re	sported result	s do not agree wi	thin 10,0% of the

LCSCLC.1SB

recalculated results.

LDC #: 19097B/ SDG #: <u>pu coner</u>

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	/_of	_
Reviewer:	P1	
2nd reviewer:	0/	_
	V	

METH Y N Y N	N/A	GC/MS VOA (EPA SW 846 Method 8260B) Were all reported results recalculated and v Were all recalculated results for detected ta	verified for all level IV samples? arget compounds agree within 10.0% of the reported results?
Conc	entratio	$n = \frac{(A_x)(I_s)(DF)}{(A_s)(RRF)(V_o)(\%S)}$	Example:
A <sub>x</sub>	=	Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D;;
$A_{is}$	=	Area of the characteristic ion (EICP) for the specific internal standard	
۱ <sub>s</sub>	=	Amount of internal standard added in nanograms (ng)	Conc. = $($ ) $($ ) $($ ) $($ )
RRF	=	Relative response factor of the calibration standard.	$\sim$
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.	

#	Sample ID	0	Reported Concentration	Calculated Concentration	
<del> </del>		Compound			Qualification
			······································		
	· ·	· · · · · · · · · · · · · · · · · · ·			
	an an Anna Anna Anna Anna Anna Anna Ann				
	·····				

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

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

LDC Report Date: July 23, 2008

Matrix: Soil

Parameters: Semivolatiles

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120167

### Sample Identification

TSB-GJ-08-10 TSB-GJ-08-20\*\* TSB-GJ-08-30\*\* TSB-GJ-08-40 TSB-GJ-08-10MS TSB-GJ-08-10MSD

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 6 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).

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

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

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

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
6/18/08	Phthalic acid	0.01422 (≥0.05)	All samples in SDG F8F120167	J (all detects) UJ (all non-detects)	А
	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	25.06818	TSB-GJ-08-30** TSB-GJ-08-40	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)	TSB-GJ-08-10 TSB-GJ-08-20** TSB-GJ-08-10MS TSB-GJ-08-10MSD F8F160000-439	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-08-30** TSB-GJ-08-40	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.

Sample "RINSATE 1" (from SDG F8F120137) was identified as a rinsate. No semivolatile contaminants were found in this blank.

### VI. Surrogate Spikes

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

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

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

Sample	Internal Standards	Area (Limits)	Compound	Flag	A or P
TSB-GJ-08-30**	1,4-Dichlorobenzene-d4 Perylene-d12 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12	53781 (82431-329724) 25394 (281395-1125580) 201776 (303781-1215124) 101990 (159543-638172) 150470 (271508-1086030) 72798 (268054-1072214)	All TCL compounds	J (all detects) UJ (all non-detects)	A
TSB-GJ-08-40	Perylene-d12	197078 (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)

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

### **XIV. System Performance**

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

### XV. Overall Assessment of Data

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

### XVI. Field Duplicates

No field duplicates were identified in this SDG.

### BRC Tronox Parcel G Semivolatiles - Data Qualification Summary - SDG F8F120167

SDG	Sample	Compound	Flag	A or P	Reason
F8F120167	TSB-GJ-08-10 TSB-GJ-08-20** TSB-GJ-08-30** TSB-GJ-08-40	Phthalic acid n-(Hydroxymethyl)phthalimide	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A	Initial calibration (RRF)
F8F120167	TSB-GJ-08-30** TSB-GJ-08-40	Phthalic acid	J- (all detects) UJ (all non-detects)	A	Continuing calibration (%D)
F8F120167	TSB-GJ-08-10 TSB-GJ-08-20** TSB-GJ-08-30** TSB-GJ-08-40	Phthalic acid n-(Hydroxymethyl)phthalimide	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	<b>, A</b>	Continuing calibration (RRF)
F8F120167	TSB-GJ-08-30**	All TCL compounds	J (all detects) UJ (all non-detects)	А	Internal standards (area)
F8F120167	TSB-GJ-08-40	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 F8F120167

No Sample Data Qualified in this SDG

### BRC Tronox Parcel G Semivolatiles - Field Blank Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

### VALIDATION COMPLETENESS WORKSHEET

LDC #: <u>19097B2</u> SDG #: <u>F8F120167</u> Laboratory: Test America

### Level III/IV

	Date:	7	/21/	08
	Page:_		f/	,
	Reviewer:		17	
2nd	Reviewer:		q-	
			1	

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
١.	Technical holding times	А	Sampling dates: 6/11/08
11.	GC/MS Instrument performance check	Δ	
III.	Initial calibration	SW	°/0 psp, r2 Z0,990
IV.	Continuing calibration/ICV	SW	
V.	Blanks	A	
VI.	Surrogate spikes	Δ	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	دىت	LCS
IX.	Regional Quality Assurance and Quality Control	N	
Х.	Internal standards	SU	
XI.	Target compound identification	۵	Not reviewed for Level III validation.
XII.	Compound quantitation/CRQLs	A	Not reviewed for Level III validation.
XIII.	Tentatively identified compounds (TICs)	Å	Not reviewed for Level III validation.
XIV.	System performance	A	Not reviewed for Level III validation.
XV.	Overall assessment of data	A	
XVI.	Field duplicates	N	
XVII.	Field blanks	ND	R = Rinsate / SDG F8F120137

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

	<u>soil</u>				
Ŧ	TSB-GJ-08-10	11		21	31
2	TSB-GJ-08-20**	12		22	 32
$\frac{1}{2}$ $\frac{1}{3}$ $\frac{1}{4}$	TSB-GJ-08-30**	13		23	33
+ 4	TSB-GJ-08-40	14		24	34
5	TSB-GJ-08-10MS	15		25	35
6	TSB-GJ-08-10MSD	16		26	36
7	F8F160000-439	17	8168439	27	37
8		18		28	38
9		19		29	39
10		20		30	40

LDC #:\_\_\_ SDG #:\_\_\_ 19097B2 per cover

### **VALIDATION FINDINGS CHECKLIST**

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

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

Validation Area	Yes	No	NA	Findings/Comments
All technical holding times were met.	Ĺ			
Cooler temperature criteria was met.	/	ſ		
IN STANDARD PROPAGATION CONTRACTOR				
Were the DFTPP performance results reviewed and found to be within the specified criteria?	-	+		
Were all samples analyzed within the 12 hour clock criteria?		1		
Mercinol-submissi				
Did the laboratory perform a 5 point calibration prior to sample analysis?	$\leq$			
Were all percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?	_			
Was a curve fit used for evaluation?				
Did the initial calibration meet the curve fit acceptance criteria of $\geq$ 0.990?		-		
Were all percent relative standard deviations (%RSD) $\leq$ 30% and relative response factors (RRF) $\geq$ 0.05?		/		
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?				narana na historia da barana kana na na na da balan na da baha na historia da baran da baha da baha na baha da Internet da baha
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?	$\backslash$	-		
Were all percent differences (%D) $\leq$ 25% and relative response factors (RRF) $\geq$ 0.05?		/		
Was a method blank associated with every sample in this SDG?	_	-		
Was a method blank analyzed for each matrix and concentration?	_			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		11-12-1-14-14		
Were all surrogate %R within QC limits?	_	_		
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?			4	-
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?			and the second	
A spannacker and set and the set of the set				
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?	1			
W. LONG 20 No. 19 NO. STREET				
Was an LCS analyzed for this SDG?	$\square$			

.

	Page:_	<u></u>	2
	Reviewer:	P	7
2nd	Reviewer:	9	-

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?		-	-	
PC Restant Train Assessed State Cranges and				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
Contract Statements And				
Were internal standard area counts within -50% or +100% of the associated calibration standard?	8	1		
Were retention times within + 30 seconds from the associated calibration standard?				
Ker Terretaringeringer Benniteringer				
Were relative retention times (RRTs) within + 0.06 RRT units of the standard?				-
Did compound spectra meet specified EPA "Functional Guidelines" criteria?			-	_
Were chromatogram peaks verified and accounted for?		_		
AN SULUERINARIZINARING A SCALE				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?			4	-
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	+			
MD TELEVISION AND AND AND AND AND AND AND AND AND AN				
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?	ł	_		
Were relative intensities of the major ions within $\pm$ 20% between the sample and the reference spectra?	7	-		
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?				
System performance was found to be acceptable.				
	1			
Overall assessment of data was found to be acceptable.	$\leq$			
			i in a a carta	
Field duplicate pairs were identified in this SDG.		$\leq$	-	·
Farget compounds were detected in the field duplicates.			+	
Field blanks were identified in this SDG.		and the second	ener ildere het e	en e en estador las estados e tomas a famolas de las dels fois de stados, en termo e en estado con contra en es
arget compounds were detected in the field blanks.		7		

1

VALIDATION FINDINGS WORKSHEET

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

A. Phenol**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachiorophenoi**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroanline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	VV. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichiorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenoi	XX. Di-n-butyiphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene*⁺	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	000. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	TTT.
M. Isophorone	BB. 2-Nitroanlline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	UUU
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	ww.
0. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachiorobenzene	HHH. Benzo(k)fluoranthene	www.

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

709782	Le coner
2/	
#	#
g	SDG #

### VALIDATION FINDINGS WORKSHEET **Initial Calibration**

ō Page:

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

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". MAN N/A Did the laboratory conduct an acceptable 5 point calibration prior to sample analysis? YN NA YN NA

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

Was a curve fit used for evaluation? If yes, what was the acceptance criteria used for evaluation? Did the initial calibration meet the acceptance criteria? Y/N N/A Y N N/A

	<b></b>	1		T	-	-	-	-	1	 	-	 1	-	÷	 	 	 	-	 -	-		-	
	Qualifications	J/4J/4																					
	Associated Samples	A/1 +B/	7																				
nd ≥0.05 RRF ?	Finding RRF (Limit: ≥0.05)	0.01422	0. D 4408																				
criteria of ≤30 %RSD a	Finding %RSD (Limit: <u>≤</u> 30.0%)		Ŋ																		•	-	
is within the validation o	Compound	Ph tha lic Acid	hy out & arph H)-N	Ph thall mide	/																		
Were all %RSDs and RRFs within the validation criteria of ≤30 %RSD and ≥0.05 RRF ?	Standard ID	JICAL.SPEC																					
V N/A V	te	6/18/08																					
<del>ال</del> ر	) *																						

1999 - J. J.

14097132 the coner LDC #: / SDG #:

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

2nd Reviewer: Reviewer: Page:

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

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

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

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

 Y N N/A V N/A

	Qualifications		1/47/4		7			-	1/uu/A		J-/4J A											
	Associated Samples	F &FI 60000-439,	1, 2, 5, 6	4				ح ر/		Ń												
KRF ? Finding RRF	(Limit: >0.05)							0.01066	C 00170 0	C7240.0												
a <u>or ≾∠5 %U and ≥0.05 RRF ?</u> Finding %D F	(Limit: <25.0%)		ļ	1) 0.043311						20 11 11	01800.00											
	Compound	1 1 1 10	VN Mall G Hud	Howha wohth )-N	ghthall midy	/		/	1	Ph thalic. Rind	the solution of											
	Standard ID	1011 101	16107407					1eAL5229														
	# Date	1 110/00	Qn/x1/a					80/61/9		1												

CONCAL.2S

SDG #: Fer cours

## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

•

d ď Page: 2nd Reviewer: Reviewer: \_

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

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

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

		_	-	-	-	-	-	-	_		-	-				_	 		_								
			-/ Pial P	1.d/sm /and cu																							
	Associated Samules		VIG + IIH																								
	RPD (Limits)		-	(	( )	( 	-	(	( )	()	~	(		· · ·					~ ~	)	(	(	<b>^</b>	( )			)
-	LCSD %R (Limits)	(		<b>^</b>	(	( )	( )	( )	~	( )	(	( )			, - ,	) ~					~	(	( )	(	(	~	)
	LCS %R (Limits)	(16-75) 61			~	<b>^</b>	(	( )	( )	( )	)	( )	~ ~	(			`````			-	~ ~	<b>^</b>	- -	(	( )	(	( )
	Compound	НН																									
	rcs/rcsd ID	8168439-10>																									
	Date																										
2	*			1		T												$\uparrow$	$\uparrow$	╢	1		+				

LCSLCSD.2S

LDC #:   SDG #:		19097BJ	٩٧	VALIDATION FINDINGS WORKSHEET Internal Standards	SHEET	Page: of A	·
METH Please	ETHOD: GC/M See quality N N/A	METHOD: GC/MS BNA (EPA SW 846 Method 8270) Please qualifications below for all questions answered "N". Not ap <u>X (N N/A</u> Were all internal standard area counts within -50 to + <u>Y N N/A</u> Were the retention times of the internal standards with	rod 8270) tions answered "N" area counts within - f the internal standa	VS BNA (EPA SW 846 Method 8270) iffications below for all questions answered "N". Not applicable questions are identified as "N/A". Were all internal standard area counts within -50 to +100 of the associated calibration standard? Were the retention times of the internal standards within +/- 30 seconds of the retention times of the associated calibration standard?	ified as "N/A". ation standard? tention times of the associated cali	2nd Reviewer:	
*	Date	Sample ID	Internal Standard	Area (Limits)	RT (Limits)	Qualifications	<u></u>
		6	вра	53781 (82431 - 324	( hel f	111 1 mm 4/ 5 mm 4/1	22
			РРУ	-56E187/16Esc 8to Lot	(0858711-56k	1	
			NPT	201776 (203781-1215124	(1-21-2-1)		
			ANT	159543-	638172)		
			PHN	=1-Basize ) 02 hasi	1686030)	<b>*</b>	
			ery	$\sim$	( //224		
		×		$\left  \right $			
		~					
		4	PRY	.26218c / 820261	+// 25560)	J/45/4 044)	
							L
÷ 0 *	* OC limite are advison						_

\* GC 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

INTST.2S

SDG #: Les gones LDC # 1909782

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

5 R Page: / of , 2nd Reviewer: Reviewer:

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

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

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

l

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

A<sub>s</sub> = Area of associated internal standard

intration of internal standard	f the RRFs
C <sub>is</sub> = Concentration	X = Mean of the R

Rended         Rend         Rend         Rend	
RRF         RRF         Average RRF         Average RRF         "ARD $(S^{20}$ std) $(J^{2}C)$ std)	
1.87x53       1.87x53       1.87x53       1.87x53       1.87x53       1.670/       1.970/       1.970/       1.32         1.0943¥       1.0943¥       1.1090/       1.1090/       1.1090/       1.925       0.532       0.533         1.111       1.4171       1.41229       0.1943       0.543       0.533       0.533         0.20260       0.20260       0.20263       0.1943       0.543       9.534         1.1350%       1.1350%       1.1182       1.1182       6.1486         1.1350%       1.1182       1.1182       6.1486         0.51976       0.51976       0.5177       0.71577       0.71577         0.51976       0.5177       0.57777       0.7158       8.41339         0.04162       0.04162       0.04162       0.04183       8.41339	Calibration         Calibration           Standard ID         Date         Compound (Reference Internal Standard)
1.0943 X       1.0943 X       1.0943 X       1.0943 X       1.990/       1.909/       1.332         1.171 X       1.4171 X       1.4129       0.533       0.533       0.533       0.533         0.20260       0.20260       0.20263       0.90763       0.90763       0.953       9.534         0.20260       0.20263       0.90763       0.863       9.534       9.534         1.13260       1.182       1.182       1.182       6.48       9.534         0.51976       0.51974       0.51974       0.71/51       0.71/51         0.51976       0.51974       0.51774       0.71/51       0.71/51         0.01/62       0.51774       0.51774       0.71/51       8.41/339	1.09.1 - 3.07.09 Phenol (1st internal standard)
1. 4/71 X       1. 4/71 X       1. 4/11 X       1. 4/11 X       1. 4/11 X       1. 4/11 X       0. 741 × 10       0. 57-3       0. 54-3 <t< td=""><td>Naphthalene (2nd internal standard)</td></t<>	Naphthalene (2nd internal standard)
0.257       0.7434       0.7434       0.257         0.7063       0.70763       0.8343       9.524         0.7063       0.7874       0.8543       9.524         1.13508       1.1822       1.182       6.483         1.182       1.182       6.484       6.484         0.51976       0.51976       0.7157       6.486         0.51976       0.51976       0.7157       6.484         0.51976       0.51976       0.7157       6.486         0.51976       0.51976       0.7157       6.486         0.51976       0.51976       0.7157       6.486         0.51976       0.51976       0.7157       6.41339         0.04162       0.04408       8.41339       8.41339	Fluorene (3rd internal standard)
Controls 0.90%3 CSA3 CSA3 CSA3 CSA24 1.13562 1.1350 1.1210 0.51976 0.51217 0.51274 0.51374 0.51716 0.51976 0.51217 0.51217 0.51716 0.51976 0.51217 0.51239 0.04162 0.04162 0.04123 0.04162 0.04162 0.04162 0.04162 0.04163 0.0416	Pentachlorophenol (4th internal standard)
0.51976 0.51976 1.11/8 1.11/8 0.51374 0.71/51 0.51976 0.51976 0.51274 0.51737 0.21976 0.21976 0.51274 0.517359 0.24162 0.24162 0.24193 0.241339	Bis(2-ethylhexyl)phthalate (5th internal standard)
0.7/57/ 2. 1/2/ 2.	11
0.04162 0.04408 8.4/339	LCHL-JAPPa 1/2 / 1
0. evyox	Naphthalene (2nd internal standard)
0.04/b3	Fluorene (3rd internal standard)
0.04162 0.04162	Pentachlorophenol (4th internal standard)
0. evites	Bis(2-ethylhexyl)phthalate (5th internal standard)
D. D. U. V.	
	1 CHL-JSPEC Phenol (13 internal standard) 7 Dr Hali mid C
	Naphthalene (2nd internal standard)
	Fluorene (3rd internal standard)
	Pentachlorophenol (4th internal standard)
	Bis(2-ethylhexyl)phthalate (5th internal standar
	Benzo(a)pyrene (6th 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

1909782	Les coner
LDC #:	SDG #:

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

Ġ Page: Reviewer: 2nd Reviewer:

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

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

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF = (A\_x)(C\_a)/(A\_a)(C\_x)

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

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

*					Reported	Recalcutated	Renorted	Recalculated
+ +	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q\$%	Q%
-	JCAL5195	6/18/03	Phenol (1st internal standard)	1. 855 37	1-87174	1-8717	0. 8×21U	0.821
╉			Naphthalene (2nd internal standard)	1.10 901	1.10/35	101.1	0.69070	0.6907
╈			Fluorene (3rd internal standard)	1.41229	10865-1	RE.1	1. 0/05	10/01
			Pentachlorophenol (4th internal standard)	0.19634	010370	0.2037	3.74920	3.749
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0-56343	0.870 82	0.879	0.8222	0-822
╢			Benzo(a)pyrene (6th internal standard)	1.11182	1-11 507	1.1151	0.27280	0.2728
2	7615107	80/81/2	Phenol (1st internal standard)	0.51274	0.52/85	0.52/27	1.77632	(-776 2 2
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
4	16/57801	6/18/00	Rentachlorophenol (411) internal (Sandard)	1 0.04408	0.0433/	0.0133/	1.72419	618261
╉			Bis(2-ethylhexyl)phthalate (5th internal standard)				, 102 / . 1	1 100 1-1
			Renzo(a)pyrene (6th internal standard)					
е М			Phenol (1st internal standard)					
╉			Naphthalene (2nd internal standard)					
╉			Fluorene (3rd internal standard)					
╉			Pentachlorophenol (4th internal standard)					
+			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					

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

And or LDC #: 1909782 SDG #:

**Continuing Calibration Results Verification** VALIDATION FINDINGS WORKSHEET

∕ of Page: / Reviewer: 2nd Reviewer:

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

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

Where: % Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF = (A\_x)(C\_s)/(A\_s)(C\_x)

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

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

					Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q%	Ω%
	Leest 425	119/08	Phenol (1st internal standard)	1. 85537	1.80162	1.2016	2-89721	2-897
			Naphthalene (2nd internal standard)	10601.1	212801	1.087	1.97399	1. 974
			Fluorene (3rd internal standard)	1.41239	1. 4087X	1.408	0. 24 8.53	0.2486
			Pentachlorophenol (4th internal standard)	0.19634	05208.0	0.2073	625 25-5	5-585-5
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.56343	R8/28-0	0-8719	0.97842	0.978
			Benzo(a)pyrene (6th internal standard)	1.11/82	1.12694	1.1267	1.36062	1-36/
2	JCALSZZ	6/19/0X		0.5/274	0.51326	0.5/33	0.10062	D.100 6
		•	Naphtbalene (2nd internal standard)					
	JCAL5229	X0/61/9	6/17/0X Howener (3rd interpal standard) / phthallmit 0. 04400	9 0. 04408	@.04Sa3	0.0452	2.61460	2-6/5
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			<u>Benzo(a)pyrene (6th internal standard)</u>					
m			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					

Comments: Refer to 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 #: 1909182

SDG #: ele comen

### VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

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

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Sample ID:  $\# \mathcal{L}$ 

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	50	33.3/03	67	67	0
2-Fluorobiphenyl		34.9043	70	70	
Terphenyl-d14	1	33.6734	67	67	1
Phenol-d5	75	48.9853	65	65	
2-Fluorophenol		48.52	45	65	
2,4,6-Tribromophenol	L	SD.6613	68	68	
2-Chlorophenol-d4			· · · · · · · · · · · · · · · · · · ·		
1,2-Dichlorobenzene-d4					

### Sample ID:

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

### Sample ID:

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

LDC #: 1909782 SDG #: per court

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

Page: / of / C 2nd Reviewer. Reviewer:

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

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

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

Where: SSC = Spiked sample concentration SA = Spike added

MS = Matrix spike percent recovery

RPD = I MS - MSD I \* 2/(MS + MSD)

MS/MSD samples: S + C

MSD = Matrix spike duplicate percent recovery

SC = Sample concentation

	S St	Spike Added	Sample	Spiked Sample	ample	Matrix Spike	Spike	Matrix Spike Duplicate	Duplicate	USM/SM	ds
Compound	, , ,	alka	( white here allow	Concentivation	uon	Percent Recovery	PCOVEDV	Dorocad D			
		δ	Ь						ecovery	RPD	
	WS	MSD		MS	MsD	Reported	Docalc	Tettorog	-		
					İΓ			Danootaa	Recal	Reported	Recalculated
Phenol	3570	2630	52	obhe	ashe	20	2	69	63	1.7	1.7
N-Nitroso-di-n-propylamine			-	2730	02.75	77	17	252	X	2.2	( ) · · ·
4-Chloro-3-methylphenol				2760	2690	17	17	54	75		1.
Acenaphthene				2640	21.20	76	17	7 ( - T		8.4	4.8
							5	c )	62	Ċ	Ċ
Pentachiorophenol				2300	2230	64	64	62	62	a, S	3.0
Pyrene	الا	$\checkmark$		2460	0622	69	61	67	1	) [   	1
				+     			à	<u>}</u>	•	, 8	0.
									**		

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

MSDCLC.wpd

LDC #: 1909187	SDG #: Are coner
Ц	Ŋ

# VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

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

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

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

% Recovery = 100 \* (SC/SA

SSC = Spike concentration SA = Spike added Where:

LCS = Laboratory control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery RPD = ILCS - LCSD I \* 2/(LCS + LCSD)

8168439-105 LCS/LCSD samples: \_

	s	ike '	Sp	Spike		CS				435
			- 							CS/LCSD
Compound	ри 9 И 9	(ug/bg	Concentration	itration	Percent Recovery	Recovery	Percent Recoverv	ecoverv	ŭ	Cax
	5	0								
	108	I CSD	ICS	1 CSD	Reported	Recalc	Reported	Recalc	Renorted	Recalculated
Phenol	3330	NД	2360	۸A ۸	1 4	lt.				
N-Nitroso-di-n-propytamine	-		2570	-	77	77				
4-Chloro-3-methylphenol			2260		77	11				
Acenaphthene			210		75	75				
Pentachlorophenol			Ohte		67	67				
Pyrene	~	<b>A</b>	Usee	J	04	70				

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 #: 19097B2 SDG #: <u>recener</u>

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

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

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



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?

Conc	entratio	on = $(A_{i})(I_{i})(V_{i})(DF)(2.0)$ $(A_{u})(RRF)(V_{u})(V_{i})(%S)$	Example:
A,	=	Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D;:
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. = $() () () () () ()$
V <sub>o</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)	N///
Df	=	Dilution Factor.	I IV P
%S	=	Percent solids, applicable to soil and solid matrices only.	
2.0	=	Factor of 2 to account for GPC cleanup	

### 

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

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

Collection Date: June 11, 2008

LDC Report Date: August 11, 2008

Matrix:

Parameters: Chlorinated Pesticides

Soil

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120167

### Sample Identification

TSB-GJ-08-10 TSB-GJ-08-20\*\* TSB-GJ-08-30\*\* TSB-GJ-08-40 TSB-GJ-08-10MS TSB-GJ-08-10MSD

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 6 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8081A for Chlorinated Pesticides.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

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

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

### III. Initial Calibration

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

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

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

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

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

### V. Blanks

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

Sample "RINSATE 1" (from SDG F8F120137) was identified as a rinsate. No chlorinated pesticide contaminants were found in this blank.

### VI. Surrogate Spikes

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

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Pesticide Cleanup Checks

### a. Florisil Cartridge Check

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

### b. GPC Calibration

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

### XI. Target Compound Identification

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

### XII. Compound Quantitation and Reported CRQLs

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

### XIII. Overall Assessment of Data

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

### **XIV. Field Duplicates**

No field duplicates were identified in this SDG.

BRC Tronox Parcel G Chlorinated Pesticides - Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

计分子操作性 化分子的 化氯化物 化化物化物 化化物化物 化化物化物 化化物化物化物化物化物化物

BRC Tronox Parcel G Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

BRC Tronox Parcel G Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

### VALIDATION COMPLETENESS WORKSHEET

LDC #: <u>19097B3a</u> SDG #: <u>F8F120167</u> Laboratory: <u>Test America</u>

### Level III/IV

Date: 1/21/08 Page: / of Reviewer: 2nd Reviewer

New work that a tradition of the Repair

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
١.	Technical holding times	A	Sampling dates: 6/11/08
11.	GC/ECD Instrument Performance Check	Δ	
111.	Initial calibration	A	
IV.	Continuing calibration/ICV	SIA	101 £15
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	Δ	
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	Δ	Not reviewed for Level III validation.
XII.	Compound quantitation and reported CRQLs	A	Not reviewed for Level III validation.
XIII.	Overall assessment of data	N	1
XIV.	Field duplicates	N	
XV.	Field blanks	ND	R = Rinsati / SDG # F8F/2013)

Note:

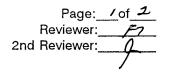
A = Acceptable N = Not provided/applicable SW = See worksheet ND = No compounds detected R = Rinsate FB = Field blank D = Duplicate TB = Trip blank EB = Equipment blank

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

1	TSB-GJ-08-10	11		21	31	
2	TSB-GJ-08-20**	12		22	32	
12 13 14	TSB-GJ-08-30**	13		23	33	
4	TSB-GJ-08-40	14		24	34	
5	TSB-GJ-08-10MS	15		25	35	
5 6	TSB-GJ-08-10MSD	16		26	36	
7	F8F160000-164	17	8168164	27	37	
8		18		28	38	
9		19		29	39	
10		20		30	40	

LDC #:\_\_\_\_\_\_ SDG #:<u>su coneg\_\_\_\_</u>

### VALIDATION FINDINGS CHECKLIST



-

### Method: Pesticides/PCBs (EPA SW 846 Method 8081/8082)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	/	f 		
Cooler temperature criteria was met.	/	F		
II. GC/ECD Instrument performance check				
Was the instrument performance found to be acceptable?	<	ł		
III. 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) $\leq$ 20%?	-	-		
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?		_	-	
Did the initial calibration meet the curve fit acceptance criteria?			/	-
Were the RT windows properly established?		-		
Were the required standard concentrations analyzed in the initial calibration?				
IV. Continuing calibration				
What type of continuing calibration calculation was performed?%D or%R	/	-		
Were Evaluation mix standards analyzed prior to the initial calibration and sample analysis?				
Were endrin and 4,4'-DDT breakdowns $\leq$ 15%.0 for individual breakdown in the Evaluation mix standards?	/	-		
Was a continuing calibration analyzed daily?		/		
Were all percent differences (%D) $\leq$ 15%.0 or percent recovieries 85-115%?		-	-	
Were all the retention times within the acceptance windows?	_	_		
V. Blanka				
Was a method blank associated with every sample in this SDG?	_			
Was a method blank analyzed for each matrix and concentration?	$\square$			
Were extract cleanup blanks analyzed with every batch requiring clean-up?			_	-
Was there contamination in the method blanks or clean-up blanks? If yes, please see the Blanks validation completeness worksheet.		_	_	
VI. Surrogate spikes				
Were all surrogate %R within the QC limits?	$\square$			
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?			_1	-

LDC #: 1909783a SDG #: <u>su cone</u>

### VALIDATION FINDINGS CHECKLIST

	Page:_	of	2.
R	eviewer:	1	2
2nd Re	eviewer:	9	-
	-		

Validation Area	Yes	No	NA	Findings/Comments
VII: Matrix spike/Matrix spike duplicates		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?	$\leq$			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII. Laboratory control samples				
Was an LCS analyzed for this SDG?				
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?			<	
Were the performance evaluation (PE) samples within the acceptance limits?			_	
X. Target compound identification				
Were the retention times of reported detects within the RT windows?			_	—
XI, Compound quantitation/CRQLs				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions, dry weight factors, and clean-up activities applicable to level IV validation?	/	-		
XII. System performance				
System performance was found to be acceptable.	-	-		
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/	-		
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.		-	-	
Target compounds were detected in the field duplicates.			7	
XV: Field blanks		······	I	
Field blanks were identified in this SDG.	7	-		
Target compounds were detected in the field blanks.		1		

VALIDATION FINDINGS WORKSHEET

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

	Q. Endrin ketone R. Endrin aldehyde S. alpha-Chiordane	Y. Aroclor-1242 Z. Aroclor-1248	66.
		Aroclor-1248	
			HH.
0		AA. Aroclor-1254	II.
	T. gamma-Chlordane	BB. Arocior-1260	JJ.
E. reptachior M. 4,4'-DDD	U. Toxaphene	CC. DB 608	KK.
F. Aldrin N. Endosulfan sutfate	V. Aroclor-1016	DD. DB 1701	LL.
G. Heptachlor epoxide O. 4,4:-DDT	W. Aroclor-1221	EE.	MM.
H. Endosulfan I P. Methoxychlor	X. Aroclor-1232	FF.	NN.

Notes:

LDC #: / 9097/334 SDG #: Jul contr

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: Lof Reviewer: 2nd Reviewer:

LPLC D METHOD: GC

The calibration Pactor (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

# Standard ID 1 1 CAL	$\frac{\text{Calibration}}{\text{Date}}$	compound endosulgan / eh D(B) ne Hoxychlet (A)	CF CF CF CF Average CF (Initial) D(B) 285001720 255 001720 2.735334/2 AV217640 44217640 42222360 C(A) 5302/6040 5.10 995/40	CE DO2%Id) 24 27640 4427640 5 302/6040	Average CF         Average CF           (initial)         (initial)           2.73 5 334/2         273 5 3 4 2 2 2 3 4 2 2 2 2 2 2 2 2 2 2 2 2 2		%RSD 2.96581 6.0/863	%RSD 2.96 6,02
		lot ch	ortie prin Ofort Cats Otortos	01/100 Sh 01/10/17 01/10/17 01/10/17	273533412		2.98/0.2	2.96
	6/16/02	c y	artie min Ofor/ Cats Officiel	0770/17/ 0/09/202 S 079/2040	09E 2211		6. 0/863	6.02
┍╾╢──┬──╢──	6/16/02		aton/caes	11/10/2040				
	6/11e/02		artie min	09/202 S				
			11-11 31-1-10	111/91400	0h/566 a15	2/10/2015	3.1482	3. 1480
			12001 1101	10/1/0/	1914 2008 1914 34680 1522 720 20	15227620	6.2551 1	6.255/5
		-						
						-		
<b>Г</b>								
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#. 19097834 SDG #: per contr

**Continuing Calibration Results Verification** VALIDATION FINDINGS WORKSHEET

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

HPLC METHOD: GC\_

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

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

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

$ \begin{array}{c c c c c c c c c c c c c c c c c c c $						Reported	Recalculated	Reported	Recalculated
kentoov       6/14/08       endosulpan       (14 A       2000       0,0352       0.0352         kentoos       L/14/08       N       V       0,0352       0.0352       0.0352         kentos       L/14/08       V       V       0.0357       0.0352       0.0357         kentos       L/14/08       V       V       0.0357       0.0357       0.0357         kentos       V       V       V       V       V       V       V         kentos       V       V       V       V       V       V       V         v       V       V       V       V       V       V       V       V         v       V       V       V       V       V       V       V       V       V         v       V<	#	Standard ID	Calibration Date	Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc. CCV	Q%	۵%
$FCALOSO \ U/IX/08 \qquad MARXJCATE \qquad V  0.0322  0.032  1.0 \\ U & U & U & U & U & U \\ U & U & U & U$	-	K 24 2064	80/\$1/9		0,020	6.5200	00269	3.7	3.7
$\frac{kcaloso}{1} \frac{1}{12} \frac{1}{$				mu thoxy chier	7	0, 0252	232000	0.8	0.8
kcaloso     l     u.u.s.2     aox2     l       v     v     v     v     v       v     v     v <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>									
		KCAL 080	BO/ 81/7			L.Sr.0.0	0.0252	1.0	01
			-	de la	1	6.025 J	00257	ر. د ا	2.7
	<i>т</i>						-		
			1						
	4								

CONCLC.1S

recalculated results.

### LDC #: <u>1909783</u>~ SDG #: <u>pu cour</u>

### VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page:	of/
Reviewer:	n
2nd reviewer:	$\nu'$

### METHOD: GC Pesticides/PCBs (EPA SW 846 Method 8081/8082)

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

. . .

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

### Sample ID: #2

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene						
Tetrachloro-m-xylene	ch A	0.02	001676	84	84	0
Decachlorobiphenyl	ł	V	0.01750	87	87	V
Decachlorobiphenyl						

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

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachioro-m-xylene						
Tetrachloro-m-xylene						
Decachlorobiphenyl						
Decachlorobiphenyl						

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

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene						·
Tetrachloro-m-xylene						
Decachlorobiphenyl						
Decachlorobiphenyl						

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

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene						
Tetrachloro-m-xylene						
Decachlorobiphenyl						
Decachlorobiphenyl						

Notes:

19097832	au cand
LDC #	SDG #:

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



METHOD: GC Pesticides/PCBs (EPA SW 846 Method 8081/8082)

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

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

RPD = I MS - MSD I \* 2/(MS + MSD)

MS/MSD samples: 5 +

1

Where: SSC = Spiked sample concentration SC = SA = Spike added MS = Matrix spike percent recovery MSD

MSD = Matrix spike duplicate percent recovery

SC = Concentration

Compound		DINE STORE	Sample	Spiked	Spiked Sample	Matrix	Matrix Spike	Matrix Spik	Matrix Spike Duplicate	M	MS/MSD
	2	All (manny	Concentration	Conce (	ntration )	Percent	Percent Recovery	Percent	Percent Recoverv		RPD
	WS	MSD		WS	MSD	Reported	Recalc.	Reported	Recalc	Denortod	Lotol-cloced
gamma-BHC	17.7	17.5	0r	15.6	15,3	×.	o K	87	R 7	Z - ()	
4,4'-DDT	<i>.</i> ,	Ņ		9:51 .	16.3	2	2	, e 6	93	4.0	1.1
									T		
							-				

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

5	
Q	
<u>г</u>	3
16061	કુ
0	2
	₹
#	#
B	Ő
<b>_</b>	S

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

Page: 6f / Reviewer: 7 2nd Reviewer: 7

METHOD: GC Pesticides/PCBs (EPA SW 846 Method 8081/8082)

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

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

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

LCS = Laboratory control sample percent recovery

SSC = Spiked sample concentration SA = Spike added

Where:

LCSD = Laboratory control sample duplicate percent recovery

SC = Concentration

	•		Spike	Spiked Sample	רו	LCS	<u>۲</u>	LCSD	ILCS/	LCS/LCSD
Compound	Аа ( И5	Addeg	Zonce Conce	Concentration	Percent	Percent Recovery	Percent	Percent Recovery	R	RPD
	LCS	LCSD	rcs	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
gamma-BHC	16.7	44	15:0	NT	90	90				
4,4'-DDT	1	7	16.8	1	/01	/0/	AN .			

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

LDC #: 1909783~ SDG #: <u>pu cone</u>

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	of_/
Reviewer:	רק
2nd reviewer:	<u> </u>
	Ý

METHOD: GC Pesticides/PCBs (EPA SW 846 Method 8081/8082)



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?

Exam	ple:		
Samp	le I.D	 _:	
Conc.	. = <u>(</u> (	 	<u>)</u>
=	NP		

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

Note:

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

Project/Site Name:	BRC Tronox Parcel G
Collection Date:	June 11, 2008
LDC Report Date:	July 22, 2008
Matrix:	Soil
Parameters:	Polychlorinated Biphenyls
Validation Level:	EPA Level III & IV
Laboratory:	TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120167

### Sample Identification

TSB-GJ-08-10 TSB-GJ-08-20\*\* TSB-GJ-08-30\*\* TSB-GJ-08-40 TSB-GJ-08-10MS TSB-GJ-08-10MSD

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 6 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.

Sample "RINSATE 1" (from SDG F8F120137) was identified as a rinsate. No polychlorinated biphenyl contaminants were found in this blank.

### VI. Surrogate Spikes

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

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Pesticide Cleanup Checks

### a. Florisil Cartridge Check

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

### **b. GPC Calibration**

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

### XI. Target Compound Identification

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

### XII. Compound Quantitation and Reported CRQLs

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

### XIII. Overall Assessment of Data

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

# XIV. Field Duplicates

No field duplicates were identified in this SDG.

BRC Tronox Parcel G Polychlorinated Biphenyls - Data Qualification Summary - SDG F8F120167

a da serie de la companya de la comp

# No Sample Data Qualified in this SDG

BRC Tronox Parcel G Polychlorinated Biphenyls - Laboratory Blank Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

BRC Tronox Parcel G Polychlorinated Biphenyls - Field Blank Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

# VALIDATION COMPLETENESS WORKSHEET

LDC #: 19097B3b SDG #: F8F120167 Laboratory: Test America

# Level III/IV

21/08 Date: Page: 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
١.	Technical holding times	A	Sampling dates: 6/11/08
II.	GC/ECD Instrument Performance Check	A	
111.	Initial calibration	A	
IV.	Continuing calibration/ICV	A	1CY = 15
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	107
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	Α	
XV.	Field blanks	ND	R= Rinsate / SPG # P8F/2013

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:

	30/2					
1	TSB-GJ-08-10	11		21	31	
2	TSB-GJ-08-20**	12		22	32	
3	TSB-GJ-08-30**	13		23	33	
4	TSB-GJ-08-40	14		24	34	
5	TSB-GJ-08-10MS	15		25	35	
6	TSB-GJ-08-10MSD	16		26	36	
7	F8F160000-162	17	8168162	27	37	
8		18		28	38	
9		19		29	39	
10		20		30	40	

\*\* Indicates sample underwent Level IV validation

LDC # 19097 B36 SDG #: pu coner

# VALIDATION FINDINGS CHECKLIST

Page: /of 1 Reviewer: \_\_\_\_7 2nd Reviewer: \_\_\_\_6

Method:GCHPLC				
Validation Area	Ye	s No	NA	Findings/Comments
L. Technical holding times			(MAR)	
All technical holding times were met.		1		
Cooler temperature criteria was met.		1		
III initial calumations				
Did the laboratory perform a 5 point calibration prior to sample analysis?	1-	-		
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) $\leq$ 20%?		1		
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?		V	ł	
Did the initial calibration meet the curve fit acceptance criteria?			-	
Were the RT windows properly established?	1/	1		
IV-Counting Calibration				
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%?	/	1		
Were all the retention times within the acceptance windows?		1.		
VaBlanks MALE IN THE REAL PROPERTY OF THE REAL		<b>探</b> 影		
Was a method blank associated with every sample in this SDG?	/			
Was a method blank analyzed for each matrix and concentration?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
VI Simogate spikes				
Were all surrogate %R within the QC limits?	$\mathbf{P}$			
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?			/	
f any %R was less than 10 percent, was a reanalysis performed to confirm %R?			7	
VII. Matox spike/Matox spike duplicates				
Nere a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each natrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	~			
Vas a MS/MSD analyzed every 20 samples of each matrix?	7			
Vere the MS/MSD percent recoveries (%R) and the relative percent differences RPD) within the QC limits?	-			
Illistaboratory control samples				
Vas an LCS analyzed for this SDG?				
Vas an LCS analyzed per extraction batch?	イ	[	Ē	
Vere the LCS percent recoveries (%R) and relative percent difference (RPD) ithin the QC limits?	7			

LDC #: 1909 7836 SDG #: 41 Cone

# VALIDATION FINDINGS CHECKLIST

Page: 20f 2 Reviewer: 77 2nd Reviewer:

		T	T	
Validation Area	Yes	No	NA	Findings/Comments
X. Regional Quality Assurance and Quality Control	H-Sec.			
Were performance evaluation (PE) samples performed?			/ /	
Were the performance evaluation (PE) samples within the acceptance limits?			/	-
X Target source and a deminication and a start of the second start of the second start of the second start of the				
Were the retention times of reported detects within the RT windows?	Π			-
Ki-Genpoond quantitation/GRGIs				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions		85.3 BEA		
and dry weight factors applicable to level IV validation?				
subsystem performance of the second				
System performance was found to be acceptable.			Ī	
Xultoveraltassessment of data	$\Box$	Sisters P		
Autospecial assessment of data at a second state of the second state of				
Overall assessment of data was found to be acceptable.			T	
Field duplicate pairs were identified in this SDG.				
	<u>l</u> :	$\leq$		
Target compounds were detected in the field duplicates.			1	
SV: diedolaats			t nie se	
Field blanks were identified in this SDG.				
Farget compounds were detected in the field blanks.	4			
	Y		1	

N.

LDC# 19097836 cart 3 SDG #:

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Reviewer. 2nd Reviewer: Page:

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

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

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

Recalculated 9.582 (2 · C) **%RSD** 5.2 Reported. 9.582 %RSD Average CF (initial) Recalculated 27977 3 9/6 5 Average CF (initial) Reported 77875 39/64 Recalculated CF. (SV)std) 3926 91.shz CF S20std) 81062 **Reported** 24576 . ch A 5 4 S Compound 120-1 1260-, Calibration Date 20/12/s Standard ID 7401 ₩ 2 ო 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

1909728061 Certer 3 SDG #: LDC #:

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

5 Page: Reviewer. 2nd Reviewer:

HPLO 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

% 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

ú <u>**Recalculated</u></u></u>** 4 ð ھ 6,3 4-8 Reported å 952.19 Recalculated 937.33 CF/Conc. CCV 937. 3342 752. 1 902 CF/Conc. CCV Reported. Average CF(Ical)/ CCV Conc. o dral 022/ Compound Arecler 6/18/08 6/18/08 Calibration Date 680782 Standard ID PCALIDO ¥Ł 2 c d 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

DC # 19097830 SDG #: Let con

# VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

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

METHOD: GC HPLC

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Percent Difference 0 Recalculated Percent Recovery B Percent Recovery Reported Z 16.0769 Surrogate Found Surrogate Spiked Å Column/Detector C1 A Surrogate DcB Sample ID:

Sample ID:

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

Sample ID:

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

SURRCALCNew.wpd

7,636	oner
6061	202
LDC #:	SDG #:_

# <u>Matrix Spike/Matrix Spike Duplicates Results Verification</u> VALIDATION FINDINGS WORKSHEET

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

HPLC 3 **METHOD:** 

The percent recoveries (%R) and relative percent differences (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation: %Recovery = 100 \* (SSC - SC)/SA

Where

RPD =(({SSCMS - SSCMSD} + 2) / (SSCMS + SSCMSD))+100

e 1

5

MS/MSD samples:

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

MSD = Matrix spike duplicate SC = Sample concentration

	S.	Spike	Sample	Spike :	Spike Sample	Matrip	Matrix soike	Matrix Snik	- Dunlland		
Compound	Adde Adde	24 / 27	Coner ( WX //Sr	Concei	Concentration	Dercent	0				20
「「「「「「「「」」」」では、「」」」では、「」」」では、「」」」では、「」」」」では、「」」」」では、「」」」」では、「」」」」」では、「」」」」」」」」」、「」」」」」」」」」」	Ň	0			×,			Percent	Percent Recovery	RPD	0
		- MOU		WS	TMSD	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)			-								
Arocher 1260	771	178	ON	181	194	107	201.	0 \	0	) : r	
								/2/	201	× · /	7.7
											Ī
Comments: Refer to Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and construct of the list of an alternation of the second	ike/Matrix	Spike Dup	licates finding	s workshaat f	or tiet of availe						
of the recalculated results.			<b>D</b>	100110110110		ICALIOUS AND A	<u>issociated san</u>	<u>nples when rel</u>	ported results	s do not agree	<u>within 10.0%</u>

MSDCLCNew.wpd

SDG # HOD:	S S	GCHPLC	ry Contr	va ol Sample/	Laborator	y Control	VALIUA I I UN FINUINGS WURKSHEE I ple/Laboratory Control Sample Duplic	VALIDATION FINDINGS WORKSHEET Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification HPLC	<u>esults Ver</u>	ification	Page:_ Reviewer:_ 2nd R	age: of ewer: 2nd Reviewer:
The percent recoveries (%R) and relative percent differences (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:	veries (%R) a ified below us	and relativ sing the fc	e percent illowing ca	differences (I Iculation:	RPD) of the I	aboratory co	ntrol sample	and laborator	y control sam	ple duplicate	were recalcu	lated for the
%Recovery = 100 * (SSC - SC)/SA	sc - sc)/sA		Where S	SSC = Spiked concentration SA = Spike added	centration		SC = Sample concentration	ncentration				
RPD =(({SSCLCS - SSCLCSD} * 2) / (SSCLCS + SSCLCSD))*100 LCS/LCSD samples:	sclcsd} • 2) / (ssclc <i>¥   6 &amp;   6 2</i>	162 + 5	رعم دعم	8 1	LCS = Laborat	LCS = Laboratory Control Sample percent recovery _	ple percent recov		LCSD = Laboratory Control Sample duplicate percent recovery	Control Sample	duplicate percent	recovery
		Spike	ke	Sample	Spike Sample	Sample		LCS	rcsD	SD C	rcs/rcsd	CSD
Compound	nd	Added 1	Ex)	Cong.	Concentration	itration	Percent	Percent Recovery	Percent Recovery	ecovery	Qda	
		rcs -	LCSD	1	rcs	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (	(8015)											
Diesel (8	(8015)											
Benzene (8	(8021B)											
Methane (	(RSK-175)											
2,4-D (8	(8151)											
Dinoseb (8	(8151)											
Naphthalene (	(8310)											
Anthracene (	(8310)											
) XMH	(8330)											
2,4,6-Trinitrotoluene (8330)	iene (8330)											
haclor 1	260	167	<i>ч</i> 4	C	17/	44	£0]	601	KAN.			
Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicate findings worksheet for list of gualifications and associated samples when reported	er to Laborato	ry Control	Sample/L	aboratory Co	ntrol Sample	Duplicate fin	dinas worksh	leet for list of c	ualifications a	and associate	ed samples w	hen reported
results do not agree within 10.0% of the recalculated results.	ree within 10.	0% of the	recalculat	ted results.								

LCSCLCNew.wpd

۲

oncerturion LCSD Rep NA-	Sample Spike Sample	LCS	rcsD	rcs/rcsD
LCS         LCS <thls< th=""> <thls< th=""> <thls< th=""></thls<></thls<></thls<>		Percent Recovery	Percent Recovery	RPD
(8015)       (8015)       (8015)         (8015)       (8015)       (8015)         (8015)       (8015)       (9015)         (8021B)       (8021B)       (9015)         (8151)       (8151)       (9015)         (8151)       (8151)       (9015)         (8151)       (8151)       (9015)         (8151)       (8151)       (9015)         (8151)       (8151)       (9015)         (8151)       (8151)       (9015)         (8151)       (8151)       (9015)         (8151)       (8151)       (9015)         (8151)       (8151)       (9015)         (8151)       (8151)       (9015)         (8151)       (8151)       (9015)         (8130)       (8110)       (9015)         (8130)       (9015)       (9015)         (8130)       (1015)       (117)         (117) $N4-$ (117)		Reported Recalc.	Reported Recalc.	Reported Recalc.
(8015)       (8015)       (8015)         (8021B) $(8021B)$ $(8021B)$ (8021E) $(8021E)$ $(8021E)$ (8151) $(8151)$ $(8151)$ (8151) $(8151)$ $(8151)$ (8151) $(8151)$ $(8151)$ (8151) $(8151)$ $(8151)$ (8151) $(8151)$ $(8151)$ (8151) $(8151)$ $(8151)$ (8151) $(8151)$ $(8151)$ (8151) $(8151)$ $(8151)$ (810) $(810)$ $(810)$ (810) $(810)$ $(810)$ (810) $(810)$ $(810)$ (810) $(810)$ $(810)$ (810) $(810)$ $(810)$ (810) $(810)$ $(91)$ (810) $(91)$ $(91)$ (810) $(91)$ $(91)$ (810) $(91)$ $(91)$ (810) $(91)$ $(91)$ (810) $(91)$ $(91)$ (810) $(91)$ $(91)$ (91)				
(8021B)       (8021B)         (RSK-175)       (RSK-175)         (RSK-175)       (RSK-175)         (8151)       (RSK-15				
(RSK-175)       (RSK-175)       (RSK-175) $(8151)$ $(810)$ $(810)$ $(810)$ $(8310)$ $(8310)$ $(810)$ $(8310)$ $(8310)$ $(8310)$ $(8310)$ $(8310)$ $(810)$ $(8310)$ $(8310)$ $(810)$ $(810)$ $(8310)$ $(8310)$ $(810)$ $(810)$ $(810)$ $(8310)$ $(8310)$ $(810)$ $(810)$ $(810)$ $(810)$ $(8310)$ $(810)$ </td <td></td> <td></td> <td></td> <td></td>				
(3151)       (3151)         (8151)       (8151)         (8151)       (8151)         (8151)       (8151)         (8151)       (8151)         (810)       (810)         (810)       (810)         (8310)       (810)         (810)       (810)<				
(8151)       (8151)         (8151)       (8151)         (8310)       (8310)         (8310)       (8310)         (8310)       (810)         (8310)       (810)         (8310)       (810)         (8310)       (810)         (8310)       (810)         (8310)       (810)         (8310)       (810)         (8310)       (810)         (8310)       (810)         (8310)       (810)         (8310)       (810)         (8310)       (810)         (8310)       (810)         (8310)       (810)         (8310)       (810)         (8310)       (810)         (810)       (810)         (810)       (810)         (810)       (810)         (810)       (810)         (810)       (810)         (811)       (810)         (811)       (810)         (811)       (810)         (811)       (810)         (811)       (810)         (811)       (810)         (811)       (810)         (811)       (810) </td <td></td> <td></td> <td></td> <td></td>				
(8310)       (8310)       (8310)       (8310)       (8310)       (9000000000000000000000000000000000000				
ne       (8310)       ne       ne       (8330) $(8330)$ (8330)       (8330)       (8330)       (8330)         nitrotoluene (8330)       (8330)       (8330)       (8330)       (8330) $e^{-r}$ $12e^{-r}$ $N\Phi$ (7) $N\Phi$ (7)				
(8330) itrotoluene (8330) <i>box 1</i> とつ 167 NA ひ 17/ NA				
nitrotoluene (8330) レイ レビン ノセフ ハイ ジ 17/ ルイ				
6× 1200 167 NA 0 171 NA				
	17/	(03 /03	NA +	

õ Č	LDC #: 19097 125 b SDG #: 224 conor	VALIDATI Sampl	VALIDATION FINDINGS WORKSHEET Sample Calculation Verification	HEET <u>ion</u>	Page: Reviewer:
ME	METHOD: GC HPLC			•	iamainan niz
	N N/A N/N/A Were all recalculate	Were all reported results recalculated and verified for all level IV samples? Were all recalculated results for detected target compounds within 10% of	ed for all level IV samples? compounds within 10% of the reported results?	ported results?	
Cor	Concentration= (A)(Fv)(Df) (RF)(Vs or Ws)(%S/100)				
A= DV= f=	Area or heigh Final Volume Dilution Fact	asured Sample ID	Com	Compound Name	
н 1 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	<b>4</b>	d Concentration =		Ŵ	
		· · · · · ·			
	* Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications
	· · · · · · · · · · · · · · · · · · ·				
Com	Comments:				

SAMPCALew.wpd

:

ALAS 2020 (1010)

# Laboratory Data Consultants, Inc. Data Validation Report

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

Collection Date: June 11, 2008

LDC Report Date: July 24, 2008

Matrix: Soil

Parameters:

Validation Level: EPA Level III & IV

Metals

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120167

## Sample Identification

TSB-GJ-08-10 TSB-GJ-08-20\*\* TSB-GJ-08-30\*\* TSB-GJ-08-40 TSB-GJ-08-10MS TSB-GJ-08-10MSD

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 6 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Methods 6010B, 6020, and 7000 for Metals. The metals analyzed were Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Lithium, Magnesium, Manganese, Molybdenum, Mercury, Nickel, Niobium, Palladium, Phosphorus, Platinum, Potassium, Selenium, Silicon, Silver, Sodium, Strontium, Sulfur, Thallium, Tin, Titanium, Tungsten, Uranium, Vanadium, and 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 an EPA Level IV review. An EPA Level III review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Level III criteria since this review is based on QC data.

The following are definitions of the data qualifiers:

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

# I. Technical Holding Times

All technical holding time requirements were met.

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

# II. Calibration

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/L	All samples in SDG F8F120167

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-08-10	Mercury	19.1 ug/Kg	35.7U ug/Kg
TSB-GJ-08-20**	Thallium Tungsten	0.40 mg/Kg 0.70 mg/Kg	0.48U mg/Kg 1.2U mg/Kg
TSB-GJ-08-30**	Lithium	65.0 mg/Kg	180U mg/Kg

Sample "RINSATE 1" (from SDG F8F120137) was identified as a rinsate. No metal contaminants were found in this blank with the following exceptions:

Rinsate ID	Sampling Date	Analyte	Concentration	Associated Samples
RINSATE 1	6/11/08	Calcium Iron Magnesium Manganese Silicon Sodium Strontium	131 ug/L 154 ug/L 17.9 ug/L 0.84 ug/L 38.6 ug/L 39.2 ug/L 1.5 ug/L	All samples in SDG F8F120167

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

# 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-08-10MS/MSD (All samples in SDG F8F120167)	Sulfur Phosphorus	140.1 (75-125) 134.8 (75-125)	135.4 (75-125) -	-	J+ (all detects) J+ (all detects)	A
TSB-GJ-08-10MS/MSD (All samples in SDG F8F120167)	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-08-10MS/MSD (All samples in SDG F8F120167)	Niobium	40.6 (75-125)	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 an EPA Level IV review was performed with the following exceptions:

Sample	Internal Standard	%R (Limits)	Analyte	Flag	A or P
TSB-GJ-08-20**	Scandium-45	127.557 (30-120)	Silicon Strontium	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A
TSB-GJ-08-30**	Scandium-45	129.653 (30-120)	Silicon Strontium	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A

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

# IX. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

# X. ICP Serial Dilution

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

Diluted Sample	Analyte	%D (Limits)	Associated Samples	Flag	A or P
TSB-GJ-08-10L	Iron	10.4 (≤10)	All samples in SDG F8F120167	J (all detects)	A

# XI. Sample Result Verification

All sample result verifications were 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.

# 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 F8F120167

SDG	Sample	Analyte	Flag	A or P	Reason
F8F120167	TSB-GJ-08-10 TSB-GJ-08-20** TSB-GJ-08-30** TSB-GJ-08-40	Sulfur Phosphorus	J+ (all detects) J+ (all detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8F120167	TSB-GJ-08-10 TSB-GJ-08-20** TSB-GJ-08-30** TSB-GJ-08-40	Antimony Copper Silicon Vanadium Lithium Nickel Tungsten Zinc	J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8F120167	TSB-GJ-08-10 TSB-GJ-08-20** TSB-GJ-08-30** TSB-GJ-08-40	Niobium	J- (all detects) R (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8F120167	TSB-GJ-08-20** TSB-GJ-08-30**	Silicon Strontium	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A	Internal standards (%R)
F8F120167	TSB-GJ-08-10 TSB-GJ-08-20** TSB-GJ-08-30** TSB-GJ-08-40	Iron	J (all detects)	Α.	ICP serial dilution (%D)

# BRC Tronox Parcel G Metals - Laboratory Blank Data Qualification Summary - SDG F8F120167

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8F120167	TSB-GJ-08-10	Mercury	35.7U ug/Kg	А
F8F120167	TSB-GJ-08-20**	Thallium Tungsten	0.48U mg/Kg 1.2U mg/Kg	A
F8F120167	TSB-GJ-08-30**	Lithium	180U mg/Kg	A

# BRC Tronox Parcel G Metals - Field Blank Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

# VALIDATION COMPLETENESS WORKSHEET

LDC #: 19097B4 SDG #: F8F120167

#### Laboratory: Test America

Level III/IV

Date	<u>. 1/~4/.8</u>
Page:	<u>    l of                               </u>
Reviewer	
2nd Reviewer	: <u>A</u>
	/-

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
Ι.	Technical holding times	Ą	Sampling dates: 6/11/48
11.	Calibration	4	
111.	Blanks	ふと	
IV.	ICP Interference Check Sample (ICS) Analysis	A	
V.	Matrix Spike Analysis	sw	1 Ms/hiso
VI.	Duplicate Sample Analysis	2	, , , , , , , , , , , , , , , , , , ,
VII.	Laboratory Control Samples (LCS)	A	Lus
VIII.	Internal Standard (ICP-MS)	SW	Not verienced for ferre 3
IX.	Furnace Atomic Absorption QC	N	Not veriend for fere 3
<b>X</b> .	ICP Serial Dilution	5W	σ
XI.	Sample Result Verification	A	Not reviewed for Level III validation.
XII.	Overall Assessment of Data	A	
XIII.	Field Duplicates	2	
XIV.	Field Blanks	5W	R=RINSATZ 1 (F8F 120/37)

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-08-10	11	21	31	
2	TSB-GJ-08-20**	12	22	32	
3	TSB-GJ-08-30**	13	23	33	
4	TSB-GJ-08-40	14	24	34	
5	TSB-GJ-08-10MS	15	25	35	
6	TSB-GJ-08-10MSD	16	26	36	
7	OB	17	27	37	
8	•	18	28	38	
9		19	29	39	
10		20	30	40	

Notes:

LDC #: 19097134 SDG #: <u>Cet win</u>

#### VALIDATION FINDINGS CHECKLIST



Ť4

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times	e	i, i		
All technical holding times were met.	1			
Cooler temperature criteria was met.				
II -Calibration				
Were all instruments calibrated daily, each set-up time?	/		<b></b>	
Were the proper number of standards used?		ļ	<b> </b>	
Were all initial and continuing calibration verification %Rs within the 90-110% (80- 120% for mercury and 85-115% for cyanide) QC limits?	1			
Were all initial calibration correlation coefficients > 0.995? (Level IV only)			-	
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.				
IV-ICR Interference Check Sample				
Were ICP interference check samples performed daily?	/			
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?			er/046762	
V-Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	<			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.	~			
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq$ 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 Laboratory control samples		<b>1</b> 023		
Was an LCS anaylzed for this SDG?	/			
Was an LCS analyzed per extraction batch?	_			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?	/			
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% QC limits?			1	

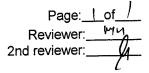
LDC # (9097B4 SDG # <u>Sel women</u>

## VALIDATION FINDINGS CHECKLIST

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

Validation Area	Yes	No	NA	Findings/Comments
VILICR Senal Dilution				
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the IDL?	1		<b> </b>	7 wax much for Zephy
Were all percent differences (%Ds) < 10%?		4		
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.		/		
VIII. Internal Standards (EPA SW 845 Method 8020)				
Were all the percent recoveries (%R) within the 30-120% of the intensity of the internal standard in the associated initial calibration?		1		
If the %Rs were outside the criteria, was a reanalysis performed?	198 A 19 19 19		No. of Concession	
IX: Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?	CRIMINAN	10-10-10-10-10		
X. Sample Result Ventication				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XI Overall assessment of deta				
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.	$\checkmark$			
Target analytes were detected in the field blanks.	_/			

LDC #:<u>(9 • 97</u> SDG #:<u>\_\_\_\_\_</u>



1

All circled elements are applicable to each sample.

		Target Analyte List (TAL)
Sample ID	1	
1-4	'Soi)	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, 11, V, Zn, Mo, B, Si,
mino	Soil	
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
_		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
1-4	( Coil	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr, /
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
MSib	50:1	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Z,
		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,
		Analysis Method
ICP		Li, S
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 <u>, B. Si</u>
ICP-MS		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Zr,
GEAA		AL Sb. As. Ba. Be. Cd. Ca. Cr. Co. Cu. Fe. Pb. Mg. Mn. Hg. Ni, K. Se. Ag. Na, TI, V. Zn. Mo, B. Si, CN.

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

Sample Concentration units, unless otherwise noted: <u>mg/Kg ex</u>	incentratio	nn units, un			Sample Concentration units, unless otherwise noted: _mg/Kg except Hg: ug/Kg	pt Hg: ug/Kg	Associated	: ug/Kg Associated Samples: All					/
									Sample Id	Sample Identification			
Analyte	Maximum PBª (ma/Ka)	Maximum PB <sup>a</sup> (uo/I )	Maximum ICB/CCB <sup>a</sup>	Blank Action I imit	-	2	е						
<u> </u>			1.3										
			1.1	0.22		0.40 / 0.48							
L			1.4			0.70 / 1.2							
			2.7		-								
 			8.0				65.0 / 180						
<u> </u>			0.1		19.1/35.7								
<u> </u>					-								
<u> </u> 													
<u> </u> 													
<u>L</u> T													
<u> </u>													
1													
<u> </u>													
<u> </u>													
L 													
<u> </u>													
<u> </u>													
<u> </u> 													
1													
<u> </u>													
l													
Ì			Ī									•	

qualified as not detected, "U". Note : a - The listed analyte concentration is the highest ICB, CCB, or PB detected in the analysis of each element.

19097B.wpd

LDC #: <u>19097B4</u> SDG #: <u>See Cover</u>

# VALIDATION FINDINGS WORKSHEET <u>Field Blanks</u>

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

METHOD: Trace Metals (EPA SW846 6010B/6020/7000)Y) N N/AWere field blanks identified in this SDG?Y N N/AWere target analytes detected in the field blanks?Y N N/AWere target analytes detected in the field blanks?Y N N/ASociated sample units: mg/KqSampling date: 6/11/08Soil factor applied 200XField blank type: (circle one) Field Blank / Rinsate / Other: R

													-				
														-			
10X  or  > RL	ion																
mples: All (>	Sample Identification																
Associated Samples: All (>10X or > RL)	Sar																
R																	
te / Other:																	
ield blank type: (circle one) Field Blank / Rinsate / Other:																	
one) Field I		Action Level	262	308													
type: (circle	Blank ID	RINSATE 1	131	154	17.9	0.84	38.6	39.2	1.5								
ield blank	Analyte		Ca	Fe	Mg	Mn	Si	Na	ა								

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

9057B4	sel com
LDC #:	SDG #:

VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

đ 5 Page: 2nd Reviewer: Reviewer:

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

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

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

<u>Y (N) N/A</u> We

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

W/SW	di QSW/SW	Matrix	Analyte	MS %Recovery	MSD %Recovery	RPD (Limits)	Associated Samples	Qualifications
2/12		(:05	\$	(to,)	7.581		411	T+ J+ /A
			5 h	5tiz	79.4	×		-M
			cu	12.5	60,4	Ĩ		L.
			٩/٦	9101	1.96			J-/R/A
			9	134.8				T+ 1+/A
			, S	129	44.6			J-/wj/A
			<u> </u>	7,89	Shartho	0		A
			· 1		69,8			J-/47/A
			N.Y		71.1			
			M		60.6			
			N-2		62.2			Y
			1.1			2019		No gard ( Lesin)
			2 P			33.1		<b>_</b>
			Bo∽			20,2		•
			ع			29,45		
			Co			N.'0		
			1;			20.9		
	-		لم) ا			28-50		
			5			226		$\boldsymbol{\lambda}$
								-
Comments:	Å.	Can Fe	Ha Mn	52, 71	74X			
			/ 0	· · · ·				

MSD.4S2

LDC #: 1909754 SDG #: See win

VALIDATION FINDINGS WORKSHEET Internal Standards (ICP-MS)

Reviewer: of | Reviewer: Page:

METHOD: Metals (EPA SW 846 Method 6020)

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

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

- T			T	, ,		 _	-	 _	-	 	 		 		 	
	<b>Qualifications</b>	J/1/1/2		Å												
	Associated Samples	ト		3												
	%R (Limits)	125621		129,643	-											
	Associated Metals	Si.Sr.		$\Lambda$												
-	Internal Standard	5c45		Sc45												
1L	*			7												

INTST.4S2

METHOD: Tr	METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)	Method 6010/6	(020/7000)			
Please see qualific N N/A If V N/A V V N/A Is V N/A Is V N/A Is	ualifications below for all q If analyte concentratior Were ICP serial dilutior Is there evidence of ne	questions answerns were > 50X ti n percent differu gative interfere	ared "N". Not al he MDL (ICP) , ∋nces (%D) ≤1 <sup>(</sup> nce? If yes, pr	pplicable questions or >100X the MDL 0%? ofessional judgeme	Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". N N/A If analyte concentrations were > 50X the MDL (ICP) ,or >100X the MDL (ICP/MS), was a serial dilution analyzed? Y (N N/A Vere ICP serial dilution percent differences (%D) ≤10%? Y (N N/A Is there evidence of negative interference? If yes, professional judgement will be used to qualify the data.	n analyzed? lata.
	Were recalculated resu	ults acceptable(	See Level IV	Recalculation Wor	Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations.	
<u>*</u> Date	Diluted Sample ID	Matrix	Analyte	%D (Limits)	Associated Samples	Qualifications
		507	ц	Vert	14	TL+/A
-						
Comments:	Ne. U < lock man	Jew.				
	1					

VALIDATION FINDINGS WORKSHEET **ICP Serial Dilution** 

Cer cover

LDC #: 1909784

SDG #:

Reviewer: U Page:

of /

SDILICPMS.wpd

ł

LDC #: (909) B &

VALIDATION FINDINGS WORKSHEET initial and Continuing Calibration Calculation Verification

Page: \_\_\_\_\_of \_/ Reviewer: \_\_\_\_\_\_2nd Reviewer: \_\_\_\_\_\_

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 = Found × 100 True

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%К	%R	Acceptable (Y/N)
TeV	ICP (Initial calibration)	S	aolith	4000	(06. S	(10,4%)	٢
	GFAA (Initial calibration)			-			
Tev	CVAA (Initial calibration)	K+1	2133	2.5	43.2	922	Y
ced	ICP (Continuing calibration)	<u>ار</u>	4920	Sv. o	98.4	98.4	->
	GFAA (Continuing calibration)						
col	CVAA (Continuing calibration)	H	4.98	5-0	49.6	9-66	λ
NT	ICP/MS (Initial calibration)	, J	(01), \$	( 6° O	~/o)	لر ( ٥)	
eed	ICP/MS (Continuing callbation)	Μ	8-3901	0.01	( o f a s	6,901	Ţ

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.

CALCLC.4SW

9998 y	Set cover
	SDG #:

# VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

£ Ъ 2nd Reviewer: Page: Reviewer:

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

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

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 × 100 True

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

 RPD = [S-D]
 x 100
 Where,
 S = Original sample concentration

 (S+D)/2
 D = Duplicate sample concentration

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

%D = <u>1-SDR</u> × 100 Wher

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

reis Element Found S I True I D I SDR (unite) reck Ay $g_{\mathcal{L}}, b_{\mathcal{C}} \mathcal{T}$ [ $\circ \circ$ sample $M_{\mathcal{D}}$ $vb_{\mathcal{C}} g_{\mathcal{T}}$ b $v_{\mathcal{L}} g_{\mathcal{T}}$ [ $\circ \circ$ k (ISR-SR) R (ISSR-SR) (35.8-6 R) $3$ ( $g_{\mathcal{L}}$ $g_{\mathcal{T}}$ $g_{\mathcal{L}} g_{\mathcal{T}}$						Recalculated	Reported	
ICP interference check     As     9.5, 6 ° S     ( - o       Laboratory control sample     Vb     24, 8 ° S     700       Matrix spike     V     (sSR-SR)     (10), 3     (358-6       Duplicate     P     31, 8 ° S     74.9	Sample ID	Type of Analysis	Element	Found / S / I (units)	True / D / SDR (units)	%R / RPD / %D	%R / RPD / %D	Acceptable (Y/N)
Laboratory control sample     Matrix spike     Matrix spike     Matrix spike     Matrix spike       Matrix spike     K     (SSR-SR)     (SSR-SR)       Duplicate     R     3 (.gsf     34g			A.5	95,6.5	٩ ه }	95.6	95%	4
Matrix spike $k$ (SSR-SR) (10), 3 (358-6 Duplicate $p_{1}$ 3 ( $g_{1}$ $g_{1}$ $g_{2}$ $g_{1}$ $g_{2}$	119	Laboratory control sample	AN AN	rb.87	stro	2.5 م)	107.5	
Duplicate P 31,85 34.9	4	Matrix spike	X	(ssr-sr) 1 1 0   _ }	1338-6	Errs	82.3	
	5/16	Duplicate	R	31,85	34.9	4,2	9.0	
My 715.8 725.54		ICP serial dilution	Ηv	413.81	458.52	م <i>،</i> کر	5-6	~

Comments: Refer to appropriate worksheet for list of gualifications and associated samples when reported results.

TOTCLC.4SW

. 2.05

LDC #: 1909134 SDG #: Ser com

#### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	<u></u>
Reviewer:	My
2nd reviewer:	$\sim$
	1

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". $\underline{N}$  N N/AHave results been reported and calculated correctly? $\underline{N}$  N N/AAre results within the calibrated range of the Instruments and within the linear range of the ICP? $\underline{N}$  N N/AAre all detection limits below the CRDL?

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

Concentration = (RD)(FV)(Dil) (In. Vol.)(%S) RD = Raw data concentration FV = Final volume (ml) In Vol = Initial volume (ml) or we

Recalculation: Mg = 51418.7 y/LX0, lex= = 1502 3 mg/mg

In. Vol. = Initial volume (ml) or weight (G) Dil = Dilution factor

%S = Decimal percent solids

Sample ID	Analyte	Reported Concentration ( W. J. 44 )	Calculated Concentration ( My My )	Acceptable (Y/N)
7	L	73.50	125	Ч
	S	6030	6030	1
	AL	11900	11900	
	As	24.4	43,2	
	Ba	43.4		
	Be	p.s.y	0.54	
	B	2211	22.1	
	Ca	9510	9172	
	Gr	30,3	30.3	
	6	4.8	4,8	
	<u> </u>	1,4	И.Ф	
· · · · · · · · · · · · · · · · · · ·	Fe	(1200	11200 J	
	рЬ	7.8	7.8	
	<u> </u>	2000	25000	
	My	153	52	
	Mo	0.56	0.56	
	Nì nl	11-6	11.6	
	pd P	0.74	0.21	·
		484	483	
· · · · · · · · · · · · · · · · · · ·	K	3190	7190	
	£ì	373	323	/
	Ag	0-17	0.17	

RECALC.4S2

LDC #: <u>19097 B4</u> SDG #: Sue lover

#### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	V or V
Reviewer:	<u>` MH'</u>
2nd reviewer:	l

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". $\underline{Y \ N \ N/A}$ Have results been reported and calculated correctly? $\underline{Y \ N \ N/A}$ Are results within the calibrated range of the instruments and within the linear range of the ICP? $\underline{Y \ N \ N/A}$ Are all detection limits below the CRDL?

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

Recalculation:

 Concentration =
 (RD)(FV)(Dil) (In. Vol.)(%S)

 RD =
 Raw data concentration

 FV =
 Final volume (ml)

 In. Vol. =
 Initial volume (ml) or weight (G)

 Dil
 =

%S = Decimal percent solids

Sample ID	Analyte	Reported Concentration (Wy Kg )	Calculated Concentration ( Inc. ( Inc. )	Acceptable (Y/N)
	Na	185	186	Ч
		106	1.6	
	Tl	0.40	0,40	
	SN	0.51	0.5	
	ī`	528	528	
	W	0.70	0.70	
	<u> </u>	try	5.4	
	V	42.3	.42,3	
	<u></u>	32.8	3217	
		29.8	29.8	<u>у</u>
	······································			
	· · · · · · · · · · · · · · · · · · ·			
	· · · · · · · · · · · · · · · · · · ·		· · ·	······································

RECALC.4S2

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	BRC Tronox Parcel G

Collection	Date:	June 11, 2008

LDC Report Date: July 23, 2008

Matrix: Soil

Parameters: Wet Chemistry

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120167

# Sample Identification

TSB-GJ-08-10 TSB-GJ-08-20\*\* TSB-GJ-08-30\*\* TSB-GJ-08-40 TSB-GJ-08-10MS TSB-GJ-08-10DUP

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 6 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
CCB1	Orthophosphate as P	0.260 mg/L	TSB-GJ-08-10
CCB2	Orthophosphate as P	0.212 mg/L	TSB-GJ-08-20** TSB-GJ-08-30** TSB-GJ-08-40

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

Sample "RINSATE 1" (from SDG F8F120137) was identified as a rinsate. No contaminant concentrations were found in this blank with the following exceptions:

Rinsate ID	Sampling Date	Analyte	Concentration	Associated Samples
RINSATE 1	6/11/08	Sulfate	0.12 mg/L	All samples in SDG F8F120167

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

### IV. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) samples were reviewed for each matrix as applicable. Percent recoveries (%R) were within QC limits.

### V. Duplicates

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

### VI. Laboratory Control Samples

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

### VII. Sample Result Verification

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

### VIII. Overall Assessment of Data

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

### **IX. Field Duplicates**

No field duplicates were identified in this SDG.

BRC Tronox Parcel G Wet Chemistry - Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

BRC Tronox Parcel G Wet Chemistry - Laboratory Blank Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

BRC Tronox Parcel G Wet Chemistry - Field Blank Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

LDC #: <u>19097B6</u>	VALIDATION COMPLETENESS WORKSHEET		Date: 7/23/08
SDG #: F8F120167	Level III/IV		Page:of
Laboratory: Test America			Reviewer:
, <u> </u>	(/ ,	./	2nd Reviewer:

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/11/08
lla.	Initial calibration	Å	
lib.	Calibration verification	Á	
	Blanks	SW	
IV	Matrix Spike/Matrix Spike Duplicates	A	2M3/pup
v	Duplicates	A	
VI.	Laboratory control samples	A	163
VII.	Sample result verification	A	Not reviewed for Level III validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N	
x	Field blanks	SW	R=RWSATZ 1 (FSF 120137)

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

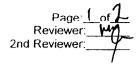
Validated Samples: \*\* Indicates sample underwent Level IV validation

1	TSB-GJ-08-10	11	21	31	
2	TSB-GJ-08-20**	12	22	32	
3	TSB-GJ-08-30**	13	23	33	
4	TSB-GJ-08-40	14	24	34	
5	TSB-GJ-08-10MS	15	25	35	
6	TSB-GJ-08-10DUP	16	26	36	
7	MB	17	27	37	
8		18	28	38	
9		19	29	39	
10		20	30	40	

Notes:\_

LDC #: 1909736 SDG #: 500 circle

### VALIDATION FINDINGS CHECKLIST



### Method: Inorganics (EPA Method See WWW

Validation Area	Yes	No	NA	Findings/Comments
( . Technical holding times		$\mathbf{F}_{\mathbf{F}}$		A PERSONAL PROPERTY AND A PERSON
All technical holding times were met.	1			
Coolor temperature criteria was met.	1			
Il Californion	制度	和社		
Were all instruments calibrated daily, each set-up time?	1			
Were the proper number of standards used?	1			
Were all initial calibration correlation coefficients $\geq$ 0.995?	/			· · · · · · · · · · · · · · · · · · ·
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?	1			
Were titrant checks performed as required? (Level IV only)			1	
Were balance checks performed as required? (Level IV only)		a with the		
Was a method blank associated with every sample in this SDG?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/		
IVIMAID Spike Manusoke uppicites and Triplicanese 1. 411 House the sector.				新發 和相同的相關
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.	/			
2 Eastanty Expression anotes	ų,			
Was an LCS anaytzed for this SDG?	-			
Was an LCS analyzed per extraction batch?	-4			
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 Orality Assurance and Quality Control (1). Section 44, 44 (4) (4)				
Were performance evaluation (PE) samples performed?			4	
Were the performance evaluation (PE) samples within the acceptance limits?			1	

LDC # 19097136 SDG #: 500 #:

### VALIDATION FINDINGS CHECKLIST

 $\begin{array}{c} Page: \sum_{of} Y\\ Reviewer: \mu 4 \\ 2nd Reviewer: \\ \end{array}$ 

Validation Area	Yes	No	NA	Findings/Comments
VII. Sample Result Verification		ie i i i	í.	
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	~			
Were detection limits < RL?	V			
	u, j			
Overall assessment of data was found to be acceptable.	1			
				<b>ehe</b> Politika di Antonia
Field duplicate pairs were identified in this SDG.		$\checkmark$		
Target analytes were detected in the field duplicates.			~	
Field blanks were identified in this SDG.	~			
Target analytes were detected in the field blanks.	1			

### LDC #: 1909186 SDG #: <u>See</u> cover

### VALIDATION FINDINGS WORKSHEET Sample Specific Analysis Reference

Page:_	of/
Reviewer:	$\sim$
2nd reviewer:	¢
	- /

All circled methods are applicable to each sample.

ample ID	Matrix	Parameter
1-4	Goi)	Br Bromine CI Chlorine F NO3 NO2 SO4 O-PO4 Chlorate CIO4 O+O/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
nsib	∽i)	(Br,)Bromine (C) Chlorine (F) NO3 NO2 (SO) O (PO) Chlorate CIO4 (+C)/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 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 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 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 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 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 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 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 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 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 CIO <sub>4</sub> O+G/TPH

Sec cores (909782 LDC #: SDG #:

### VALIDATION FINDINGS WORKSHEET Blanks

Page: of 2nd Reviewer: Reviewer:

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

(													
(an) /-2=200	ation												
cett = 1, ccbs	Sample Identification					- -							
Associated Samples:													
Associate													
	Blank	Action Limi											
/Kg		ICB/CCB M &/L	erto	0.27									
	Blank ID												
Conc. units: 144	Analyte		o-bot-o	a-bot-o	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1								

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".

BLANKS.6

Page: Baviewer	2nd Reviewer:																U".
																	were qualified as not detected, "
VALIDATION FINDINGS WORKSHEET <u>Field Blanks</u>	Associated Samulae.	Sample Identification				-				Associated Samples;	Sample Identification						Samples with analyte concentrations within five times the associated field blank concentration are listed above, these sample results were qualified as not detected, "U".
VALIDATION FIN <u>Fiel</u>	is SDG? Ithe field blanks? htts: 1_MS/MS or applied ate / Other: R								its: r applied le / Other								ociated field blank concentration ar
	Were field blanks identified in this SDG? Were target analytes detected in the field blanks? Associated sample units: 1, 4, 4, 4, 5, 5, 5, 5, 1, 1, 0, 5, 5, 1, 1, 0, 5, 5, 1, 1, 1, 0, 5, 1, 1, 1, 0, 5, 1, 1, 1, 0, 1, 1, 1, 1, 0, 1, 1, 1, 0, 1, 1, 1, 1, 1, 0, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1,	Blank	Limit						Associated sample units: Soil factor applied one) Field Blank / Binsate / Othor		Blank Action	Limit				aualified. All results	ions within five times the asso
LDC #: 19097 8/6	METHOD: Inorganics, EPA Method $\underbrace{(v,v,v)}_{Were}$ field blanks identified in this SDG? $\underbrace{(M \ N \ NA}_{Ware}$ Were field blanks identified in the field	Analyte Blank ID	RINSATE	21.0 705				Rank tinite.	tte: type: (circle		Analyte Blank ID					IL I	Samples with analyte concentral

·:.

FBLKASC.6

•							2nd Reviewer:
Method: Inorganics, Method <u>کرنے دہ</u> The correlation coefficient (r) for the calibration of	<u>غر) ()</u> for the calibra	5	<i>√<sup>0</sup>2−√</i> was recalculated.Calibration date:	lated.Calibration	date: 6/18/08	ès	
An initial or continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:	bration verificati	ion percent recc	very (%R) was re	scalculated for e	ach type of analysis	using the followi	ng formula:
%R = <u>Found X 100</u> True		Where,	Found = concent True = concent	tration of each a tration of each a	= concentration of each analyte <u>measured</u> in the analysis = concentration of each analyte in the ICV or CCV source	the analysis of th CCV source	Found = concentration of each analyte <u>measured</u> in the analysis of the ICV or CCV solution True   = concentration of each analyte in the ICV or CCV source
					Recalculated	Reported	Acceptable
Type of analysis	Analyte	Standard	Conc. (ug/L)	Area	r orr²	r or r²	(N/N)
Initial calibration		s1	20	0.01			
	NO3-N	s2	100	0.046	0.99997	06666.0	7
		s3	200	0.087			-
		S4	500	0.227			
		s5	1000	0.454			
لمعالم Calibration verification	رمی	4 000	3865.9		96.59	MK	Y
Calibration verification	μ	010	9455		94.55	94.55	
Cultibration varification	n	2 640	9.481)		96.83	96.83	~

5 10.0% of the recalculated results.\_\_

097 Bb	Lee correr
LDC #: ( (	SDG #:

VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

Page: of Reviewer: wun 2nd Reviewer:

METHOD: Inorganics, Method Sel Court

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

concentration of each analyte <u>measured</u> in the analysis of the sample. For the matrix spike calculation, Found = SSR (spiked sample result) - SR (sample result).	concentration of each analyte in the source
Found =	True =
Where,	
%R = <u>Found</u> x 100 True	

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

Original sample concentration Duplicate sample concentration	
။ ။ ပ	
RPD = <u>IS-D1</u> × 100 Where, (S+D)/2	

	•				Receiculated	Reported	
Sample ID	Type of Analysis	Element	Found / S (unite)	True / D (units)	%R / RPD	%R / RPD	Acceptable (Y/N)
	Laboratory control sample						
rcy		0-pop-r	19.14	80	99	99	<u>&gt;</u>
	Matrix spike sample		(SSR-SR)				
4		<del>8</del>	d'art	オート	96	Ħ	~
þ	Duplicate sample	tos	しょう	14.4	2.	e t	>
						)	

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.

TOTCLC.6

 N N/A
 Have results been reported and calculated correctly?

 N N/A
 Are results within the calibrated range of the instruments?

 ON N/A
 Are all detection limits below the CRQL?

Concentration =

Recalculation:

Soy= 0.314 × 10 × 0.0412 = 284 mg/mg 904 = Are XDF

reported with a positive detect were

#	Sample ID	Analyte	Reparted Concentration (Mg/73)	Calculated Concentration ( WS/W)	Acceptable (Y/N)
1	2	chionti il il F	1.2	1~0	Y
		l	14.6	14.6	↓ · · · · ·
		U <sub>2</sub>	29.2	29,2	
		F	1-0	10	
		N03-N 504	1.3	1.3	
		504	285	284	M
			-		
		· · · · · · · · · · · · · · · · · · ·			ļ
		· · · · · · · · · · · · · · · · · · ·			
			-		
					1
					1
		······································	- <u> </u>		
			· ·	1	

Note:

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

Project/Site Name:	BRC Tronox Parcel G

Soil

Collection Date: June 11, 2008

LDC Report Date: July 22, 2008

Matrix:

Parameters: Gasoline Range Organics

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120167

### Sample Identification

TSB-GJ-08-10 TSB-GJ-08-10RE TSB-GJ-08-20\*\* TSB-GJ-08-30\*\* TSB-GJ-08-40 TSB-GJ-08-10MS TSB-GJ-08-10DUP

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 7 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.

Sample "RINSATE 1" (from SDG F8F120137) was identified as a rinsate. No gasoline range organic contaminants were found in this blank.

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

### a. Surrogate Recovery

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

### b. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) samples were reviewed for each matrix as applicable. Percent recoveries (%R) were within QC limits.

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable. Results 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 F8F120167

No Sample Data Qualified in this SDG

BRC Tronox Parcel G Gasoline Range Organics - Laboratory Blank Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

BRC Tronox Parcel G Gasoline Range Organics - Field Blank Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

LDC #: <u>19097B7</u> SDG #: <u>F8F120167</u> Laboratory: <u>Test America</u>

### Level III/IV

Date: <u>7/21/08</u> Page: <u>/ of /</u> viewer: Reviewer: 2nd Reviewer:

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

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

	Validation Area		Comments
١.	Technical holding times	A	Sampling dates: 6/(1/0 ¥
lla.	Initial calibration	4	1
IIb.	Calibration verification/ICV	Å	$ c  \leq  c $
	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates /Dup	A/A	NO MSD
IVc.	Laboratory control samples	A	Laslp
V.	Target compound identification	4	Not reviewed for Level III validation.
VI.	Compound Quantitation and CRQLs	4	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	
Χ.	Field blanks	DU	R: Pinsate 1

Note:

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

SOIL

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

7 1	TSB-GJ-08-10	11	F8F130000-269	21 /	8165269	31	6/14
21	TSB-GJ-08-10RE	12	F8F170000-178	22	8169178	32	6/17
31	TSB-GJ-08-20**	13		23		33	
4/	TSB-GJ-08-30**	14		24		34	
52	TSB-GJ-08-40	15		25		35	
6 /	TSB-GJ-08-10MS	16		26		36	
7-	TSB-GJ-08-10MSD	17		27		37	
8 /	TSB-GJ-08-10DUP	18		28		38	
9		19		29		39	
10		20		30		40	

Notes:

LDC #: 19097B7 SDG #: peu coner

VALIDATION FINDINGS CHECKLIST

Page: \_/of \_2 Reviewer: \_\_\_\_7 2nd Reviewer: \_\_\_\_7

Method:	GC	HPLC					_			
Va	lidation Area			Yes	No	NA		Findings/0	Comments	
1 Technical holding times							1.1			
All technical holding times were me				/	1					
Cooler temperature criteria was met				-	ſ			·		
11 Initial calunation									Directory	
Did the laboratory perform a 5 point	calibration prior	to sample anal	ysis?							
Was a linear fit used for evaluation? deviations (%RSD) < 20%?	If yes, were all	percent relative	standard	-	ł					
Was a curve fit used for evaluation? used?	If Yes, what wa	s the acceptanc	ce criteria		_	-				
Did the initial calibration meet the cu	rve fit acceptan	ce criteria?				-				
Were the RT windows properly estat	lished?			-	f				_	
MitConficting Calibration						Таруй				
What type of continuing calibration c %R	alculation was p	erformed?	_%D or	/	-					
Was a continuing calibration analyze	d daily?									
Were all percent differences (%D) <	15%.0 or percer	nt recoveries 85	-115%?							
Were all the retention times within the	e acceptance w	indows?		<						
V Blanks				¥ 2.		ЭН)			- <b>(.</b> 34-34)	
Was a method blank associated with	every sample ir	this SDG?		$\langle \rangle$						
Was a method blank analyzed for each	ch matrix and co	oncentration?		_	-					
Was there contamination in the methor validation completeness worksheet.	od blanks? If ye	s, please see th	ie Blanks		1	-				
n VI Simogate spikes						99£				
Were all surrogate %R within the QC	limits?			7						
If the percent recovery (%R) of one or a reanalysis performed to confirm %R	more surrogate	es was outside (	QC limits, was			_				
If any %R was less than 10 percent, w	as a reanalysis	performed to c	onfirm %R?			7	-			
VII. Matux spike/Matux spike duplicate	S OF LESSING		和初期	sų.					Andria	
Were a matrix spike (MS) and matrix s matrix in this SDG? If no, indicate whi MS/MSD. Soil / Water.	spike duplicate ( ch matrix does r	MSD) analyzed tot have an ass	for each ociated	7	-					
Was a MS/MSD analyzed every 20 sa	mples of each n	natrix?		イ			<u></u>			
Were the MS/MSD percent recoveries (RPD) within the QC limits?			differences	7					<u> </u>	
All Laboratory control samples									16.2 · · ·	
Nas an LCS analyzed for this SDG?				7	Τ					x of the state of the
Vas an LCS analyzed per extraction b	atch?			7	_					
Vere the LCS percent recoveries (%R vithin the QC limits?	) and relative pe	ercent difference	e (RPD)	7					<u> </u>	

LDC #: <u>[9097B7</u> SDG #: <u>eu coner</u>

### VALIDATION FINDINGS CHECKLIST

Page: 2 of 2 Reviewer: 7 2nd Reviewer: 7

Validation Area	Yes	No	NA	Findings/Comments
IX. Regional Quality Assurance and Quality Control	in see			
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?			a	
X larger compound identification as 2 and 2 an				
Were the retention times of reported detects within the RT windows?			/	
XI Compound quantitation/GROLs				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?		-		
Missister responses as a second s				
System performance was found to be acceptable.		-		
Overall assessment of data was found to be acceptable.			T	
KIV Feldiupicates				
Field duplicate pairs were identified in this SDG.		7	-	
Farget compounds were detected in the field duplicates.			コ	-
W. Elelapians				
ield blanks were identified in this SDG.	$\neg$		Τ	
arget compounds were detected in the field blanks.	_	7		

"New

~	$\overline{\mathbf{x}}$
70	Tra
60	2
6 1	Fr I
#	 ₩
Ö	SDG

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

of 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

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

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

Standard ID     Catholic Data     Compound     (人ご Catholic Mineral final)     Average CF     Average CF     Average CF     Merage CF     M				Reported	Recalculated	Reported	Recalculated	Reported	b a tablication of the second s
	# Standard ID	Calibration Date		CF (/· <sup>O</sup> std)	CF (/ Ostd)	Average CF (initial)	Average CF (initial)	KSD	
		Onlist c	0×5	64252011	61952061	17/82732-		3-715	3-515
	-			·					
			· · ·			A			
	<u> </u>								
				-					
						-			

282606 Care 3 LDC #: SDG #:

**Continuing Calibration Results Verification** VALIDATION FINDINGS WORKSHEET

ð 2nd Reviewer: Reviewer. Page:

HPLO 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

				<u></u>				
					Reported	Recalculated	Reported	Persirilated
Standard ID Date		Calibration Date	Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc.	۵%	a%
1CAL396 B	_	80/21/2	GRU	1.0	0.9982	0.9982	4.0	9.2
-								
		<b>4</b> .						
	- 11							
		<b>.</b>						
		·						
	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

SDG # Level

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

Page: ot / ſ 2nd reviewer: Reviewer:

METHOD: \_\_\_\_ GG\_\_\_ HPLC

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Percent Difference

Percent Recovery

Percent Recovery

I Į 0

Recalculated

Reported

ر) ل

3

Sample ID: # 3		-		
Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	
797	Vitingenton	0.04	0 0 > 300	

Sample ID:

Surrogate Surrogate Surrogate ColumnDetector Spiked ColumnDetector Spiked Recovery Recovery Recovery Recovery Country and ColumnDetector Country and ColumnDetector ColumnDetec							
	Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
					Reported	Recalculated	

Sample ID:

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

LDC #: 1 909710/ cores SDG #: 11

## <u>Matrix Spike/Matrix Spike Duplicates Results Verification</u> VALIDATION FINDINGS WORKSHEET

Page: / of Z Reviewer: 2nd Reviewer:

HPLC 90 **METHOD:** 

The percent recoveries (%R) and relative percent differences (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using Where the following calculation: %Recovery = 100 \* (SSC - SC)/SA

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

ل

MS/MSD samples:

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

SC = Sample concentration

MSD = Matrix spike duplicate

		Spike		Sample	Spike S	Sample	Matrix	Matrix enika	Mateir Catt			
~		Added	4.	Conc.	Concentration	tration		opino.	matrix spike pupiicate	• Uuplicate	DSW/SW	sp
Compound		ž	EX	KI Ku	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	14	Percent Recovery	Recovery	Percent Recovery	ecoverv	Uda	
		MS	MSD	)   1	WS	ر MSD	Reported	Recalc	Penorted	Baada		
Gasoline (8015)	1	1.07	44	0	2 0.1	41	ò	16	nationav.	Vecalc.	реподех	Recalc.
Diesel (8015)					)		10	9	24			
Benzene (8021B)												
Methane (RSK-175)	5)											
2,4-D (8151)												
Dinoseb (8151)												
Naphthalene (8310)												
Anthracene (8310)												
HMX (8330)												
2,4,6-Trinitrotoluene (8330)	30)											
												T
Comments: Refer to Matrix Spike/Matrix Spike Duplicates findings worksheet for list of gualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.	ix Spike/N	latrix Sc	oike Dupli	cates findings	worksheet fo	or list of qualif	cations and a	ssociated sam	i ples when rep	) orted results	do not agree	within 10.0%

MSDCLCNew.wpd

	Laboratory Con
LDC #: 1909787	SDG #: M comen

GC HPLC

METHOD:

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

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

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

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

Where SSC = Spiked concentration SA = Spike added

SC = Sample concentration

LCS = Laboratory Control Sample percent recovery

RPD =(({ssclcs - ssclcsD} \* 2) / (ssclcs + ssclcsD))\*100

LCS/LCSD samples: 8/6 526 7 - 103 11

LCSD = Laboratory Control Sample duplicate percent recovery

		Spike	e l	Sample	Spike S	Spike Sample		LCS	rcsd	٥	rcs/rcsd	SD
Compound	nnd	Ku -	[ks	Lone.	Concer ( My	tration ///	Percent F	Percent Recovery	Percent Recovery	ecovery	RPD	
		LCS	LCSD		rcs		Reported	Recaic.	Reported	Recalc.	Reported	Recalc.
Gasoline	(8015)	1.0	1.0		0.1	0-944	100	0el	76	74	62	5.2
Diesel	(8015)											
Benzene	(8021B)			-								
Methane	(RSK-175)											
2,4-D (	(8151)											
Dinoseb	(8151)											
Naphthalene	(8310)											
Anthracene	(8310)											
HMX	(8330)											
2,4,6-Trinitrotoluene (8330)	uene (8330)											
Comments: Refer to Laboratory Control Sample/Laboratory Contr	er to Laborator	y Control	Sample/L	aboratory Col	ntrol Sample	Duplicate fine	dings worksh	eet for list of c	ol Sample Duplicate findings worksheet for list of qualifications and associated samples when reported	nd associate	d samples wt	ien reported

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

soc LDC	LDC #: 1909 7 B 7 SDG #: 244 Conor	VALIDATI Sampl	VALIDATION FINDINGS WORKSHEET Sample Calculation Verification	HEET on	Page: <u>of</u> Reviewer:
Ш М	METHOD: GC HPLC				2nd Reviewer:
	NIA	Were all reported results recalculated and verified for all level IV samples? Were all recalculated results for detected target compounds within 10% of the reported results?	or all level IV samples? pounds within 10% of the rep	oorted results?	
Con	Concentration <del>=</del> ( <u>A)(Fv)(Df)</u> (RF)(Vs or Ws)(%S/100)				
A F T T T T	Area or height of the compound to be measured Final Volume of extract Dilution Factor	asured Sample ID	Comp	Compound Name	
RF= VS= %S=	RF= Average response factor of the compound in the initial calibration Vs= initial volume of the sample Ws= Initial weight of the sample %S= Percent Soild	d Concentration =	14	(IN)	
	•	• • • •		-	
#	Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications
<u> </u>					
<u> </u>					
Comr	Comments:				
.				•	

SAMPCALew.wpd

ŝ.

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

**Diesel Range Organics** 

<b>Project/Site</b>	Name:	BRC Tronox Parcel G
---------------------	-------	---------------------

Soil

Collection Date: June 11, 2008

LDC Report Date: July 22, 2008

Matrix:

Parameters:

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120167

### Sample Identification

TSB-GJ-08-10 TSB-GJ-08-20\*\* TSB-GJ-08-30\*\* TSB-GJ-08-40 TSB-GJ-08-10MS TSB-GJ-08-10MSD

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 6 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.

Sample "RINSATE 1" (from SDG F8F120137) was identified as a rinsate. No diesel range organic contaminants were found in this blank.

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

### a. Surrogate Recovery

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

Sample	Surrogate	%R (Limits)	Compound	Flag	A or P
TSB-GJ-08-20**	ortho-Terphenyl	41 (75-150)	Diesel range organics	J- (all detects) UJ (all non-detects)	Ρ

### 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 F8F120167

SDG	Sample	Compound	Flag	A or P	Reason
F8F120167	TSB-GJ-08-20**	Diesel range organics	J- (all detects) UJ (all non-detects)	Ρ	Surrogate recovery (%R)

BRC Tronox Parcel G

Diesel Range Organics - Laboratory Blank Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

**BRC Tronox Parcel G** 

Diesel Range Organics - Field Blank Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

LDC #: 19097B8	VALIDATION COMPLETENESS WORKSHEET	Date: 7/21/08
SDG #: F8F120167	Level III/IV	Page:_/of/
Laboratory: Test America		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
1.	Technical holding times	Δ	Sampling dates: 6/11/0 ¥
lla.	Initial calibration	Α	
llb.	Calibration verification/ICV	4	101 = 15
.	Blanks	A	
IVa.	Surrogate recovery	SW	
IVb.	Matrix spike/Matrix spike duplicates	Δ	
IVc.	Laboratory control samples	A	Les
V.	Target compound identification	<u>A</u>	Not reviewed for Level III validation.
VI.	Compound Quantitation and CRQLs	A	Not reviewed for Level III validation.
VII.	System Performance	A	Not reviewed for Level III validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N	
Х.	Field blanks	ND	R = Pinsate / SDG # F8F/2013

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

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

valiue	ited Samples. Indic		ample underwent Leventy v			
1	TSB-GJ-08-10	11	•	21	31	
2	TSB-GJ-08-20**	12		22	32	
3	TSB-GJ-08-30**	13		23	· 33	
4	TSB-GJ-08-40	14		24	34	
5	TSB-GJ-08-10MS	15		25	35	
6	TSB-GJ-08-10MSD	16		26	36	
7	P8 F130000-291	17	8165291	27	37	
8	F8F180000-312	18	8170312	28	38	
9		19		29	39	
10		20		30	40	

Notes:

LDC #: 1909788 SDG #: per comer

**VALIDATION FINDINGS CHECKLIST** 

T

Т

Ť

7

Page: /of <u>1</u> Reviewer: <u>7</u> 2nd Reviewer: <u>7</u>

Method:

ſŕ

GC

HPLC

Validation Area	Ye	s N	<u>0 N</u>	A Findings/Comments
1. Technical holding times				
All technical holding times were met.		1		
Cooler temperature criteria was met.	/	1		
III. Tradital realistments				
Did the laboratory perform a 5 point calibration prior to sample analysis?	-	1_		
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) $\leq$ 20%?	/	1		
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?		-	1	
Did the initial calibration meet the curve fit acceptance criteria?			-	
Were the RT windows properly established?	/ /	$\vdash$		
IN-Continuing calibration				
What type of continuing calibration calculation was performed?%D or %R	/	t		
Was a continuing calibration analyzed daily?	/-	Ł		
Were all percent differences (%D) $\leq$ 15%.0 or percent recoveries 85-115%?				
Were all the retention times within the acceptance windows?		$\mathbf{k}$		
V Blanks				
Was a method blank associated with every sample in this SDG?	$\left \right $	L		
Was a method blank analyzed for each matrix and concentration?	/	Ĺ		
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
W Simogate spikes				
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. Malox sokerMatrix spike duplicates/ t	题的:		È.	
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			
Was a MS/MSD analyzed every 20 samples of each matrix?	7	_		
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	/	_		
VIII Laboratory control samples		<b>医</b> 静		
Nas an LCS analyzed for this SDG?	$\neg$			
Nas an LCS analyzed per extraction batch?	7			
Nere the LCS percent recoveries (%R) and relative percent difference (RPD) vithin the QC limits?	/			

LDC #: 19097B8 SDG #: 41 Coner

### VALIDATION FINDINGS CHECKLIST

Page: 20f 2 Reviewer: 7 2nd Reviewer: 6

Validation Area	Yes	No	NA	Findings/Comments
IX. Regional Quality Assurance and Quality Control	in since	b P		r mongsconments
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				-
Xt liarget compound identification 22/2 at 12 and 20 at 20				
Were the retention times of reported detects within the RT windows?			/	· · ·
N. Lompound quantitation/GRQLs				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
System performance was found to be acceptable.	/	-		
XUN executions essential data (2007) and a second data (2007) and a second data (2007) and a second data (2007)				
Overall assessment of data was found to be acceptable.	1		T	
AV Fold publicates of the second s				
Field duplicate pairs were identified in this SDG.	Ţ		-	
Farget compounds were detected in the field duplicates.			7	
AV Ejelopianis				and the second
ield blanks were identified in this SDG.	T	-1	Ţ	
arget compounds were detected in the field blanks.		オ	$\neg$	

a second

τ. Α

DG #: Le Control
SDG 1

## VALIDATION FINDINDS WORKSHEET Surrogate Recovery



METHOD: \_\_\_\_\_GC \_\_\_ HPLC Are surrogates required by the method? Yes\_\_\_

NA Were surroates spiked into all samples and blanks?

									وللتركين والمتحدث والمستعد والمستعم والمستعد والمستعد والمست والمستعم والمستوال والمستعم والمستعم والمستعم والمستعم والمستعم والمستعم والمستعم والمستوال والمستوال والمستعم والمستوال والمستوال والمستوال والمستوال والمستوال والمستوال والمستوال والمست والمستوال والمستوال والمست والمستوال والمست والمستوال والمستوال	
) #	Sample ID	Detector/ Column	stor/ mn	Surrogate Compound		%R (Limits)			ğ	alifications
	2 ·	Not	+ specifit	H V		· ) //	1-54	ר   י <u>הי</u> י	2/ [n]	/ <del> </del>
			0 1					(		
								(		
						)		(		
						)		(		
								) (		
								(		
						)				
						}				
								(		
						)		) (		
						<u>,</u>		(		
								] (		
								) (		
								(		
						· •		(		
								(		
								) (		
	Surrogate Compound		Surrog	Surrogate Compound		Surrogate Compound		Surrogate Compound		
٩	Chiorobenzene (CBZ)	U	ő	Octacosane	Σ	Benzo(e)Pyrene	s	1-Chloro-3-Nitrobenzene		Tetrachloro-m- xylene
8	4-Bromofluorobenzene (BFB)	I	PO	Ortho-Terphenyl	z	Terphenyl-D14	F	3,4-Dinitrotoluene		
υ	a,a,a-Trifluorotoluene	-	Fluoro	Fluorobenzene (FBZ)	0	Decachlorobiphenyl (DCB)	Э	Tripentyltin		
٥	Bromochlorobenene		ė	n-Triacontane	۵.	1-methvinaphthalene	>	Trl-n-propyltin		
ω	1,4-Dichlorobutane	×	Ī	Hexacosane	σ	Dichlorophenyl Acetic Acid (DCAA)	₹	Tributyl Phosphate		
u.	1.4-Difluorobenzene (DFB)	_	, Bro	Bromobenzene	œ	4-Nitrophenol	×	Triphenyl Phosphate		

2

882606 LDC #: SDG #:

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

ر of Page: Reviewer. 2nd Reviewer

THEC METHOD: GC

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

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

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

L									
	-			Reported	Recalculated	Reported	Receletidated	Denot	
#	Standard ID	Calibration Date	Compound	CF (/02/ Attd)	CF		Average CF		Kecalculated
	1041	5/16/08	DR ( )		Incont	(initial)	(initial)	%RSD	%RSD
	1			16236	16236	16023	16023	3.432	3.45%
	-								
м									
¢									
2					·				
T									
4									
Γ									
Corri esult	nents: <u>Refer to</u> s.	Initial Calibratio	Comments: <u>Refer to Initial Calibration findings worksheet for list of qualifications and associated samples when reported results do not arme within 10 0% of the module </u>	ons and associa	ated samples w	then reported re-	sults do not anner	within 10,0%	of the received
								0/ 0.01 11111111	OI ILLE LECAICULS

INICLU 18B

881606 Cere 3 LDC #: SDG #:

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

0 2nd Reviewer. Page: 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

% Difference = 100 ⁺ (ave. CF - CF)/ave. CF CF ≈ A/C

Where:

ave. CF = initial calibration average CF CF = continuing calibration CF

A = Area of compound C = Concentration of compound

					Reported	Recalculated	Reported	Recalculated
¥	Standard ID	Calibration Date	Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc. CCV	۵%	Q%
-	ECALS 15	6 /17/0X	DRO	0 000/	996.53	996.53	0,3	e. 9
	R41527	6/17/m						
		8-1.112	×		1034.562/	1034.5621	3,5	3.5
2								
T								
ო								
		<b>.</b>						
Τ								
Τ							2	
4		£.						
		- <b></b> - <b>I</b>						

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

88	5	
160	3	
61	77	
ן כי גי		
Ч	5	

## VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page: \_\_\_\_\_ot \_\_\_\_ 

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

#2 Sample ID:

	Spiked	Found	Recovery	Recovery	Difference
			Reported	Recalculated	
0-Terphen, 100 - Terphen	y.	10.2	//	17	0
					-

Sample ID:

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

Sample ID:

	Reported	Recalculated	
			-
	·		

SURRCALCNew.wpd

ROL 1909 108 coner SDG #: 20

## <u>Matrix Spike/Matrix Spike Duplicates Results Verification</u> VALIDATION FINDINGS WORKSHEET

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

METHOD: GC HPLC The percent recoveries (%R) and relative percent differences (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation: %Recovery = 100 \* (SSC - SC)/SA

Where

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

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

MSD = Matrix spike duplicate SC = Sample concentration

> 310 MS/MSD samples:\_

Compound( $M_{2}$ )( $M_{2}$ )( $M_{2}$ ) $M_{2}$	Percent Recovery     Percent Recovery       orted     Recalc.       Reported     Recalc.       S     S	RPD Reported Recalc.
WS WSD WS WSD WS WSD	Recalc. Reported	Reported Reca
87.2 x.8 wD 74.5 76./	×8 ×8	
87.2 K. & WD 74.5 76./	8	5
(8330)		
(8330)		

MSDCLCNew.wpd

METHOD: GC HPLC The percent recoveries (%R) and relative percent differences (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:			I Sample/	Laborator	y control	Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification	<u>iplicates F</u>	<u>tesults Veri</u>	IICAUOII		
The percent recoveries (%R) and r compounds identified below using	C									7	ZNO KEVIGWER
	relative pe the follow	ercent d ing calc	ifferences (F sulation:	RPD) of the I	aboratory co	trol sample a	and laborator	y control sam	ole duplicate	were recalcu	lated for the
%Recovery = 100 * (SSC - SC)/SA	Where		SSC ≖ Spiked concentration SA ≂ Spike added	entration		SC = Sample concentration	ncentration				
KPD =(({ssclcs - ssclcsD} * 2) / (ssclcs + ssclcsD))*100	LCS + SSCL	csD))*10	Q	LCS = Laborat	ory Control Samp	LCS = Laboratory Control Sample percent recovery		LCSD = Laboratory Control Sample duplicate percent recovery	Control Sample o	Juplicate percent	recovery
LCS/LCSD samples: 8//0 529/	527-										
	Spike		Sample	Spike Sample	ample	Ľ	LCS	rcsD	9	rcs/rcsD	csd
Compound	Added/	X	Conc.	Concen (パイ)	tration FVT	Percent F	Percent Recovery	Percent Recovery	ecovery	RPD	
		LCSD	0	LCS		Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)											
Diesel (8015) 83	ý	44	О	68.7	ケイ	8 8	83	× 4			
Benzene (8021B)			-								
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)		1									
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	control Sar	mple/La	Iboratory Co	ntrol Sample	Duplicate fin	dings worksh	eet for list of	qualifications	and associate	ed samples w	hen reported
results do not agree within 10.0% of the recalculated results	of the rec	alculate	ed results.								

and a strategy and the

 $\odot$ 

LCSCLCNew.wpd

	#: 1909 7.128	VALIDATI	VALIDATION FINDINGS WORKSHEET Sample Calculation Verification	HEET <u>on</u>	Page: Reviewer: 2nd Reviewer:
N N MET	HOD: N/A	GCHPLC Were all reported results recalculated and verified for all level IV samples? Were all recalculated results for detected target compounds within 10% of the reported results?	r all level IV samples? pounds within 10% of the re	ported results?	K
Conc Pfr Ss= %Ss= %Ss= %	Concentration= (A)(Fv)(Df) (RF)(Vs or Ws)(%S/100) A= Area or height of the compound to be measured Fv= Final Volume of extract Df= Dilution Factor RF= Average response factor of the compound In the initial calibration Vs= Initial volume of the sample Ws= Percent Solid	Concentration =		Compound Name	
#	Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications
<u> </u>					
Comr	Comments:				
	SAMPCALew.wpd			•	

ыâ

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

Polynuclear Aromatic Hydrocarbons

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

Soil

Collection Date: June 11, 2008

LDC Report Date: July 22, 2008

Parameters:

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120167

### Sample Identification

Matrix:

TSB-GJ-08-10 TSB-GJ-08-20\*\* TSB-GJ-08-30\*\* TSB-GJ-08-40 TSB-GJ-08-10MS TSB-GJ-08-10MSD

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 6 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	All samples in SDG F8F120167	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 F8F120167	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.

Sample "Rinsate 1" (from SDG F8F120137) was identified as a rinsate. No polynuclear aromatic hydrocarbon contaminants were found in this blank.

### IV. Accuracy and Precision Data

### a. Surrogate Recovery

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

### b. Matrix Spike/Matrix Spike Duplicates

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

### c. Laboratory Control Samples

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

### V. Target Compound Identification

All target compound identifications were within validation criteria for samples on which 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 F8F120167

SDG	Sample	Compound	Flag	A or P	Reason
F8F120167	TSB-GJ-08-10 TSB-GJ-08-20** TSB-GJ-08-30** TSB-GJ-08-40	Benzo(g,h,i)perylene	J+ (all detects)	A	Continuing calibration (%D)
F8F120167	TSB-GJ-08-10 TSB-GJ-08-20** TSB-GJ-08-30** TSB-GJ-08-40	Benzo(k)fluoranthene	J+ (all detects)	A	Continuing calibration (ICV %D)

### BRC Tronox Parcel G Polynuclear Aromatic Hydrocarbons - Laboratory Blank Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

### BRC Tronox Parcel G Polynuclear Aromatic Hydrocarbons - Field Blank Data Qualification Summary -SDG F8F120167

No Sample Data Qualified in this SDG

VALIDATION	COMPL	<b>ETENESS</b>	WORKSHEET
• • • • • • • • • • • • • • • • • • • •			

LDC #: <u>19097B9</u> SDG #: <u>F8F120167</u> Laboratory: <u>Test America</u>

### Level III/IV

Date:	80/12/08
Page:_	<u></u>
Reviewer:	
2nd Reviewer:	"A

1

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/11/08
lla.	Initial calibration	A	
llb.	Calibration verification/ICV	SW	$ \alpha  = 15$
111.	Blanks	Δ	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	A	
IVc.	Laboratory control samples	A	LCS
V.	Target compound identification	A	Not reviewed for Level III validation.
VI.	Compound Quantitation and CRQLs	A	Not reviewed for Level III validation.
VII.	System Performance	4	Not reviewed for Level III validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N	
Х.	Field blanks	ND	R = Rinsati / SDG # 18 F/20137

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

	2012			<u> </u>			
1	TSB-GJ-08-10	11	F8 1=160000-153	21	8168158	31	
2	TSB-GJ-08-20**	12		22		32	
3	TSB-GJ-08-30**	13		23		33	
4	TSB-GJ-08-40	14		24		34	
5	TSB-GJ-08-10MS	15		25		35	
6	TSB-GJ-08-10MSD	16		26		36	
7		17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	

Notes:

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

Validation Area     Yza     No     Nz     Findings/Comments       Lite/Activation     Lite/Activation     Lite/Activation     Lite/Activation     Lite/Activation       All lechnical holding times were met.	Method:GCHPLC				
All technical holding times were met. Coder temperature criteria was met. Did the laboratory perform a 5 point calibration prior to sample analysis? Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) 5 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 property established? Was a curve fit used for evaluation analyzed billing? Was a curve fit used for evaluation calculation was performed? Was a curve fit used for evaluation calculation was performed? Was a curve fit used for evaluation calculation was performed? Was a continuing calibration analyzed daily? Was a continuing calibration analyzed daily? Were all the retention times within the acceptance windows? Was a method blank associated with every sample in this SDG? Was a method blank associated with every sample in this SDG? Was there contamination in the method blanks? If yes, please see the Blanks talication completeness workshort. Were all surogate %R within the QC limits? If the percent efformed to confirm %R? If the percent efformed to confirm %R? Was a method blank associated with every sample in this SDG? Was an method blank associated with every sample in this SDG? Was an embod blank associated with every sample in this SDG? Was an embod blank associated with every sample in this SDG? Was an embod blank associated with every sample in this SDG? Was an embod blank associated with every sample in this SDG? Was a method blank associated with every sample in this SDG? Was a method blank associated with every sample in this SDG? Was an anethod blank associated with every sample was outside QC limits, was a a canadysis performed to confirm %R? Were all have been associated with every as a reanalysis performed to confirm %R? Was a Mint be QC limits? Was have the Mint be QC limits? Was a Mint be QC limits? Was have the Mint be QC limits? Was a metho	Validation Area	Ye	s No	NA	
Cooler temperature criteria was met.	I ledinical fielding imes				
Did the taboratory perform a 5 point calibration prior to sample analysis?       Image: Construction of the evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?	All technical holding times were met.	$\leq$	-		
Did the taboratory perform a 5 point calibration prior to sample analysis?         Was a linear fit used for evaluation? If yes, what was the acceptance criteria         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?         Wore the RT windows property established?         What type of continuing calibration calculation was performed?         Was a continuing calibration analyzed daily?         Were all percent differences (%D) < 15%. 0 or percent recoveries 85-115%?	Cooler temperature criteria was met.		1		
Was a linear fit used for evaluation? If yes, were all percent relative standard         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?         What type of continuing calibration calculation was performed?         What type of continuing calibration calculation was performed?         Was a continuing calibration analyzed daily?         Were all percent differences (%D) < 15% 0 or percent recoveries 85-115%?					
deviations (%RSD) < 20%?	Did the laboratory perform a 5 point calibration prior to sample analysis?	//	1	$\perp$	
used?	Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) $\leq$ 20%?	/~	+		
Were the RT windows property established?			-	4	T
We settletter and the settletter and th	Did the initial calibration meet the curve fit acceptance criteria?			-	
What type of continuing calibration calculation was performed?%D or      %D or         Was a continuing calibration analyzed daily?	Were the RT windows properly established?		Ł		
%R       ✓         Was a continuing calibration analyzed daily?       ✓         Were all percent differences (%D) ≤ 15%.0 or percent recoveries 85-115%?       ✓         Were all the retention times within the acceptance windows?       ✓         Was a method blank associated with every sample in this SDG?       ✓         Was a method blank analyzed for each matrix and concentration?       ✓         Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.       ✓         Were all surrogate %R within 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?       ✓         If any %R was less than 10 percent, was a reanalysis performed to confirm %R?       ✓         If any %R was less than 10 percent, was a reanalysis performed to confirm %R?       ✓         If any %R was less than 10 percent, was a reanalysis performed to confirm %R?       ✓         If any %R was less than 10 percent, was a reanalysis performed to confirm %R?       ✓         If any %R was less than 10 percent, was a reanalysis performed to confirm %R?       ✓         If any %R was less than 10 percent, was a reanalysis performed to confirm %R?       ✓         If any %R was less than 10 percent, was a reanalysis performed to confir	IN ASsound any contraction that the second state of the second state of the second state of the second state of				
Were all percent differences (%D) ≤ 15%.0 or percent recoveries 85-115%?         Were all the retention times within the acceptance windows?         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.         With the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?         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?         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 wisMSD. Soil / Water.         Nas a MS/MSD analyzed every 20 samples of each matrix?         Vere the MS/MSD precent recoveries (%R) and the relative percent differences RPD) within the QC limits?		-			
Were all the retention times within the acceptance windows?         Value         Was a method blank associated with every sample in this SDG?         Was a method blank analyzed for each matrix and concentration?         Was a method blank analyzed for each matrix and concentration?         Was a method blank analyzed for each matrix and concentration?         Was a method blank analyzed for each matrix and concentration?         Was a method blanks analyzed for each matrix and concentration?         Was a method blank analyzed for each matrix and concentration?         Was a method blank analyzed for each matrix and concentration?         Was a method blank analyzed for each matrix and concentration?         Was a method blank analyzed for each matrix and concentration?         Was a method blank analyzed for each matrix and concentration?         Was 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.         Nas a MS/MSD paralyzed every 20 samples of each matrix?         Vere the MS/MSD percent recoveries (%R) and the relative percent differences RPD) within the OC limits?         It Alaborator controls simple	Was a continuing calibration analyzed daily?	-			
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.         Wishing indeps         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?         Winditian spike duplicate?         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 percent recoveries (%R) and the relative percent differences RPD) within the QC limits?	Were all percent differences (%D) $\leq$ 15%.0 or percent recoveries 85-115%?		-	t	
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.         Wishing the spore         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?         Windux specedult which 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?         Was a MS/MSD percent recoveries (%R) and the relative percent differences         Were the MS/MSD percent recoveries (%R) and the relative percent differences         Was a MS/MSD panalyzed every 20 samples of each matrix?         Was a MS/MSD panalyzed samples	Were all the retention times within the acceptance windows?	/	t		
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.         With organize pake.         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?         VillMation spice Matrix spike duplicates         Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix spike (MS) and 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 CC limits?			<b>1</b> 23	民族	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.  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?  If any %R was less than 10 percent, was a reanalysis performed to confirm %R?  Were a 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.  Nas a MS/MSD analyzed every 20 samples of each matrix?  Nere the MS/MSD percent recoveries (%R) and the relative percent differences RPD) within the QC limits?	Was a method blank associated with every sample in this SDG?	-			
validation completeness worksheet.	Was a method blank analyzed for each matrix and concentration?				
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?         If any %R was less than 10 percent, was a reanalysis performed to confirm %R?         VillMator spice duplicates         Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDC? If no, indicate which matrix does not have an associated         MS/MSD. Soil / Water.         Nas 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?         Ill a bioratory control samples			-	ł	
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? With any spikentiatix 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. Nas 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?	Wishing the solution of the so				
a reanalysis performed to confirm %R? If any %R was less than 10 percent, was a reanalysis performed to confirm %R? VIIMator 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. Nas 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?	Were all surrogate %R within the QC limits?	1-1			
VIII Maliox 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.         Nas a MS/MSD analyzed every 20 samples of each matrix?         Nere the MS/MSD percent recoveries (%R) and the relative percent differences RPD) within the QC limits?         III Teboratory control samples	If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?			7	
VIII Maliox 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.         Nas a MS/MSD analyzed every 20 samples of each matrix?         Nere the MS/MSD percent recoveries (%R) and the relative percent differences RPD) within the QC limits?         III Teboratory control samples	If any %R was less than 10 percent, was a reanalysis performed to confirm %R?				/
natrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water. Nas a MS/MSD analyzed every 20 samples of each matrix? Were the MS/MSD percent recoveries (%R) and the relative percent differences RPD) within the QC limits?					
Was a MS/MSD analyzed every 20 samples of each matrix?         Nere the MS/MSD percent recoveries (%R) and the relative percent differences         RPD) within the QC limits?         IIII Teboratory control samples	natrix in this SDG? If no, indicate which matrix does not have an associated		-		
Vere the MS/MSD percent recoveries (%R) and the relative percent differences RPD) within the QC limits?  IIII aboratory control samples		7	-		
RPD) within the QC limits?		17	-		
	RPD) within the QC limits?	Sandara Maria	1000 0000		
Vas an LCS analyzed for this SDG?	All Aslocatory control samples				
	Vas an LCS analyzed for this SDG?	-1			
Vas an LCS analyzed per extraction batch?	Vas an LCS analyzed per extraction batch?	$\square$			
Vere the LCS percent recoveries (%R) and relative percent difference (RPD)	Vere the LCS percent recoveries (%R) and relative percent difference (RPD) ithin the QC limits?	-			

LDC #: 1909739 SDG #: <u>fu coner</u>

### VALIDATION FINDINGS CHECKLIST

Page: 20f 2 Reviewer: 9 2nd Reviewer: 4

Validation Area	Yes	No	NA	Findings/Comments
IX. Regional Quality Assurance and Quality Control Assurance and Quality Control	196			
Were performance evaluation (PE) samples performed?			/	
Were the performance evaluation (PE) samples within the acceptance limits?			~	
A appendix an addition of the second s				
Were the retention times of reported detects within the RT windows?	Γ			
d Compound quantization (c) (q)				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
system performance was found to be acceptable.		-	Τ	
verall assessment of data was found to be acceptable.	7			
eld duplicate pairs were identified in this SDG.		7	-	
arget compounds were detected in the field duplicates.				
eld blanks were identified in this SDG.				
rget compounds were detected in the field blanks.		$\geq$	+	

"New

GC\_HPLC-SW.wpd version 1.0

LDC #: 1 902789 SDG #: ALL LOWIN

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

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

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

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

 Y
 W/A
 Were continuing calibration standards analyzed at the required frequencies?
 Y

 Y
 N/A
 Did the continuing calibration standards meet the %D / RPD validation criteria of <15.0%?</td>

ever IV Only N N/A

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

					_		 			_	-	-	-		 	 		_		 	_	
	Qualifications	1+ /A clt			1-122.1	~ // ~																
	Associated Samples	A11 + B1K			1	*																
	RT (limit)	( )	(				1	1 multis low	() pary/ene)				( )	~ 		( )	)	(	(			
%D/RPD	(Limit < 15.0)	16-6			15.2		Repres (x)		Behzer (9, hU													
	Compound	H			5		H - D0		G = Be	-												
Detector/	Column	not grugen	1 1		>																	
	Standard ID	\$ 10 V 76 8			6/16/08 BCAL873																	
	Date	e/4/00		1	6/16/08																	
	# -	•			+																	

97.89	could
190.	AL
	"# 00S

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

of 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

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

- 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 Recalculated %RSD 7.2 17. P. Reported KRSD ろう 1-821 Recalculated Average CF (initial) 23507 012908 Average CF (initial) Reported OILDAR 225 527 **Recalculated** CF (の-S std) 815134 Jacke CF (0-5 std) Reported りってんそ 81573Y A = Area of compound, C = Concentration of compound, S = Standard deviation of the CF X = Mean of the CFs Compound Nophalene Anthrace Calibration 6/4/08 Date Standard ID 1001 \* Q ო 4

INICI D 1SB

en caner LDC #: 1909789 SDG #:

**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

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

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

					Reported	Recalculated	Reported	Recalculated
*			Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc. CCV	۵%	a%
-	QCA 4873	6/16/08	nghthalen-	r.0	X265 3	S. 3978	0.3	6.0
			anthracer	a D	0.5307	0.5307	6./	6,1
								)
2								
0								
						•		
4								
					·			
		•						
				-				
	ments: <u>Refer to</u>	Continuing Ca	Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported monito do and in the second samples when reported monito do and an associated samples when reported monito do associated samples when reported monitor do associated samples when reported monitor do associated samples when reported monitor do as	qualifications and	associated samp	les when reported s		
	המומובת ובסחווס.					1 101100010010000	ESUIS UN HOL BOLE	<u>se witnin 10,0% o</u>

CONCLC.1S

÷.

METHOD: \_\_\_\_\_\_\_\_ HPLC DC #: 1707107 SDG #: LLL COM

### VALIDATION FINDINGS WURKSHEEI **Surrogate Results Verification**

rage: 01 Reviewer: 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

y Sample ID:

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
p - terpheny /	not specifical	X	21. 3996	98	876	ତ
	1 1					

### Sample ID:

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

### Sample ID:

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

SURRCALCNew.wpd

coner SDG #: 20

## <u>Matrix Spike/Matrix Spike Duplicates Results Verification</u> VALIDATION FINDINGS WORKSHEET

∠of ∕ Reviewer: Page: 2nd Reviewer:\_\_

METHOD: GC HPLC The percent recoveries (%R) and relative percent differences (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using Where the following calculation: %Recovery = 100 \* (SSC - SC)/SA

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

و

4 ()

MS/MSD samples:

SC = Sample concentration

MSD = Matrix spike duplicate

	S	Spike	Sample	Spike	Spike Samole						
Compound	¥ :	Add90	Confr	Conc	Concentration	matr	matrix spike	Matrix Spik	Matrix Spike Duplicate	DSW/SW	SD
こうちょう うちがい たちまち あいたい ほうぼう		9		<u> な</u>	121	Percent	Percent Recovery	Percent 1	Percent Recovery	Caa	
「「「「「「「」」」」」「「」」」」」」」「「「」」」」」」」」」」」」」」	WS	MSD		MS	C MSD	Reported	Recalc				
Gasoline (8015)								Veported	Kecalc.	Reported	Recalc.
Diesel (8015)											
Benzene (8021B)											
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)	69 X	601	an	145	17 - 5	72	1				
Anthracene (8310)	67.X	70.9		3 5	10/1		12	(3	73	л Г С	2,0
HMX (8330)	2			1.70	77.6	16	16	20	70	6.5	6.5
2,4,6-Trinitrotoluene (8330)											
Comments: Refer to Matrix Sp	ike/Matrix :	Spike Dupli	cates finding	e workshoot							
of the recalculated results.				1991 ICU ICU C	ini list of gual	lications and a	<u>ssociated sam</u>	ples when rep	orted results	do not agree v	<u>vithin 10.0%</u>

MSDCLCNew.wpd

LDC #: 1909789 SDG #: 1909789	aborator	v Conti	VALID Laboratory Control Sample/Lal	VALIDATION <u>pie/Laborato</u> i	FINDINGS	ATION FINDINGS WORKSHEET boratory Control Sample Duplic	IEET Iplicates R	ATION FINDINGS WORKSHEET boratory Control Sample Duplicates Results Verification	<u>ification</u>	Page: Reviewer;	, of
METHOD: GC	HPLC		·							2nd	2nd Reviewer:
The percent recoveries (%R) and relative percent differences (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:	and relative using the fol	e percent lowing ca	differences ( alculation:	(RPD) of the	laboratory co	ntrol sample	and laborator	y control sam	ple duplicate	were recalcula	ted for the
%Recovery = 100 * (SSC - SC)/SA		Where S	SSC = Spiked concentration SA = Spike added	ncentration		SC = Sample concentration	ncentration				
RPD =(((SSCLCS - SSCLCSD) * 2)/(SSCLCS + SSCLCSD))*100 LCS/LCSD samples:	- 2) / (SSCLCS + SS	scLcsD))*	8	LCS = Labora	tory Control Sam	LCS = Laboratory Control Sample percent recovery		SD = Laboratory	Control Sample o	LCSD = Laboratory Control Sample duplicate percent recovery	covery
	Spike		Sample	Spike 5	Spike Sample		LCS	rcsD	0	rcs/rcsD	a
Compound	1 49/PX	PEX	ung // Kr		Concentration	Percent F	Percent Recovery	Percent Recovery	ecovery	QAN	
	LCS	LCSD	<b>)</b>	LCS		Reported	Recalc.	Reported	Recalc.	Reported	Recalc
Gasoline (8015)											
Diesel (8015)											
Benzene (8021B)											
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)	267	рЛ		484	42	73	37				
Anthracene (8310)	66.7	7		21.2	1	77	77	NA/			
HMX (8330)	,										
2,4,6-Trinitrotoluene (8330)											
Comments: <u>Refer to Laboratory Control Sample/Laboratory Control</u> results do not agree within 10.0% of the recalculated results	ory Control S	Sample/L	aboratory Co		Duplicate fin	<u>dings workshe</u>	set for list of g	ualifications a	and associate	Sample Duplicate findings worksheet for list of qualifications and associated samples when reported	n reported

LCSCLCNew.wpd

	LDC #: 19097 137 SDG #: 224 conor	VALIDAT Sampl	VALIDATION FINDINGS WORKSHEET Sample Calculation Verification	HEET <u>on</u>	Page: <u>of</u> Reviewer:
ME	METHOD: GC HPLC			•	2nd Reviewer:
≻≻	N N/A Were all reported re N N/A Were all recalculate	Were all reported results recalculated and verified for all level IV samples? Were all recalculated results for detected target compounds within 10% of the reported results?	or all level IV samples? 1pounds within 10% of the rep	ported results?	
ပိ	Concentration= (A)(Fv)(Df) (RF)(Vs or Ws)(%S/100)	5) Example:			
A P T T T T T T	Area or heigh Final Votume Dilution Fact	asured Sample ID.		Compound Name	
R F	RF≖ Average response factor of the compound In the initial calibration	d Concentration =	11		
°S 88 88 88	Vs≖ Initial volume of the sample Ws≖ Initial weight of the sample %S≡ Percent Solid				
<u> </u>	# Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications
	· ·				
<u> </u>					
<u> </u>					
Con I	Comments:				

SAMPCALew.wpd

r

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

<b>Project/Site</b>	Name:	BRC Tronox Parcel G
---------------------	-------	---------------------

Collection Date: June 11, 2008

LDC Report Date: July 23, 2008

Matrix:

Parameters: Dioxins/Dibenzofurans

Soil

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120167

### Sample Identification

TSB-GJ-08-10 TSB-GJ-08-20\*\* TSB-GJ-08-30\*\* TSB-GJ-08-40

\*\*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.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

### III. Initial Calibration

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

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

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

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

### IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

### V. Blanks

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

Sample "RINSATE 1" (from SDG F8F120137) was identified as a rinsate. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank.

### 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)	All samples in SDG F8F120167	J+ (all detects) J+ (all detects)	Р

### VIII. Regional Quality Assurance and Quality Control

Not applicable.

### IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
TSB-GJ-08-20**	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	37 (40-135) 39 (40-135)	OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Ρ
TSB-GJ-08-30**	<sup>13</sup> C-OCDD	29 (40-135)	OCDD	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Ρ
TSB-GJ-08-40	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	26 (40-135) 33 (40-135)	OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	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 F8F120167

SDG	Sample	Compound	Flag	A or P	Reason
F8F120167	TSB-GJ-08-10 TSB-GJ-08-20** TSB-GJ-08-30** TSB-GJ-08-40	1,2,3,7,8,9-HxCDD OCDD	J+ (all detects) J+ (all detects)	Ρ	Laboratory control samples (%R)
F8F120167	TSB-GJ-08-20** TSB-GJ-08-40	OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Ρ	Internal standards (%R)
F8F120167	TSB-GJ-08-30**	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Ρ	Internal standards (%R)

### **BRC Tronox Parcel G**

Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

BRC Tronox Parcel G Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG F8F120167

No Sample Data Qualified in this SDG

LDC #: <u>19097B21</u> SDG #: <u>F8F120167</u> Laboratory: <u>Test America</u>

### Level III/IV

Date:	7/19/08
Page:_	<u>of</u>
Reviewer:	n
2nd Reviewer:	A

÷.

### 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: 6/11/08
11.	GC/MS Instrument performance check	Å	
111.	Initial calibration	A	
IV.	Routine calibration/ <del>IGV</del>	A	
V.	Blanks	A	
VI.	Matrix spike/Matrix spike duplicates	N	client specified
VII.	Laboratory control samples	SV	Us
VIII.	Regional quality assurance and quality control	N	-
IX.	Internal standards	SW	
Х.	Target compound identifications	4	Not reviewed for Level III validation.
XI.	Compound quantitation and CRQLs	L L	Not reviewed for Level III validation.
XII.	System performance	4	Not reviewed for Level III validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	И	
XV.	Field blanks	ND	R= RINSATE 1 (+8F120137)

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-08-10	11	8170493MB	21	 31	
2	TSB-GJ-08-20**	12		22	 32	
3	TSB-GJ-08-30**	13		23	 33	
4	TSB-GJ-08-40	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:

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technicat 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?				
Were the retention time windows established for all homologues?				
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers $\leq$ 25% ?	/			
Is the static resolving power at least 10,000 (10% valley definition)?				
Was the mass resolution adequately check with PFK?				
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?				
III, Initial calibration				
Was the initial calibration performed at 5 concentration levels?	/			
Were all percent relative standard deviations (%RSD) $\leq$ 20% for unlabeled standards and $\leq$ 30% for labeled standards?	/			
Did all calibration standards meet the Ion Abundance Ratio criteria?	/			
Was the signal to noise ratio for each target compound $\geq$ 2.5 and for each recovery and internal standard $\geq$ 10?	/			
IV. Continuing calibration	·		r	
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?	1			
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	/			
V. Blanks				
Was a method blank associated with every sample in this SDG?	/			
Was a method blank performed for each matrix and concentration?	/		ļ	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?		/		
VI. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.		/		
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?			<b> </b>	
VII. Leboratory control samples		Γ	T	
Was an LCS analyzed for this SDG?	<u>11</u>	L	<u> </u>	

LDC #: 19097321 SDG #: F8F120167

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?	<u> </u>			
X. Target compound identification				
For 2,3,7,8 substituted congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard?				/
For 2,3,7,8 substituted congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration?				
For non-2,3,7,8 substituted congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution?			/	
Did compound spectra contain all characteristic ions listed in the table attached?			/	
Was the Ion Abundance Ratio for the two quantitation ions within criteria?			/	
Was the signal to noise ratio for each target compound and labeled standard $\geq$ 2.5?			/	
Does the maximum intensity of each specified characteristic ion coincide within $\pm$ 2 seconds (includes labeled standards)?				
For PCDF identification, was any signal (S/N $\geq$ 2.5, at <u>+</u> seconds RT) detected in the corresponding PCDPE channel?			/	
Was an acceptable lock mass recorded and monitored?				
XI: Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?			/	
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XII. System performance				
System performance was found to be acceptable.	/			
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.				

### VALIDATION FINDINGS CHECKLIST

LDC #: 19097821 SDG #: F8F120167

Pa	age:	<u>2of </u> ₹_
Revie	wer:	K
2nd Revie	wer:	1

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

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:

1287	20102
600	ESF
*	*
Ц Ц	SDG

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

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

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

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

Was a LCS required? Was a LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed? Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the OC limited

ts?	Associated Samples	, <del>,</del>	HC JAM	*																							
relative percent difference (HPD) within the QC limits?	RPD (Limits)		~					^ ^	· · ·	. ~					( )	( )						-	)	(			· · · ·
	LCSD %R (Limits)	(				( )			( )	( )	( )				(	( )	(	· ·	~ ~				~	()	( )		
	%R (Limits)	137 (12/2) (12/2)	308 ( TIL-11/1)		· ·	(	)	( )	( )	)	(	) (		· · ·		<pre>(</pre>	( )	<b>^</b>	· ·	(	( )		-	(	( )	( )	( )
	Compound		J				-											÷									
	Lab ID/Reference	81724924CS																									
∦	# Date																										

LCS90.21

128290A	F8F120167
#	#
БС	SDG

### VALIDATION FINDINGS WORKSHEET Internal Standards

Page: tof 2nd Reviewer: Reviewer:

も

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/A</u> Mos the C/N ratio of internal standard recoveries were within the 40-135% criteria?

		was ure o/n railo all internal standard peaks <u>&gt;</u>	stariuaru peaks ≥ 10?		
#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		4	T	SE1-07) 12	) I/47/4 (C, 2)
			Ċ	<i>34</i>	(0,1
				)	
		4	+-1	) 65	
				)	
		4	H	) 92	
			Э	1) 25	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~
				}	
				)	
				)	(
				)	
				)	(
				)	
	¥ v			)	(
				)	)
				)	
				)	
				)	(
				)	(
				)	
		Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used
Ä	<sup>13</sup> C-2,3,7,8-TCDF	DF		K. 1 <sup>13</sup> C-1,2,3,4-TCDD	
В	_	DD			
Ċ	-	PeCDF		M.	
<u> </u>	┿	<sup>p</sup> eCDD		N.	
ш	<sup>13</sup> C-1,2,3,6,7,8-HxCDF	3-HxCDF		0	
u.'	<sup>13</sup> C-1,2,3,6,7,8-HxCDD	3-HxCDD		Ġ	
<u>ଓ</u> :	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	7,8-HpCDF		Ö	
Ξŀ	<sup>13</sup> C-1,2,3,4,6,1	7,8-HpCDD		Ľ.	

INTST90.21

SDG #: FSF120167 LDC #: (9097 B21

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

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

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

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

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

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

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

#         Calibration Bate         Compound (Reference Internal Standard)         A           1         \C/L         L/lb/pcf         2.3.7.8-TCDF (*C-2,3.7,6-TCDF)         2.3.7.8-TCDF)           1         \C/L         L/lb/pcf         2.3.7.8-TCDF (*C-2,3.7,6-TCDF)         2.3.7.8-TCDF)           1         1         2.3.7.8-TCDF (*C-2,3.7,6-TCDF)         2.3.7,8-TCDF)         2.3.7,8-TCDF)           1         1.2.3.6.7,8-HpCDD (*C-1,2.4.6,7,8,-HpCDD)         1.2.3.6.7,8-HpCDD)         2.3.7,8-TCDF)         2.3.7,8-TCDF)           1         1.2.3.6.7,8-HpCDD (*C-1,2.4.6,7,8,-HpCDD)         1.2.3.6,7,8-HpCDD)         1.2.3.6,7,8-HpCDD)         2.3.7,8-TCDF)         2.3.7,8-TCD										
#         Standard ID         Calibration Date         Calibration Compound (Reference Internal Standard)         Average RRF (initia)         RRF         RRF (initia)         RRF (initia)					Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
VAL         L/lb/pg         23.7.вТОБГ (*C.23.7.вТОВ)         0.79%         0.79%         0.82         0.82         0.82         0.13         0.13           12.86.7.84MOD0 (*C.23.7.вТОВ)         0.79%         0.79%         0.87         0.82         0.79%         0.79%         0.79%         0.79%         0.13         0.14         0.15         0.14         0.15         0.14         0.15         0.13         0.14         0.15         0.14         0.15         0.14         0.15         0.14         0.15         0.14         0.14         0.15         0.14         0.15         0.14         0.15         0.14         0.15         0.14         0.15         0.14         0.15         0.15         0.14         0.15         0.14         0.15         0.14         0.15         0.15         0.15	*	Standard ID	Calibration Date	Compound (Reference Internal Standard)		Average RRF (initial)	RRF ( CS > stat)			
Zал.6-ТОD (*C-2ал.6-ТСD)         2-9[1-2]         0.93 <th0.93< th="">         0.93         0.93         &lt;</th0.93<>	-	<b>ICAL</b>	20/91/2	2,3,7,8-TCDF ( <sup>18</sup> C-2,3,7,8-TCDF)	_	0 796		┛Ĺ	%HSD	RSD
1.2.36.7 внисор (*C-1.2.8.7,6-нисор)         2. §2-1         0. §7-0         0. §7-0         0. 5.7         0. 5.7         0. 5.7         0. 5.7         0. 5.7         0. 5.7         1.2.3         0. 5.7         1.2.3         0. 5.7 <th< th=""><th>Ţ</th><td></td><td></td><td>2.3.7,8-TCDD (<sup>13</sup>C-2,3,7,8-TCDD)</td><td>2912</td><td>2150</td><td>0.97</td><td>10.0</td><td>2.5</td><td>ν.α</td></th<>	Ţ			2.3.7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	2912	2150	0.97	10.0	2.5	ν.α
1.23.46.7.8-HpCDD ( <sup>1</sup> C-1.24.6.7.8HpCDD)         0.84µ         0.87         0.87         0.88         0.5.7           CODF ( <sup>1</sup> PC-CODD)         (1.72)         (1.72)         (1.72)         0.88         0.88         0.58 <th>T</th> <td></td> <td></td> <td>1,2,3,6,7,8-HxCDD (<sup>13</sup>C-1,2,3,6,7,8-HxCDD)</td> <td>128.0</td> <td>0.820</td> <td>0010</td> <td>2 2 2</td> <td>44</td> <td>8.0</td>	T			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	128.0	0.820	0010	2 2 2	44	8.0
CODF (*C-COD)         (.721         (.72.)         (.72.         (.62         (.62         (.72.)         (.72.)         (.72.)         (.62.)         (.53.)	T			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	0 844	0 צחת	00 00	18.0	12.7	
2.3.7.8-TCDF (*C-2.3.7.8-TCDF)         2.3.7.8-TCDF (*C-2.3.7.8-TCDF)         1.0.0           2.3.7.8-TCDP (*C-2.3.7.8-TCDD)         2.3.7.8-TCDD (*C-1.2.4.6.7.8.+HACDD)         1.2.3.6.7.8-HACDD)         1.2.3.6.7.8-HACDD)           1.2.3.6.7.8-HACDD (*C-1.2.4.6.7.8.+HACDD)         1.2.3.6.7.8-HACDD)         1.2.3.6.7.8-HACDD)         1.2.3.6.7.8-HACDD)           1.2.3.6.7.8-HACDD (*C-1.2.4.6.7.8HACDD)         1.2.3.6.7.8-HACDD)         1.2.3.6.7.8-HACDD)         1.2.3.6.7.8-HACDD)           1.2.3.6.7.8-HACDD (*C-1.2.4.6.7.8HACDD)         1.2.3.6.7.8-HACDD)         1.2.3.6.7.8-HACDD)         1.2.3.6.7.8-HACDD)           1.2.3.6.7.8-HACDD (*C-1.2.4.6.7.8HACDD)         1.2.3.6.7.8-HCDD)         1.2.3.6.7.8-HCDD)         1.2.3.6.7.8-HCDD)           1.2.3.6.7.8-HCDD (*C-1.2.3.6.7.8-HCDD)         1.2.3.6.7.8-HCDD)         1.2.3.6.7.8-HCDD)         1.2.3.6.7.8-HCDD)           1.2.3.6.7.8-HCDD (*C-1.2.3.6.7.8-HCDD)         1.2.3.6.7.8-HCDD)         1.2.3.6.7.8-HCDD)         1.2.3.6.7.8-HCDD)           1.2.3.6.7.8-HCDD (*C-1.2.3.6.7.8-HCDD)         1.2.3.6.7.8-HCDD)         1.2.3.6.7.8-HCDD)         1.2.3.6.7.8-HCDD)           1.2.3.6.7.8-HCDD (*C-1.2.4.6.7.8-HDCDD)         1.2.3.6.7.8-HCDD)         1.2.3.6.7.8-HCDD)         1.2.3.6.7.8-HCDD)				OCDF ("C-OCDD)	122.1	1.7.7	101	80.1	3.5	2.2
23.7.8-TCDD (*C-23.7.8-TCDD)         23.7.8-TCDD (*C-23.7.8-TCDD)         1           1.23.8.7.8-HxCDD (*C-1.23.8.7.8-HxCDD)         1.23.8.7.8-HxCDD (*C-1.23.8.7.8-HxCDD)         1           1.23.4.8.7.8-HxCDD (*C-1.23.8.7.8-HxCDD)         1         1         1           3         0CDF (**C-0CDD)         1         1         1         1           3         23.7.8-TCDP (*C-1.2.4.6.7.8HxCDD)         1         1         1         1           3         23.7.8-TCDF (*C-0CDD)         1         1         1         1         1           3         23.7.8-TCDP (*C-23.7.8-TCDD)         1         1         1         1         1           3         23.7.8-TCDP (*C-23.7.8-TCDD)         1         1         1         1         1           3         23.7.8-TCDD (*C-1.2.4.6.7.8HxCDD)         1         1         1         1         1           3         23.7.8-TCDD (*C-1.2.4.6.7.8HxCDD)         1         1         1         1         1           1         23.7.8-TCDD (*C-1.2.4.6.7.8HxCDD)         1         1         1         1         1           1         1         1         1         1         1         1         1         1         1         1	~			2.3.7,8-TCDF ( <sup>1</sup> C-2,3,7,8-TCDF)			101	301	16.2	(6.2
				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)						
2.3.7,8-TCDD (*C-2,3,7,8-TCDD)       2.3.7,8-TCDD (*C-2,3,7,8-TCDD)         1,2.3,6,7,8-HPCDD (*C-1,2,3,6,7,8-HPCDD)       1.2.3,4,6,7,8,HPCDD (*C-1,2,4,6,7,8,HPCDD)         OCDF (*C-0CDD)       0CDF (*C-0CDD)	。			2,3,7,8-TCDF ( <sup>18</sup> C-2,3,7,8-TCDF)						
1,2,3,6,7,8-HxCDD (*C-1,2,3,6,7,8-HxCDD)     1,2,3,6,7,8-HxCDD)       1,2,3,4,6,7,8,+HpCDD (*C-1,2,4,6,7,8,-HpCDD)       OCDF (*C-0CDD)	+			2,3,7,8-TCDD ( <sup>11</sup> C-2,3,7,8-TCDD)						
1.2.3.4.6.7.8HpCDD (*C-1,2,4.6.7.8,HpCDD) OCDF (*C-OCDD)	_			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)						
	$\uparrow$		<b>I</b>	1.2.3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)						
	$\neg$			OCDF (#C-OCDD)						

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

· SDG #: F8+120167 LDC #: 19097321

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

Page: 1 of / 2nd Reviewer: Reviewer:

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: % Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF =  $(A_n)(C_n)(A_n)(C_n)$ 

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

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

	Standard IDCalibrationCompound (fadewore Internal Standard)Average RRFRRFRRFRRF(CC)CC		-14)				Reported	Recalculated	Reported	Recalculated
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	*		Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	۵%	0%
$\frac{23.78-TCDD}{1.23.67.84+KCDD} (*C-23.78-TCDD) 0.824 0.61 0.87 0.87 0.87 0.87 0.87 0.87 0.87 0.87$	Comments:         23.7.8-TCDD (*C-2.3.7.8-TCDD)         0. (7) 3         0. (8)         0. (1)           12.36.7.8+McDD (*C-1.2.46.7.8.+McDD)         0. (8)         0. (8)         0. (8)         0. (8)         0. (1)           2         5706/30         (1.7)         1. 58         0. (8)         0. (3)         0. (1)         0. (3)         0. (3)	-	ST26072	\$ 9/2=/2		0.798	0.83	0, 83	4.0	
$\frac{1.2.3.6.7.8+McDD ("C-1.2.3.6.7.8+McDD)}{1.2.3.6.7.8+McDD ("C-1.2.3.6.7.8+McDD)} \frac{0.824}{0.844} \frac{0.82}{1.58} \frac{0.87}{1.58} \frac{1.5}{1.58} \frac{1.5}{$	Image: constraint of the section of the secting the section of the section of the section of the sectio				2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0,913	0.81	18.0	1.7	1.7
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	Comments:         1.23,4.6.7.8+HpCDD (*C-1.24.6.7.8.+HpCDD)         0.2 以山         0.2 以山         0.2 公         0.				1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	0.821	0.87	0,87	6.5	64
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	2         うせもうっ         0 coEF (*2-c37,8-TCDF)         0.7 つち         1. 下を         1. 5g         1. 1, 5g         1				1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	0.844	0.82	0.63		
分でも次         23.7.8-TCDF ("C-23.7.8-TCDF)         0.79(2)         0.85         E.9           2.3.7.8-TCDP ("C-23.7.8-TCDP)         0.913         0.85         E.9         E.9           2.3.7.8-TCDP ("C-23.7.8-TCDP)         0.913         0.82         0.85         E.9         E.9           1.2.3.6,7.8-HCDD ("C-12.3.6,7.8-HCDD)         0.92         0.92         0.92         0.92         1.0.32         1.1.6           1.2.3.6,7.8-HPCDD ("C-1.2.4.6,7.8HPCDD)         0.82         0.92         0.92         1.1.6         1.1.6           1.2.3.6,7.8-HPCDD ("C-1.2.4.6,7.8HPCDD)         0.82         0.92         0.92         1.1.6         1.1.6           2.3.7.8-TCDF ("C-0CDD)         1.7.724         1.65         1.65         1.1.5           2.3.7.8-TCDF ("C-2.3.7.8-TCDF)         1.7724         1.65         1.1.5         1.1.5           2.3.7.8-TCDF ("C-0CDD)         1.7724         1.65         1.1.5         1.2.5           2.3.7.8-TCDF ("C-2.3.7.8-TCDF)         1.1.754         1.1.65         1.1.5           2.3.7.8-TCDF ("C-2.2.7.8-TCDF)         1.1.754         1.1.65         1.1.5           2.3.7.8-TCDF ("C-2.3.7.8-TCDF)         1.1.754         1.1.65         1.1.5           2.3.7.8-TCDF ("C-2.2.7.8-TCDF)         1.1.754	2       分でも次       2.3.7.8-TCDF ("3-C.2.3.7.8-TCDF)       0.79(2)       0.85       5.9         2       2.3.7.8-TCDD ("3-C.2.3.7.8-TCDD)       0.31(3)       0.31(3)       0.85       5.9         2       3.3.7.8-TCDD ("3-1.2.3.6.7.8-HxCDD)       0.32(1)       0.82       0.82       10.3         1       1.2.3.6.7.8-HxCDD ("3-1.2.3.6.7.8-HxCDD)       0.82(4)       0.92       0.92       10.3         3       1.2.3.4.6.7.8-HxCDD ("3-1.2.3.6.7.8-HxCDD)       0.82(4)       0.92       0.92       11.4         0       2.3.7.8-TCDE ("3-2.3.7.8-TCDD)       0.82(4)       0.82       0.92       11.4         3       0       2.3.7.8-TCDE ("3-2.3.7.8-TCDD)       1.7794       (65       11.65       11.4         3       2.3.7.8-TCDE ("3-2.3.7.8-TCDD)       1.7794       0.82       0.82       5.3         3       2.3.7.8-TCDE ("3-2.3.7.8-TCDD)       1.7794       0.65       1.65       1.65         1       2.3.7.8-TCDE ("3-2.3.7.8-TCDD)       1.7794       0.82       6.9       6.3         1       2.3.7.8-TCDE ("3-2.3.7.8-TCDD)       1.7794       0.65       1.65       1.65         2.3.7.8-TCDE ("3-2.3.7.8-TCDD)       1.236.7.8-HCDD)       1.236.7.8-HCDD)       1.236.7.8-HCDD)       1.236.7.8-HCDD)<				OCDF (13C-OCDD)	142.1	1.58	1,58	₹.3	2.2
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	Comments:         East of the contraction         Contraction<	2		80/62/9	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.798	0.85	b.85	5.9	5.4
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	Image: Comments:         1,23,4,5,7,8+HxCDD (*0-1,23,6,7,8,+HxCDD)         0.324         0.42. <th0.42.< th=""> <t< td=""><th></th><td></td><td></td><td>2,3,7,8-TCDD (<sup>13</sup>C-2,3,7,8-TCDD)</td><td>0.913</td><td>0.82</td><td>0.82</td><td>10.3</td><td>10.2</td></t<></th0.42.<>				2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.913	0.82	0.82	10.3	10.2
1.2.3.4.6.7.8-HpCDD (*C-1.2.4.6.7.8.+HpCDD)         0.S44         0.89         0.S9         5.2         1           OCDF (*C-OCDD)         1.7724         1.7724         1.65         1.65         1.65         1.5           2.3.7.8-TCDF (*C-2.3.7.8-TCDF)         1.7724         (.65         1.65         1.65         1.55           2.3.7.8-TCDF (*C-2.3.7.8-TCDF)         1.7724         (.65         1.65         1.65         1.55           2.3.7.8-TCDF (*C-2.3.7.8-TCDF)         1.23,67,8-HxCDD)         1.1,73,67,78,4-HyCDD)         1.1,73,67,78,4-H	Image: Section of the section of t				1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	0.821	0.92	260	11.6	11.6
OCDF ( <sup>a</sup> C-OCDD)         1.7)2-1         (. しこ         1, しご $-1$ 2.3.7.8-TCDF ( <sup>a</sup> C-2.3.7,8-TCDF)         2.3.7,8-TCDD ( <sup>a</sup> C-2.3.7,8-TCDD)         1.0         1.0         1.0           2.3.7,8-TCDD ( <sup>a</sup> C-2.3.7,8-TCDD)         1.2.3,6.7,8+TCDD)         1.0         1.0         1.0         1.0           1.2.3,6.7,8+TCDD ( <sup>a</sup> C-1,2.3,6.7,8-HCDD)         1.2.3,6.7,8-HCDD ( <sup>a</sup> C-1,2.4,6.7,8,-HpCDD)         1.0         1.	a         ocdp ( <sup>a</sup> C-OCDD)         1.72月         1.65 <th1.65< th="">         1.65</th1.65<>				1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	0.844	0.89	0 29	5.2	2 
2,3,7,8-TCDF (*C-2,3,7,8-TCDF)       2,3,7,8-TCDD (*C-2,3,7,8-TCDD)         2,3,7,8-TCDD (*C-2,3,7,8-TCDD)       1,2,3,6,7,8+TCDD)         1,2,3,6,7,8+TCDD (*C-1,2,3,6,7,8+TCDD)       1,2,3,4,6,7,8+TCDD)         1,2,3,4,6,7,8+TPCDD (*C-1,2,4,6,7,8,-TPCDD)       0000         0000 (*CODD)       0000	3     2:3.7.8-TCDF (*C-2,3.7,8-TCDF)     2:3.7,8-TCDF (*C-2,3.7,8-TCDD)       1     2:3.7,8-TCDD (*C-2,3.7,8-TCDD)     1.2.3,6.7,8-HxCDD (*C-1,2.3,6.7,8-HxCDD)       1     1.2.3,6.7,8-HxCDD (*C-1,2.3,6.7,8-HxCDD)     1.2.3,6.7,8-HxCDD (*C-1,2.4,6.7,8,-HpCDD)       0     0     0       1     2.3,7,8-TCDD)     1.2.3,6.7,8-HxCDD (*C-1,2.4,6.7,8,-HpCDD)       0     0     0       1     0     0       1     0     0       1     0     0       0     0     0<				OCDF (13C-OCDD)	122.1	1.65	1.65	4 7	
2.3.7.8-TCDD ( <sup>13</sup> C-2,3.7,8-TCDD)       2.3.7,8-TCDD ( <sup>13</sup> C-1,2,3,6,7,8-HXCDD)         1.2.3,6,7,8-HXCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HPCDD)       1.2.3,4,6,7,8-HPCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HPCDD)         0 CDF ( <sup>13</sup> C-0CDD)       0 CDF ( <sup>13</sup> C-0CDD)	Comments:     Refer to Routine Calibration findings worksheet for list of qualifications and associated samples when reported results do not	ო			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)					
1,2,3,6,7,8+hxCDD ( <sup>1</sup> C-1,2,3,6,7,8+hxCDD)     1,2,3,6,7,8+hxCDD ( <sup>1</sup> C-1,2,4,6,7,8,+hyCDD)       0CDF ( <sup>1</sup> 3C-0CDD)     0CDF ( <sup>1</sup> 3C-0CDD)	Image: Comments:     Image: Sector Sect				2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
1,2,3,4,6,7,8-HpCDD ( <sup>3</sup> C-1,2,4,6,7,8,-HpCDD)       0CDF ( <sup>3</sup> C-0CDD)	Comments: <u>Refer to Routine Calibration findings worksheet for list of gualifications and associated samples when renorted results do not</u>				1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
OCDF ("C-OCDD)	Comments: <u>Refer to Routine Calibration findings worksheet for list of gualifications and associated samples when renorted results do not</u>				1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)					
	Comments: <u>Refer to Routine Calibration findings worksheet for list of gualifications and associated samples when reported results do not</u>				OCDF ("C-OCDD)					

C:\WPDOCS\WRK\DIOXIN90\CONCLC90.21

recalculated results.

1909792	[3102] = 100 [0]
	SDG #

### VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Results Verification

Page: Lof Reviewer: A 2nd Reviewer: C

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 ID: SITO493LCS

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

LCS/LCSD	RPD	-	Recalculated									
I CS		1										
csn	Recovery										-	
	Percent Recovery		namo									
CS	Percent Recovery	Dooolo	92	Ī	102	50	50			-		
T	Percent	Donortod	45	5	102	50)	te					
Spiked Sample	entration	l oxí										
Spiked	Concen ( P.4/	501	13.4	11	22	لاما	209					
oike	Added (75/15)	1 CSN										
ц С	94 (1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-	1 CS	2	5		->	202					
	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.

Analyte	HPCDF HPCDF HPCDF (S) HPCDD HPCDD HPCDD HPCDD HPCDD S) NCDPE (S) NCDPE S)	OCDF OCDF OCDD OCDD (S) OCDD (S) DCDPE PFK
Elemental Composition	C <sub>12</sub> H <sup>45</sup> Cl <sub>3</sub> <sup>37</sup> ClO C <sub>12</sub> H <sup>45</sup> Cl <sub>3</sub> <sup>37</sup> ClO 13 C <sub>12</sub> H <sup>45</sup> Cl <sub>3</sub> <sup>37</sup> ClO 13 C <sub>12</sub> H <sup>45</sup> Cl <sub>3</sub> <sup>37</sup> ClO 13 C <sub>12</sub> H <sup>45</sup> Cl <sub>3</sub> <sup>37</sup> ClO 14 H <sup>45</sup> Cl <sub>3</sub> <sup>37</sup> ClO 14 H <sup>45</sup> Cl <sub>3</sub> <sup>37</sup> ClO 14 H <sup>45</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O 14 H <sup>45</sup> Cl <sub>3</sub> <sup>47</sup> ClO 13 C <sub>12</sub> H <sup>45</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O 14 H <sup>45</sup> Cl <sub>3</sub> <sup>47</sup> Cl <sub>2</sub> O 14 Cl <sub>3</sub> <sup>47</sup> Cl <sub>2</sub> O 14 Cl <sub>3</sub> <sup>47</sup> Cl <sub>3</sub> <sup>47</sup> Cl <sub>2</sub> O 14 Cl <sub>3</sub> <sup>47</sup> Cl <sub>2</sub> O 14 Cl <sub>3</sub> <sup>47</sup> Cl <sub>3</sub> CCl <sub>2</sub> O 14 Cl <sub>3</sub> <sup>47</sup> Cl <sub>3</sub> CCl <sub>2</sub> O 14 H <sup>45</sup> Cl <sub>3</sub> <sup>47</sup> Cl <sub>2</sub> O 14 Cl <sub>3</sub> <sup>47</sup> Cl <sub>3</sub> CCl <sub>2</sub> O 14 H <sup>45</sup> Cl <sub>3</sub> <sup>47</sup> Cl <sub>2</sub> O 14 Cl <sub>3</sub> <sup>47</sup> Cl <sub>3</sub> CCl <sub>2</sub> O 14 H <sup>45</sup> Cl <sub>3</sub> <sup>47</sup> Cl <sub>2</sub> O 14 Cl <sub>3</sub> <sup>47</sup> Cl <sub>3</sub> CCl <sub>2</sub> O 14 H <sup>45</sup> Cl <sub>3</sub> <sup>47</sup> Cl <sub>3</sub> O 14 Cl <sub>3</sub> <sup>47</sup> Cl <sub>3</sub> CCl <sub>2</sub> O 14 H <sup>45</sup> Cl <sub>3</sub> <sup>47</sup> Cl <sub>3</sub> O 14 Cl <sub>3</sub> <sup>47</sup> Cl <sub>3</sub> CCl <sub>2</sub> O 14 H <sup>45</sup> Cl <sub>3</sub> CCl <sub>2</sub> O 14 H <sup>45</sup> Cl <sub>3</sub> CCl <sub>2</sub> O 14 H <sup>45</sup> Cl <sub>3</sub> CCl <sub>2</sub> O 14 Cl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> O 14 Cl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> O 14 Cl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> O 14 Cl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> O 14 Cl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> O 14 Cl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> CC 14 Cl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> CCCl <sub>3</sub> CC 14 Cl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> CCl <sub>3</sub> CCCl <sub>3</sub> CCCl <sub>3</sub> CCCl <sub>3</sub> CCCCCCCCCC	C <sub>12</sub> <sup>36</sup> Cl, <sup>37</sup> ClO C <sub>12</sub> <sup>36</sup> Cl, <sup>37</sup> ClO C <sub>12</sub> <sup>36</sup> Cl, <sup>37</sup> ClO <sub>2</sub> C <sub>10</sub> F <sub>17</sub> C <sub>10</sub> F <sub>17</sub>
lon ID Eler	$ \begin{array}{c} M \\ \mathsf$	M + 2 M + 4 M + 2 M + 2
Accurate Mass <sup>(a)</sup>	407.7818 409.7788 419.7.8250 419.8220 425.7737 425.7737 425.7737 435.7737 435.8169 437.8140 437.8140 430.9728]	441.7428 443.7399 457.7377 459.7348 459.7348 449.7780 513.6775 [422.9278]
Descriptor	4	n 44444407
Analyte	TCDF TCDF (S) TCDF (S) TCDD (S) TCDD (S) TCDD (S) HXCDPE PFK	PecDF PecDF (S) PecDF (S) PecDD (S) PecDD (S) PecDD (S) PecDD (S)
Elemental Composition	C <sub>12</sub> H, <sup>33</sup> C1,0 C <sub>12</sub> H, <sup>43</sup> C1,37C10 <sup>13</sup> C <sub>12</sub> H, <sup>43</sup> C1,37C10 <sup>13</sup> C <sub>12</sub> H, <sup>43</sup> C1,02 C <sub>15</sub> H, <sup>43</sup> C1,37C10 C <sub>15</sub> H, <sup>43</sup> C1,37C10 C <sub>15</sub> H, <sup>43</sup> C1,37C10	င <sub>12</sub> H3ီငါ, TCIO င <sub>12</sub> H3 <sup>&amp;</sup> ငါ, TCIO 13C12H3 <sup>&amp;</sup> CI, TCIO 13C12H3 <sup>&amp;</sup> CI, TCIO 13C12H3 <sup>&amp;</sup> CI, TCIO 12H3 <sup>&amp;</sup> CI, TCIO C12H3 <sup>&amp;</sup> CI, TCIO C12H3 <sup>&amp;</sup> CI, TCIO 13C12H3 <sup>&amp;</sup> CI, TCIO 13C12H3 <sup>&amp;</sup> CI, TCIO 13C12H3 <sup>&amp;</sup> CI, TCIO 5, F13 C2F13
lon ID	CCK 2 2 M 2 M 2 M 2 M 2 M 2 M 2 M 2 M 2 M	M M M M M M M M M M M M M M M M M M M
Accurate mass <sup>(a)</sup>	303.9016 305.8987 315.9419 317.9389 319.8965 321.8936 331.9368 333.9338 333.9338 375.8364 [354.9792]	339.8597 341.8567 351.9000 353.8970 355.8546 357.8516 367.8949 369.8919 409.7974 [354.9792]
Descriptor		N

The following nuclidic masses were used:

**a**)

H = 1.007825 C = 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

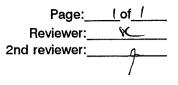
C:\WPDOCS\WRK\DIOXIN90\TCl90.21

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

LDC #: 19097821 SDG #: F8F120167

### VALIDATION FINDINGS WORKSHEET

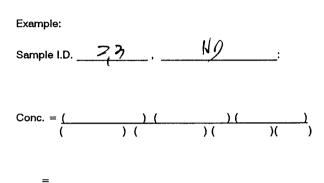
Sample Calculation Verification



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

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

$Concentration = \frac{(A_{.})(I_{.})(DF)}{(A_{is})(RRF)(V_{o})(\%S)}$						
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				
I <sub>s</sub>	Ξ	Amount of internal standard added in nanograms (ng)				
V.	H	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.				



				T	
		<b>0</b>	Reported Concentration	Calculated Concentration	Qualification
#	Sample ID	Compound	()	( )	Qualification
				· · · · · · · · · · · · · · · · · · ·	
		L			
					<u></u>