

LABORATORY DATA CONSULTANTS, INC.

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March 19, 2008

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

SUBJECT: BRC Tronox Parcel H, Data Validation

Dear Ms. Barajas-Albalawi

Enclosed are the final validation reports for the fractions listed below. This SDG was received on March 3, 2008. Attachment 1 is a summary of the samples that were reviewed for each analysis.

### LDC Project # 18386:

SDG # Fraction

F8A250221,Volatiles, Semivolatiles, Chlorinated Pesticides, PolychlorinatedF8A290158Biphenyls, Metals, Gasoline Range Organics, Diesel Range<br/>Organics, Dioxins/Dibenzofurans, Wet Chemistry

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

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

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

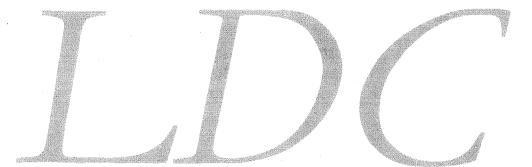
Sincerely,

Erlinda T. Rauto Operations Manager/Senior Chemist

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### BRC Tronox Parcel H Data Validation Reports LDC# 18386

Volatiles



### LDC Report# 18386A1

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

TSB-HR-08-10'MS

TSB-HR-08-10'MSD

Project/Site Name: BRC Tro	nox Parcel H
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Collection Date: January 24, 2008

LDC Report Date: March 14, 2008

Matrix: Soil/Water

Parameters: Volatiles

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

### Sample Delivery Group (SDG): F8A250221

TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HJ-04-0' TSB-HR-04-0'\*\* TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'\*\* TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'\*\* TSB-HJ-07-0'-FD TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-10' TSB-TB-3 TSB-TB-2 TSB-TB-1 TSB-HR-08-0'MS TSB-HR-08-0'MSD

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 19 soil samples and 3 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 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
1/21/08	Dibromomethane	0.04510 (≥0.05)	All water samples in SDG F8A250221	J (all detects) UJ (all non-detects)	А
1/30/08	Ethanol	0.00855 (≥0.05)	All water samples in SDG F8A250221	J (all detects) UJ (all non-detects)	A

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
2/4/08	Ethanol Acetonitrile	0.00291 (≥0.05) 0.01869 (≥0.05)	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HR-04-0'** TSB-HR-07-10' TSB-HR-07-10'** TSB-HR-06-0' TSB-HR-06-10' TSB-HR-06-10' TSB-HJ-07-10' TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-0' TSB-HR-08-0'MS TSB-HR-08-0'MSD 8038049-Blank	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A
2/6/08	Ethanol	0.00366 (≥0.05)	TSB-HJ-07-0'-FD TSB-HR-08-10' TSB-HR-08-10'MS TSB-HR-08-10'MSD 8038277-Blank	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
1/30/08	Bromomethane	48.37592	All water samples in SDG F8A250221	J+ (all detects)	A

Date	Compound	%D	Associated Samples	Flag	A or P
2/5/08 (09:12)	1,1-Dichloroethane Iodomethane Carbon tetrachloride 2-Nitropropane	89.46153 32.31122 28.82024 33.49971	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HR-04-0'** TSB-HR-04-0'** TSB-HR-07-0' TSB-HR-07-10'** TSB-HR-06-0' TSB-HR-06-10' TSB-HR-06-0' TSB-HJ-07-10' TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-0' TSB-HR-08-0'MS TSB-HR-08-0'MS TSB-HR-08-0'MSD 8038049-Blank	J+ (all detects) J+ (all detects) J+ (all detects) 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
1/21/08	lodomethane Vinyl acetate	33.60319 31.00872	All water samples in SDG F8A250221	J+ (all detects) J+ (all detects)	A
2/4/08	1,1-Dichloroethane	72.08435	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HR-04-0' TSB-HR-04-0'** TSB-HR-07-0' TSB-HR-07-10'** TSB-HR-06-0' TSB-HR-06-10' TSB-HR-06-10' TSB-HR-07-10' TSB-HR-08-0' TSB-HR-08-0' TSB-HR-08-0'MS TSB-HR-08-0'MSD 8038049-Blank	J+ (all detects)	A
2/6/08	Bromomethane	34.53645	TSB-HJ-07-0'-FD TSB-HR-08-10' TSB-HR-08-10'MS TSB-HR-08-10'MSD 8038277-Blank	J+ (all detects)	A
2/6/08	Acetonitrile	27.60270	TSB-HJ-07-0'-FD TSB-HR-08-10' TSB-HR-08-10'MS TSB-HR-08-10'MSD 8038277-Blank	J- (all detects) UJ (all non-detects)	a

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

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
1/30/08	Dibromomethane	0.04735 (≥0.05)	All water samples in SDG F8A250221	J (all detects) UJ (all non-detects)	A
2/5/08 (09:12)	Acetonitrile	0.01921 (≥0.05)	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HJ-04-10' TSB-HJ-04-0' TSB-HJ-04-0'** TSB-HR-07-0' TSB-HR-07-10'** TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'** TSB-HJ-07-10' TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-0' TSB-HR-08-0'MSD 8038049-Blank	J (all detects) UJ (all non-detects)	A
2/5/08 (10:14)	Ethanol	0.00259 (≥0.05)	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HJ-04-0' TSB-HR-04-0'** TSB-HR-07-0' TSB-HR-07-10'** TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-10' TSB-HJ-07-10' TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-0'MS TSB-HR-08-0'MSD 8038049-Blank	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 with the following exceptions:

Method Blank ID	Analysis Date	Compound TIC (RT in minutes)	Concentration	Associated Samples
8031135-Blank	1/30/08	Dichloromethane	0.16 ug/L	All water samples in SDG F8A250221
8032877-Blank	2/6/08	Dichloromethane	2.8 ug/Kg	TSB-HJ-07-0'-FD TSB-HR-08-10'

Sample concentrations were compared to concentrations detected in the method 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 method blanks with the following exceptions:

Sample	Compound TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
TSB-TB-3	Dichloromethane	0.20 ug/L	1.0U ug/L
TSB-TB-2	Dichloromethane	0.14 ug/L	1.0U ug/L
TSB-TB-1	Dichloromethane	0.18 ug/L	1.0U ug/L
TSB-HJ-07-0'-FD	Dichloromethane	6.1 ug/Kg	6.1U ug/Kg
TSB-HR-08-10'	Dichloromethane	4.2 ug/Kg	5.5U ug/Kg

Samples TSB-TB-3, TSB-TB-2, and TSB-TB-1 were identified as trip blanks. No volatile contaminants were found in these blanks with the following exceptions:

Trip Blank ID	Sampling Date	Compound	Concentration	Associated Samples
TSB-TB-3	1/24/08	Dichloromethane Acetone	0.20 ug/L 4.3 ug/L	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HJ-04-0' TSB-HR-04-0'** TSB-HJ-04-10'
TSB-TB-2	1/24/08	Dichloromethane	0.14 ug/L	TSB-HR-07-0' TSB-HR-07-10'** TSB-HR-06-0' TSB-HR-06-10'
TSB-TB-1	1/24/08	Dichloromethane Acetone	0.18 ug/L 4.9 ug/L	TSB-HJ-07-0'** TSB-HJ-07-0'-FD TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-10'

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

Sample	Compound	Reported Concentration	Modified Final Concentration
TSB-HJ-05-0'	Acetone	17 ug/Kg	21U ug/Kg
TSB-HR-04-0'**	Acetone	6.8 ug/Kg	21U ug/Kg
TSB-HJ-04-10'	Acetone	14 ug/Kg	21U ug/Kg
TSB-HJ-07-10'	Acetone	19 ug/Kg	21U ug/Kg
TSB-HR-08-0'	Acetone	7.4 ug/Kg	21U ug/Kg

### VI. Surrogate Spikes

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

Sample	Surrogate	%R (Limits)	Compound	Flag	A or P
8036136-Blank	Bromofluorobenzene	126 (66-115)	All TCL compounds	J+ (all detects)	Р
TSB-TB-3	Bromofluorobenzene	120 (66-115)	Nonanal	J+ (all detects)	A

### VII. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Although the MS/MSD percent recoveries and relative percent differences (RPD) were not within QC limits for some compounds, the LCS/LCSD 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 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.

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

### XVI. Field Duplicates

Samples TSB-HJ-07-0'\*\* and TSB-HJ-07-0'-FD were identified as field duplicates. No volatiles were detected in any of the samples with the following exceptions:

	Concentrat	lion (ug/Kg)				
Compound	TSB-HJ-07-0'**	TSB-HJ-07-0'-FD	RPD (Limits)	Difference (Limits)	Flag	A or P
Dichloromethane	5.8	6.1	-	0.3 (≤5.4)	-	-
1,2,4-Trimethylbenzene	0.41	5.3U	-	4.89 (≤5.4)	_	-

### BRC Tronox Parcel H Volatiles - Data Qualification Summary - SDG F8A250221

SDG	Sample	Compound	Flag	A or P	Reason
F8A250221	TSB-TB-3 TSB-TB-2 TSB-TB-1	Dibromomethane Ethanol	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A	Initial calibration (RRF)
F8A250221	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HJ-04-0' TSB-HR-04-0'** TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'** TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'** TSB-HJ-07-10' TSB-HR-08-0'	Ethanol Acetonitrile	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A	Initial calibration (RRF)
F8A250221	TSB-HJ-07-0'-FD TSB-HR-08-10'	Ethanol	J (all detects) UJ (all non-detects)	A	Initial calibration (RRF)
F8A250221	TSB-TB-3 TSB-TB-2 TSB-TB-1	Bromomethane	J+ (all detects)	A	Continuing calibration (%D)
F8A250221	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HJ-04-0' TSB-HR-04-0'** TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'** TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'** TSB-HJ-07-10' TSB-HR-08-0'	1,1-Dichloroethane lodomethane Carbon tetrachloride 2-Nitropropane	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	A	Continuing calibration (%D)
F8A250221	TSB-TB-3 TSB-TB-2 TSB-TB-1	lodomethane Vinyl acetate	J+ (all detects) J+ (all detects)	A	Continuing calibration (ICV %D)

SDG	Sample	Compound	Flag	A or P	Reason
F8A250221	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HR-04-0' TSB-HR-04-0'** TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'** TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'** TSB-HJ-07-10' TSB-HR-08-0'	1,1-Dichloroethane	J+ (all detects)	A	Continuing calibration (ICV %D)
F8A250221	TSB-HJ-07-0'-FD TSB-HR-08-10'	Bromomethane	J+ (all detects)	A	Continuing calibration (ICV %D)
F8A250221	TSB-HJ-07-0'-FD TSB-HR-08-10'	Acetonitrile	J- (all detects) UJ (all non-detects)	A	Continuing calibration (ICV %D)
F8A250221	TSB-TB-3 TSB-TB-2 TSB-TB-1	Dibromomethane	J (all detects) UJ (all non-detects)	A	Continuing calibration (RRF)
F8A250221	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HJ-04-0' TSB-HR-04-0'** TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'** TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-10' TSB-HJ-07-10' TSB-HJ-07-10'	Acetonitrile Ethanol	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A	Continuing calibration (RRF)
F8A250221	TSB-TB-3	Nonanal	J+ (all detects)	A	Surrogate recovery (%R)

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### BRC Tronox Parcel H Volatiles - Laboratory Blank Data Qualification Summary - SDG F8A250221

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P
F8A250221	TSB-TB-3	Dichloromethane	1.0U ug/L	A
F8A250221	TSB-TB-2	Dichloromethane	1.0U ug/L	A
F8A250221	TSB-TB-1	Dichloromethane	1.0U ug/L	A

### VALIDATION COMPLETENESS WORKSHEET

LDC #: <u>18386A1</u> SDG #: <u>F8A250221</u> Laboratory: <u>Test America</u>

### Level III/IV

80/01 Date: Page: / of Reviewer: 2nd Reviewer:

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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
<u>I.</u>	Technical holding times	Δ_	Sampling dates: 1 22/08
<u> </u>	GC/MS Instrument performance check	A	
.	Initial calibration	ري	% PSP, 12
IV.	Continuing calibration/ICV	SW	101575
V.	Blanks	SW	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	sw	Lesp
IX.	Regional Quality Assurance and Quality Control	N	
Х.	Internal standards	<u> </u>	
XI.	Target compound identification	<u> </u>	Not reviewed for Level III validation.
XII.	Compound quantitation/CRQLs	$\triangle$	Not reviewed for Level III validation.
XIII.	Tentatively identified compounds (TICs)	$\mathbf{\nabla}$	Not reviewed for Level III validation.
XIV.	System performance	Δ	Not reviewed for Level III validation.
xv.	Overall assessment of data	A	
XVI.	Field duplicates	لاسى	P = 114 12
XVII.	Field blanks	SW	TB= 16, 14, 18

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

(	SOIL 7 WAT	<u></u>								
13	TSB-HJ-05-10'	1	113	TSB-HJ-07-0'**		3	214	TSB-HR-08-10'MS	371	8031135-Blank 1/30
23	TSB-HJ-05-0'	ł	124	TSB-HJ-07-0'-FD		3	224	TSB-HR-08-10'MSD	<u>3</u> 2 2	8036136 - Blank 2/4
33	TSB-HR-04-10'	1	13 <b>3</b>	TSB-HJ-07-10'		3	23		333	803849-Blank 2/5/08
4 3	TSB-HJ-04-0'	1	14 <b>3</b>	TSB-HR-08-0'		3	24		<del>1</del> 34 4	8038277-Blank 2/6/08
53	TSB-HR-04-0'**	1	15 <b>4</b>	TSB-HR-08-10'		3	25		3 <del>5 S</del>	8031135 -Bknt 1/30-
63	TSB-HJ-04-10'	1	16	≁ TSB-TB-3	Ś	1	26		36	
7 <b>z</b>	TSB-HR-07-0'	2	17	≁ TSB-TB-2	1	2	27		37	
\$ 3	TSB-HR-07-10'**	2	<sub>18</sub>	≁ TSB-TB-1		3	28		38	
9 <b>7</b>	TSB-HR-06-0'	2	19 <b>3</b>	TSB-HR-08-0'MS			29		39	
10 <b>3</b>	TSB-HR-06-10'	r	20 <b>3</b>	TSB-HR-08-0'MSD			30		40	

Batch #14 analyzed after ICAL

LDC #: 18386A1 SDG #: pre coner

### VALIDATION FINDINGS CHECKLIST

Page: / of 2 Reviewer: 77 2nd Reviewer:

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

Validation Area	Yes	s No	NA	Findings/Comments
1 Technical holding times (S. 1996) and the second state of the second state of the second state of the second				
All technical holding times were met.	<	1		
Cooler temperature criteria was met.		1		
III GOMS Instrument certormance codek				
Were the BFB performance results reviewed and found to be within the specified criteria?		Į		
Were all samples analyzed within the 12 hour clock criteria?				
Did the laboratory perform a 5 point calibration prior to sample analysis?	/_	-		
Were all percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?				
Was a curve fit used for evaluation?	-			
Did the initial calibration meet the curve fit acceptance criteria of $\geq 0.990$ ?	$\square$			
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?	/_	-		
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 alidation completeness worksheet.		-		
Vere all surrogate %R within QC limits?			-	
f the percent recovery (%R) for one or more surrogates was out of QC limits, was a			-	
eanalysis performed to confirm samples with %R outside of criteria?	<u>.</u>	$\leq$		
Vere a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each hatrix in this SDG? If no, indicate which matrix does not have an associated IS/MSD. Soil / Water.	_			
/as a MS/MSD analyzed every 20 samples of each matrix?	7			
/ere the MS/MSD percent recoveries (%R) and the relative percent differences RPD) within the QC limits?	JAA	7	/	
II in a Social City control stamples, and a state of the				
as an LCS analyzed for this SDG?				

### VALIDATION FINDINGS CHECKLIST

LDC #:	(X386A)
SDG #:	su cover
7	

Page:	2_of_ ≁	
Reviewer:	PT	
2nd Reviewer:	in	-

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per analytical batch?	$\perp$			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		-	$\vdash$	
Reported Challey Assurance and Cotally Control				
Were performance evaluation (PE) samples performed?			/	· · ·
Were the performance evaluation (PE) samples within the acceptance limits?				
Xeniemal standards				
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?				
Were relative retention times (RRT's) within ± 0.06 RRT units of the standard?		-		
Did compound spectra meet specified EPA "Functional Guidelines" criteria?				
Were chromatogram peaks verified and accounted for?		-	ļ	
XII. competine quantitation CROE				
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?				
Mit enclosely identified companies (TICs) 2 and 2 an				
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?	/	-		
Were relative intensities of the major ions within $\pm$ 20% between the sample and the reference spectra?	1	-		
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?				
SV. System performances and				
System performance was found to be acceptable.	1			
W. Cverally SSESSmen Condenses			ч <b>с</b>	
Overall assessment of data was found to be acceptable.	7		Ι	
Field duplicate pairs were identified in this SDG.	7	-		
Target compounds were detected in the field duplicates.	$\mathcal{A}$	-		
SVID EPEID NEWS				
Field blanks were identified in this SDG.	$\downarrow$			
arget compounds were detected in the field blanks.	7			

TARGET COMPOUND WORKSHEET

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

A. Chloromethane*	U. 1,1,2-Trichloroethane	OO. 2,2-Dichloropropane	III. n-Butylbenzene	CCCC.1-Chlorohexane
B. Bromomethane	V. Benzene	PP. Bromochloromethane	JJJ. 1,2-Dichlorobenzene	DDDD. Isopropyl alcohol
C. Vinyi choride**	W. trans-1,3-Dichloropropene	QQ. 1,1-Dichloropropene	KKK. 1,2,4-Trichlorobenzene	EEEE. Acetonitrile
D. Chloroethane	X. Bromoform*	RR. Dibromomethane	LLL. Hexachlorobutadiene	FFF. Acrolein
E. Methylene chloride	Y. 4-Methyl-2-pentanone	SS. 1,3-Dichloropropane	MMM. Naphthalene	GGGG. Acrylonitrile
F. Acetone	Z. 2-Hexanone	TT. 1,2-Dibromoethane	NNN. 1,2,3-Trichlorobenzene	HHHH. 1,4-Dioxane
G. Carbon disulfide	AA. Tetrachloroethene	UU. 1,1,1,2-Tetrachloroethane	000. 1,3,5-Trichlorobenzene	IIII. Isobuty! alcohol
H. 1,1-Dichloroethene**	BB. 1,1,2,2-Tetrachloroethane*	VV. Isopropylbenzene	PPP. trans-1,2-Dichloroethene	JJJJ. Methacrylonitrile
1. 1,1-Dichloroethane*	CC. Toluene**	WW. Bromobenzene	QQQ. cis-1,2-Dichloroethene	KKKK. Propionitrile
J. 1,2-Dichioroethene, 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. Benzył 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	.dada
O. Carbon tetrachloride	II. 2-Chloroethylvinyl ether	CCC. tert-Butylbenzene	WWW. Ethanol	<u>aaaa.</u>
P. Bromodichloromethane	JJ. Dichlorodifiuoromethane	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	LL. Methyl-tert-butyl ether	FFF. 1,3-Dichlorobenzene	ZZZ. tert-Butyl alcohol	TTTT.
S. Trichloroethene	MM. 1,2-Dibromo-3-chloropropane	GGG. p-Isopropyltoluene	AAAA. Ethyl tert-butyl ether	nuuu.
T. Dibromochloromethane	NN. Methyl ethyl ketone	HHH. 1,4-Dichlorobenzene	BBBB. tert-Amyl methyl ether	VVVV.

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

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LDC #: 18 3 80 A) re con SDG #:

## VALIDATION FINDINGS WORKSHEET **Initial Calibration**

৾৾ 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". 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? A/A N/A

	<b></b>		1	1		1	T		Т	1 7	1		1	ī—	1-	T	T	1	1	1	1	 	<del></del>	-
	Qualifications	A/Ln/			Ţ							A/ m/r												
2		2031135-Blank	- All water		<b>^</b>		303849 - Blank		1			8038277-15/ant,	12, 15, 21, 22											
eria? criteria of ≤30 %RSD and ≥0.05 RRF ?	Finding RRF (Limit: <u>&gt;</u> 0.05)	0.04510	6-000-17		0.00 & SS		0. 60 29	0.01869				0.00366												
e criteria? ttion criteria of ≤30 %	Finding %RSD (Limit: <30.0%)							ttee)		-	-													
n meet the acceptanc RRFs within the valida	Compound	βŖ	-butter		33		TAMMAN	10				223												
Did the initial calibration meet the acceptance criteria? Were all %RSDs and RRFs within the validation criteri	Standard ID	JCAL			ICAL		ICA L					1041												
	# Date	80 12/1	-		20/06/1		24/08					3/9/00	-											

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## VALIDATION FINDINGS WORKSHEET **Continuing Calibration**

**7** 2nd Reviewer: Page: Reviewer:

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

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

Was a continuing calibration standard analyzed at least once every 12 hours for each instrument? Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's ?

	Qualifications	Jt/A dut					J+/Add	2/4J/A			2+/Adut			`	11/A dut	\$/ [n/-[ \$				
	Associated Samples	403 1135-8 Ank	+ all water					~			803849-Blank,	1-7 1. 19. 20	-	-	8038277-BANK,	12, 15, 21, 28				
l ≥0.05 RRF ?	Finding RRF (Limit: >0.05)							o. 04735											and a second	
iteria of ≤25 %D and	Finding %D (Limit: <u>&lt;</u> 25.0%)	33.60319	31.00872				48.37592				12.08435				74.53645	27. 6027U				
Were all %D and RRFs within the validation criteria of ≤25 %D and ≥0.05 RRF ?	Compound	Ludomethane	HH				B	RR			F-4			A	ĥ	EFEE				
ere all %D and RRFs	Standard ID	٨٤٧					CCV				ICV			+ 0.2/	201					
Y/N N/A We	# Date	4 1/21/08	-	 ,		4	1 130/00	14:32			4 2/4/02			211.109	2010			· · · ·		

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## VALIDATION FINDINGS WORKSHEET **Continuing Calibration**

Page: Z of Z 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> Ware percent differences (%D) and relative records for for the percent differences (%D) and relative records for for the percent differences (%D) and relative records for for the percent differences (%D) and relative records for for the percent differences (%D) and relative records for the percent differences (

SE SE	M/A	Vere all %D and RRF:	Were all %D and RRFs within the validation criteria of ≤25 %D and	iteria of ≤25 %D and	d ≥0.05 RRF ?	Were all %D and RRFs within the validation criteria of ≤25 %D and ≥0.05 RRF ?	
) #	Date	Standard ID	Compound	Finding %D (Limit: ≤25.0%)	Finding RRF (Limit: >0.05)	Associated Samples	Qualifications
+	2/5 00	cev	J	×19.46153		203849 - Black	17/0 1.4
+	9.12		Iodomethane	32. 31122		171 61 11 4-1	
+	-		φ	24.82024		19.20	
4			2. Nitropropane	33.4997			
			EEEE 1		0. 01921		1/uJ/2
	3/5/08	eev	<b>3 3 3 3 3 3 3 3 3 3</b>	0-00-0	65000.0		1/11/
	, 10.14						4
	-						

LDC #: 18 386 A) SDG #: <u>fre cone</u> /			VALIDA'	TION FIND	VALIDATION FINDINGS WORKSHEET <u>Blanks</u>	RSHEET			Page:_ Reviewer:	10
	VIS VOA (EPA SW 846 Method 8260B) ifications below for all questions answered " Was a method blank associated with every Was a method blank analyzed at least once Was there contamination in the method blan date: 1(3, b)00	ethod 8260B estions answ ociated with ulyzed at leas in the metho	ered " every t once od blau	N". Not applicable qu sample in this SDG? • every 12 hours for e nks? If yes, please se	AS VOA (EPA SW 846 Method 8260B) ifications below for all questions answered "N". Not applicable questions are identified as "N/A". 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 qualifications below. date: 1(3,0)00	identified as and concentr cations belov	"N/A". ation? v.		2nd Reviewer:	
	-		Ř	Associated Samples:	mples:	All V	ia lut			-
Compound	Blank ID					Sample Identification	ation			
	8031135-Blank	ank	Ĩ Č	11	1					
y icu jorom cryam Methylene chleride	0.16		0.20/1.01	0.14/1.04	0.18/1.0M					
Acetone			-	-				     		
-										
						\ \				
-										
CROL										
Blank analysis date: $2 6 69$	5									
			Aı	Associated Samples:		12, 15				
Compound	Blank ID					Sample identification	ation			
	8038277-8 ank	Ank	2	Ē						
Vicuitorow ethane Methytene chloride	2.0		6.1/4	4.7 /C CIA						
Aaetone			4			-				
				_						
ICHUL						-			-	
All results were qualified using the criteria stated below except those circled.	criteria stated	oelow except the	ose circled.	•						
Note: Common contaminants such as Methylene chloride. Acetone, 2-Butanone, Carbon disulfide and TICs that were detected in samples within ten times the associated method blank concentration were qualified as not detected. "U". Other contaminants within five times the method blank concentration were also qualified as not detected in samples within ten times the associated method blank concentration were	) as Methylene c er contaminants	hloride, Acetone within five time	a, 2-Butanone, C s the method bli	arbon disulfide c ank concentratio	and TiCs that wer on were also qual	a detected in sar filed as not date	nples within ten ti Mad = = =	mes the associa	tted method blank	concentration were

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LDC # 18 280 +1/ SDG # <u>put count</u>			VALIDAT	ION FINDINGS V Field Blanks	LIDATION FINDINGS WORKSHEET Field Blanks	SHEET		Page: Zof Z
; GC/N	A SW 846 Me lanks identifie	AS VOA (EPA SW 846 Method 8260B) Were field blanks identified in this SDG?	~					2nd Reviewer:
Y N N/A Vere target compounds detected in the field blanks? Blank units: wall Associated sample units: wall of their Field blank true! (circle one) Field Blank / Binstel / Trin Blank), Other	compounds d clated samp	Were target compounds detected in the <u>AIL</u> Associated sample units: <u>wo</u> re <u>AICircle one</u> ) Field Blank / Binsete / <u>Win</u>	field blanks?	i.	icces Vessor	Accordated Cameloc.	9 4- 1	(ND + 75x)
Compound	Blank ID   6	Blank ID			DOREC	arou Oarripico. Samole Identification	-	1
	1/24/08		4	১	و			
Vi UNOTO me thane Nothytene chioride	0.20			(	۱			
Acetone	4.3		17 / al u	6.8 AN	14 klu			
<del>Chlerofo</del> rm			-	-	-			
CROL								
Blank units: <u>va   _</u> Associated sample units: Field blank type: (circle one) Field Blank / Rinsate	Associated sample units: e one) Field Blank / Rinsate	le units: wa / Rinsate/ Trip	Blank/Other:	er: .	Associ	Associated Samples:	) (1 <del>4</del> L	(xSZ T ON,
Compound	Blank ID 17	Biank ID				Sample Identification		
	124/08							
VICINIO ROME YARANE Methytene shloride	0.14							
Acetone								
Chloroform								
CRQL								
CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT: Common contaminants such as Methylene chloride, Acetone, 2-Butanone and Carbon disulfide that were detected in samples within ten times the associated field blank concentration were qualified as not	DUALIFIED, ALI thylene chloride,	- RESULTS NOT Acetone, 2-Butar	CIRCLED WER	RE QUALIFIED B n disulfide that w	Y THE FOLLOW! are detected in sal	NG STATEMENT: mples 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".

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DC #			VALIDAT	TON FIND	VALIDATION FINDINGS WORKSHEET <u>Field Blanks</u>	KSHEET				5 K
: GC/N	W 846 Met s identified	thod 8260B) d in this SDG								
Y_N_N/A	npounds d t <b>ed sampl</b> eld Blank /	C 1 ~	the field blanks?	2 161:	Asso	Associated Samples:	s: 	514	XmaHo 2< + 0N) 51 4-	X
Compound Bla	Blank ID 18					Sample Id	Sample Identification			
Alley January	24 00		13	4						
Menyiana chlorida	0.15		t							
	4.9		19/214	1-4 /2IM	5					
<u>ehtorofor</u> m	-		-							
CROL.										
Blank units:Associated sample units:_ Field blank type: (circle one) Field Blank / Rinsate	Associated sample units: e one) Field Blank / Rinsate		' Trip Blank / Other:	Jer:	Asso	Associated Samples:	9S:			
ound	Blank ID	Blank ID				Sample ic	Sample identification			
allessin the										
Methylene chloride										
Acetone										
Chloroform										
										T
CROL										
CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT: Common contaminants such as Methylene chloride, Acetone, 2-Butanone and Carbon disulfide that were detected in samples within ten times the associated field blank concentration were qualified as not detected, "U". Other contaminants within five times the field blank concentration were qualified as not detected, "U". Other contaminants within five times the times times the times the times the times the times the times	ALIFIED. ALI lene chloride, lin five times i	- RESULTS NO Acetone, 2-Butz the field blank oc	r CIRCLED WE inone and Carbo prcentration wer	RE QUALIFIED on disulfide that re also qualified	BY THE FOLLO were detected in as not detected,	WING STATEME samples within te "U".	NT: In times the assoc	ciated field blank o	oncentration were qualifi	ed as not

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## VALIDATION FINDINGS WORKSHEET Surrogate Spikes

of 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>\*\* N\_NA</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?

				لم	5																	_
Qualifications	J+/P dut			1+/2 dut ever nonanal only																		
(Limits)	(-2-132-)	( 511-97)	) (	( (وه-۱۱ ک)	(	( )	•	( )	( )	( )	( )	) (	( )	( )	( )	( )	( )	) (	( )	( )	)	
%Recovery (Limits)	124			120																		OC I imits (Water)
Surrogate	BFB			BFB				-														C
Sample ID	8036136 - BRNK			اله																		OC Limits (Soil)
Date												•										
*																						

QC Limits (Soil) 81-117 74-121 80-120 80-120 SMC1 (TOL) = Toluene-d8 SMC2 (BFB) = Bromofluorobenzene SMC3 (DCE) = 1,2-Dichloroethane-d4 SMC4 (DFM) = Dibromofluoromethane

88-110

86-115 80-120 86-118

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LDC #:	SDG #:

## VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

∕ of Page: Reviewer: 2nd Reviewer:

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

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

Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water. AN 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?

기								
#	Date	di QSW/SW	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		P420	Н	198 (55-139)		42 (30)	7-	WO DI LAT 148 IN
			EEEE	363 (30-150)	(	1-		
			HH	( )	( )	( 02 ) Gh		
				( )	( )	( )		
				( )	(	( )		
					( )	( )		
		2422	AA	152 ( 25-150	151 ( )	(	اک	WO DUAL LOS IN
			EFEE		( )	31 (20)		
		Todd	Todomethane	( )	( )			
			HH	( )	( )	~		
				(	( )			
				(	( )	( )		
				( )	( )	(		
				( )	( )	· ·		
				( )	( )	)		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
		Compound	und	QC Limits (Soil)	s (Soil)	RPD (Soil)	QC Limits (Water)	RPD (Water)
	Η	1,1-Dichloroethene		59-172%	2%	< 22%	61-145%	< 14%
	Ś	Trichloroethene		62-137%	37%	< 24%	71-120%	< 14%
	<u>,</u>	Benzene		66-142%	2%	< 21%	76-127%	< 11%
	CO	Toluene		59-139%	6%	< 21%	76-125%	< 13%
	DD.	Chlorobenzene		60-133%	33%	< 21%	75-130%	< 13%

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VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

ð Page: Reviewer: 2nd Reviewer:

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

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

A/A AN

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

		2																							
	Qualifications	10/SM JAND on																							
	Associated Samples	so Joyg-Blank,	71. 81 1 1 4-1																						
	RPD (Limits)	( )	(	( )	()	(	( )	( )	( )	(	(	( )	( )	( )	( )	· · ·	( )	( )	(	(	( )	( )	( )	)	( )
rcsD	%R (Limits)	( )			( )	( )	( )	( )	( )	( )	( )	( )	(	( )	( )	( )	( )	( )	(	)	( )	( )	()	( )	
LCS	%R (Limits)	ne 142/40-140)		(	(	(	( )	( )	( )	( )	( )	( )	(	( )	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
	Compound	2 - Nitrograganc	<b>r</b> 1																						
	LCS/LCSD ID	8038049-Les																							
	Date																		_						
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LCSLCSD.1SB

LDC #: 18386 A ) SDG #: See Cover

### VALIDATION FINDINGS WORKSHEET Field Duplicates

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

YN N/A YN N/A

Were field duplicate pairs identified in this SDG? Were target compounds detected in the field duplicate pairs?

	Concentrat	tion ( ug Kg		ene
Compound	<u> </u>	12	Virre	rene
dichloromethane	5.8	6.1	0.3	(4 5.4)
POD	0.4	5.34	4.89	(= 5.4)
			1 .	· · · /

	Concentration ()	
Compound		RPD

	Concentration ()	
Compound		RPD
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	Concentration ()	
Compound		RPD

ell cover LDC #: 18 386A ) SDG #:

## VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

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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

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

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

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

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	RRF (20 std)	RRF ( 72/ std)	Average RRF (initial)	Average RRF (initial)		USO%
-	ICAL	30/4/08	Vinyl Chloride	0.77522	0.71822	6.13013	043013	6.16160	011-91-9
			200 internal standard)	61562.1	1-79313	1.80952	1-8025	18120.1	1912.4
			Contraction (1) (1) (1) (1) (1) (1) (1) (1) (1) (1)	1.53477	1-53477	1.54561	1-24-22-1	3.5403)	3,5408
7			しして、 (1st internal standard)	0.00342		6000	16200.0	11-51035	52012-11
			Vimethy (2nd internal standard)	0.50639	6.5063.0	o.s 4575	542 20	hSazt:El	
			1. 2 JA to the floor	0.5%190	0-88190	0.899-71	(L7922-0	9.47511	11512.0
б			Thi chlor the Mis com- (1st internal standard)						
			(2nd internal standard)						
			(3rd internal standard)						
4			(1st internal standard)						
			(2nd internal standard)						
			(3rd internal standard)						

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

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

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

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

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF = (A<sub>x</sub>)(C<sub>s</sub>)/(A<sub>s</sub>)(C<sub>x</sub>)

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

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

1.36954 0.89307 3.575 Recalculated 1221-E 1143218 6-716765 0% 12132101 0.89307 5-76-765 -3695-3.571S 1727.E Reported 0% Recalculated 12628.0 1-87686 1.53 80 1.09104 65000.0 0.58269 RRF (CC) 1-87 639 2628.0 0.582 69 Reported 1.531 80 0.00 20 halba. RRF (CC) Average RRF 61061.0 1. 209SZ 1. 0 7630 ومعلاكات 0.0029 (initial) 1.5456 Compound (Reference internal Standard) Crit ch longle his en a Ethyl Bens en standard) - DCB [3rd internal standard) Dineting (2nd internal standard) (2nd internal standard) (1st internal standard) (2nd internal standard) (3rd internal standard) (1st internal standard) (3rd internal standard) (1st internal standard) (1st internal standard) Vingl chiende 33 13.5-ہ -Calibration 2/5/00 h1:01 Date 80/2/c 21:12 Standard ID 3 3 2 # ი

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.

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### VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

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### 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: 45

Percent Percent Recovery Surrogate Surrogate Recovery Percent Spiked Found Reported Recalculated Difference 0 109 50 54.6314 109 Toluene-d8 52.9992 106 106 Bromofluorobenzene 97 97 48.7043 1,2-Dichloroethane-d4 48.6525 97 97 J 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			· · ·		

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

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

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

٩ Page: 2nd Reviewer: 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: 19 + 20

	<i>•</i> •••••••••••••••••••••••••••••••••••	Spike	Sample	Spiked Sample	ample	Matrix Spike	Spike	Matrix Spike Duplicate	Duplicate	WS	MS/MSD
Compound	loopy )	1 (ka)	Concentration (ver ker)	Concentration	tration	Percent Recovery	ecovery	Percent Recovery	acovery		RPD
	WS	J MSD		SM NS		Reported	Recalc.	Reported	Recalc.	Renorted	Becalculated
1,1-Dichloroethene	52.4	51.9	n D	ج 19	59-1	01	011	ہم =	113	<i>p. c</i>	۲. <i>1</i>
Trichloroethene			-	50.4	55. 1	a c	96	104	hal	8.8	2.2
Benzene				56.0	र <u>न</u> .0	و ر ه	90	102	101	2.0	, , r
Toluene				21.2	يد. ك	103	103	Sol	201	6.1	2.0
Chlorobenzene	->	<u> </u>			54.2	100	001	201	503	- ~ ~	7.6

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.

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## Laboratory Control Sample Results Verification VALIDATION FINDINGS WORKSHEET

Page: / of 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: SSC = Spiked sample concentration SA = Spike added

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

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

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	5 S	Spike	Spiked :	sample		CS	USU I			
Compound	Add Add	ded Kel	Concentration	tration						100
「「「「「「「」」」、「「」」、「」」、「「」」、「「」」、「」、「」、「」、「				X	rercent	rercent kecovery	Percent Recovery	tecovery	RPD	0
	1 CS	1 CSD	L CS		Reported	Recalc	Renorted	Recalc	Reported	Bocalculated
1,1-Dichloroethene	20.0	うや	ча. S	<b>₹</b> 2	60	99				
Trichloroethene	_	l	47.	-	94	94				
Benzene			47.24		96	96				
Toluene			48.4		76	47				
Chlorobenzene		~~	48.2		31	96	NA			
-										
		i								
Comments: <u>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</u>	y Control Sa	mple findings	worksheet for	list of qualifi	cations and a	ssociated sa	nples when re	ported results	L do not agree wit	L IO.0% of the

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recalculated results.

LDC # SDG #	<u>العرام:</u> برمر :#	(386A) coner		FINDINGS WORKSHEET       Page:of         Iculation Verification       Reviewer:2         2nd reviewer:2       Page:of
<u>YN</u>	OD: N/A N/A		ults recalculated and	d verified for all level IV samples? target compounds agree within 10.0% of the reported results?
Conce	ntratior	$n = \frac{(A_{s})(I_{s})(DF)}{(A_{is})(RRF)(V_{o})(\%S)}$		Example:
A <sub>x</sub>		Area of the characteristic ion ( compound to be measured	EICP) for the	Sample I.D. #5, Autone
$A_{is}$	=	Area of the characteristic ion ( internal standard	EICP) for the specific	
l <sub>s</sub>	=	Amount of internal standard ac	ded in nanograms	Conc. = () () ()

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(	only				
#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		× = (10043 \/1	- 0.13125		
		$\frac{x}{50} = \frac{10043}{619429} \left( \frac{1}{0.061} \right)$			
		X = 6.529			
		finn = 6.529 =	6-8 ug ks		
		0.959	00		
		, , , , , , , , , , , , , , , , , , ,			
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	•				

(ng)

Dilution factor.

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Relative response factor of the calibration standard.

Volume or weight of sample pruged in milliliters (ml) or grams (g).

Percent solids, applicable to soils and solid matrices

RRF

V,

Df

%S

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

Project/Site Name: BRC 1	Tronox Parcel H
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Collection Date: January 28, 2008

LDC Report Date: March 12, 2008

Matrix: Soil/Water

Parameters: Volatiles

Validation Level: EPA Level III

Laboratory:

TestAmerica, Inc.

### Sample Delivery Group (SDG): F8A290158

TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10' TSB-TB-1-1/28/08 TSB-TB-2-1/28/08 RINSATE-2 TSB-TB-03-1/28/08 TSB-HR-05-10'MS TSB-HR-05-10'MSD

### Introduction

This data review covers 11 soil samples and 4 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8260B for Volatiles.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

Raw data were not reviewed for this SDG. The review was 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
1/21/08	Dibromomethane	0.04510 (≥0.05)	All water samples in SDG F8A290158	J (all detects) UJ (all non-detects)	A
1/30/08	Ethanol	0.00855 (≥0.05)	All water samples in SDG F8A290158	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
1/30/08	Bromomethane	48.37592	All water samples in SDG F8A290158	J+ (all detects)	A
2/11/08	Ethanol 2-Methylhexane 3-Ethylpentane n-Heptane	28.63874 27.07574 25.56456 26.94019	All soil samples in SDG F8A290158	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	

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
1/21/08	lodomethane Vinyl acetate	33.60319 31.00872	All water samples in SDG F8A290158	J+ (all detects) J+ (all detects)	A
2/11/08	Acetonitrile 4-Methyl-2-pentanone 4-Chlorotoluene	27.39844 27.93055 25.96800	All soil samples in SDG F8A290158	J+ (all detects) J+ (all detects) J+ (all detects)	A

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

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
1/30/08	Dibromomethane	0.04735 (≥0.05)	All water samples in SDG F8A290158	J (all detects) UJ (all non-detects)	A
2/11/08	Ethanol	0.00649 (≥0.05)	All soil samples in SDG F8A290158	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 with the following exceptions:

Method Blank ID	Analysis Date	Compound TIC (RT in minutes)	Concentration	Associated Samples
8031135-Blank	1/30/08	Dichloromethane	0.16 ug/L	All water samples in SDG F8A290158

Sample concentrations were compared to concentrations detected in the method 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 method blanks with the following exceptions:

Sample	Compound TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
TSB-TB-1-1/28/08	Dichloromethane	0.21 ug/L	1.0U ug/L
TSB-TB-2-1/28/08	Dichloromethane	0.19 ug/L	1.0U ug/L
TSB-TB-03-1/28/08	Dichloromethane	0.22 ug/L	1.0U ug/L

Samples TSB-TB-1-1/28/08, TSB-TB-2-1/28/08, and TSB-TB-03-1/28/08 were identified as trip blanks. No volatile contaminants were found in these blanks with the following exceptions:

Trip Blank ID	Sampling Date	Compound	Concentration	Associated Samples
TSB-TB-1-1/28/08	1/28/08	Dichloromethane Acetone	0.21 ug/L 4.0 ug/L	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10'
TSB-TB-2-1/28/08	1/28/08	Dichloromethane Acetone	0.19 ug/L 5.1 ug/L	TSB-HJ-08-0' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10'
TSB-TB-03-1/28/08	1/28/08	Dichloromethane Acetone	0.22 ug/L 4.4 ug/L	RINSATE-2

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

Rinsate Blank ID	Sampling Date	Compound	Concentration	Associated Samples
RINSATE-2	1/28/08	Dichloromethane Acetone	12 ug/L 8.0 ug/L	All soil samples in SDG F8A290158

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

Sample	Compound	Reported Concentration	Modified Final Concentration
TSB-HJ-10-0'	Dichloromethane	6.1 ug/Kg	6.1U ug/Kg
TSB-HJ-10-10'	Dichloromethane	3.9 ug/Kg	5.2U ug/Kg
TSB-HR-06-0'	Dichloromethane	7.4 ug/Kg	7.4U ug/Kg
TSB-HR-06-0'-FD	Dichloromethane	3.0 ug/Kg	5.4U ug/Kg
TSB-HR-06-10'	Dichloromethane	7.3 ug/Kg	7.3U ug/Kg
TSB-HJ-08-0'	Dichloromethane	8.8 ug/Kg	8.8U ug/Kg
TSB-HJ-08-10'	Dichloromethane	5.8 ug/Kg	5.8U ug/Kg
TSB-HR-05-0'	Dichloromethane Acetone	7.8 ug/Kg 15 ug/Kg	7.8U ug/Kg 15U ug/Kg
TSB-HR-05-10'	Dichloromethane	6.5 ug/Kg	6.5U ug/Kg
RINSATE-2	Acetone	8.0 ug/L	8.0U ug/L

### 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
8036136-Blank	Bromofluorobenzene	126 (66-115)	All TCL compounds	J+ (all detects)	Р

### VII. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Although the MS/MSD percent recoveries and relative percent differences (RPD) were not within QC limits for some compounds, the LCS/LCSD 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. Percent recoveries (%R) and relative percent differences (RPD) were within QC limits.

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

All internal standard areas and retention times.

### XI. Target Compound Identifications

Raw data were not reviewed for this SDG.

### XII. Compound Quantitation and CRQLs

Raw data were not reviewed for this SDG.

### XIII. Tentatively Identified Compounds (TICs)

Raw data were not reviewed for this SDG.

### XIV. System Performance

Raw data were not reviewed for this SDG.

### XV. Overall Assessment of Data

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

### XVI. Field Duplicates

Samples TSB-HR-06-0' and TSB-HR-06-0'-FD were identified as field duplicates. No volatiles were detected in any of the samples with the following exceptions:

	Concentration (ug/Kg)			<b>.</b>		
Compound	TSB-HR-06-0'	TSB-HR-06-0'-FD	RPD (Limits)	Difference (Limits)	Flag	A or P
Dichloromethane	7.4	3.0	-	4.4 (≤5.4)	-	-
Toluene	0.54	5.4U	-	4.86 (≤5.4)	-	-

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### BRC Tronox Parcel H Volatiles - Data Qualification Summary - SDG F8A290158

SDG	Sample	Compound	Flag	A or P	Reason
F8A290158	TSB-TB-1-1/28/08 TSB-TB-2-1/28/08 RINSATE-2 TSB-TB-03-1/28/08	Dibromomethane Ethanol	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A	Initial calibration (RRF)
F8A290158	TSB-TB-1-1/28/08 TSB-TB-2-1/28/08 RINSATE-2 TSB-TB-03-1/28/08	Bromomethane	J+ (all detects)	A	Continuing calibration (%D)
F8A290158	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10'	Ethanol 2-Methylhexane 3-Ethylpentane n-Heptane	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	A	Continuing calibration (%D)
F8A290158	TSB-TB-1-1/28/08 TSB-TB-2-1/28/08 RINSATE-2 TSB-TB-03-1/28/08	lodomethane Vinyl acetate	J+ (all detects) J+ (all detects)	A	Continuing calibration (ICV %D)
F8A290158	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10'	Acetonitrile 4-Methyl-2-pentanone 4-Chlorotoluene	J+ (all detects) J+ (all detects) J+ (all detects)	A	Continuing calibration (ICV %D)
F8A290158	TSB-TB-1-1/28/08 TSB-TB-2-1/28/08 RINSATE-2 TSB-TB-03-1/28/08	Dibromomethane	J (all detects) UJ (all non-detects)	А	Continuing calibration (RRF)
F8A290158	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10'	Ethanol	J (all detects) UJ (all non-detects)	A	Continuing calibration (RRF)

### BRC Tronox Parcel H Volatiles - Laboratory Blank Data Qualification Summary - SDG F8A290158

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P
F8A290158	TSB-TB-1-1/28/08	Dichloromethane	1.0U ug/L	A
F8A290158	TSB-TB-2-1/28/08	Dichloromethane	1.0U ug/L	Α
F8A290158	TSB-TB-03-1/28/08	Dichloromethane	1.0U ug/L	А

### BRC Tronox Parcel H Volatiles - Field Blank Data Qualification Summary - SDG F8A290158

SDG	Sample	Compound	Modified Final Concentration	A or P
F8A290158	TSB-HJ-10-0'	Dichloromethane	6.1U ug/Kg	А
F8A290158	TSB-HJ-10-10'	Dichloromethane	5.2U ug/Kg	A
F8A290158	TSB-HR-06-0'	Dichloromethane	7.4U ug/Kg	А
F8A290158	TSB-HR-06-0'-FD	Dichloromethane	5.4U ug/Kg	A
F8A290158	TSB-HR-06-10'	Dichloromethane	7.3U ug/Kg	A
F8A290158	TSB-HJ-08-0'	Dichloromethane	8.8U ug/Kg	A
F8A290158	TSB-HJ-08-10'	Dichloromethane	5.8U ug/Kg	А
F8A290158	TSB-HR-05-0'	Dichloromethane Acetone	7.8U ug/Kg 15U ug/Kg	A
F8A290158	TSB-HR-05-10'	Dichloromethane	6.5U ug/Kg	А
F8A290158	RINSATE-2	Acetone	8.0U ug/L	Α

### VALIDATION COMPLETENESS WORKSHEET

LDC #: <u>18386B1</u> SDG #: <u>F8A290158</u> Laboratory: <u>Test America</u>

### Level III

	3/7/08
Date:_	,
Page:_	<u>/of/</u>
Reviewer:	<u> </u>
2nd Reviewer:	<u></u>

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

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

	Validation Area		Comments
١.	Technical holding times	Δ	Sampling dates: \2808
11.	GC/MS Instrument performance check	$\triangle$	, ·
111.	Initial calibration	SN	% PSD ( <sup>2</sup> ZO.990
IV.	Continuing calibration/ICV	لىبى	INEN
V.	Blanks	SW	
VI.	Surrogate spikes	<i>s</i> ω	
VII.	Matrix spike/Matrix spike duplicates	sW	
VIII.	Laboratory control samples	4	LCS/D
IX.	Regional Quality Assurance and Quality Control	N	
Х.	Internal standards	A	
XI.	Target compound identification	N	
XII.	Compound quantitation/CRQLs	N	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	N	
XV.	Overall assessment of data	A	
XVI.	Field duplicates	3	D = 3,4
XVII.	Field blanks	SW	R = 12 TB = 10 TB=
lote:		o compounds	= 11, 13

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:		
	SOIL	4	water

13	TSB-HJ-10-0'	11	≁ TSB-TB-2-1/28/08 ₩ 7	21 1	8031135	31	1130
23	TSB-HJ-10-10'	12	RINSATE-2	22 Z	8036136	32	nonanal only 2/4
3 ~	TSB-HR-06-0' 👂 1	13 l	2 TSB-TB-03-1/28/08	<u>2</u> 33	8043263	33	2/11
4 <del>3</del>	TSB-HR-06-0'-FD D 1	14	TSB-HR-05-10'MS	24	8.	34	
5 °)	TSB-HR-06-10'	15	TSB-HR-05-10'MSD	25		35	
63	TSB-HJ-08-0' · •	16		26		36	
73	TSB-HJ-08-10'.	17		27		37	
83	TSB-HR-05-0'	18		28		38	
9 J	۲SB-HR-05-10'	19		29		39	
10	Т́SB-ТВ-1-1/28/08 W	20		30		40	

TARGET COMPOUND WORKSHEET

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

L					
<u>*  </u>	A. Chloromethane*	U. 1,1,2-Trichloroethane	OO. 2,2-Dichloropropane	III. n-Butylbenzene	CCCC.1-Chlorohexane
ш 	B. Bromomethane	V. Benzene	PP. Bromochloromethane	JJJ. 1,2-Dichlorobenzene	DDDD. Isopropyl alcohol
<u> </u>	C. Vinyl choride**	W. trans-1,3-Dichloropropene	QQ. 1,1-Dichloropropene	KKK. 1,2,4-Trichlorobenzene	EEEE. Acetonitrile
1	D. Chloroethane	X. Bromoform*	RR. Dibromomethane	LLL. Hexachlorobutadiene	FFFF. Acrolein
<u> </u>	E. Methylene chloride	Y. 4-Methyl-2-pentanone	SS. 1,3-Dichloropropane	MMM. Naphthalene	GGGG. Acrylonitrile
<u> </u>	F. Acetone	Z. 2-Hexanone	TT. 1,2-Dibromoethane	NNN. 1,2,3-Trichlorobenzene	HHHH. 1,4-Dioxane
0	G. Carbon disulfide	AA. Tetrachloroethene	UU. 1,1,1,2-Tetrachloroethane	000. 1,3,5-Trichlorobenzene	III. Isobutyl alcohol
<u> </u>	H. 1,1-Dichloroethene**	BB. 1,1,2,2-Tetrachloroethane*	W. isopropylbenzene	PPP. trans-1,2-Dichloroethene	JJJJ. Methacrylonitrile
<u> </u>	I. 1,1-Dichloroethane*	CC. Toluene**	WW. Bromobenzene	QQQ. cis-1,2-Dichloroethene	KKKK. Propionitrile
<u>ا</u> ۲	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.
2	M. 2-Butanone	GG. Xylenes, total	AAA. 1,3,5-Trimethylbenzene	UUU. 1,2-Dichlorotetrafluoroethane	0000.
z	N. 1,1,1-Trichloroethane	HH. Vinyl acetate	BBB. 4-Chlorotoluene	VVV. 4-Ethyltoluene	PPPP.
0	O. Carbon tetrachloride	II. 2-Chloroethylvinyl ether	CCC. tert-Butylbenzene	WWW. Ethanol	<u>aaaa.</u>
<u> </u>	P. Bromodichloromethane	JJ. Dichlorodifiuoromethane	DDD. 1,2,4-Trimethylbenzene	XXX. Di-isopropyl ether	RRR.
σ	Q. 1,2-Dichloropropane**	KK. Trichlorofluoromethane	EEE. sec-Butylbenzene	YYY. tert-Butanol	SSSS.
∝	R. cis-1,3-Dichloropropene	LL. Methyl-tert-butyl ether	FFF. 1,3-Dichlorobenzene	ZZZ. tert-Butyl alcohol	TTT.
S	S. Trichloroethene	MM. 1,2-Díbromo-3-chloropropane	GGG. p-Isopropyltoluene	AAAA. Ethyl tert-butyl ether	UUUU.
F	T. Dibromochloromethane	NN. Methyl ethyl ketone	HHH. 1,4-Dichlorobenzene	BBBB. tert-Amyl methyl ether	VVVV.

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

LDC #: 1 X 3 26 B)

# VALIDATION FINDINGS WORKSHEET **Initial Calibration**

ò 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".

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

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

	( <u> </u>		_	 		_		 	 	 	·····	 	 	 	 
	Qualifications	-2/m/A-	7												
	Associated Samples	-2021135-Blank,	All water	>											
RSD and ≥0.05 RRF ?	Finding RRF (Limit: <u>&gt;</u> 0.05)	01240.0	<del>0.00477</del>	0. UOB 53											
e criteria r ion criteria of ≤30 %I	Finding %RSD (Limit: <u>≤</u> 30.0%)														
RFs within the validat	Compound	RR	Record	3											
Were all %RSDs and RRFs within the validation criteria of ≤30 %RSD and ≥0.05 RRF ?	Standard ID	IGAL		141											
Y (N/N/A V	# Date	89/12/1	-	1 20/00											

LDC #: 1836 0 SDG #: Ser cover

# VALIDATION FINDINGS WORKSHEET **Continuing Calibration**



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

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

Was a continuing calibration standard analyzed at least once every 12 hours for each instrument? Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's ? Were all %D and RREs within the validation criterio of 75 %D and 50 method

	Qualifications	1+ / A dot						1+// 1.+	$\sim$	$\downarrow$		J/m/L	1+/A dut	- E		<u> </u>	14/2 0.4	1/ A aur		Å	
	Associated Samples	8031135.8)ank,	All water						/~	Þ		504 3263 - Blank	KII soils			$\overline{\gamma}$				>	
í ≥0.05 RRF ?	Finding RRF (Limit: >0.05)								7.4140.0			0.006491									
iteria of ≤25 %D and ≥0.05 RRF ?	Finding %D (Limit: ≤25.0%)	33.60319	31.00872			•		48.27592					28.63874	27.07574	25.56456	26.94019	hiser LC	1 9 7 7 7	1010 10	22 7 68UU	
Were all %D and RRFs within the validation criteria	Compound	Icolomethane !	НH					8	RR			3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3	۲ 3 3	2 + Mekry / hyane	34Ethylpentane	- heptane		4	000	6190	
Vere all %D and RRFs	Standard ID	101						ce V				eev		¢	3	v	Mer 10,1				
/N N/A V	Date	80 121	-					1 30 08	14:32			39/11/2	11:81				2/11/00	1	));.a		
Ş.	) #	+	+					4					+	+	+	+	+	+	- 4	-	

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LDC #: 15386B) SDG #: 200 conol			VALIDA	TION FIND Bl	VALIDATION FINDINGS WORKSHEET <u>Blanks</u>	KSHEET		Rev.	Page: / of/ Reviewer: 75
METHOD: GC/MS VOA (EPA SW 846 Method 8260B) Please see qualifications below for all questions answered " $\underline{Y}$ N/A Was a method blank associated with every i $\underline{Y}$ N/A Was a method blank analyzed at least once $\underline{Y}$ N/A Was there contamination in the method blank Blank analysis data: $\frac{12.0 \ 10.0}{12.0 \ 10.0}$	(EPA SW 846 M s below for all qu nethod blank ass nethod blank ans re contamination	VIS VOA (EPA SW 846 Method 8260B) liftications below for all questions answered * Was a method blank associated with every Was there contamination in the method blan date. 1/2.0108	ered "† every ( t once od blar	N". Not applicable qu sample in this SDG? every 12 hours for e nks? If yes, please se	AS VOA (EPA SW 846 Method 8260B) fifcations below for all questions answered "N". Not applicable questions are identified as "N/A". Was a method blank associated with every sample in this SDG? Was there contamination in the method blanks? If yes, please see the qualifications below.	dentified as " nd concentre cations below	N/A". ttion?	2nd Reviewer:	:Mer:
Conc. units: wa/L	onta		As	Associated Samples:	nples:	<b>A</b> 0	ū	all walt sample	(aga
Compound	Blank ID				ÿ	Sample Identification	tion		
	8031135 - Blan K	10	11	2-	13	•			
Methylene chloride	0.16	0.21/1.0 V	no.1/61.0	(2)	N.0.1/ 12-0				
Acetone					1				
-									
CROL.									
Blank analysis date:									
Cone, units:			As	Associated Samples:	:88				
Compound	Blank ID				S	Sample identification	tion		
Methylene chloride									
Acetone						-			
ICRAL								-	
. All results were qualified using the criteria stated below except those circled.	s criteria stated	selow except tho	se circled.	•					
Note: Common contaminants such as Methylene chloride, Acetone, 2-Butanone, Carbon disulfide and TICs that were detected in samples within ten times the associated method blank concentration were given and an event of the start of the store	n as Methylene c ier contaminants	hioride, Acetone within five times	, 2-Butanone, Ce the method ble	arbon disulfide ar Ink concentration	nd TiCs that were 1 were also qualif	detected in sam lied as not detect	oles within te ted e e	n times the associated method bi	ank concentration were

BLANKS2.1SB

ı										
METHOD: GC/MS VOA (EPA SW 846 Method 8260B)Y N/NAWere field blanks identified in this SDG?Y N N/AWere target compounds detected in theBlank units: way bAssociated sample units: way	V SW 846 Me anks identifie compounds c clated samp	thod 8260B) d in this SDC letected in th	AS VOA (EPA SW 846 Method 8260B) Were field blanks identified in this SDG? Were target compounds detected in the field blanks?	~						
Field Diank type: (circle one)	) Field Blank Blank ID [7	Rinsate I Blank ID	rip Blank / Oth	ler:	Assoc	Associated Samples:	tification	C1100 M		
	1		-	4	e	1	ە	9	٢	8
Dichlerom ethane			10.1/11	3.915.24	1.4/2	3.0/5.44	7,3/M	8.5/ N	5.8/4	n/8°L. \$\$
Acetone	۵,۵		١	- \				. (	- \ \	15/01
Chlorefotm										_
CROL										
Blank units: we be Associated sample units: we K	ciated samp ) Field Blank	le units:/ / Rinsate / T	wg ∐rey Trip Blank / Other:	ler: .	Assoc	Associated Samples:		All soils	~	
Compound	Blank ID_12	Blank ID				Sample Id	Sample Identification			
	1/24/08		6							
Pichlorome them	12		6+5/4							
Acetone	4.0		T-ta-5/4							
Chloroform										
CRQL						-				

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

FBLKASC2.1SB

METHOD:: GC/MS VOA (EFA SW 846 Metrica S00B)     2nd Revious Jamifican Inits SDC9       VINIXA. Were field branch starting from this SDC9     Vinixa Were field branch starting from this SDC9       VINIXA. Were field branch starting from this SDC9     Vinixa Were field branch starting from this SDC9       VINIXA. Were field branch starting from this SDC9     Vinixa Were field branch starting from this SDC9       Refer unix Target of the starting from this SDC9     Vinixa Were field branch starting from this SDC9       Refer unix Target of the starting from this SDC9     Vinixa Were field branch starting from this SDC9       Refer unix Target of the starting from this SDC9     Vinixa Were field branch starting from this SDC9       Refer unix Target of the starting from this SDC9     Vinixa Were from the starting from the	nks?	2nd X
Other:     Associated Samples:     Image: Sample identification       Sample identification     Sample identification       Sample identification     Sample identification	Other: Associated	X012 5 4-1
Sample Identification	Other:	
	Image: Second state     Image: Second state       Image: Second state     Image: Second state       Image: Second state     Image: Second state	Sample Identification
This Blank / Other: Associated Samples: 6 7 1 Sample identification	Trip Blank / Other: Associate	-
vy let vy le	Minipelies Associated	
vg/k     vg/k     k - v       vg/k     Associated Samples:     k - v	Valley Valley Trip Blank / Other: Associated	
valler valler Samples: 6 − P ⊂ 1 Samples: 6 − P ⊂ 1 Samples: 6 − P ⊂ 1 Samples: 6 − P ⊂ 1	Valley Valley X Associate	
Market     Market       Market     Associated Samples.       Sample Identification       Sample Identification	Trip Blank / Other: Associated	
Market     Associated Samples:     6 - P - P       Market     Associated Samples:     6 - P - P       I     15 / M     15 / M	Ver Lev Ver Lev Trip Blank / Other: Associated	
Market     Associated Samples:     6     9       Associated Samples:     6     9       Associated Samples:     6     9	Val Ka Val Ka Trip Blank / Other: Associate A 15/V	
Marky     Associated Samples:     6 - P       R     Sample Identification	Var Leon Associated	
Variation     Associated Samples:     Associated Samples:       R     Sample Identification       I     I	Trip Blank / Other: Associate	
	- - 15/4	) b 4-9
12 ¥108       Jacro we Heart       I.a. or Me       ene chloride       0. M       ene chloride       form       form	o me them to the stand of the s	Sample identification
Loro we then	omethan 0.9	
CRQL		
	CROL	

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

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# VALIDATION FINDINGS WORKSHEET **Field Blanks**



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		þlanks?	_1
METHOD: GC/MS VOA (EPA SW 846 Method 8260B)	Were field blanks identified in this SDG?	Were target compounds detected in the field blanks?	Associated sample units: VA
C/MS VOP	Were fi	Were to	
METHOD: G(	Y N/N/A	Y/N N/A	Blank units: v& ∏

Blapk / Other: Associated Samples:	Sample Identification			8.0/u				
e units: Rinsate //Trip	Blank ID							
Field Blank /	Blank ID 13 Blank ID	1 26/08	0.22	4.4				
Blank units: <u>why besectated sample units: why besectated sample units: why besectated blank type:</u> Citcle one) Field Blank / Rinsate / (Trip Blank / Other:	Compound		DUTULOR METURE	Acetone	Chlenoform			CRQL

# Associated sample units: Blank units:\_ Field blank ty

9

Blank ID	Blank ID	Sample i	Sample identification		
			-		

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

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# VALIDATION FINDINGS WORKSHEET Surrogate Spikes

ð 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-A/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 N/N/N

	Qualifications	J+/Part																			
	%Recovery (Limits)	128 124 ( <del>63-133</del>	( 57-112)	)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	
	%Rec	428																			<u>OC Limits (Water)</u> 88-110 86-115 80-120 86-118
	Surrogate	BFB				-															
	Sample ID	8036136 - B)ank																			<u>OC Limits (Soil)</u> 81-117 74-121 80-120 80-120
oi criteria?	Sam	803613																			SMC1 (TOL) = Toluene-d8 SMC2 (BFB) = Bromofluorobenzene SMC3 (DCE) = 1,2-Dichloroethane-d4 SMC4 (DFM) = Dibromofluoromethane
0 10	Date											-									TOL) = Toluer BFB) = Bromc DCE) = 1,2-Di DFM) = Dibro
- [	*																				MC1 ( MC2 ( MC3 (

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# VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

3 õ Page: Reviewer: 2nd Reviewer:

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

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

Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water. N/N/ N A/N N/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?

1								
#	Date	DI DSW/SW	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Dualifications
		14+ 15	Dichlerometh	Dichleronzthane 157 (47-145)	( )	( )	6	WO COURT I E SI'N
			ΔA	( OS-1-3C ) 291	160 (22-12)	( )		-
			HH	( )		× (20)		
				( )	( )			7
				(	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				(	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	(		
				(	( )	( )		
				( )	)	( )		
				( )	(	( )		
				( )	)	( )		
				(	(	( )		
				( )	(	( )		
				( )	( )	( )		
		Compound	puno	QC Limits (Soil)	ts (Soil)	RPD (Soil)	OC Limits (Water)	PDD (Weter)
	т	1,1-Dichloroethene		59-1	59-172%	< 22%	61-1460.	(1916) / 140/
	S.	Trichloroethene		62-1	62-137%	< 24%	71-170/0	14 /0
	,	Benzene		66-1	66-142%	< 21%	0/ 0-1-1 / 1-120 /0 76 1-779/	14.70
	CC.	Toluene		59-1	59-139%	< 21%	75 1759/	11%
	DD.	Chlorobenzene		60-1	60-133%	< 21%	75-120%	< 13%

LDC #: 18386B1 SDG #: See cover

### VALIDATION FINDINGS WORKSHEET Field Duplicates

Page:	<u></u>	/
Reviewer:_	مر	7
2nd reviewer:_	-1~	

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

YN N/A YN N/A Were field duplicate pairs identified in this SDG? Were target compounds detected in the field duplicate pairs?

	Concentrat	ion ()	Difference
Compound	3	4	( \ RPD
Dichloro methane	7.4	3,0	4.4 ( 5.4 )
Toluene	0.54	5,41	4.86 (£ 5.4)

	Concentration ()	
Compound		RPD

	Concentration ( )	
Compound		RPD

	Concentration (	
Compound		RPD

### BRC Tronox Parcel H Data Validation Reports LDC# 18386

Semivolatiles



### LDC Report# 18386A2

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

Project/Site Name:	BRC Tronox Parcel H		
Collection Date:	January 24, 2008		
LDC Report Date:	March 12, 2008		
Matrix:	Soil		
Parameters:	Semivolatiles		
Validation Level:	EPA Level III & IV		
Laboratory:	TestAmerica, Inc.		

Sample Delivery Group (SDG): F8A250221

### Sample Identification

TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HJ-04-0' TSB-HR-04-0'\*\* TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'\*\* TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'\*\* TSB-HJ-07-0'-FD TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-10' TSB-HR-08-0'MS TSB-HR-08-0'MSD

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 17 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.

### 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) with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
2/6/08	Pentachlorophenol	22.53156	All samples in SDG F8A250221	None	Р

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.

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.

### V. Blanks

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

Method Blank ID	Extraction Date	Compound TIC (RT in minutes)	Concentration	Associated Samples
8029394-Blank	1/29/08	Unknown aldol condensate (4.254)	8600 ug/Kg	All samples in SDG F8A250221

Sample concentrations were compared to concentrations detected in the method 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 method blanks with the following exceptions:

Sample	Compound TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
TSB-HJ-05-10'	Unknown aldol condensate (4.2676)	8400 ug/Kg	8400U ug/Kg
TSB-HJ-05-0'	Unknown aldol condensate (4.2526)	7600 ug/Kg	7600U ug/Kg
TSB-HR-04-10'	Unknown aldol condensate (4.2534)	7800 ug/Kg	7800U ug/Kg
TSB-HJ-04-0'	Unknown aldol condensate (4.2519)	9400 ug/Kg	9400U ug/Kg
TSB-HR-04-0'**	Unknown aldol condensate (4.2572)	7700 ug/Kg	7700U ug/Kg
TSB-HJ-04-10'	Unknown aldol condensate (4.2632)	8300 ug/Kg	8300U ug/Kg
TSB-HR-07-0'	Unknown aldol condensate (4.2583)	8000 ug/Kg	8000U ug/Kg
TSB-HR-07-10'**	Unknown aldol condensate (4.2472)	9300 ug/Kg	9300U ug/Kg
TSB-HR-06-0'	Unknown aldol condensate (4.2524)	8200 ug/Kg	8200U ug/Kg

Sample	Compound TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
TSB-HR-06-10'	Unknown aldol condensate (4.2627)	9400 ug/Kg	9400U ug/Kg
TSB-HJ-07-0'**	Unknown aldol condensate (4.2678)	9600 ug/Kg	9600U ug/Kg
TSB-HJ-07-0'-FD	Unknown aldol condensate (4.269)	8600 ug/Kg	8600U ug/Kg
TSB-HJ-07-10'	Unknown aldol condensate (4.2555)	9200 ug/Kg	9200U ug/Kg
TSB-HR-08-0'	Unknown aldol condensate (4.2578)	9300 ug/Kg	9300U ug/Kg
TSB-HR-08-10'	Unknown aldol condensate (4.2595)	9100 ug/Kg	9100U ug/Kg

No field blanks were identified in this SDG.

### VI. Surrogate Spikes

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

### VII. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Although the MS/MSD percent recoveries (%R) and relative percent differences (RPD) were not within QC limits for some compounds, the LCS 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. Percent recoveries (%R) were within QC limits.

### 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

Samples TSB-HJ-07-0'\*\* and TSB-HJ-07-0'-FD were identified as field duplicates. No semivolatiles were detected in any of the samples.

### BRC Tronox Parcel H Semivolatiles - Data Qualification Summary - SDG F8A250221

SDG	Sample	Compound	Flag	A or P	Reason
F8A250221	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HR-04-0' TSB-HR-04-0'** TSB-HR-07-0' TSB-HR-07-10'** TSB-HR-06-10' TSB-HR-06-10' TSB-HJ-07-0'** TSB-HJ-07-0'-FD TSB-HJ-07-10' TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-10'	Pentachlorophenol	None	Ρ	Continuing calibration (CCC %D)

### BRC Tronox Parcel H

### Semivolatiles - Laboratory Blank Data Qualification Summary - SDG F8A250221

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P
F8A250221	TSB-HJ-05-10'	Unknown aldol condensate (4.2676)	8400U ug/Kg	A
F8A250221	TSB-HJ-05-0'	Unknown aldol condensate (4.2526)	7600U ug/Kg	A
F8A250221	TSB-HR-04-10'	Unknown aldol condensate (4.2534)	7800U ug/Kg	A
F8A250221	TSB-HJ-04-0'	Unknown aldol condensate (4.2519)	9400U ug/Kg	A
F8A250221	TSB-HR-04-0'**	Unknown aldol condensate (4.2572)	7700U ug/Kg	A
F8A250221	TSB-HJ-04-10'	Unknown aldol condensate (4.2632)	8300U ug/Kg	A
F8A250221	TSB-HR-07-0'	Unknown aldol condensate (4.2583)	8000U ug/Kg	A
F8A250221	TSB-HR-07-10'**	Unknown aldol condensate (4.2472)	9300U ug/Kg	A
F8A250221	TSB-HR-06-0'	Unknown aldol condensate (4.2524)	8200U ug/Kg	A
F8A250221	TSB-HR-06-10'	Unknown aldol condensate (4.2627)	9400U ug/Kg	A
F8A250221	TSB-HJ-07-0'**	Unknown aldol condensate (4.2678)	9600U ug/Kg	A

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P
F8A250221	TSB-HJ-07-0'-FD	Unknown aldol condensate (4.269)	8600U ug/Kg	A
F8A250221	TSB-HJ-07-10'	Unknown aldol condensate (4.2555)	9200U ug/Kg	A
F8A250221	TSB-HR-08-0'	Unknown aldol condensate (4.2578)	9300U ug/Kg	A
F8A250221	TSB-HR-08-10'	Unknown aldol condensate (4.2595)	9100U ug/Kg	A

### BRC Tronox Parcel H Semivolatiles - Field Blank Data Qualification Summary - SDG F8A250221

No Sample Data Qualified in this SDG

VALIDATION COMPL	<b>_ETENESS WORKSHEET</b>

LDC #: <u>18386A2</u> SDG #: <u>F8A250221</u> Laboratory: <u>Test America</u>

Level III/IV

3/11/08 Date: Page: **Reviewer**: 2nd Reviewer

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

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

	Validation Area		Comments
١.	Technical holding times	А	Sampling dates:
П.	GC/MS Instrument performance check	Δ	1/ 1/
111.	Initial calibration	Δ	% PSD, 12 20.990
IV.	Continuing calibration/ICV	SW	ICIENS
V.	Blanks	SW	
VI.	Surrogate spikes	Δ	
VII.	Matrix spike/Matrix spike duplicates	لىكى	
VIII.	Laboratory control samples	A	LCS
IX.	Regional Quality Assurance and Quality Control	N	-
Х.	Internal standards	$\wedge$	
XI.	Target compound identification	< <u>`</u>	Not reviewed for Level III validation.
XII.	Compound quantitation/CRQLs	$\wedge$	Not reviewed for Level III validation.
XIII.	Tentatively identified compounds (TICs)	Δ	Not reviewed for Level III validation.
XIV.	System performance	Λ	Not reviewed for Level III validation.
XV.	Overall assessment of data	Ā	
XVI.	Field duplicates	NP	p = 11 + 12
XVII.	Field blanks	λ	

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-HJ-05-10'	11	TSB-HJ-07-0'**	<b>†</b> 21	8029394-BIK	31	
2	TSB-HJ-05-0'	12	TSB-HJ-07-0'-FD	22		32	
3	TSB-HR-04-10'	13	TSB-HJ-07-10'	23		33	
4	TSB-HJ-04-0'	14	TSB-HR-08-0'	24		34	
5	TSB-HR-04-0'**	15	TSB-HR-08-10'	25		35	
6	TSB-HJ-04-10'	16	TSB-HR-08-0'MS	26		36	
7	TSB-HR-07-0'	17	TSB-HR-08-0'MSD	27		37	
8	TSB-HR-07-10'**	18		28		38	
9	TSB-HR-06-0'	19		29		39	
10	TSB-HR-06-10'	20		30		40	

### VALIDATION FINDINGS CHECKLIST

Page: / of 2 Reviewer: <u>F7</u> 2nd Reviewer: <u>/</u>

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

Validation Area	Yes	No	NA	Findings/Comments
All technical holding times were met.				
Cooler temperature criteria was met.		1000-1000 Mer 184 1	Maina	
1) Several strategier conferences er cat				
Were the DFTPP performance results reviewed and found to be within the specified criteria?				
Were all samples analyzed within the 12 hour clock criteria?		Children V agos a		
Did the laboratory perform a 5 point calibration prior to sample analysis?	$\leq$	<u> </u>		
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?		<b></b>		
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?	~			
in Commune Editedica.	inner van seine			
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?	/			
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?	/			
Were all percent differences (%D) $\leq$ 25% and relative response factors (RRF) $\geq$ 0.05?		•		
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.	/			
MUSER REPORT KE				
Were all surrogate %R within QC limits?				
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?				
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?		21 2 20 20 20 20 20 20 20 20 20 20 20 20 2		
M. Manu-Schelmesenki-duchenes				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			
Was a MS/MSD analyzed every 20 samples of each matrix?				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				New York David Street and S
Was an LCS analyzed for this SDG?	1			

LDC #: 18386A2 SDG #: pu coner

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?		ł	•	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		-	ł	
Response Shall (Assumed ve and Compared and Compared and Compared and Compared and Compared and Compared and Co				
Were performance evaluation (PE) samples performed?			-	- · · ·
Were the performance evaluation (PE) samples within the acceptance limits?			/	-
X INCREASES				
Were internal standard area counts within -50% or +100% of the associated calibration standard?	/			
Were retention times within + 30 seconds from the associated calibration standard?		1		· · · · · · · · · · · · · · · · · · ·
ext Frite compositiou dentine iron				
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?				
Musicance nemificave toles				
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?				
All Tenetive Prote militari espinotives Alfoes)				
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?				
Were relative intensities of the major ions within $\pm$ 20% between the sample and the reference spectra?			-	· ·
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?				
System performance was found to be acceptable.				
System performance was found to be acceptable.				
			a a conservation de la conservation La conservation de la conservation d	
Overall assessment of data was found to be acceptable.				
Field duplicate pairs were identified in this SDG.	-	-		
Target compounds were detected in the field duplicates.	Ì	1	-	
AMD PREMIMENTS				
Field blanks were identified in this SDG.				
Farget compounds were detected in the field blanks.			F	

VALIDATION FINDINGS WORKSHEET

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

P. tast/2-finoreentoxymentane         E.E. 2.6.0.Introduenee         T.T. Pentachiorophenoi*           Interpreting         Q. 2.4.Dicknorphenoi*         F.F. 3.Mitroaniline         UU         Phenanittrene           Annol         R. 1.3.4.Trichlorobenzene         GG. Acenaphthene**         UU         Prenanittrene           Annol         R. 1.3.4.Trichlorobenzene         GG. Acenaphthene**         WY. Anthracene         WY. Anthracene           Annol         R. 1.3.4.Trichlorobenzene         GG. Acenaphthene**         WY. Fluoranittrene         MY. Chinoranittrene           Annol         R. 1.3.4.Trichlorobenzene         GG. Acenaphthene**         WY. Fluoranittrene         MY. Eluoranittrene           Annol         U. Hexachlorobuladiene**         IL. Altrophenoi**         MY. 2.4.Dintrophenoi**         MY. Eluoranittene**           Annol         V. 4.Chlorobuladiene**         JJ. Dibenzofura         YY. Fluoranithene***         MY. Eluoranithene***           Annol         V. 4.Chlorobrobuladiene**         IL. Diethylphthalate         ZZ. Pyrene         ZZ. Pyrene           Annol         V. 4.Chlorophenoi***         MM. 4.Chlorophenoi***         MM. 4.Chlorophenoi****         ZZ. Pyrene           Annol         V. 4.Chlorophenoi************************************					
Q. 2.4-Dichlorophenol*Fr. 3-NtroantifieeUU. PhenantifieneeR. 1.2.4-TrichlorobenzeneeGG. Acenaphthene*WV. AntirazeneS. NaphthaleneHH. 2.4-Dinitrophenol*WW. CarbazoleJ. S. NaphthaleneHH. 2.4-Dinitrophenol*WW. CarbazoleU. Hexachlorobutadiene*JJ. DibenzofuranYY. Fluoranthene*U. Hexachlorobutadiene*JJ. DibenzofuranYY. Fluoranthene*Name)U. Hexachlorobutadiene*JJ. DibenzofuranYY. Fluoranthene*Name)W. 2-MethylmaphthaleneLL. DiethylphthalateZ. PyreneAmeditymaphthaleneLL. DiethylphthalateBBB. 3.3-DichlorobenzidineAmeditymaphthaleneNM. 4-Chlorophenyl-thenyl etherBBB. 3.3-DichlorobenzidineAmeditymaphthaleneNM. 4-Chlorophenyl-thenyl etherBBB. 3.3-DichlorobenzidineAmeditymaphthaleneNM. 4-Chlorophenyl-thenyl etherBBB. 3.3-DichlorobenzidineAmeditymaphthaleneNM. 4-Chlorophenyl-thenyl etherDD. ChryseneAmeditymaphthaleneOO. 4-NitroantlineDD. ChryseneAmeditymaphthaleneOO. 4-NitroantlineDD. ChryseneBB. 2-NitroantlineOO. 4-NitroantlineCC. Dimethylphthalate*BB. 2-NitroantlineRR. 4-Bronophenyl-thenetCG. Benzolphthalate*DD. AcenaphthyleneS. HexachlorobenzeneDD. ChryseneDD. AcenaphthyleneBB. 2-NitroantlineCG. Benzolphthalate*DD. AcenaphthyleneS. 4-Bronophenyl-thereCG. Benzolphthalate*DD. AcenaphthyleneBB. 2-NitroantlineCG. Benzolphthalate*DD. Acenaphthylene	A. Pnenol	P. Bis(2-chioroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenoi**	ill. Benzo(a)pyrene**
R. 1.2.4.Trichlorobenzene         GG. Acenaphthene**         VV. Anthracene           S. Naphthalene         HH. 2.4.Dinitrophenol*         WV. Carbazole           J. Uberachlorobutadiene**         H. 4.Nitrophenol*         WV. Carbazole           U. Hexachlorobutadiene**         J.J. Dibenzofuran         XX. Di-n-butyphthalate           U. Hexachlorobutadiene**         J.J. Dibenzofuran         XY. Fluoranthene**           V. 4.Chloro-3-methyphenol**         KK. 2.4.Dinitrotoluene         ZP. Pyrene           V. 4.Chloro-3-methyphenol**         KK. 2.4.Dinitrotoluene         ZP. Pyrene           V. 4.Chloro-3-methyphenol**         KK. 2.4.Dinitrotoluene         ZP. Pyrene           V. 4.Chloro-3-methyphenol**         IL. Dietryphthalate         AA. Butylhenzyphthalate           Pine*         V. 2.46-Trichlorophenol**         IN. Fluoraphtene         ZP. Pyrene           Mine*         Y. 2.46-Trichlorophenol**         IN. Fluoraphtene         EB. 3.3 'Dichlorobenzidine           Mine*         Y. 2.46-Trichlorophenol***         IN. Fluoraphtene         ED. Chrysene           Mine*         Y. 2.46-Trichlorophenol***         IN. Fluoraphtene         ED. Chrysene           Mine*         Y. 2.46-Trichlorophenol***         IN. Fluoraphtene         ED. Chrysene           Mine*         Y. 2.46-Trichlorophenol***         IN. Fluora	B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
S. NaphthaleneHH. 2.4-Dinitrophenol*WW. Carbazole1. 4-ChloroanilineH. 4Nitrophenol*WW. Carbazole1. 4-ChloroanilineJ. DibenzofuranXX. Din-butylphthalateU. Hexachlorobutadlene*J. DibenzofuranYY. Fluoranthene*U. Hexachlorobutadlene*J. DibenzofuranYY. Fluoranthene*U. Hexachlorobutadlene*J. DibenzofuranYY. Fluoranthene*V. 4-Chloro-3-methylphenol**KK. 2.4-DinitrotolueneZZ. PyreneV. 4-Chloro-3-methylphenol**KY. 2.4-DinitrotolueneZZ. PyreneN. 2-Methylphenol**M. 4-Chlorophenyl etherBB. 3.3-DichlorobenzidineNine*Y. 2.4.6-Trichlorophenol**NN. FluoreneCC. Benzola)anthraceneNine*Y. 2.4.6-Trichlorophenol**NN. FluoreneDD. ChryseneA. 2-Chlorophenol**NN. FluoreneDD. ChryseneDD. ChryseneA. 2-Chlorophenol**DO. 4-NitroadilineDD. ChryseneB. 2-NitroanilineDD. ActionalineCC. Benzola)anthraceneB. 2-NitroanilineCC. DimethylphenolEEE. Bis (2-ethylheade***********************************	C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	VV. Anthracene	KKK. Dibenz(a,h)anthracene
T. 4-Chloroanilne         II. 4-Nitrophenol*         X.X. Di-n-buty/phthalate           U. Hexachlorobutadiene**         JJ. Diberzofuran         YY. Fluoranthene**           U. Hexachlorobutadiene**         JJ. Diberzofuran         YY. Fluoranthene**           V. 4-Chloro-3-methylphenol**         KX. 2,4-Dintrotoluene         ZZ. Pyrene           opane)         W. 2-Methylnaphthalene         LL. Diethylphthalate         AA. Butylberzylphthalate           opane)         W. 2-Methylnaphthalene         LL. Diethylphthalate         AA. Butylberzylphthalate           opane)         W. 2-Methylnaphthalene         LL. Diethylphthalate         AA. Butylberzylphthalate           opane)         W. 2-Methylnaphthalene         NM. 4-Chlorophenyl-phenyl ether         BBB. 3,3'-Dichlorobenzidine           mine*         Y. 2.4.6-Trichlorophenol**         NM. Fluorene         CC. Benzo(a)anthracene           Mine*         Y. 2.4.6-Trichlorophenol**         NM. Fluorene         CC. Benzo(a)anthracene           Mine*         Y. 2.4.6-Trichlorophenol**         OO. 4-Nitroaniline         DD. Chrysene           AA. 2-Chloronaphthalene         PP. 4.6-Dinitro-2-methylphenol         EE. Bis/2-ethylhexylphthalate**           AA. 2-Chloronaphthalene         PP. 4.6-Dinitro-2-methylphenol         EE. Bis/2-ethylphthalate**           BB. 2-Nitroaniline         PP. 4.6-Dinitro-2-meth	D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
U. Hexachlorobutadiene**JJ. DibenzofuranYY. Fluoranthene**U. Hexachlorobutadiene*JJ. DibenzofuranYY. Fluoranthene**V. 4-Chloro-3-methylphenol**KK. 2,4-DinitrotolueneZZ. PyreneW. 2-MethylnaphthaleneLL. DiethylphthalateAA. ButylbenzylphthalateW. 2-Methylnaphthalene*LL. DiethylphthalateAA. ButylbenzylphthalateY. 2,4,6-Trichlorophenol**MM. 4-Chlorophenyl-phenyl etherBBB. 3,3-DichlorobenzidineY. 2,4,6-Trichlorophenol**NN. FluoreneCCC. Benzo(a)anthraceneY. 2,4,6-Trichlorophenol**OO. 4-NitroanilineDDD. ChryseneY. 2,4,6-Trichlorophenol**OO. 4-NitroanilineDDD. ChryseneY. 2,4,6-Trichlorophenol**BBB. 3,3-DichlorobenzidineBBB. 3,3-DichlorobenzidineY. 2,4,6-Trichlorophenol**NN. FluoreneBCC. Benzo(a)anthraceneY. 2,4,6-Trichlorophenol**DO. 4-NitroanilineDDD. ChryseneY. 2,4,6-Trichlorophenol**BBB. 2,2-NitroanilineDD. ChryseneY. 2,4,6-Trichlorophenol**BBB. 2,2-NitroanilineDD. ChryseneY. 2,4,6-Trichlorophenol**BBB. 2,NitroanilineDD. Acenaphthalate***********************************	E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenol⁺	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
V. 4-Chloro-3-methylphenol**KK. 2,4-DinitrotolueneZ. PyreneW. 2-MethylnaphthaleneL.L. DiethylphthalateAAA. ButylbenzylphthalateW. 2-Methylnaphthalene*L.L. DiethylphthalateAAA. ButylbenzylphthalateX. Hexachlorocyclopentadiene*MM. 4-Chlorophenyl-phenyl etherBBB. 3,3'-DichlorobenzidineY. 2,4,6-Trichlorophenol**NN. FluoreneCCC. Benzo(a)anthraceneY. 2,4,6-Trichlorophenol**NN. FluoreneCCC. Benzo(a)anthraceneA. 2-Chlorophenol**OO. 4-NitroanilneDD. ChryseneA. 2-ChloronaphthalenePP. 4,6-Dinitro-2-methylphenolEEE. Bis(2-ethylhexyl)phthalateBB. 2-NitroanilneQQ. N-Nitrosodiphenylamine (1)**FFF. Di-n-octylphthalate*BB. 2-NitroanilneCC. DimethylphthalateGGG. Benzo(b)fluorantheneDD. AcenaphttyleneSS. HexachlorobenzeneHHH. Benzo(h)fluoranthene	F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
W. 2-MethylnaphthaleneLL. DiethylphthalateAdA. ButylbenzylphthalateX. Hexachlorocyclopentadlene*MM. 4-Chlorophenyl-phenyl etherBBB. 3,3*DichlorobenzidineY. 2,4,6-Trichlorophenol**NN. FluoreneCCC. Ben2o(a)anthraceneY. 2,4,6-Trichlorophenol**NN. FluoreneDD. ChryseneZ. 2,4,5-Trichlorophenol*OO. 4-NitroanilineDD. ChryseneAn 2-ChloronaphthalenePP. 4,6-Dinitro-2-methylphenolEEE. Bis(2-ethylhexyl)phthalateBB. 2-NitroanilineQQ. N-Nitrosodiphenylamine (1)**FFF. Di-n-octylphthalate*DD. AcenaphthyleneRR. 4-Bromophenyl-phenyletherGGG. Benzo(b)fluorantheneDD. AcenaphthyleneSS. HexachlorobenzeneHHL. Benzo(k)fluoranthene	G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	000. N-Nitrosodimethylamine
X. Hexachlorocyclopentadiene*MM. 4-Chlorophenyl-phenyl etherBBB. 3,3'-DichlorobenzidineY. 2,4,6-Trichlorophenol**NN. FluoreneCGC. Benzo(a)anthraceneY. 2,4,5-TrichlorophenolOO. 4-NitroanilineDDD. ChryseneA. 2-ChloronaphthalenePP. 4,6-Dinitro-2-methylphenolEEE. Bis(2-ethylhexyl)phthalateBB. 2-NitroanilineQQ. N-Nitrosodiphenylamine (1)**FFF. Di-n-octylphthalate**DD. AcenaphthyleneSS. HexachlorobenzeneHHH. Benzo(k)fluorantheneDD. AcenaphthyleneSS. HexachlorobenzeneHHH. Benzo(k)fluoranthene	H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzolc Acid
Y.2,4,6-Trichlorophenol**NN. FluoreneCCC. Benzo(a)anthraceneZ.2,4,5-TrichlorophenolOO. 4-NitroanilineDDD. ChryseneA. 2-ChloronaphthalenePP. 4,6-Dinitro-2-methylphenolEEE. Bis(2-ethylhexyl)phthalateBB. 2-NitroanilineQQ. N-Nitrosodiphenylamine (1)**FFF. Di-n-octylphthalate**CC. DimethylphthalateRR. 4-Bromophenyl-phenyletherGGG. Benzo(b)fluorantheneDD. AcenaphthyleneSS. HexachlorobenzeneHHH. Benzo(k)fluoranthene	I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
haneZ. 2,4,5-TrichlorophenolOO. 4-NitroanilineDDD. ChryseneA. 2-ChloronaphthalenePP. 4,6-Dinitro-2-methylphenolEEE. Bis(2-ethylhexyl)phthalateB. 2-NitroanilineQQ. N-Nitrosodiphenylamine (1)**FFF. Di-n-octylphthalate****CC. DimethylphthalateRR. 4-Bromophenyl-phenyletherGG. Benzo(b)fluoranthenehenolDD. AcenaphthyleneSS. HexachlorobenzeneHHH. Benzo(k)fluoranthene	J. N-Nitroso-di-n-propylamine⁴	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
AA. 2-Chloronaphthalene     PP. 4,6-Dinitro-2-methylphenol     EEE. Bis(2-ethylhexyl)phthalate       BB. 2-Nitroaniline     QQ. N-Nitrosodiphenylamine (1)**     FFF. Di-n-octylphthalate**       **     CC. Dimethylphthalate     GG. Benzo(b)fluoranthene       **     DD. Acenaphthylene     SS. Hexachlorobenzene     HHH. Benzo(k)fluoranthene	K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
BB. 2-Nitroaniline     QQ. N-Nitrosodiphenylamine (1)**     FFF. Di-n-octylphthalate**       CC. Dimethylphthalate     RR. 4-Bromophenyl-phenylether     GGG. Benzo(b)fluoranthene       DD. Acenaphthylene     SS. Hexachlorobenzene     HHH. Benzo(k)fluoranthene	L. Nitrobenzene	AA. 2-Chioronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	тп.
CC. Dimethylphthalate     RR. 4-Bromophenyl-phenylether     GGG. Benzo(b)fluoranthene       DD. Acenaphthylene     SS. Hexachlorobenzene     HHH. Benzo(k)fluoranthene	M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	UUU
DD. Acenaphthylene SS. Hexachlorobenzene HHH. Benzo(k)fluoranthene	N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	vvv.
	O. 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.

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### VALIDATION FINDINGS WORKSHEET Continuing Calibration

Page: 7\_of\_ Reviewer:

Rease see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". <u>N N/A</u> Was a continuing calibration standard analyzed at least once every 12 hours of sample analysis for each instrument? METHODF GC/MS BNA (EPA SW 846 Method 8270C)

Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's ? Were all %D and RRFs within the validation criteria of ≤25 %D and ≥0.05 RRF ?	Finding %D Finding RRF (Limit: ≤25.0%) (Limit: ≥0.05)	TT (ccc) 2253156 A114 B1K W														
Vere percent difference Vere all %D and RRFs	Standard ID	ecv														
N N/A N N/A N	Date	2/6/08	24:00					 								
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# VALIDATION FINDINGS WORKSHEET <u>Blanks</u>

Page: 2nd Reviewer: Reviewer:

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

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

Was a method blank analyzed for each matrix? A/N

2

Was a method blank analyzed for each concentration preparation level? N N/A

Was a method blank associated with every sample? Y N N/A

2

₩ A

4.25.24 8200 σ 4.2472 9300 8 4. 2583 80% ٢ 4.2632.4 4300 e Sample Identification 4,2532) 120011 0.52.1 9400 -Associated Samples: 1.1 Ň 3 1800 1 4. 2526 2 0000 8400 H. 2 676 9 8029394 - Blank Blank ID <del>(اعد ا</del> ا shoo contentration Conc. units: ug / Ka Compound NUMANAN AIdol

Blank extraction date: <u>\\xe\ob</u> Blank analysis date: <u>2 \L \ob</u> Conc. units: مم الك

 $\exists \forall$ Associated Samples:

-				 	 	
					1	
5	1	9100 4.2595	•			•
Sample Identification	+1	1300 1	×			
2	13	9600 8600 9200 9200 9200 9200 - 14.269 94.269 94.269	/			
	12	8600 (4.269)	/ .	1	·	
		9600 (4.267B)				
	10	9400				
Cl Juela	- Horbrog	Seed 14.254	-			
Compand			•			

Common contaminants such as the phthalates and TICs noted above that were detected in samples within ten times the associated method blank concentration were qualified as not detected, "U". Other contaminants CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT: within five times the method blank concentration were also qualified as not detected, "U".

BNA\_blank.wpd

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# Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

of Page: 2nd Reviewer: Reviewer:

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

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". <u>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. <u>Y N N/A</u> Was a MS/MSD analyzed every 20 samples of each matrix?

$\star$ Date         MSMSD ID         Compound         RM Mission         Ren (Limits)         Ren (Limits)         Ascotated Samples         Qualifications $1$ $1 \leftarrow 1 \top$ $H \downarrow$ $+$ $(1 \leftarrow 3)$ $2$ $(1 \leftarrow 3)$										
HH $+$ $(P-q1)$ $2$ $(1-q-1)$ $2$ $(1-q-1)$ $2$ $(1-q-1)$ $2$ $(1-q-1)$ $2$ $(1-q-1)$ $2$ $(1-q-1)$	$\leftarrow$		Compound	MS %R (Lin	lits)	MSD %R (Limits)	RPD (Lmits)	Associated Samples	Qualifications	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		16 1 17	HH		116-	~	11	14	Sal	S
			PP	)	(66-0	)	( )	7	11" <b>P</b>	
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				)	(	( )	- - ,	1		
				•	^	( )	· ·			
				~	<b>^</b>	( )	( )			
				-	<b>^</b>	( )	( )			
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	_			•	<b>^</b>	( )	( )			
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				~	(	( )	( )			
				)	(	( )	( )			
				-	(	( )	( )			
				~	(	( )	(			

	Compound	QC Limits (Soll)	RPD (Soil)	QC Limits (Water)	RPD (Water)		Compound	QC Limits (Soil)	RPD (Soll)	QC Limits (Water)	RPD (Water)
Ä	Phenol	26-90%	35%	12-110%	<u>&lt;</u> 42%	IJ IJ IJ	Acenaphthene	31-137%	<u>&lt;</u> 19%	46-118%	<u>&lt;</u> 31%
Ċ	2-Chiorophenol	25-102%	₹ 50%	27-123%	< 40%	=	4-Nitrophenol	11-114%	< 50%	10-80%	< 50%
ш	1,4-Dichlorobenzene	28-104%	≤ 27%	36-97%	≤ 28%	¥	2,4-Dinitratoluene	28-89%	< 47%	24-96%	<ul><li>38%</li></ul>
-;	N-Nitroso-di-n-propylamine	41-126%	<u>&lt;</u> 38%	41-116%	≤ 38%	Ę	Pentachlorophenol	17-109%	<u>&lt;</u> 47%	9-103%	1 50%
œ.	1,2,4-Trichlorobenzene	38-107%	~52%	39-98%	≤ 28%	Ŋ	Pyrene	35-142%	< 36%	26-127%	≤ 31%
۲.	4-Chloro-3-methylphenof	26-103%	<u>&lt;</u> 33%	23-97%	< 42%						

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## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

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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:

RRF = (A<sub>x</sub>)(C<sub>s</sub>)/(A<sub>s</sub>)(C<sub>x</sub>) average RRF = sum of the RRFs/number of standards %RSD = 100 \* (S/X)

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

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

I standard	
C <sub>k</sub> = Concentration of internal standard	RFs
ncentratio	X = Mean of the RRFs
ت ت"	X = Mea

#         Standard ID         Compound (Reference Internel Standard)         RPF         Resolutation         Reported         Resolutation         Reported         Resolutation           1         1         LOLL         1         Varage RFF         Average RFF         Varage         Varage RFF         Varage RF										
Calibration Data         Compound (Raference Internal Standard) Data         Reportant         Reportant<			•							
Standard ID         Calibration         Campound (Reference Internal Standard)         RRF         RRF         Average RRF         Averaft Average RRF         Averaft Average RRF					Reported	Recalcutated	Reported	Recalculated	Reported	Recalculated
$ \frac{1 (\Delta L)}{1 (\Delta L)} \frac{1}{2} (\Delta L) \frac{1}{2}$	#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	RRF ( らつ std)	RRF ( SOstd)	Average RRF (initial)	Average RRF (initial)	%RSD	%RSD
Nephthelee (2nd internal standard)         1. $1.0271$ 1. $1.0271$ 1. $0757$ 10.33P072           Flucene (3nd internal standard)         1. $3.6773$ 1. $3.4737$ 1. $3.4737$ 1. $3.4737$ 1. $3.37972$ Pentachicrophenol (4th internal standard)         0. $1.3677$ 1. $3.4737$ 1. $3.4737$ 1. $4.74450$ Pentachicrophenol (4th internal standard)         0. $1.3607$ $0.15020$ $0.15020$ $0.15020$ $1.94745$ $1.0071$ Pentachicrophenol (4th internal standard)         0. $1.2057$ $1.21873$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ Pentachicrophenol (4th internal standard) $1.121571$ $1.21187$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ Pentachicrophenol (4th internal standard)         Pentachicrophenol (4th internal standard) $1.121877$ $1.121877$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ $1.16779$ $1.16779$	-	ICAL	go lor/	Phenol (1st internal standard)	2. Levey	2.66254	2.69741	1419.2	6. 25.349	6.25249
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$			3	Naphthalene (2nd internal standard)	1.10277	1-10277	1. 09527	1. 097277	1039092	26062.01
Pentachlorophenol (4th internal standard)         e. \\S_O_V         c.\\S_O_V         c.\\S_O^O_V         c.\\S_O^O_V         c.\\S_O^O_V         c.\\S_O^O_V         c.\\S_O^O_V         r.\\S_A^A_A           Bic(2-ethylhexyl)phthalate (5th internal standard)         C.\&\S_T_167         c.\\S_O^O_V         c.\\S_O^O_V         c.\\S_O^O_V         c.\\S_O^O_V         r.\\S_A^A_A         r.\S_A^A_A         r.\S_				Fluorene (3rd internal standard)	1.36978	1.36978	81846.1	1.34876	U22422-41	14.54450
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$				Pentachlorophenol (4th internal standard)	0. 1Sb02	0.1Stool	0.15050	Nas1.0	2PT 24795	11. 84793
$\left( \begin{array}{c c c c c c c c c c c c c c c c c c c $				Bis(2-ethylhexyl)phthalate (5th internal standard)	0 sorted	0.40769	0 81480	95418-0	7.069797	7.069.79
Phenol (1st internal standard)         Phenol (1st internal standard)           Nopthhalene (2nd internal standard)         Nopthhalene (2nd internal standard)           Fluorene (3rd internal standard)         Penol (4th internal standard)           Penol (4th internal standard)         Penol (4th internal standard)           Bis(2-ethytheyt)hext)phthalate (5th internal standard)         Penol (4th internal standard)           Penol (1st internal standard)         Penol (1st internal standard)				Benzo(a)pyrene (6th internal standard)	Lse12.1	1.21257	1.18939		3.60258	3.60050
	7			Phenol (1st internal standard)						
				Naphthalene (2nd internal standard)						
				Fluorene (3rd internal standard)						
				Pentachiorophenol (4th internal standard)						
				Bis(2-ethylhexyl)phthalate (5th internal standard)						
				Benzo(a)pyrene (6th internal standard)						
Naphthalene (2nd internal standard)         Naphthalenene (2nd internal standard)         Naphtha	m			Phenol (1st internal standard)						
Fluorene (3rd internal standard)     Fluorene (3rd internal standard)       Pentachlorophenol (4th internal standard)     Pentachlorophenol (4th internal standard)       Bis(2-ethylhexyl)phthalate (5th internal standard)     Pentachlorophenol (4th internal standard)       Benzo(a)pyrene (6th internal standard)     Pentachlorophenol (4th internal standard)				Naphthalene (2nd internal standard)						
Pertachlorophenol (4th internal standard)     Pentachlorophenol (4th internal standard)       Bis(2-ethylhexyl)phthalate (5th internal standard)     Bis(2-ethylhexyl)phthalate (5th internal standard)				Fluorene (3rd internal standard)						
Bis(2-ethylhexyl)phthelate (5th internal standard) Benzo(a)pyrene (6th internal standard)				Pentachlorophenol (4th internal standard)						
Benzo(a) pyrene (6th internal standard)				Bis(2-ethylhexyl)phthalate (5th internal standard)						
				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 results.

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

1 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:

% 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, Where:

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

aStandard IDCompound (Reference Internal Standard)Average RKFRRFRRFRRF $w$ $w$ 1 $k$ $k$ $k$ $k$ $k$ $k$ $k$ $k$ $w$ $w$ $w$ $w$ 1 $k$ $w$ $w$ $w$ 1 $k$ $w$ $w$ $w$ 1 $k$ <th></th> <th></th> <th></th> <th></th> <th></th> <th>Reported</th> <th>Recalculated</th> <th>Reported</th> <th>Recalculated</th>						Reported	Recalculated	Reported	Recalculated
$\chi c\Delta L t_3 SD$ $2/L \left  OD$ Pnenol (tar internal standard) $2. L c_3 t_1 t_1$ $2. L c_1 CBL$ $2. c_3 t_1 CBL$ $2. c_4 t_1 CBL$ $2. c_5 t_1 CBL$ $2. c_4 t_1 CBL$	#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q%	C1%
Nephthalene (2nd internal standard) $1.03327$ $1.03339$ $0.03399$ $0.42152$ $0$ Fluorene (3rd internal standard) $1.34879$ $1.24939$ $0.42152$ $0$ Pentachlorophenol (4th internal standard) $0.15950$ $0.18441$ $0.09441$ $2.2.53154$ $0$ Bis(2-sthylhosylphthalte (5th internal standard) $0.18450$ $0.18441$ $0.019441$ $2.2.53154$ $0.25866$ Pentachlorophenol (4th internal standard) $0.81450$ $0.19750$ $0.73590$ $2.3.54440$ Pentachlorophenol (2th internal standard) $1.184339$ $1.19732$ $1.19732$ $0.78502$ Pentachlorophenol (3th internal standard) $1.184359$ $1.19732$ $0.73590$ $2.3.54440$ Pentachlorophenol (3th internal standard) $1.184359$ $1.19732$ $0.79502$ $2.364490$ Pentachlorophenol (4th internal standard) $1.184359$ $1.19732$ $0.79502$ $2.354440$ Pentachlorophenol (4th internal standard) $1.184359$ $1.19732$ $1.19732$ $0.868233$ Pentachlorophenol (4th internal standard) $1.184359$ $1.19732$ $0.79530$ $2.354440$ Pentachlorophenol (4th internal standard) $1.184359$ $1.19732$ $0.79530$ $2.7547402$ Pentachlorophenol (4th internal standard) $1.184336$ $1.19732$ $0.79530$ $2.354440$ Pentachlorophenol (4th internal standard) $1.184356$ $1.19732$ $0.19732$ $0.868233$ Pentachlorophenol (4th internal standard) $1.184326$ $1.19732$ $0.197326$ $0.197326$ Pentach	-	KCAL4350		Phenol (1st internal standard)	2.6974)	2.64086	2.64086	2.09649	2.09649
Index<				Naphthalene (2nd internal standard)	1.09527	1. 09989	1-099,89	0.42132	26124.0
Pertectionspheric $0 \cdot 1SOSO$ $0 \cdot 1Sq41$ $0 \cdot 1G441$ $1 \cdot 1G941$ $2 \cdot 2 \cdot 53 \cdot 1S_{1}$ Bis(2-etty/hrex)(pythelate (5th internal standard) $0 \cdot 8 \cdot 14SO$ $0 \cdot 1G \cdot 23 \cdot 24 \cdot 4O$ $2 \cdot 34 \cdot 4O$ Bana(c)pueza (ani internal standard) $0 \cdot 8 \cdot 14SO$ $0 \cdot 1G \cdot 23 \cdot 24 \cdot 4O$ $2 \cdot 34 \cdot 4O$ Prenol (1st internal standard) $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ $2 \cdot 34 \cdot 4O$ Prenol (1st internal standard) $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ $2 \cdot 34 \cdot 4O$ Prenol (1st internal standard) $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ $2 \cdot 34 \cdot 4O$ Prenol (1st internal standard) $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ $2 \cdot 34 \cdot 4O$ Prenol (1st internal standard) $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ $2 \cdot 34 \cdot 4O$ Prenol (1st internal standard) $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ $2 \cdot 34 \cdot 4O$ Prenol (1st internal standard) $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ Prenol (1st internal standard) $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ Prenol (1st internal standard) $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ Prenol (1st internal standard) $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ Prenol (1st internal standard) $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ Prenol (1st internal standard) $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ Prenol (1st internal standard) $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ Prenol (1st internal standard) $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ $1 \cdot 1G \cdot 32$ Prenol (1st				Fluorene (3rd internal standard)	1. 348 76	62542.1	6254 2-1	0. 25866	0.25866
Bis(2-ethylhex/)phthate (5th internal standard) $\alpha$ St (45 $\omega$ $\alpha$ -7955 $\omega$ $2.3\omega$ 44 $\omega$ Image: Phenol (1st internal standard)Image: Phenol Phenol Phenol Phenol (1st internal standard)Image: Phenol				Pentachlorophenol (4th internal standard)	asas1.0	0.18441	0.1844)	75125-22	2253352
Image: line line line line line line line line				Bis(2-ethylhexyl)phthalate (5th internal standard)	0. 81450	0.79520	OESTELO	2.30440	2-3644
Phenol (1st internal standard)Phenol (1st internal standard)Naphthalene (2nd internal standard)Naphthalene (2nd internal standard)Fluorene (3nd internal standard)Pentachlorophenol (4th internal standard)Pentachlorophenol (4th internal standard)Pentachlorophenol (4th internal standard)Bis(2-ethylhexy/)phthalate (5th internal standard)Pentachlorophenol (4th internal standard)Phenol (1st internal standard)P				Benzo(a)pyrene (6th internal standard)	1.18939	25601.1	1-19972	0 86823	0-81-823
	~			Phenol (1st internal standard)					
				Naphthalene (2nd internal standard)					
				Fluorene (3rd internal standard)					
				Pentachlorophenol (4th internal standard)					
				Bis(2-ethylhexyl)phthalate (5th internal standard)					
				. Banzo(a)pyrene (6th internal standard)					
Naphthalene (2nd internal standard)         Naphthalene (2nd internal standard)         Naphthalene (3nd internal standard) <thnaphthalene (3nd="" internal="" standard)<="" th="">         Naphthalen</thnaphthalene>	m			Phenol (1st internal standard)					
Fluorene (3rd internal standard)         Fluorene (3rd internal standard)           Pentachlorophenol (4th internal standard)         Pentachlorophenol (4th internal standard)           Bis(2-ethylhexyl)phthalate (5th internal standard)         Pentachlorophenol (4th internal standard)           Bis(2-ethylhexyl)phthalate (5th internal standard)         Pentachlorophenol (4th internal standard)				Naphthalene (2nd internal standard)					
Pentachlorophenol (4th internal standard)     Pentachlorophenol (4th internal standard)       Bis(2-ethylhexyl)phthalate (5th internal standard)     Enzo(a)       Benzo(a)pyrene (6th internal standard)     Enzo(a)				Fluorene (3rd internal standard)					
Bis(2-ethylhexyl)phthalate (5th internal standard)     Bis(2-ethylhexyl)phthalate (5th internal standard)       Benzo(a)pyrene (6th internal standard)     Benzo(a)pyrene (6th internal standard)				Pentachlorophenol (4th internal standard)					
Benzo(a)pyrene (6th 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 #: 18386A2

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## 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: #5

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	50. J	36.4457	13	13	Ø
2-Fluorobiphenyl		37.8084	710	76	
Terphenyl-d14		465125	93	93	
Phenol-d5		54.5613	73	73	
2-Fluorophenol		52089)	67	67	
2.4,6-Tribromophenol		54.1507	72	72	
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 #: 18380 A2 SDG #: per court

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

Page: \_\_\_\_\_\_0f/\_ 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

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MS/MSD samples:

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

MSD = Matrix spike duplicate percent recovery

SC = Sample concentation

		ated									
SD	0	Recalculated	Sis	74	09	6.t	5	6.6			
USM/SM	RPD	Renorted	۲۰۶	4.8	6.0	6.1	13	6,6			
e Duplicate	ecoverv	Recalc	69	<u>6</u>	13	89	19	16	-		
Matrix Snike Duplicate	Percent Recovery	Renorted	69	19	51	١٣	19	<b>٦</b> ٢			
Spike	lecoverv	Recalc	12	8	17	13	9	<i>R</i>			
Matrix Spike	Percent Recovery	Reported	12	. 8	17	13	16	sv)			
Sample	Concentration	0 MSD	2400	oste	2552	2370	<b>[</b> 99	arn			
Spiked	Conce	WS	01556	2460	مالح	atse	581	02.82			
Sample	Concentration		β								
oike	Added War Ken	Msn	3470								
ŝ	Add (	D W	s syo						, 		·
	Compound		Phenol	N-Nitroso-di-n-propylamine	4-Chloro-3-methylphenol	Acenaphthene	Pentachlorophenol	Pyrene			

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.

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

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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

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

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

LCS/LCSD samples: なっょえろうオーレこう

	Sr	oike '	Spi	Spike		CS	1 CSD		I CS/I CSD	csD
Compound	Ad (Ve	Added ( Ug/Ker)	Concentration	tration	Percent Recovery	солегу	Percent Recovery	ecovery	RPD	0
			1 LS	) I CSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenol	3330	NA	2.300	A	69	69				
N-Nitroso-di-n-propylamine			2480		74	- <b>7</b> L	•			
4-Chloro-3-methylphenol			1520		76					
Acenaphthene			2410		22	12				
Pentachlorophenol			2690		81	81				
Pyrene		>	0292	<b>~</b>	6	01	NA NA			
	-									

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

## VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	
Reviewer:	[7]
2nd reviewer:	-h

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



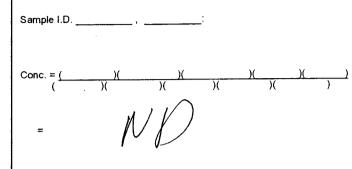
Were all reported results recalculated and verified for all level IV samples? Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

Example:

Conc	entratio	$n = (A_{\chi})(I_{\chi})(V_{\chi})(DF)(2.0) - (A_{\chi})(RRF)(V_{\chi})(V_{\chi})(\%S)$
A,	Ξ	Area of the characteristic ion (EICP) for the compound to be measured
$A_{is}$	=	Area of the characteristic ion (EICP) for the specific internal standard
l <sub>s</sub>	=	Amount of internal standard added in nanograms (ng)
V <sub>o</sub>	=	Volume or weight of sample extract in milliliters (ml) or grams (g).
$\vee_{i}$	=	Volume of extract injected in microliters (ul)
V,	=	Volume of the concentrated extract in microliters (ul)
Df	=	Dilution Factor.

%S = Percent solids, applicable to soil and solid matrices only.

2.0 = Factor of 2 to account for GPC cleanup



#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration (  )	Qualification
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	· · · · · · · · · · · · · · · · · · ·				
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## LDC Report# 18386B2

## Laboratory Data Consultants, Inc. Data Validation Report

<b>Project/Site</b>	Name:	BRC Tronox Parcel H

Collection Date: January 28, 2008

LDC Report Date: March 12, 2008

Matrix: Soil/Water

Parameters: Semivolatiles

Validation Level: EPA Level III

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8A290158

## Sample Identification

TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10' RINSATE-2 TSB-HR-05-10'MS TSB-HR-05-10'MSD

## Introduction

This data review covers 11 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 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.

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.

## 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) with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
2/8/08	Pentachlorophenol	22.52510	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HJ-08-0' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HR-05-10' RINSATE-2 TSB-HR-05-10'MS TSB-HR-05-10'MSD 8031299-Blk	None	Ρ
2/6/08	Pentachlorophenol	22.53156	8029233-Bik	None	Р

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
2/8/08 (KCAL4410)	N-Hydroxymethylphthalimide	74.84218	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HJ-08-0' TSB-HJ-08-10' TSB-HJ-08-10' TSB-HR-05-10' RINSATE-2 TSB-HR-05-10'MS TSB-HR-05-10'MSD 8031299-Blk	J+ (all 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.

## V. Blanks

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

Method Blank ID	Extraction Date	Compound TIC (RT in minutes)	Concentration	Associated Samples
8031299-Blank	1/31/08	Unknown (3.8408) Unknown aldol condensate (4.2522) Unknown aldol condensate (4.749)	1100 ug/Kg 20000 ug/Kg 320 ug/Kg	All soil samples in SDG F8A290158

Sample concentrations were compared to concentrations detected in the method 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 method blanks with the following exceptions:

Sample	Compound	Reported	Modified Final
	TIC (RT in minutes)	Concentration	Concentration
TSB-HJ-10-0'	Unknown aldol condensate (4.2487)	22000 ug/Kg	22000U ug/Kg
	Unknown aldol condensate (4.7401)	350 ug/Kg	350U ug/Kg
TSB-HJ-10-10'	Unknown aldol condensate (4.2597)	21000 ug/Kg	21000U ug/Kg
	Unknown aldol condensate (4.7565)	350 ug/Kg	350U ug/Kg
TSB-HR-06-0'	Unknown aldol condensate (4.2595)	23000 ug/Kg	23000U ug/Kg
	Unknown aldol condensate (4.751)	380 ug/Kg	380U ug/Kg
TSB-HR-06-0'-FD	Unknown aldol condensate (4.2594)	22000 ug/Kg	22000U ug/Kg
	Unknown aldol condensate (4.7509)	360 ug/Kg	360U ug/Kg
TSB-HR-06-10'	Unknown aldol condensate (4.2583)	23000 ug/Kg	23000U ug/Kg
	Unknown aldol condensate (4.7497)	370 ug/Kg	370U ug/Kg
TSB-HJ-08-0'	Unknown aldol condensate (4.2624)	23000 ug/Kg	23000U ug/Kg
	Unknown aldol condensate (4.7486)	380 ug/Kg	380U ug/Kg
TSB-HJ-08-10'	Unknown aldol condensate (4.2588)	23000 ug/Kg	23000U ug/Kg
	Unknown aldol condensate (4.7443)	380 ug/Kg	380U ug/Kg
TSB-HR-05-0'	Unknown aldol condensate (4.2682)	22000 ug/Kg	22000U ug/Kg
	Unknown aldol condensate (4.7597)	350 ug/Kg	350U ug/Kg
TSB-HR-05-10'	Unknown aldol condensate (4.247)	22000 ug/Kg	22000U ug/Kg
	Unknown aldol condensate (4.7438)	370 ug/Kg	370U ug/Kg

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

## VI. Surrogate Spikes

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

## VII. Matrix Spike/Matrix Spike Duplicates

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

## VIII. Laboratory Control Samples (LCS)

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

## IX. Regional Quality Assurance and Quality Control

Not applicable.

## X. Internal Standards

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

## **XI. Target Compound Identifications**

Raw data were not reviewed for this SDG.

## XII. Compound Quantitation and CRQLs

Raw data were not reviewed for this SDG.

## XIII. Tentatively Identified Compounds (TICs)

Raw data were not reviewed for this SDG.

## XIV. System Performance

Raw data were not reviewed for this SDG.

## XV. Overall Assessment of Data

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

## XVI. Field Duplicates

Samples TSB-HR-06-0' and TSB-HR-06-0'-FD were identified as field duplicates. No semivolatiles were detected in any of the samples.

## BRC Tronox Parcel H Semivolatiles - Data Qualification Summary - SDG F8A290158

SDG	Sample	Compound	Flag	A or P	Reason
F8A290158	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10' RINSATE-2	Pentachiorophenol	None	Ρ	Continuing calibration (CCC %D)
F8A290158 TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HR-05-10' TSB-HR-05-10' RINSATE-2		N-Hydroxymethylphthalimide	J+ (all detects)	A	Continuing calibration (%D)

## BRC Tronox Parcel H Semivolatiles - Laboratory Blank Data Qualification Summary - SDG F8A290158

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P
F8A290158	TSB-HJ-10-0'	Unknown aldol condensate (4.2487) Unknown aldol condensate (4.7401)	22000U ug/Kg 350U ug/Kg	A
F8A290158	TSB-HJ-10-10'	Unknown aldol condensate (4.2597) Unknown aldol condensate (4.7565)	21000U ug/Kg 350U ug/Kg	A
F8A290158	TSB-HR-06-0'	Unknown aldol condensate (4.2595) Unknown aldol condensate (4.751)	23000U ug/Kg 380U ug/Kg	A
F8A290158	TSB-HR-06-0'-FD	Unknown aldol condensate (4.2594) Unknown aldol condensate (4.7509)	22000U ug/Kg 360U ug/Kg	A
F8A290158	TSB-HR-06-10'	Unknown aldol condensate (4.2583) Unknown aldol condensate (4.7497)	23000U ug/Kg 370U ug/Kg	A
F8A290158	TSB-HJ-08-0'	Unknown aldol condensate (4.2624) Unknown aldol condensate (4.7486)	23000U ug/Kg 380U ug/Kg	A

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P
F8A290158	TSB-HJ-08-10'	Unknown aldol condensate (4.2588) Unknown aldol condensate (4.7443)	23000U ug/Kg 380U ug/Kg	A
F8A290158	TSB-HR-05-0'	Unknown aldol condensate (4.2682) Unknown aldol condensate (4.7597)	22000U ug/Kg 350U ug/Kg	A
F8A290158	TSB-HR-05-10'	Unknown aldol condensate (4.247) Unknown aldol condensate (4.7438)	22000U ug/Kg 370U ug/Kg	A

## BRC Tronox Parcel H Semivolatiles - Field Blank Data Qualification Summary - SDG F8A290158

No Sample Data Qualified in this SDG

## VALIDATION COMPLETENESS WORKSHEET Level III

SDG #: <u>F8A290158</u> Laboratory: <u>Test America</u>

LDC #: 18386B2

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## Date: <u>3/6/</u>08 Page: <u>/</u>of/\_ Reviewer: <u></u> 2nd Reviewer: <u></u>

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
Ι.	Technical holding times	Δ	Sampling dates: () 20 00
11.	GC/MS instrument performance check	۵	
III.	Initial calibration	Д	% RD '(2 IO.990
IV.	Continuing calibration/ICV	دىر	$ c  \in \mathcal{M}$
V.	Blanks	sW	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	STA/A	
VIII.	Laboratory control samples	SW	
IX.	Regional Quality Assurance and Quality Control	N	
Х.	Internal standards	Δ	
XI.	Target compound identification	N	
XII.	Compound quantitation/CRQLs	N	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	N	
XV.	Overall assessment of data	4	
XVI.	Field duplicates	ND	D= 314
XVII.	Field blanks	ND	REID

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

	GOIL + NO						
1	TSB-HJ-10-0'	11	TSB-HR-05-10'MS	21	8029233-Blank	31	
2	TSB-HJ-10-10'	12	TSB-HR-05-10'MSD	22	8031299-13 Jank	32	
3	TSB-HR-06-0'	13		23		33	
4	TSB-HR-06-0'-FD	14		24		34	
5	TSB-HR-06-10'	15		25		35	
6	TSB-HJ-08-0'	16		26		36	
7	TSB-HJ-08-10'	17		27		37	
8	TSB-HR-05-0'	18		28		38	
9	TSB-HR-05-10'	19		29		39	
10	RINSATE-2	20		30		40	

# VALIDATION FINDINGS WORKSHEET

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

A. Phenol**     P. Bis(2-ch       B. Bis (2-chloroethyl) ether     Q. 2,4-Dich       C. 2-Chlorophenol     R. 1,2,4-Tri	P. Bis(2-chloroethoxv)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenol**	lll. Benzo(a)pyrene**
	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	VV. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene S. Naphthalene		HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	roaniline	II. 4-Nitrophenol⁺	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene U. Hexachi	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol V. 4-Chloro	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	000. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane) W. 2-Methy	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol X. Hexachl	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine <sup>*</sup> Y. 2,4,6-Tri	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane Z. 2,4,5-Tri	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-Nitroaniline	oaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	UUU
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	VVV.
0. 2,4-Dimethylphenol DD. Acenal	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	www.

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

Page: Reviewer: 2nd Reviewer:	es Qualifications	/ oran		1 <sup>+</sup> /A dut		BIK was/p							
for each instrument? CCC's and SPCC's ?	Associated Samples	\$031299-B1K,	+ All samples			8029233 CB							
applicable questions are identified as "N/A".         least once every 12 hours of sample analysis for all CC a of ≤25 %D and ≥0.05 RRF ?	Finding RRF (Limit: >0.05)												
<b>Continuing Calit</b> applicable questions are identif least once every 12 hours of s se factors (RRF) within metho a of ≤25 %D and ≥0.05 RRF ?	Finding %D (Limit: ≤25.0%)	01525.22		214.84218		22.53150							
od 8270C) ions answered "N". Not a on standard analyzed at (%D) and relative respor thin the validation criteri	Compound	TT (cec)		N - (Hydroxymethy) phthalimide	-	TT (202)							
SDG #. Continuing Calibration METHOD: GC/MS BNA (EPA SW 846 Method 8270C) Blease see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". N/A Was a continuing calibration standard analyzed at least once every 12 hours of sample analysis for each instrument? V N N/A Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's ? V N N/A Were all %D and RRFs within the validation criteria of ≤25 %D and ≥0.05 RRF ?	Standard ID	KEAL 4400		KCAL 4410		kea Lysso							
SDG # Conserved to the second	Date	218 00	HZ:ST	2 8 03	B1:91	2/16/08							

CONCAL.2S

## VALIDATION FINDINGS WORKSHEET

LDC #: 18 380 82 are could SDG #:

## VALIDATION FINDINGS WORKSHEET **Blanks**

Page: 2nd Reviewer: Reviewer:

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

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

Was a method blank analyzed for each matrix? VN N/A

Was a method blank analyzed for each concentration preparation level? N N/A

Was a method blank associated with every sample? V N/A

<u>\/\/\/\/\/\</u> Was the blank contaminated? If yes, please see qualification below. Blank extraction date: ↓૱\\୭안 Blank analysis date: <u>2\08\0</u>®

<u>.</u>

Conc. units: walkay Blank ID Blank ID Blank ID Blank ID Compound Blank ID Blank ID 2021299- 1 2 Blank ID 1000 1000 1000 1000 1000 1000 1000 1	Blank ID 8031299- 1000 1000 1000 1000 1000 1000 1000 1	Blank ID 031299- 8100K - 3.8408) - 3.8408) - 3.8408) - 1.2487) - (4.7487) - (4.7491) (4.7401)	Associated Sample 2 Associated Sample 2 Associated Sample 2 Associated Sample 2 Associated Sample 2 Associated Sample		All xp 1 5 sample Identification 5 1, 23 coc 23 1, 23 coc 23 1, 23 coc 23 1, 23 coc 23 1, 23 coc 24 1, 24 coc 25 1, 24 coc 25 1, 25	6 1 1 1 1 1 8 6 9	7 7 280 (6+4-1) (6+4-1)	8 22200 (4.262) 750 750 (14.702)	9 (142.47) (1442.47) (1424)
			\ \						

Blank analysis date: Blank extraction date:

Associated Samples:

Conc. units:		Associated Samples:
	Blank ID	
Compound		

Common contaminants such as the phthalates and TICs noted above that were detected in samples within ten times the associated method blank concentration were qualified as not detected, "U". Other contaminants within five times the method blank concentration were also qualified as not detected, "U". CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:

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## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Page: \_/ 2nd Reviewer: Reviewer:

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

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

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	Qualifications	MO OUAL WSDIN																								
	Associated Samples	8029233-BANK,	# 10																							
	RPD (Limits)	(	(	( )	( )	( )	· ·		( )	( )	( )	(		( )	(	(	(	)	( )	( )	( )	()	(	(	(	(
1.060	ACSU %R (Limits)	· · ·	( )	()	( )	( )	( )	( )	( )	)	( )	)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(
1	LCS %R (Limits)	1 20-92 ) Sr	( . )	( )	( )	. (	( )	( )	( )	( )	( )	( )	( )	(	(	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	<b>^</b>
	Compound	HH																								
	LCS/LCSD ID	501- Estbus										-														
	Date																									
ľ	*																									

## BRC Tronox Parcel H Data Validation Reports LDC# 18386

Chlorinated Pesticides



## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	BRC Tronox Parcel H
Collection Date:	January 24, 2008
LDC Report Date:	March 13, 2008
Matrix:	Soil
Parameters:	Chlorinated Pesticides
Validation Level:	EPA Level III & IV
Laboratory:	TestAmerica, Inc.

Sample Delivery Group (SDG): F8A250221

## Sample Identification

TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HJ-04-0' TSB-HR-04-0'\*\* TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'\*\* TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'\*\* TSB-HJ-07-0'-FD TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-10' TSB-HR-08-0'MS TSB-HR-08-0'MSD

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 17 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 with the following exceptions:

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
2/8/08	KCAL587	RTX-CLP1	Toxaphene	29.5	TSB-HJ-07-0'-FD	J+ (all detects)	A
2/8/08	KCAL587	RTX-CLP2	Toxaphene	27.4	TSB-HJ-07-0'-FD	J+ (all detects)	A
2/9/08	KCAL661	RTX-CLP1	Toxaphene	25.4	TSB-HJ-05-0' TSB-HJ-05-10' TSB-HR-04-10' TSB-HJ-04-0' TSB-HR-04-0'** 8029397-Blank	J+ (all detects)	A

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
2/9/08	KCAL677	RTX-CLP1	Toxaphene	31.6	TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'** TSB-HJ-07-10' TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-0'MS TSB-HR-08-0'MSD	J+ (all detects)	A
2/9/08	KCAL689	RTX-CLP1	Toxaphene Endosulfan II 4,4'-DDT Endrin aldehyde Endosulfan sulfate Endrin ketone	16.8 16.0 15.2 15.6 17.6 19.4	TSB-HR-08-10'	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	A
2/9/08	KCAL691	RTX-CLP1	Toxaphene	32.8	TSB-HR-08-10'	J+ (all detects)	A
2/9/08	KCAL692	RTX-CLP1	2,4'-DDE 2,4'-DDD	16.1 16.6	TSB-HR-08-10'	J+ (all detects) J+ (all detects)	A

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

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

Retention times (RT) of all compounds in the calibration standards were within QC limits for samples on which 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.

No field blanks were identified in this SDG.

## VI. Surrogate Spikes

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

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
TSB-HJ-05-0'	Not specified	Decachlorobiphenyl	128 (63-117)	All TCL compounds	J+ (all detects)	Ρ

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
TSB-HR-08-10'	Not specified	Decachlorobiphenyl	123 (63-117)	All TCL compounds	J+ (all detects)	Р

## VII. Matrix Spike/Matrix Spike Duplicates

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

Samples TSB-HJ-07-0'\*\* and TSB-HJ-07-0'-FD were identified as field duplicates. No chlorinated pesticides were detected in any of the samples with the following exceptions:

	Concentr	ation (ug/Kg)	RPD	Difference		
Compound	TSB-HJ-07-0'**	TSB-HJ-07-0'-FD	(Limits)	(Limits)	Flag	A or P
beta-BHC	1.8U	3.4	-	1.6 (≤1.8)	-	-

## BRC Tronox Parcel H Chlorinated Pesticides - Data Qualification Summary - SDG F8A250221

SDG	Sample	Compound	Flag	A or P	Reason
F8A250221	TSB-HJ-07-0'-FD TSB-HJ-05-0' TSB-HJ-05-10' TSB-HR-04-10' TSB-HR-04-0'** TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'** TSB-HR-06-0' TSB-HR-06-10' TSB-HR-06-10' TSB-HJ-07-10' TSB-HJ-07-10' TSB-HR-08-0'	Toxaphene	J+ (all detects)	A	Continuing calibration (%D)
F8A250221	TSB-HR-08-10'	Toxaphene Endosulfan II 4,4'-DDT Endrin aldehyde Endosulfan sulfate Endrin ketone 2,4'-DDE 2,4'-DDD	J+ (all detects)	A	Continuing calibration (%D)
F8A250221	TSB-HJ-05-0' TSB-HR-08-10'	All TCL compounds	J+ (all detects)	Ρ	Surrogate spikes (%R)

## **BRC Tronox Parcel H**

Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG F8A250221

No Sample Data Qualified in this SDG

BRC Tronox Parcel H

Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG F8A250221

No Sample Data Qualified in this SDG

LDC #: <u>18386A3a</u> SDG #: <u>F8A2502**91**</u> Laboratory: <u>Test America</u>

Level III/IV

108 Ð Date: Page: Reviewer: 2nd Reviewer:

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

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

	Validation Area		Comments
I.	Technical holding times	Δ	Sampling dates: 12408
11.	GC/ECD Instrument Performance Check	Δ	
111.	Initial calibration	Δ	
IV.	Continuing calibration/ICV	<u>ي</u> ت	
V.	Blanks	$\Delta$	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	4	Les
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	
Xb.	GPC Calibration	N	
XI.	Target compound identification	$\mathbf{A}$	Not reviewed for Level III validation.
XII.	Compound quantitation and reported CRQLs		Not reviewed for Level III validation.
XIII.	Overall assessment of data	Δ	
XIV.	Field duplicates	sω	$D =    +  ^2$
XV.	Field blanks	N	

Note:

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

FB = Field blank

D = Duplicate TB = Trip blank EB = Equipment blank

Validated Samples: \*\* Indicates sample underwent Level IV validation SO(V)

1	TSB-HJ-05-10'	11	TSB-HJ-07-0'**	21	8029397-Blank	31	
2	TSB-HJ-05-0' /	+ 12	TSB-HJ-07-0'-FD	22		32	
3	TSB-HR-04-10'	13	TSB-HJ-07-10'	23		33	
4	TSB-HJ-04-0'	14	TSB-HR-08-0'	24		34	
5	TSB-HR-04-0'**	15	TSB-HR-08-10' 🖌	25		35	
6	TSB-HJ-04-10'	16	TSB-HR-08-0'MS	26		36	
7	TSB-HR-07-0'	17	TSB-HR-08-0'MSD	27		37	
8	TSB-HR-07-10'**	18		28	· · · · · · · · · · · · · · · · · · ·	38	
9	TSB-HR-06-0'	19		29		39	
10	TSB-HR-06-10'	20		30		40	

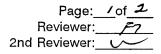
LDC #: 18386+39 SDG #: en coney

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## VALIDATION FINDINGS CHECKLIST

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

Validation Area	Yes	No	NA	Findings/Comments
L Technical holding times				
All technical holding times were met.	/			
Cooler temperature criteria was met.	-			
II. GC/ECD Instrument performance check				
Was the instrument performance found to be acceptable?	/			
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 Blanks				
Was a method blank associated with every sample in this SDG?	-			
Was a method blank analyzed for each matrix and concentration?	-			
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?		/		
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?			$\ge$	

LDC #: 18386A3a SDG #: pu cones

## VALIDATION FINDINGS CHECKLIST

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Validation Area	Yes	No	NA	Findings/Comments
VII. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			
Was a MS/MSD analyzed every 20 samples of each matrix?				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (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			<u> </u>	
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.	7			
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.	1	ſ	T	
Target compounds were detected in the field duplicates.	1			
XV. Field blanks				
Field blanks were identified in this SDG.		7		
Farget compounds were detected in the field blanks.			2	

VALIDATION FINDINGS WORKSHEET

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

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

Notes:

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LDC # 18 380 839 SDG #: Lev cover BC HPLC

METHOD:

## VALIDATION FINDINGS WORKSHEET **Continuing Calibration**

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2nd Reviewer:

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

ever IV Only AN NA NA Y N/N/A

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

Y								
#	Date	Standard ID	Detector/ Column	Compound	%D / RPD (Limit ≤ 15.0)	RT (limit)	Associated Samples	Qualifications
+	2	KCA1587	RTX-CUP!	5	عطرح	(	12	)+/A dut
	- -		RIX-CLPZ	γ	9-1-6	(	7	, L ,
			-					
						( )		
+	2908	Ked L661	RTX-UP!	1784	ж,ц	(	8009397-BAnk,	1-1/A dit
L	-			•	-	( )	1.2.3.4.5 (1-25)	Ι
							. 11	
+	2900	KCAL677	RTX - 41P	5	31.6	(	6-0 B, 16, 17	_ J <sup>+</sup> /A dut_
L						( )	1 13,14	
						( )	-	
						( )		
+	2010	Keal 689	Rtx-er PI	Ч	16.8	(	اك	1+/A ext
+	-			لہ	16.0	(		
+				Q	15.2	(		
+	-			R	15.6	( )		
+				ν	0-LI	( )		
+				ବ	19.4	(	L V	L
		}				( )		
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LDC #: 18 386A3A SDG #: Les cover

## VALIDATION FINDINGS WORKSHEET **Continuing Calibration**

Page: /of / 62 Reviewer: 2nd Reviewer:

/GC HPLC METHOD:

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 Were continuing calibration standards analyzed at the required frequencies? % Did the continuing calibration standards meet the %D / RPD validation criteria of  $\leq 15.0\%$ ?

Y N NIA Leveriv Only Y N NIA

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

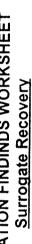
Г			Ī	1			Τ	T		-	-		1					Π	-		T	
	Qualifications	1+/A eut	~				1+/A dit	\ ,														
	Associated Samples	آکر				15	5															
	RT (limit)	)	( )	) (	( )	) )	)	( )	( )	(	(	· · · · · · · · · · · · · · · · · · ·	) (		( )	( )	( )	( )	( )	( )	) (	(
	%D / RPD (Limit ≤ 15.0)	32.25				1.91	16.6															
	Compound	γ				2,4'- PDE	Jdd-14.6															
	Detector/ Column	RIX-erp				RTX-CLP1	7															
	Standard ID	KCALC91				KCAL692																
	Date	2/9/08				3/9/00																
$\mathbb{R}$	*																<u> </u>					

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## VALIDATION FINDINDS WORKSHEET





METHOD:

METHOD: GC HPLC Are surrogates required by the method? Yes or No Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". Y N N/A Were surrogates spiked into all samples and blanks?

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LDC #. 18 386 A3 < SDG #. 14 LOULD	VALIDATION F	VALIDATION FINDINGS WORKSHEET Field Duplicates	ET.	Page: /of / Reviewer: /
METHOD:         GC         HPLC           Y         N         N/A         Were field duplicate pairs identified in this SDG?           Y         N         N/A         Were target compounds detected in the field dupli	d in this SDG? in the field duplicate pairs?			2nd reviewer:
	ncentration (	nglky)	%RPD	Qualification
bunoon	-	2	Di klovenca	Parent only / All Samples
ß	1.24	3.4	1.6 12. 12	
	Concentration (	(	%RPD	Qualification
				Parent only / All Samples
			•	
		•		

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LDC #. 18 386 A3~ ومع 3 SDG #:

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Page: Reviewer:

HPLC METHOD: GC

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

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

A = Area of compound,

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

			Reported	Recalculated	Reported	Recalculated	Renorted	Decelordated
Standard ID	Calibration Date	Compound	CF (0.6Vstd)	CF. (a ory std)	Average CF (initial)	Average CF (initial)	U28%	
IGAL	30/1/e	endosultant Rtxup)	040846 805	ahashlans Ohashl sos	45985255	5	6.294	6.274
		methowy ched J	OTHNSAK	Orgh some Orgh Nork	Organstic	1 .	6-78 7	121.7
		RTXCUPY	264234520	234520 264234520	off psa sar		5,85,2	5.585
			525 96400	Cordered	55-334490		4.736	レイレク
	•							
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LDC #: 18306A30 SDG#: Rev cont

**Continuing Calibration Results Verification** VALIDATION FINDINGS WORKSHEET

/ of Page: Reviewer: 2nd Reviewer:

НРГС 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

#     standard ID     Calibration     Calibration     Calibration     Calibration $Calibration$ <t< th=""><th></th><th></th><th></th><th></th><th></th><th>Reported</th><th>Recalculated</th><th>Reported</th><th>Recalculated</th></t<>						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	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc. CCV	MD	Ω%
KCALLETS 2 19 00 methody when V 00269 1.4 7.4 7 KCALLETS 2 19 00 V V 0.0275 0.00 74 9.6 0 V V 0.0374 9.6 0 V V 0.0374 9.6 0 V V V 0.0374 9.6 0 V V V V V V V V V V V V V V V V V V V		KULLES	30/6/2	endosultion) PTX-CUP)	٥، ەيمى	0.0262	2010.0	4.9	4.9
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$		-		methosy chief N	ł	0.0769	0.0269	h-L	7, 1
N       N		KCALLTS				0.0275	22-20.0	10. <i>0</i>	0,01
	2		-	~	ĥ	9200	h2.00.0	٩.४	ط بُا
	3								
	4								

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 recalculated results.

### LDC #: 18386A3a SDG #: <u>pu cour</u>

### VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

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### 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

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

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene						
Tetrachloro-m-xylene	REX-CIPI	0.07	0.02117	106	106	106
Decachlorobiphenyl			0.02272	114	114	114
Decachlorobiphenyl						

### Sample ID:

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachioro-m-xylene						
Tetrachloro-m-xylene		<u></u>				
Decachlorobiphenyl		<u> </u>				
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		· · · · · · · · · · · · · · · · · · ·	·			<u> </u>
Decachlorobiphenyl						
Decachlorobiphenyl						

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

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	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)

LI 7 91

MS/MSD samples:

MS = Matrix spike percent recovery

SSC = Spiked sample concentration SA = Spike added

Where:

MSD = Matrix spike duplicate percent recovery

SC = Concentration

Recalculated 8.9  $\leq$ **MS/MSD** RPD Reported 8.9 Matrix Spike Duplicate Recalc. Percent Recovery 10 96 Reported 0)/ 96 Recalc. Percent Recovery 127 901 Matrix Spike Reported 201 122 MSD Spiked Sample Concentration 17. (52) 31:4 16.6 SW Sample Concentration R ISI 0 0 MSD 11.33 6.61 X Added Spike Ş MS 2.5 Compound gamma-BHC 4,4'-DDT

Comments: Refer of 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.

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LDC #:	SDG #:

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



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/LCSD samples: 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

			Kecalc.			Ī	T	<u> </u>		
LCS/LCSD	uaa		Щ					 		
			керопеа							
LCSD	Percent Recoverv		Vecalc.	$\left  \right $						
	Percent		INFORM							
LCS	Percent Recovery	Decalo		21	-					
L L	Percent	Renorted	6.01	112						
Spiked Sample	Entration			1	>	~				
Spike	CONC ( MA	rcs	6.E1	1.81						
Spike Added	3 44		×2	⊈ →						
<u>د</u> م	× ۲ )	rcs	11	1						
	Compound		gamma-BHC	4,4'-DDT						·

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 #: 18 386 A3a SDG #: <u>en cone</u>r

Y N N/A

N N/A

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

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	$\sim$

METHOD: C 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?

Example:		
Sample I.D.	;	
Conc. = <u>(</u> (		
=	ND	

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

Note:\_

### LDC Report# 18386B3a

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

Project/Site Name:	BRC Tronox Parcel H
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Collection Date: January 28, 2008

LDC Report Date: March 14, 2008

Matrix: Soil/Water

Parameters: Chlorinated Pesticides

Validation Level: EPA Level III

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8A290158

### Sample Identification

TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10' RINSATE-2 TSB-HR-05-10'MS TSB-HR-05-10'MSD

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### Introduction

This data review covers 11 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8081A for Chlorinated Pesticides.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was 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 with the following exceptions:

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
2/8/08	KCAL617	RTX-CLP1	Toxaphene	21.7	TSB-HJ-10-0' 8035062-BLK	J+ (all detects)	A
2/8/08	KCAL631	RTX-CLP1	Toxaphene	22.4	TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10' TSB-HR-05-10'MS TSB-HR-05-10'MSD	J+ (all detects)	A

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

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

Retention times (RT) of all compounds in the calibration standards were within QC limits for samples on which 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-2" was identified as a rinsate. No chlorinated pesticide contaminants were found in this blank.

### VI. Surrogate Spikes

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

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
TSB-HJ-10-0'	Not specified	Tetrachloro-m-xylene Decachlorobiphenyl	119 (55-115) 126 (63-117)	All TCL compounds	J+ (all detects)	Ρ
TSB-HJ-10-10'	Not specified	Decachlorobiphenyl	126 (63-117)	All TCL compounds	J+ (all detects)	Р
TSB-HR-06-0'	Not specified	Decachlorobiphenyl	121 (63-117)	All TCL compounds	J+ (all detects)	Р
TSB-HR-06-0'-FD	Not specified	Tetrachloro-m-xylene Decachlorobiphenyl	122 (55-115) 127 (63-117)	All TCL compounds	J+ (all detects)	Р
TSB-HR-06-10'	Not specified	Tetrachloro-m-xylene Decachlorobiphenyl	121 (55-115) 129 (63-117)	All TCL compounds	J+ (all detects)	Р
TSB-HJ-08-0'	Not specified	Tetrachloro-m-xylene Decachlorobiphenyl	126 (55-115) 132 (63-117)	All TCL compounds	J+ (all detects)	Р
TSB-HJ-08-10'	Not specified	Tetrachloro-m-xylene Decachlorobiphenyl	119 (55-115) 123 (63-117)	All TCL compounds	J+ (all detects)	Р
TSB-HR-05-0'	Not specified	Tetrachloro-m-xylene Decachlorobiphenyl	124 (55-115) 130 (63-117)	All TCL compounds	J+ (all detects)	Р
8035062-Blank	Not specified	Decachlorobiphenyl	124 (63-117)	All TCL compounds	J+ (all detects)	Р

### VII. Matrix Spike/Matrix Spike Duplicates

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

Raw data were not reviewed for this SDG.

### XII. Compound Quantitation and Reported CRQLs

Raw data were not reviewed for this SDG.

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

Samples TSB-HR-06-0' and TSB-HR-06-0'-FD were identified as field duplicates. No chlorinated pesticides were detected in any of the samples.

### BRC Tronox Parcel H Chlorinated Pesticides - Data Qualification Summary - SDG F8A290158

SDG	Sample	Compound	Flag	A or P	Reason
F8A290158	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10'	Toxaphene	J+ (all detects)	A	Continuing calibration (%D)
F8A290158	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HJ-08-10' TSB-HR-05-0'	All TCL compounds	J+ (all detects)	Ρ	Surrogate spikes (%R)

### BRC Tronox Parcel H

Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG F8A290158

No Sample Data Qualified in this SDG

BRC Tronox Parcel H Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG F8A290158

No Sample Data Qualified in this SDG

### VALIDATION COMPLETENESS WORKSHEET

LDC #: 18386B3a SDG #: F8A290158

Level III

6/08 Date: Page: / of Reviewer: 2nd Reviewer:

Laboratory: Test America

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
Ι.	Technical holding times	А	Sampling dates: 1/28 00
11.	GC/ECD Instrument Performance Check	A	
111.	Initial calibration	$\Delta$	
IV.	Continuing calibration/ICV	sale	\$vV
V.	Blanks	Δ	
VI.	Surrogate spikes	Su	
VII.	Matrix spike/Matrix spike duplicates	Δ	
VIII.	Laboratory control samples	A	LCS
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	
Xb.	GPC Calibration	N	· · · · · · · · · · · · · · · · · · ·
XI.	Target compound identification	<u>N</u>	
XII.	Compound quantitation and reported CRQLs	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	NP	D = 3+4
XV.	Field blanks	NP	R = 10

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:

Valida	ted Samples: Soll + Wa	In _					
1	TSB-HJ-10-0'	11	TSB-HR-05-10'MS	21	8029304	31	
2	TSB-HJ-10-10'	12	TSB-HR-05-10'MSD	22	8035062	32	
3	TSB-HR-06-0' 🗘	13		23		33	
4	TSB-HR-06-0'-FD	14		24		34	
5	TSB-HR-06-10'	15		25		35	
6	TSB-HJ-08-0'	16		26		36	
7	TSB-HJ-08-10'	17		27		37	
8	TSB-HR-05-0'	18		28		38	
9	TSB-HR-05-10'	19		29		39	
10 \	RINSATE-2 W	20		30		40	

VALIDATION FINDINGS WORKSHEET

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

A sinte Bilo				
	1. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	
B. beta-BHC				
	J. 4,4. DDE	R. Endrin aldehyde	Z. Aroclor-1248	
C. delta-BHC				
	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	
D. gamma-BHC				<u></u>
	L. Endosulfan li	T. gamma-Chlordane	BB. Aroclor-1260	, Tr
E. Heptachfor	M 4.4500			
		U. Toxaphene	CC. DB 608	XX
F. Aldrin				
	N. Endosullan sulfate	V. Aroctor-1016	DD. DB 1701	
G. Heptachlor epoxide	0.4.4.001			i i i
		W. Aroclor-1221	Ë	W
H. Endosultan I				
	r. Metnoxychlor	X. Aroclor-1232	FF.	
				NN.

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Notes:

LDC #. 18 380 B 3 C SDG #: Lev cover METHOD: \_\_\_\_\_GC \_\_\_ HPLC

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

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2nd Reviewer:

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". What type of continuing calibration calculation was performed?  $\[mathcackwordsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmallewboldsmall$ 

K N N/A K N N/A Level IV Only

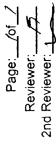
Were the retention times for all calibrated compounds within their respective acceptance windows? Y N MA

		Τ																				
Qualifications	JA AJ				JT/AWGT																	
Associated Samples	H 8-2995603 (#				4 2-49,11,12	-																
RT (limit)	( )		(	( )	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(	( )	( )	( )	( )	( )
%D / RPD (Limit ≤ 15.0)	21.7				22.4																	
Compound	N				7																	
Detector/ Column	RTX-CUPI				1																	
Standard ID	kca Lei7				KCAL631																	
Date	12/08	-			2 4 08	1 1																
#	+				+-																	

22

LDC #: 18380 B3 ~ SDG #: ev coner

### VALIDATION FINDINDS WORKSHEET **Surrogate Recovery**

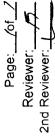


METHOD: CC HPLC Are surrogates required by the method? Yes or No ... Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". V N/A Were surrogates spiked into all samples and blanks?

) #	Sample ID	Detector/ Column	or/ nn	Surrogate Compound		%R (Limits)				Qualifications
	1 hort	1	ilied	٢		119	SS-115	15 )	1+/p dut	
		1	0	Ø		126 (	63-117	( 1/	J	
								(		
	2	-7		Ф		126 (	63-117	() () ()	1+/Pdut	
						)				
	Ś	->		Ь		121	~	(	1 1+ /Pdut	
						)		(		
	h	-		٢		) ~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	511-55	( 51	1+/part	
		<b>^</b>		Ð		127 (	63-117	() L ()		
						~		(		
	2					)   21				
		7		>		129	7	(	>	
	-O							(		
	<i>c</i>			-		1260 (		(	] J+/Pdut	
		<b>→</b>		7		132	~	(		
						)		(	,	
	L	-				119	-	(	1 1+/Paul	
		->		7		123 (	~	(		
						}		(		
	8	1				124 (		(	1-1Paut	
				1		130 (	>	(		
	Surrogate Compound		Surrogate	Surrogate Compound		Surrogate Compound		Surrogat	Surrogate Compound	
A	Chlorobenzene (CBZ)	ຍ	Octac	Octacosane	Μ	Benzo(e)Pyrene	s	1-Chlaro-	1-Chloro-3-Nitrobenzene	Tetrachloro-m- xylene
в	4-Bromofluorobenzene (BFB)	r	Ortho-	Ortho-Terphenyl	z	Terphenyl-D14	+	3,4-Dir	3,4-Dinitrotoluene	
υ	a,a,a-Trifiuorotoluene		Fluorobe	Fluorobenzene (FBZ)	0	Decachlorobiphenyl (DCB)		Trip	Tripentyltin	
٥	Bromochlorobenene	-	o-Tris	n-Triacontane	٩	1-methvinaohthalene	>	Tri-n	Tri-n-propyltin	
ω	1,4-Dichlorobutane	¥	Hexa	Hexacosane	σ	Dichlorophenyl Acetic Acid (DCAA)	3	Tributy	Tributyl Phosphate	
u	1.4-Difluorobenzene. (DFB)		, Brom	Bromobenzene	В	4-Nitrophenol	X	Triphen	Triphenyl Phosphate	

LDC #: 18 38 68 30 SDG #: 11

### VALIDATION FINDINDS WORKSHEET Surrogate Recovery



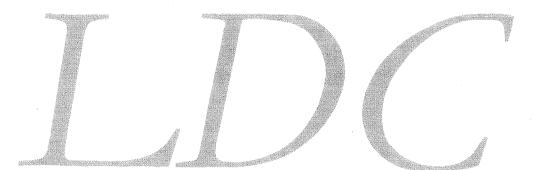
METHOD: CC HPLC Are surrogates required by the method? Yes or No Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". Y N/A Were surrogates spiked into all samples and blanks?

	Qualifications																			Tetrachloro-m- xylene					
		1Part																		\					
		1 1+/9/	. /		) (	) [	(	(		) [ (	) [	) [	) (	) [	) (		(	)	Surrogate Compound	1-Chloro-3-Nitrobenzene	3,4-Dinitrotoluene	Tripentyttin	Tri-n-oroovitin	Tributyl Phosphate	Triphenvi Phosohate
		- 117																	Surroga	1-Chloro	3,4-Di	Tri	Tdi-	Tributy	Triphen
		63-117																		s	+	<u>ر</u>	>	≥	×
	%R (Limits)	124 (				)	)	)	~	)			~	~		)			Surrogate Compound	Benzo(e)Pyrene	Terphenyl-D14	Decachlorobiphenyl (DCB)	1-methvlnaohthaiene	Dichlorophenyl Acetic Acid (DCAA)	4-Nitrophenol
5													 	 		 				Σ	z	0	٩	σ	R
meet the QC limits?	Surrogate Compound																		Surrogate Compound	Octacosane	Ortho-Terphenyl	Fluorobenzene (FBZ)	n-Triacontane	Hexacosane	Bromobenzene
s (%R)		minars	-																Surro		0	Fluo			Ш
e recoveries	Detector/ Column	not s																		U	т			¥	-
UA Did all surrogate recoveries (%R) meet the QC I	Sample ID	2 were - rade o blank																	Surrogate Compound	Chlorobenzene (CBZ)	4-Bromofluorobenzene (BFB)	a,a,a-Trifluorotoluene	Bromochlorobenene	1,4-Dichlorobutane	1.4-Difluorobenzene (DFB)
Y/N N/A	) #								_											٩	8	U	٥	ω	Ч

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### BRC Tronox Parcel H Data Validation Reports LDC# 18386

### Polychlorinated Biphenyls



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

Project/Site Name:	BRC Tronox Parcel H
Collection Date:	January 24, 2008
LDC Report Date:	March 11, 2008
Matrix:	Soil
Parameters:	Polychlorinated Biphenyls
Validation Level:	EPA Level III & IV
Laboratory:	TestAmerica, Inc.

Sample Delivery Group (SDG): F8A250221

### Sample Identification

もつい かどう しかつ ごがらい しょう かいううやう

TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HJ-04-0' TSB-HR-04-0'\*\* TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'\*\* TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'\*\* TSB-HJ-07-0'-FD TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-10' TSB-HR-08-0'MS TSB-HR-08-0'MSD

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 17 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8082 for Polychlorinated Biphenyls.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

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

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

### **III. Initial Calibration**

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

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

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

### IV. Continuing Calibration

Continuing calibration was performed at required frequencies.

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

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

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

### V. Blanks

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

No field blanks were identified in this SDG.

### VI. Surrogate Spikes

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

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### 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

Samples TSB-HJ-07-0'\*\* and TSB-HJ-07-0'-FD were identified as field duplicates. No polychlorinated biphenyls were detected in any of the samples.

BRC Tronox Parcel H Polychlorinated Biphenyls - Data Qualification Summary - SDG F8A250221

No Sample Data Qualified in this SDG

BRC Tronox Parcel H Polychlorinated Biphenyls - Laboratory Blank Data Qualification Summary - SDG F8A250221

No Sample Data Qualified in this SDG

BRC Tronox Parcel H Polychlorinated Biphenyls - Field Blank Data Qualification Summary - SDG F8A250221

No Sample Data Qualified in this SDG

### VALIDATION COMPLETENESS WORKSHEET

LDC #: 18386A3b SDG #: F8A250221

### Laboratory: Test America

Level III/IV

768 Date: Page:\_/\_of Reviewer: 2nd Reviewer:

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

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

	Validation Area		Comments
Ι.	Technical holding times	4	Sampling dates: 1/24/09
II.	GC/ECD Instrument Performance Check	NΔ	
111.	Initial calibration	4	
IV.	Continuing calibration/ICV	А	101 = 15
V.	Blanks	4	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	4	
VIII.	Laboratory control samples	SW	105
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	
Xb.	GPC Calibration	N	
XI.	Target compound identification	<u> </u>	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	ND	D=11+12
XV.	Field blanks	$\mathbb{N}$	

Note:

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

FB = Field blank

TB = Trip blank EB = Equipment blank

D = Duplicate

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

	Sell					
1	TSB-HJ-05-10'	11	TSB-HJ-07-0'**	21	8029 396-Blank	31
2	TSB-HJ-05-0'	12	TSB-HJ-07-0'-FD	22		32
3	TSB-HR-04-10'	13	TSB-HJ-07-10'	23		33
4	TSB-HJ-04-0'	14	TSB-HR-08-0'	24	· · · · · · · · · · · · · · · · · · ·	34
5	TSB-HR-04-0'**	15	TSB-HR-08-10'-	25		35
6	TSB-HJ-04-10'	16	TSB-HR-08-0'MS	26	••••••••••••••••••••••••••••••••••••••	36
7	TSB-HR-07-0'	17	TSB-HR-08-0'MSD	27		37
8	TSB-HR-07-10'**	18		28		38
9	TSB-HR-06-0'	19		29		39
10	TSB-HR-06-10'	20		30		40

### VALIDATION FINDINGS CHECKLIST

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

Method:GCHPLC				
Validation Area	Yes	No	NA	Findings/Comments
1. Technical holding times				
All technical holding times were met.	17			
Cooler temperature criteria was met.				:
11 Initial calibration				
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%?	~			
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?		/		
Did the initial calibration meet the curve fit acceptance criteria?			-	
Were the RT windows properly established?	/			
IV: Continuing calibration		. 168		
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%?	-			
Were all the retention times within the acceptance windows?	$\checkmark$			
V Blanks		K.		
Was a method blank associated with every sample in this SDG?	/			
Was a method blank analyzed for each matrix and concentration?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/		
VI Surrogate spikes	12. 12.			
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?				Γ
VII. Matrix spike/Matrix spike duplicates	i, h			
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			
Was a MS/MSD analyzed every 20 samples of each matrix?	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	/			
VIII. Laboratory control samples				
Was an LCS analyzed for this SDG?	<			
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		/		

### VALIDATION FINDINGS CHECKLIST

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

Validation Area	Yes	No	NA	Findings/Comments
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 and dry weight factors applicable to level IV validation?				
XIII.Systemperformance:				
System performance was found to be acceptable.				
XIII9Overallisssessment 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.				
XV. Field blanks			e e sis Siste	
Field blanks were identified in this SDG.	Τ			
Target compounds were detected in the field blanks.			~†	

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

è Page: 2nd Reviewer: Reviewer:

METHOD: CC HPLC

Prease see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". <u>V N N/A</u> Were a laboratory control samples (LCS) and laboratory control sample duplicate (LCSD) analyzed for each matrix in this SDG? <u>V N N/A</u> Were the LCS percent recoveries (%R) and relative percent differences (RPD) within the QC limits?

Level IV/D Only Y N N/A Wa

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

=	141														<u> </u>				<u> </u>					
Qualifications	1+1P w 1+1	m di sm																						
Associated Samples	A11 + 7 K																							
RPD (Limits)			( )	( )	( )	( )	( )		(	( )	( )		( )	( )	( )		( )	( )	( )	( )	( )	( )	( )	
LCSD %R (Limits)			( )	( )	( )	( )	(		( )	( )	( )	( )	( )	( )	( )		( . )	( )	( )	( )	( ) .	( )	( )	
LCS %R (Limits)	110 180-11/01		( )	( )	( )	( )	( )	( )	( )		( )	( ) )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	
Compound		•																						
LCS/LCSD ID	4100201	222 [2] 10 - 12																						
L#																							<u> </u>	

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LDC #: 18386A36 cert 4r SDG #:

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

ç Page: 2nd Reviewer: Reviewer:

> HPLO METHOD: GC

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

% Retrage 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 6-1-8 %RSD 9.583 Reported 8.799 %RSD 9.539 Recalculated Average CF (initial) 11104 2 2511 Average CF (initial) Reported 1205 HOI **Recalculated** CF. (Sb (Btd) ころい 12182 (Sb) std) Reported Ч RTX eup 12182 11205 RTX-CLP2 1200-Compound - Onci japant Andor Calibration Date 9/107 Standard ID 197 L -¥t 2 ო 4

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

LDC #. 18 3 8 6 A36 cover 3 SDG:#:

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

Reviewer: 2nd Reviewer: Page:

HPLC METHOD: GC

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

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

Where:

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

A = Area of compound C = Concentration of compound

۵%

**Recalculated** , L 9.8 Reported 0.1 V بر م 0% Recalculated CF/Conc. CCV 1090.29 1006.69 109 8.2936 1006.6919 CF/Conc. CCV Reported Average CF(Ical)/ 1000.000 CCV Conc. 00.000 1260-1 RTX CUP) 1260-1 RTXCVP2 Compound Americar Aroclo1 Calibration Date 1/31/08 9497 Standard ID 20 HEAT ¥± a ო 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.

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### VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

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

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

Sample ID: # 5

	Column/Detector	Spiked	Found	Recovery	Recovery	Difference
				Reported	Recalculated	
DCR ATX CUPI	- 1d	a	19.7627	lolo	99	O

### Sample ID:

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

Sample ID:

	ColumpiDetector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
Surogate				Reported	Recalculated	
			-			
						-
			-			

436	quer
98681	200
LDC #:	SDG #:

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

Page: / of Z Reviewer: 2nd Reviewer:

Š **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 HPLC

Where the following calculation: %Recovery = 100 \* (SSC - SC//SA

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

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

SC = Sample concentration MSD = Matrix spike duplicate

> Ľ ہ۔ و MS/MSD samples:\_\_

	Spike	e ike	Sample	Spike :	Spike Sample	Matrix	Matrix spike	Matrix Snike Dunlicate	e Dunlicate	MSM	69
Compound	NOV /	par ka	conc.	Concel	ntration						
					1EX	Percent	Percent Recovery	Percent Recovery	Recovery	RPD	0
	WS	MSD	1	WS	MsD	Reported	Recalc.	Reported	Recalc	Benerad	
Gasoline (8015)	;	:								natioday	Lecalc.
Diesel (8015)											
Benzene (8021B)											
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)											
Anthracene (8310)											
HMX (8330)											
2,4,6-Trinitrotoluene (8330)											
Arochor 1260	116	ЪЦ	0	212	1.		0.1			2	)
				214	1	740	071	12	141	05.0	1-0
Comments: <u>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%</u>	ike/Matrix 5	Spike Dupl	icates finding:	s worksheet f	or list of qualif	ications and a	II ssociated sam	ples when rep	)  <u>oorted results</u>	l do not agree	within 10.0%

MSDCLCNew.wpd

SDG #: pu coner [	-aboratory	Contr	ol Sample.	/Laborato	ple/Laboratory Control Sample Duplic	Sample Di	Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification	esults Veri	<u>ification</u>	Reviewer:	
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 follo	percent wing ca	differences ( lculation:	RPD) of the	laboratory co	ntrol sample	and laborator	y control sam	ple duplicate	were recalcul:	ated for the
%Recovery = 100 * (SSC - SC)/SA	3	Where SS	SSC = Spiked concentration SA = Spike added	centration		SC = Sample concentration	ncentration				
RPD =(({SSCLCS - SSCLCSD} * 2) / (SSCLCS + SSCLCSD))*100 LCS/LCSD samples: <u>8 0 2 3 2 4 5 - L<sup>C</sup> &gt;</u>	so) + 2) / (ssclcs + ssc	cLcsD))*1	8 1	LCS = Labora	S = Laboratory Control Sample percent recovery	ple percent recov		SD = Laboratory	Control Sample c	LCSD = Laboratory Control Sample duplicate percent recovery	covery
	Spike		Sample	Spike (	Spike Sample	Ľ	LCS	rcsD	Q.	rcs/rcsD	sD
Compound	Added ( v3	<u>^</u>	Conc.	Concer (	Concentration (	Percent	Percent Recovery	Percent Recovery	ecovery	RPD	
		LCSD	0 •		Lcsp	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)										•	
Diesel (8015)	45.										
Benzene (8021B)											
Methane (Rsk-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)											
Anthracene (8310)											
HMX (8330)											
2,4,6-Trinitrotoluene (8330)											
Aracler 1260	167	02	0	611	4N V	101	107	N A N			
Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicate findings worksheet for list of qualifications and associated samples when reported	lory Control S	ample/L	aboratory Co	Introl Sample	Duplicate fin	dings worksh	eet for list of g	ualifications s	and associate	d samples who	en reported
results do not agree within 10.0% of the recalculated results.	0.0% of the re	<u>scalculat</u>	ed results.								

LCSCLCNew.wpd

Page: Reviewer: 2nd Reviewer:			Qualifications				
SHEET tion sported results?	Compound Name	M	Recatculated Results Concentrations (				
VALIDATION FINDINGS WORKSHEET Sample Calculation Verification d verified for all level IV samples?		• • •	Reported Concentrations				•.
VALIDATIC Sample Sample ecalculated and verified for its for detected target comp	Example: Sample ID. Concentration =		Compound				一 一 周 雪子
LDC #. 1878LASD SDG #:	Concentration= (A)(Fv)(Df) (RF)(Vs or Ws)(%S/100) A= Area or height of the compound to be measured Fv= Final Volume of extract Df= Dilution Factor RF= Average response factor of the compound In the Initial calibration	Vs= Initial volume of the sample Ws= Initial weight of the sample %S= Percent Solid	# Sample ID			Comments:	

SAMPCALew.wpd

### LDC Report# 18386B3b

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

Project/Site Name:	BRC Tronox Parcel H
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Collection Date: January 28, 2008

LDC Report Date: March 11, 2008

Matrix: Soil/Water

Parameters: Polychlorinated Biphenyls

Validation Level: EPA Level III

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8A290158

### Sample Identification

TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0'-FD TSB-HR-06-0'-FD TSB-HJ-08-0' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10' RINSATE-2 TSB-HR-05-10'MS TSB-HR-05-10'MS

### Introduction

This data review covers 11 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 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.

Raw data were not reviewed for this SDG. The review was 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.

### IV. Continuing Calibration

Continuing calibration was performed at required frequencies.

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

Date	Standard	Column	Compound	%D	Associated Samples	Affected Compound	Flag	A or P
2/4/08	PCAL541	RTX-CLP1	Aroclor-1016	16.8	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD	Aroclor-1016 Aroclor-1221 Aroclor-1232	J+ (all detects) J+ (all detects) J+ (all detects)	А

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

### V. Blanks

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

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

### VI. Surrogate Spikes

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

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
8031455-Blank	Not specified	Dichlorophenyl acetic acid	269 (51-150)	All TCL compounds	J+ (all detects)	Р

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### 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

Raw data were not reviewed for this SDG.

### XII. Compound Quantitation and Reported CRQLs

Raw data were not reviewed for this SDG.

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

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12.14

Samples TSB-HR-06-0' and TSB-HR-06-0'-FD were identified as field duplicates. No polychlorinated biphenyls were detected in any of the samples.

### BRC Tronox Parcel H Polychlorinated Biphenyls - Data Qualification Summary - SDG F8A290158

SDG	Sample	Compound	Flag	A or P	Reason
F8A290158	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD	Aroclor-1016 Aroclor-1221 Aroclor-1232	J+ (all detects) J+ (all detects) J+ (all detects)	A	Continuing calibration (%D)

### BRC Tronox Parcel H

Polychlorinated Biphenyls - Laboratory Blank Data Qualification Summary - SDG F8A290158

No Sample Data Qualified in this SDG

BRC Tronox Parcel H Polychlorinated Biphenyls - Field Blank Data Qualification Summary - SDG F8A290158

No Sample Data Qualified in this SDG

VALIDATION COMPLETENESS WORKSHEET
 Level III

LDC #: <u>18386B3b</u> SDG #: F8A290158

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Laboratory: Test America

Date: 3/6/08 Page: \_\_\_of Reviewer: 2nd Reviewer:

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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
Ι.	Technical holding times	Δ	Sampling dates: 1/28/08
11.	GC/ECD Instrument Performance Check	N-A	
111.	Initial calibration	Δ	
IV.	Continuing calibration/ICV	~SW	100 = 15
V.	Blanks A	sa	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	sω	105
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	
Xb.	GPC Calibration	N	
XI.	Target compound identification	N	
XII.	Compound quantitation and reported CRQLs	N	
XIII.	Overall assessment of data	Δ	
XIV.	Field duplicates	ND	p = 3 + 4
XV.	Field blanks	ND	R = 10

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:

vanuat	Soil + wour	~					
				211	8029346-B1K	31	
1	TSB-HJ-10-0'	11	TSB-HR-05-10'MS				
2	TSB-HJ-10-10'	12	TSB-HR-05-10'MSD	22 7	8031455-B1/C	32	
3	TSB-HR-06-0' 0	13		23		33	
4	TSB-HR-06-0'-FD , P	14		24		34	
5	TSB-HR-06-10'	15		25		35	
6	TSB-HJ-08-0'	16		26		36	
7	TSB-HJ-08-10'	17		27		37	
8	TSB-HR-05-0'	18		28		38	
9	TSB-HR-05-10' 🗸	19		29		39	
10/	RINSATE-2 R W	20		30		40	

VALIDATION FINDINGS WORKSHEET

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

A sinha Dide				
	I. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	GG.
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Arocior-1248	HR.
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	
D. gamma-BHC	L. Endosulfan il	T. gamma-Chlordane	BB. Arocior-1260	J
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. DB 608	KK.
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD DB 4704	
G. Heptachior epoxide	0.4,4*-DDT	W. Aroclor-1221	EE.	MM.
H. Endosulfan I	P. Methoxychior	X Amelor-1939		

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Notes:

CGC HPLC

METHOD:

## VALIDATION FINDINGS WORKSHEET **Continuing Calibration**

Page: /of / Reviewer: //7

2nd Reviewer:

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

Y (N) N/A Level IV Only × N MA

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

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Qualifications	] J + / A det	ONAL V, W, X																				
	+ 4-1																					
RT (limit)	) (	) (	) (	( )	) (	) (	( )	)	( )	) (	] ( )	( )	( )	(	( )	( )	( )	(	) (	( )	)	(
%D / RPD (Limit ≤ 15.0)	16-8																					
Compound	>																					
Detector/ Column	RTX-CUP/																					
Standard ID	PCAL SY/																					
Date	00/ h/e																					
#	+																					

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## VALIDATION FINDINDS WORKSHEET **Surrogate Recovery**



METHOD: GC HPLC Are surrogates required by the method? Yes or No Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

W/N/N/A	V/A Did all surrogate recoveries (%R) meet the QC limits?	gate re	coveries (	%к) meet t	he QC limits	\. \.					
*	Sample ID		Detector/ Column		Surrogate Compound		%R (Limits)			Ğ	Qualifications
	8031415-13/ant	ton		5			269	<u>51-150</u>	) P	It/Paut	
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	Surrogate Compound	pu		Surrogate Compound	punoduu		Surrogate Compound		Surrogate Compound	punoduu	
∢	Chlorobenzene (CBZ)		د   د	Octacosane	ne	Σ	Benzo(e)Pyrene	S	1-Chloro-3-Nitrobenzene	robenzene Y	Tetrachloro-m- xylene
в	4-Bromofluorobenzene (BFB)	3FB)	г	Ortho-Terphenyl	henyl	z	Terphenyl-D14	⊢ 	3,4-Dinitrotoluene	oluene	
U	a,a.a.Trifiuorotoluene		-	Fluorobenzene (FBZ)	ie (FBZ)	0	Decachlorobiphenyl (DCB)	<u>р</u>	Tripentyltin	łtin	
٥	Bromochlorobenene			n-Triacontane	tane	٩	1-methvlnaohthalene	>	Tri-n-propyltin	vyltin	
ω	1,4-Dichlorobutane		×	Hexacosane	ane	0	Dichlorophenyl Acetic Acid (DCAA)	3	Tributyl Phosphate	sphate	
u	1.4-Difluorobenzene (DFB)	-B)		, Bromobenzene	zene	В	4-Nitrophenol	×	Triphenvl Phosphate	osphate.	

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## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

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METHOD: GC HPLC

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

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

-	l CS/LCSD ID	Compound	LCS %R (Limits)		LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
iμ	2031722-102		1200 (5	1 80-1161		( )	1/8 - ssh/Eas	Juno ou
2	22 F 1	-				( )	× 4.1 Size /2	ni di su
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### BRC Tronox Parcel H Data Validation Reports LDC# 18386

Metals



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

Project/Site Name:	BRC Tronox Parcel H
Collection Date:	January 24, 2008
LDC Report Date:	March 11, 2008
Matrix:	Soil
Parameters:	Metals
Validation Level:	EPA Level III & IV
Laboratory:	TestAmerica, Inc.

### Sample Delivery Group (SDG): F8A250221

### Sample Identification

TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HJ-04-0' TSB-HR-04-0'\*\* TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'\*\* TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'\*\* TSB-HJ-07-0'-FD TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-10' TSB-HR-08-0'MS TSB-HR-08-0'MSD

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 17 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, Zinc, and Zirconium.

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

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

Blanks are summarized in Section III.

Field duplicates are summarized in Section XIII.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. Calibration

An initial calibration was performed.

The frequency and analysis criteria of the initial calibration verification (ICV) and continuing calibration verification (CCV) were met.

### III. Blanks

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

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
PB (prep blank)	Aluminum Boron Calcium Chromium Iron Niobium Phosphorus Potassium Sodium Tin	3.1 mg/Kg 2.4 mg/Kg 14.0 mg/Kg 0.33 mg/Kg 5.6 mg/Kg 1.4 mg/Kg 3.0 mg/Kg 3.9 mg/Kg 8.2 mg/Kg 0.067 mg/Kg	All samples in SDG F8A250221
ICB/CCB	Cadmium Chromium Cobalt Nickel Niobium Thallium Titanium Tungsten Lithium	0.036 ug/L 0.5 ug/L 0.5 ug/L 0.5 ug/L 6.8 ug/L 0.4 ug/L 0.6 ug/L 0.9 ug/L 7.6 ug/L	All samples in SDG F8A250221

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	Analytə	Reported Concentration	Modified Final Concentration
TSB-HJ-05-10'	Cadmium	0.063 mg/Kg	0.54U mg/Kg

Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-HJ-05-0'	Cadmium	0.099 mg/Kg	0.26U mg/Kg
	Lithium	14.7 mg/Kg	21.0U mg/Kg
TSB-HR-04-10'	Niobium	5.5 mg/Kg	6.6U mg/Kg
TSB-HJ-04-0'	Cadmium	0.10 mg/Kg	0.55U mg/Kg
	Niobium	4.8 mg/Kg	5.5U mg/Kg
	Lithium	10.7 mg/Kg	21.8U mg/Kg
TSB-HR-04-0'**	Cadmium	0.076 mg/Kg	0.13U mg/Kg
	Niobium	3.3 mg/Kg	5.2U mg/Kg
	Tungsten	0.29 mg/Kg	1.3U mg/Kg
	Lithium	14.3 mg/Kg	20.9U mg/Kg
TSB-HJ-04-10'	Boron	15.3 mg/Kg	53.2U mg/Kg
	Cadmium	0.064 mg/Kg	0.27U mg/Kg
	Niobium	5.7 mg/Kg	6.7U mg/Kg
TSB-HR-07-0'	Cadmium	0.069 mg/Kg	0.27U mg/Kg
	Niobium	4.0 mg/Kg	5.4U mg/Kg
	Lithium	17.3 mg/Kg	21.4U mg/Kg
TSB-HR-07-10'**	Niobium	4.4 mg/Kg	6.7U mg/Kg
TSB-HR-06-0'	Cadmium	0.11 mg/Kg	0.28U mg/Kg
	Niobium	3.8 mg/Kg	5.5U mg/Kg
	Lithium	12.0 mg/Kg	22.2U mg/Kg
TSB-HR-06-10'	Cadmium	0.068 mg/Kg	0.55U mg/Kg
	Niobium	3.4 mg/Kg	5.5U mg/Kg
TSB-HJ-07-0'**	Niobium	3.4 mg/Kg	5.4U mg/Kg
	Lithium	10.2 mg/Kg	21.7U mg/Kg
TSB-HJ-07-0'-FD	Cadmium	0.081 mg/Kg	0.27U mg/Kg
	Niobium	3.5 mg/Kg	5.3U mg/Kg
	Lithium	12.0 mg/Kg	21.2U mg/Kg
TSB-HJ-07-10'	Niobium	3.3 mg/Kg	5.4U mg/Kg
	Lithium	20.6 mg/Kg	21.5U mg/Kg
TSB-HR-08-0'	Cadmium	0.099 mg/Kg	0.27U mg/Kg
	Niobium	3.1 mg/Kg	5.3U mg/Kg
	Lithium	9.4 mg/Kg	21.2U mg/Kg
TSB-HR-08-10'	Cadmium	0.076 mg/Kg	0.55U mg/Kg
	Niobium	4.9 mg/Kg	5.5U mg/Kg

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No field blanks were identified in this SDG.

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

The frequency of analysis was met.

The criteria for analysis were met.

### V. Matrix Spike Analysis

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

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
TSB-HR-08-0'MS/MSD (All samples in SDG F8A250221)	Antimony Phosphorus	60.6 (75-125) 31.3 (75-125)	54.7 (75-125) 62.5 (75-125)	-	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	A
TSB-HR-08-0'MS/MSD (All samples in SDG F8A250221)	Barium Calcium Chromium Lead Magnesium Niobium Silicon Strontium Vanadium Zinc	335.9 (75-125) 144.1 (75-125) 150.5 (75-125) 160.5 (75-125) 190.9 (75-125) 281.6 (75-125)	225.0 (75-125) 160.2 (75-125)		J+ (all detects) J+ (all detects)	A

### VI. Duplicate Sample Analysis

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

### VII. Laboratory Control Samples (LCS)

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

LCS ID	Analyte	%R (Limits)	Associated Samples	Flag	A or P
LCS	Palladium Platinum	120.5 (80-120) 121.9 (80-120)	All samples in SDG F8A250221	J+ (all detects) J+ (all detects)	Р

### VIII. Internal Standards (ICP-MS)

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

### IX. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

### X. ICP Serial Dilution

ICP serial dilution analysis was performed by the laboratory. The analysis criteria were met.

### XI. Sample Result Verification

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

### XII. Overall Assessment of Data

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

### XIII. Field Duplicates

Samples TSB-HJ-07-0'\*\* and TSB-HJ-07-0'-FD were identified as field duplicates. No metals were detected in any of the samples with the following exceptions:

	Concentr	ation (mg/Kg)		D.14		
Analyte	TSB-HJ-07-0'**	TSB-HJ-07-0'-FD	RPD (Limits)	Difference (Limits)	Flag	A or P
Aluminum	7780	8820	13 (≤50)	-	-	-
Arsenic	2.3	1.5	-	0.8 (≤2.2)	-	-
Barium	121	198	48 (≤50)	-	-	-
Beryllium	0.58	0.66	-	0.08 (≤1.1)	-	-
Cadmium	0.054U	0.081	-	0.027 (≤0.54)	-	-
Calcium	29900	13600	75 (≤50)	-	J (all detects)	Α

	Concentration (mg/Kg)		RPD	Difference		
Analyte	TSB-HJ-07-0'**	TSB-HJ-07-0'-FD	(Limits)	(Limits)	Flag	A or P
Chromium	8.2	9.8	-	1.6 (≤2.2)	-	-
Cobalt	6.1	6.0	2 (≤50)	-	-	-
Copper	16.7	17.5	-	0.8 (≤10.8)	-	
Iron	13000	14600	12 (≤50)	-	-	-
Lead	6.6	10.4	45 (≤50)	-	-	-
Magnesium	9270	7540	21 (≤50)	-	-	-
Manganese	282	402	35 (≤50)	-	-	-
Molybdenum	0.37	0.57	-	0.2 (≤1.1)	-	-
Nickel	14.0	13.5	4 (≤50)	-	-	-
Niobium	3.4	3.5	-	0.1 (≤5.4)	-	-
Palladium	0.33	0.42	-	0.09 (≤1.1)	-	-
Phosphorus	1350	1480	9 (≤50)	-	-	-
Potassium	1720	2530	38 (≤50)	-		-
Silicon	98.9	188	-	89.1 (54.1)	J (all detects)	A
Sodium	266	181	-	85 (≤217)	-	-
Strontium	157	189	18 (≤50)	-	-	-
Titanium	512	636	22 (≤50)	-	-	-
Uranium	0.93	0.82	-	0.11 (≤1.1)	-	-
Vanadium	32.1	40.2	22 (≤50)	-	-	-
Zinc	24.2	31.8	27 (≤50)	-	-	-

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	Concentration (mg/Kg)					
Analyte	TSB-HJ-07-0'**	TSB-HJ-07-0'-FD	RPD (Limits)	Difference (Limits)	Flag	A or P
Zirconium	16.5	21.3	-	4.8 (≤21.7)	-	-
Lithium	10.2	12.0	-	1.8 (≤21.7)	-	-

	Concentr	ation (ug/Kg)		D.//		
Analyte	TSB-HJ-07-0'**	TSB-HJ-07-0'-FD	RPD (Limits)	Difference (Limits)	Flag	A or P
Mercury	20.2	7.1U	-	13.1 (≤36.1)	-	-

### BRC Tronox Parcel H Metals - Data Qualification Summary - SDG F8A250221

SDG	Sample	Analyte	Flag	A or P	Reason
F8A250221	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HR-04-0'** TSB-HR-04-0'** TSB-HR-07-0' TSB-HR-07-0' TSB-HR-06-0' TSB-HR-06-10' TSB-HR-06-10' TSB-HJ-07-0'** TSB-HJ-07-0'-FD TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-10'	Antimony Phosphorus	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8A250221	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HR-04-0'** TSB-HR-04-0'** TSB-HR-07-0' TSB-HR-07-0' TSB-HR-06-0' TSB-HR-06-10' TSB-HR-06-10' TSB-HJ-07-0'** TSB-HJ-07-0'-FD TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-10'	Barium Calcium Chromium Lead Magnesium Niobium Silicon Strontium Vanadium Zinc	J+ (all detects) J+ (all detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8A250221	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HR-04-0'** TSB-HR-04-0'** TSB-HR-07-0' TSB-HR-07-10'** TSB-HR-06-0' TSB-HR-06-10' TSB-HR-06-10' TSB-HJ-07-0'** TSB-HJ-07-0'-FD TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-10'	Palladium Platinum	J+ (all detects) J+ (all detects)	Ρ	Laboratory control samples (%R)
F8A250221	TSB-HJ-07-0'** TSB-HJ-07-0'-FD	Calcium	J (all detects)	A	Field duplicates (RPD)
F8A250221	TSB-HJ-07-0'** TSB-HJ-07-0'-FD	Silicon	J (all detects)	A	Field duplicates (Difference)

### BRC Tronox Parcel H Metals - Laboratory Blank Data Qualification Summary - SDG F8A250221

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8A250221	TSB-HJ-05-10'	Cadmium	0.54U mg/Kg	A
F8A250221	TSB-HJ-05-0'	Cadmium Lithium	0.26U mg/Kg 21.0U mg/Kg	A
F8A250221	TSB-HR-04-10'	Niobium	6.6U mg/Kg	A
F8A250221	TSB-HJ-04-0'	Cadmium Niobium Lithium	0.55U mg/Kg 5.5U mg/Kg 21.8U mg/Kg	A
F8A250221	TSB-HR-04-0'**	Cadmium Niobium Tungsten Lithium	0.13U mg/Kg 5.2U mg/Kg 1.3U mg/Kg 20.9U mg/Kg	A
F8A250221	TSB-HJ-04-10'	Boron Cadmium Niobium	53.2U mg/Kg 0.27U mg/Kg 6.7U mg/Kg	A
F8A250221	TSB-HR-07-0'	Cadmium Niobium Lithium	0.27U mg/Kg 5.4U mg/Kg 21.4U mg/Kg	A
F8A250221	TSB-HR-07-10'**	Niobium	6.7U mg/Kg	А
F8A250221	TSB-HR-06-0'	Cadmium Niobium Lithium	0.28U mg/Kg 5.5U mg/Kg 22.2U mg/Kg	А
F8A250221	TSB-HR-06-10'	Cadmium Niobium	0.55U mg/Kg 5.5U mg/Kg	A
F8A250221	TSB-HJ-07-0'**	Niobium Lithium	5.4U mg/Kg 21.7U mg/Kg	A
F8A250221	TSB-HJ-07-0'-FD	Cadmium Niobium Lithium	0.27U mg/Kg 5.3U mg/Kg 21.2U mg/Kg	A
F8A250221	TSB-HJ-07-10'	Niobium Lithium	5.4U mg/Kg 21.5U mg/Kg	A

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8A250221	TSB-HR-08-0'	Cadmium Niobium Lithium	0.27U mg/Kg 5.3U mg/Kg 21.2U mg/Kg	A
F8A250221	TSB-HR-08-10'	Cadmium Niobium	0.55U mg/Kg 5.5U mg/Kg	A

### BRC Tronox Parcel H Metals - Field Blank Data Qualification Summary - SDG F8A250221

No Sample Data Qualified in this SDG

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LDC #:	18386A4	
SDG #:	F8A250221	
Labora	tory: Test America	

### VALIDATION COMPLETENESS WORKSHEET

Level III/IV

Date: <u>3151.8</u> Page: <u>of</u> Reviewer: <u>\_\_\_</u> 2nd Reviewer: <u>@ME</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
١.	Technical holding times	A	Sampling dates: 1/24/08
11.	Calibration	A	
111.	Blanks	5~	
IV.	ICP Interference Check Sample (ICS) Analysis	A	
V.	Matrix Spike Analysis	SW	2 MS MUSD
VI.	Duplicate Sample Analysis	٦, ١	
VII.	Laboratory Control Samples (LCS)	5W	204
VIII.	Internal Standard (ICP-MS)	A	Not versioned for level 3
IX.	Furnace Atomic Absorption QC	Ň	N.+ Utilies
Х.	ICP Serial Dilution	A	ð
XI.	Sample Result Verification	A	Not reviewed for Level III validation.
XII.	Overall Assessment of Data	A	
XIII.	Field Duplicates	52	(11, 12)
XIV.	Field Blanks	۲	

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

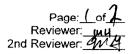
Validated Samples: \*\* Indicates sample underwent Level IV validation

1	TSB-HJ-05-10'	11	TSB-HJ-07-0'**	21	31	
2	TSB-HJ-05-0'	12	TSB-HJ-07-0'-FD	22	32	
3	TSB-HR-04-10'	13	TSB-HJ-07-10'	23	33	
4	TSB-HJ-04-0'	14	TSB-HR-08-0'	24	34	
5	TSB-HR-04-0'**	15	TSB-HR-08-10'	25	35	
6	TSB-HJ-04-10'	16	TSB-HR-08-0'MS	26	36	
7	TSB-HR-07-0'	17	TSB-HR-08-0'MSD	27	37	
8	TSB-HR-07-10'**	18	PB	28	38	
9	TSB-HR-06-0'	19		29	39	
10	TSB-HR-06-10'	20		30	40	

Notes:\_

LDC #: (8386 A4 SDG #: 40 www

### VALIDATION FINDINGS CHECKLIST



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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical fielding times		( <b>)</b> ; ()		
All technical holding times were met.	V	ļ	ļ	
Cooler temperature criteria was met.		L OF STATISTICS	100.50 MA	
II. Calibration				
Were all instruments calibrated daily, each set-up time?	1	ļ	ļ	
Were the proper number of standards used?				
Were all initial and continuing calibration verification %Rs within the 90-110% (80- 120% for mercury and 85-115% for cyanide) QC limits?	1		<b> </b>	
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.	5	915 491 201 311 4		
M. IGR Interference Check Sample	a ensen Second			
Were ICP interference check samples performed daily?	//			
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?				
IV-Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	~			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.		1		
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		<u>1</u> 17		
Was an LCS anaylzed for this SDG?	$\leq$			
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?		$\checkmark$		
W, Furnace Atomic Absorption QC	l y an Egenik			
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)			1	
Were analytical spike recoveries within the 85-115% QC limits?			/	

### VALIDATION FINDINGS CHECKLIST

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

Validation Area	Yes	No	NA	Findings/Comments
VII. ICR Senal Dilution			in die Autor	
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the IDL?	1			710 × Mor for replay
Were all percent differences (%Ds) < 10%?	/			
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.			/	
VIII. Internal Standards (EPA SW 846 Method 6020)		2 <b>4</b> (4		
Were all the percent recoveries (%R) within the 30-120% of the intensity of the internal standard in the associated initial calibration?	$\checkmark$	·		
If the %Rs were outside the criteria, was a reanalysis performed?				
IX. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?			~	· · · · · · · · · · · · · · · · · · ·
Were the performance evaluation (PE) samples within the acceptance limits?				
X. Sample Result Venfication				
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.	1			
XII: Field duplicates				
Field duplicate pairs were identified in this SDG.	~			
Target analytes were detected in the field duplicates.	-			
XIII. Field blanks				
Field blanks were identified in this SDG.		~		
Target analytes were detected in the field blanks.			~	

### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page:_	of
Reviewer:	hm
2nd reviewer:	9mA

All circled elements are applicable to each sample.

Sample ID	Matrix	Target Analyte List (TAL)
1-15	.1	(Al, Sb, As, Ba, Be, Cd, <u>Ca, Cr. Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si</u> ,)
		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,
m16,17	Sor	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	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,
1-15	Soi	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
216.17	50,-1	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U. Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Analysis Method
ICP		
ICP-MS		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, <u>Na, Tl, V, Zn, Mo, B, Si</u> ,
ICP-MS		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Zr,
GEAA		ALSh As Ba Be Cd Ca Cr Co Cu Fe Ph Mg Mn Hg Ni K Se Ag Na TI V Zn Mo B Si CN

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

LDC #: <u>18356A4</u> SDG #: <u>See Cover</u> <b>METHOD:</b> Trace Metals (EPA SW 846 Method 6010B/6020/7000) Sample Concentration units, unless otherwise noted: <u>mg/Kg</u>
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### VALIDATION FINDINGS WORKSHEET <u>PB/ICB/CCB QUALIFIED SAMPLES</u> Soil preparation factor applied: <u>ICP:100X, ICP/MS:200X, Hg:166.7X</u> Associated Sambas: A20 A.M.



AnalyteMaximun PBAI?.1AI3.1B2.4Ca14.0CdCdCr0.33				and the second se		A REAL OF A REAL WAY A REAL OF				sample Igenurication				
		Maximum PBª (ug/l)	Maximum ICB/CCB <sup>a</sup> (ug/L)	Blank Action I imit	-	N	£	4	5	9	2	8	6	10
	3.1													
	2.4									15.3 / 53.2				
	14.0													
			0.036		0.063 / 0.54	0.099 / 0.26		0.10 / 0.55	0.076 / 0.13	0.064 / 0.27	0.069 / 0.27		0.11 / 0.28	0.068 / 0.55
	0.33		0.5											
S			0.5											
Fe	5.6													
ï			0.5											
qN	1.4		6.8				5.5 / 6.6	4.8 / 5.5	3.3 / 5.2	5.7/6.7	4.0 / 5.4	4.4 / 6.7	3.8 / 5.5	3.4 / 5.5
-	3.0													
×	3.9													
Na	8.2													
			0.4											
0 ۲	0.067		0.6											
~			0.9						0.29 / 1.3					
Li			7.6			14.7/21.0		10.7 / 21.8	14.3 / 20.9		17.3/21.4		12.0 / 22.2	

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

56A4	e Cover	METHOD: Trace Metals (EPA SW 846 Method	
LDC #: 18356A4	SDG #: See Cover	METHOD: Trace Me	

# VALIDATION FINDINGS WORKSHEET PB/ICB/CCB QUALIFIED SAMPLES Soil preparation factor applied: ICP:100X. ICP/MS:200X. Hd:166.7X



2nd Reviewer: 2nd																							ple results were
2nd Review																							et. These sam
1																							ness Workshe
36.7X																							ition Complete
:200X, Hg:1(	tification																						from the Valida
100X, ICP/MS	Sample Identification	15				0.076 / 0.55					4.9 / 5.5												identifications t
Soil preparation factor applied: <u>ICP:100X, ICP/MS:200X, Hg:166.7X</u> Associated Samples: <u>6-4そ</u>		14				0.099 / 0.27 0					3.1 / 5.3							9.4 / 21.2					Samples with analyte concentrations within five times the associated ICB, CCB or PB concentration are listed above with the identifications from the Validation Completeness Worksheet. These sample results were
aration factor ed Samples: <u>6</u>		13				0					3.3 / 5.4							20.6 / 21.5	 			· · · · · · · · · · · · · · · · · · ·	tration are listed
Soil prepa Associate		12				0.081 / 0.27					3.5 / 5.3							12.0/21.2					3 or PB concen
/6020/7000) mg/Kg		11				0					3.4 / 5.4							10.2 / 21.7					ciated ICB, CCI
hod 6010B/ se noted: _		Blank Action Limit																					nes the asso
W 846 Met ess otherwi		Maximum ICB/CCBª (110/1.)				0.036	0.5	0.5		0.5	6.8				0.4	0.6	0.9	7.6					s within five tir
tals (EPA S in units, unl		Maximum PB <sup>a</sup> (110/1)						-															oncentrations
<b>METHOD</b> : Trace Metals (EPA SW 846 Method 6010B/6020/7000) Sample Concentration units, unless otherwise noted: <u>mg/Kg</u>		Maximum PBª (mơ/Kơ)	3.1	2.4	14.0		0.33		5.6		1.4	3.0	3.9	8.2		0.067							ith analyte co
<b>METHOD</b> Sample C		Analyte	AI	В	Ca	P	ບັ	ပိ	Fe	ïz	qN	٩	¥	Na	Щ	Ті	3			-			Samples v

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

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LDC #:	SDG #:

VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

216 5 Page: of Reviewer: 2nd Reviewer:

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

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". <u>Y N N/A</u> Was a matrix spike analyzed for each matrix in this SDG?

YN NA

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 NUNA

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

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

*	di QSM/SM	Matrix	Anaiyte	MS %Recovery	MSD %Recovery	RPD (Limits)	Associated Samples	OttalNicatione	
	16/17	50%	56	60, b	442		41	T-h.+/A	
			B€	123.6	9.e		112	141 H	1
			Cr Cr	33519	41.7			11411/A	
			Cr	1,441	134.7				
			gd	ک ، <i>ا</i> لح ا					-
			Ma	160.4					
			Nb 0	190,9	186.9				-
			<u>а</u>	31.3	62.5			T-/1.T/&	7
			12	2816	225.0				-
			للا ا	169.6	140.2				Y
			V	146.9	137.0				
			2 h	1314					
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## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

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

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". <u>M N/A</u> Was a laboratory control sample (LCS) analyzed for each matrix in this SDG? <u>Y M N/A</u> Were all aqueous LCS percent recoveries (%R) within the control limits of 80-120% and all soil LCS %R within laboratory established control limits.

...... ( Ware receipting and R N N/A Was <u>Y KN N/A</u> Wer <u>AEVEL IV ONLY:</u> <u>Y N N/A</u> Wer

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	Qualifications	14 14		À													
	Associated Samples	. 411		*													
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LDC#:\_<u>18386A4</u> SDG#:<u>See Cover</u>\_\_\_\_

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

<u>Yn na</u> Yn na Were field duplicate pairs identified in this SDG? Were target analytes detected in the field duplicate pairs?

	Concentratio	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Compound	11	12	RPD	Difference	Limits	(Parent Only)
Aluminum	7780	8820	13			
Arsenic	2.3	1.5		0.8	( ≤2.2)	
Barium	121	198	48			
Beryllium	0.58	0.66		0.08	( ≤1.1)	
Cadmium	0.054U	0.081		0.027	( ≤0.54)	
Calcium	29900	13600	75			J det / A
Chromium	8.2	9.8		1.6	( ≤2.2)	
Cobalt	6.1	6.0	2			
Copper	16.7	17.5		0.8	( ≤10.8)	
Iron	13000	14600	12			
Lead	6.6	10.4	45			
Magnesium	9270	7540	21			
Manganese	282	402	35			
Molybdenum	0.37	0.57		0.2	( ≤1.1)	
Nickel	14.0	13.5	4			
Niobium	3.4	3.5		0.1	( ≤5.4)	
Palladium	0.33	0.42		0.09	( ≤1.1)	
Phosphorus	1350	1480	9			
Potassium	1720	2530	38			

LDC#:<u>18386A4</u> SDG#:<u>See Cover</u>

### VALIDATION FINDINGS WORKSHEET Field Duplicates

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

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

<u>(P'N NA</u> YN NA Were field duplicate pairs identified in this SDG? Were target analytes detected in the field duplicate pairs?

	Concentratio	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Compound	11	12	RPD	Difference	Limits	(Parent Only)
Silicon	98.9	188		89.1	( ≤54.1)	J det / A
Sodium	266	181		85	( ≤217)	
Strontium	157	189	18			
Titanium	512	636	22			
Uranium	0.93	0.82		0.11	( ≤1.1)	
Vanadium	32.1	40.2	22			
Zinc	24.2	31.8	27			
Zirconium	16.5	21.3		4.8	( ≤21.7)	
Lithium	10.2	12.0		1.8	( ≤21.7)	
Mercury (ug/Kg)	20.2	7.1U		13.1	( ≤36.1)	

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LDC #: 18386A4 SDG #: 5e2 we

VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

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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 <u>measured</u> in the analysis of the ICV or CCV solution True = concentration (in ug/L) of each analyte in the ICV or CCV source	
%R = <u>Found</u> × 100 True	

Standard ID エムV ICP (I	Tune of Anshreis					REDUCED	
	I the of Autorians	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
	ICP (Initial calibration)	Л	كماود	40000	1-97	69-1	Y
GFA	GFAA (Initial calibration)						
TW CVA	CVAA (Initial calibration)	Ł41	۲.۲)	2-5	1001	1.001	٢
c U 10P (1	ICP (Continuing calibration)	L.	3995	4000	99.5	99.9	7
GFA	GFAA (Continuing calibration)						
Cev CVA	CVAA (Continuing calibration)	Ha	trob	tro.	10/~	2. [0]	λ
IW ICP/N	ICP/MS (Initial calibration)	5N	کر کر کر	cor	L'oc)	(, ۰۵۰)	_
ccv ICPIA	ICP/MS (Continuing calibation)	CU.	216.73	Cor	+،3c)	108.4	<u>_</u>

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

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Metals ss (%R) plicate	Vethod 6010/7				Ñ	2nd Reviewer: <sup>9</sup> 까성
Percent recoveries (%R) for an ICP inter %R = <u>Found</u> x 100 Where, Found = True A sample and duplicate relative percent	farence check	(000				
uplicate		sample, a laboratory con	trol sample and a matrix	spike sample were	recalculated using the	e following formula:
A sample and duplicate relative percent	Concentration of Found True = Concer	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.	analysis of the sample. For the SR (sample result). ource.	matrix spike calculation,		
	difference (RP	D) was recalculated using	g the following formula:			
RPD = <u>[S-D]</u> × 100 Where, (S+D)/2	S = Original sam D = Duplicate sa	S = Original sample concentration D = Duplicate sample concentration				
An ICP serial dilution percent difference (%D) was recalculated using the following formula:	(%D) was rec	alculated using the followi	ng formula:			
%D = <u>!!-SDR]</u> × 100 Where, 1 SDR = S	l = Initial Sample Result (mg/L) Serial Dilution Result (mg/L) (Instr	Where, I = Initial Sample Result (mg/L) SDR = Serial Dilution Result (mg/L) (Instrument Reading x 5)	(5)			
				Recalculated	Reported	
Sample ID Type of Analysis	Element	Found / S / I (units)	True / D / SDR (units)	%R / RPD / %D	%R / RPD / %D	Acceptable (Y/N)
TrisAR ICP interference check	ςb	97.93	(00	97.9	91.9	<u>}</u>
$\mathcal{LC} \mathcal{G}$ Laboratory control sample	ple L'	トロイ	0.02	(a, b)	( · f = )	
L h Matrix spike	Mo	g ft. fl p	53.079	p.01)	110.3	
16/17 Duplicate	(Hg	182	951	7.2	1,9	
( + ICP serial dilution	~¥	571773	<b>Ł</b> ttt.oS	د. لر	40	Y
Comments: Refer to appropriate worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.	heet for list of	gualifications and associe	ited samples when report	ted results do not a	gree within 10.0% of 1	the recalculated results.

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LDC #: 1838644 SDG #:

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

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

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". M N N/A Have results been reported and calculated correctly? Are results within the calibrated range of the instruments and within the linear range of the ICP? (Y) N N/A YN N/A Are all detection limits below the CRDL? were recalculated and verified using the

Detected analyte results for following equation:

Concentration =

Recalculation:

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

(RD)(FV)(Dil)

Mn = <u>s61.248/2 0.1ex 2</u> = 359.2 mg/mg

Sample ID	Analyte	Reported Concentration ( Wg/Vg)	Calculated Concentration ( Mg/kq )	Acceptable (Y/N)
t		14.3	14.3	Ч
	Al	1860	1860	
	As	1,3	1,3	
	Ba	145	145	
	Be	0.55	0.55	
· · · · · · · · · · · · · · · · · · ·	<u> </u>	0.076	0.076	
	(a	12600	1600	
	Cr	10,4	10.4	
	G	b-5	6.5	
	<u> </u>	16.4	lbit	
	Fe	12700	2700	
	Pb	1.0	1,0	
	Mg	7460	1460	
	lun lun	359	359	
	Mo	0.41	240	
	Vr	17.3	17.3	
	M_	٦, ٦	3.3	
	Pa	0.75	0,15	
	P	00	(000	
	K	1840	1840	
	Si	112	112	
	Na	203	203	J

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## LDC #: [8386AU SDG #: Let we Sample Colorier II III **Sample Calculation Verification**

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

<u>Y N N/A</u> Have results been r <u>X N N/A</u> Are results within th	eported and calculated corre	ot applicable questions are identified as "N/A". ctty? ruments and within the linear range of the ICP?
Detected analyte results for	5	were recalculated and verified using the

following equation:

<u>(RD)(FV)(Dil)</u> (In. Vol.)(%S) Concentration =

Recalculation:

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

V= 84.7394 ×0.1 × 2 0-59 × 0.959 = 35.34 mg/mg Reported Calculated Concentration Concentration Mg/Ky Acceptable Sample ID Analyte mylug) ( £ )  $(\dot{Y}/N)$ 7 5 5<u>v</u> 4 22 122 51 0.084 0.084 ĩ 5% 526 0.29 ŝ 0.29 0.90 U 0.90 N  $\frac{1}{2}$ 35.3 31.1 Zn 31.7 10g/m 40 13. Л 32 6

RECALC.4S2

## LDC Report# 18386B4

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: BRC Tronox Parcel H
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Collection Date: January 28, 2008

LDC Report Date: March 6, 2008

Matrix: Soil/Water

Parameters: Metals

Validation Level: EPA Level III

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8A290158

## Sample Identification

TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0'-FD TSB-HR-06-0'-FD TSB-HJ-08-0' TSB-HJ-08-10' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10' RINSATE-2 TSB-HR-05-10'MS TSB-HR-05-10'MSD RINSATE-2MS RINSATE-2MSD

## Introduction

This data review covers 11 soil samples and 3 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Methods 6010B, 6020, and 7000 for Metals. The metals analyzed were Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Lithium, Magnesium, Manganese, Molybdenum, Mercury, Nickel, Niobium, Palladium, Phosphorus, Platinum, Potassium, Selenium, Silicon, Silver, Sodium, Strontium, Sulfur, Thallium, Tin, Titanium, Tungsten, Uranium, Vanadium, Zinc, and Zirconium.

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

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

Blanks are summarized in Section III.

Field duplicates are summarized in Section XIII.

Raw data were not reviewed for this SDG. The review was based on QC data.

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. Calibration

An initial calibration was performed.

The frequency and analysis criteria of the initial calibration verification (ICV) and continuing calibration verification (CCV) were met with the following exceptions:

Date	Lab. Reference/ID	Analyte	%R (Limits)	Associated Samples	Flag	A or P
2/5/08	CCV (10:28)	Silver	111.4 (90-110)	PBW	J+ (all detects)	Р
2/5/08	CCV (21:53)	Boron Niobium Silver	112.3 (90-110) 111.8 (90-110) 112.6 (90-110)	All water samples in SDG F8A290158	J+ (all detects) J+ (all detects) J+ (all detects)	Ρ
2/6/08	CCV (1:47)	Silver	112.7 (90-110)	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' PBS	J+ (all detects)	Ρ
2/6/08	CCV (3.57)	Silver	112.4 (90-110)	TSB-HR-05-0' TSB-HR-05-10' TSB-HR-05-10'MS TSB-HR-05-10'MSD	J+ (all detects)	Ρ
2/6/08	CCV (18:59)	Palladium	113.6 (90-110)	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HJ-08-10' TSB-HR-05-0' PBS	J+ (all detects)	Ρ

## 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	Analytə	Maximum Concentration	Associated Samples
PB (prep blank)	Boron Cadmium Molybdenum Niobium Sodium Thallium Tin Titanium Tungsten	22.4 ug/L 0.029 ug/L 0.37 ug/L 20.1 ug/L 5.5 ug/L 1.4 ug/L 0.72 ug/L 0.80 ug/L 1.9 ug/L	All water samples in SDG F8A290158
ICB/CCB	Antimony Cadmium Molybdenum Niobium Titanium Tungsten	0.2 ug/L 0.1 ug/L 0.2 ug/L 6.1 ug/L 1.2 ug/L 0.6 ug/L	All water samples in SDG F8A290158
PB (prep blank)	Aluminum Barium Chromium Phosphorus Potassium Silver Sodium Thallium Tin Tin Titanium Zinc	1.9 mg/Kg 0.052 mg/Kg 0.15 mg/Kg 1.4 mg/Kg 1.5 mg/Kg 0.13 mg/Kg 0.073 mg/Kg 0.054 mg/Kg 0.054 mg/Kg 1.3 mg/Kg	All soil samples in SDG F8A290158
ICB/CCB	Boron Cadmium Niobium Potassium Thallium Tin Titanium Tungsten	10.6 ug/L 0.1 ug/L 6.1 ug/L 7.3 ug/L 0.5 ug/L 0.2 ug/L 0.9 ug/L 0.7 ug/L	All soil samples in SDG F8A290158

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
RINSATE-2	Cadmium	0.027 ug/L	0.50U ug/L
	Niobium	6.3 ug/L	25.0U ug/L
	Sodium	21.0 ug/L	50.0U ug/L
	Tin	0.51 ug/L	2.0U ug/L
	Titanium	1.0 ug/L	2.0U ug/L
	Tungsten	0.67 ug/L	5.0U ug/L

		Reported	Modified Final
Sample	Analyte	Concentration	Concentration
TSB-HJ-10-0'	Boron	10.4 mg/Kg	26.5U mg/Kg
	Cadmium	0.094 mg/Kg	0.13U mg/Kg
	Silver	0.072 mg/Kg	0.53U mg/Kg
	Tungsten	0.74 mg/Kg	1.3U mg/Kg
TSB-HJ-10-10'	Boron	6.7 mg/Kg	26.2U mg/Kg
	Cadmium	0.090 mg/Kg	0.13U mg/Kg
	Niobium	4.7 mg/Kg	6.6U mg/Kg
	Silver	0.092 mg/Kg	0.52U mg/Kg
	Tin	0.50 mg/Kg	0.52U mg/Kg
	Tungsten	0.46 mg/Kg	1.3U mg/Kg
TSB-HR-06-0'	Boron	4.6 mg/Kg	26.1U mg/Kg
130-114-00-0	Niobium	2.1 mg/Kg	6.5U mg/Kg
	Silver	0.090 mg/Kg	0.52U mg/Kg
	Tungsten	0.36 mg/Kg	1.3U mg/Kg
TSB-HR-06-0'-FD	Boron	3.9 mg/Kg	27.0U mg/Kg
	Cadmium	0.094 mg/Kg	0.14U mg/Kg
	Silver	0.094 mg/Kg	0.54U mg/Kg
	Tin	0.46 mg/Kg	0.54U mg/Kg
TSB-HR-06-10'	Boron	5.2 mg/Kg	26.6U mg/Kg
	Cadmium	0.077 mg/Kg	0.13U mg/Kg
	Silver	0.10 mg/Kg	0.53U mg/Kg
	Tin   Tungsten	0.47 mg/Kg 0.35 mg/Kg	0.53U mg/Kg 1.3U mg/Kg
	rungsten	0.55 mg/kg	1.50 mg/kg
TSB-HJ-08-0'	Boron	4.6 mg/Kg	27.0U mg/Kg
	Cadmium	0.10 mg/Kg	0.14U mg/Kg
	Silver	0.11 mg/Kg	0.54U mg/Kg
	Tin	0.52 mg/Kg	0.54U mg/Kg
	Tungsten	0.28 mg/Kg	1.4U mg/Kg
TSB-HJ-08-10'	Boron	E 0	07.011
130-03-00-10	Boron Cadmium	5.3 mg/Kg	27.0U mg/Kg
	Silver	0.10 mg/Kg 0.11 mg/Kg	0.14U mg/Kg 0.54U mg/Kg
	Tin	0.50 mg/Kg	0.540 mg/Kg 0.54U mg/Kg
	Tungsten	0.33 mg/Kg	1.4U mg/Kg
TSB-HR-05-0'	Cadmium	0.14 mg/Kg	0.54U mg/Kg
	Sodium	138 mg/Kg	218U mg/Kg
TSB-HR-05-10'	Boron	4.9 mg/Kg	26.8U mg/Kg
	Cadmium Silver	0.071 mg/Kg	0.13U mg/Kg
	Tin	0.11 mg/Kg 0.50 mg/Kg	0.54U mg/Kg 0.54U mg/Kg
	Tungsten	0.27 mg/Kg	1.3U mg/Kg
		1 V.27 D.9/119	noo ng/ng

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

Rinsate ID	Sampling Date	Analyte	Concentration	Associated Samples
RINSATE-2	1/28/08	Cadmium Calcium Iron Magnesium Niobium Sodium Strontium Tin Titanium Tungsten	0.027 ug/L 72.3 ug/L 32.9 ug/L 9.2 ug/L 6.3 ug/L 21.0 ug/L 0.67 ug/L 0.51 ug/L 1.0 ug/L 0.67 ug/L	All soil samples in SDG F8A290158

Sample concentrations were compared to concentrations detected in the field blanks. The sample concentrations were either not detected or were significantly greater (>5X blank contaminants) than the concentrations found in the associated field blanks with the following exceptions:

Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-HJ-10-0'	Cadmium	0.094 mg/Kg	0.13U mg/Kg
	Tungsten	0.74 mg/Kg	1.3U mg/Kg
TSB-HJ-10-10'	Cadmium	0.090 mg/Kg	0.13U mg/Kg
	Niobium	4.7 mg/Kg	6.6U mg/Kg
	Tin	0.50 mg/Kg	0.52U mg/Kg
	Tungsten	0.46 mg/Kg	1.3U mg/Kg
TSB-HR-06-0'	Niobium	2.1 mg/Kg	6.5U mg/Kg
	Tungsten	0.36 mg/Kg	1.3U mg/Kg
TSB-HR-06-0'-FD	Cadmium	0.094 mg/Kg	0.14U mg/Kg
	Tin	0.46 mg/Kg	0.54U mg/Kg
TSB-HR-06-10'	Cadmium	0.077 mg/Kg	0.13U mg/Kg
	Tin	0.47 mg/Kg	0.53U mg/Kg
	Tungsten	0.35 mg/Kg	1.3U mg/Kg
TSB-HJ-08-0'	Cadmium	0.10 mg/Kg	0.14U mg/Kg
	Tin	0.52 mg/Kg	0.54U mg/Kg
	Tungsten	0.28 mg/Kg	1.4U mg/Kg
TSB-HJ-08-10'	Cadmium	0.10 mg/Kg	0.14U mg/Kg
	Tin	0.50 mg/Kg	0.54U mg/Kg
	Tungsten	0.33 mg/Kg	1.4U mg/Kg
TSB-HR-05-0'	Cadmium	0.14 mg/Kg	0.54U mg/Kg
	Sodium	138 mg/Kg	218U mg/Kg

Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-HR-05-10'	Cadmium	0.071 mg/Kg	0.13U mg/Kg
	Tin	0.50 mg/Kg	0.54U mg/Kg
	Tungsten	0.27 mg/Kg	1.3U mg/Kg

## 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-HR-05-10'MS/MSD (All soil samples in SDG F8A290158)	Antimony	54.5 (75-125)	57.9 (75-125)	-	J- (all detects) UJ (all non-detects)	A
TSB-HR-05-10'MS/MSD (All soil samples in SDG F8A290158)	Barium	41.2 (75-125)	4.8 (75-125)	-	J- (all detects) R (all non-detects)	A
TSB-HR-05-10'MS/MSD (All soil samples in SDG F8A290158)	Niobium Palladium Magnesium	169.4 (75-125) 127.7 (75-125) -	210.0 (75-125) 128.3 (75-125) 131 (75-125)	-	J+ (all detects) J+ (all detects) J+ (all detects)	A

## VI. Duplicate Sample Analysis

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

## VII. Laboratory Control Samples (LCS)

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

LCS ID	Analyte	%R (Limits)	Associated Samples	Flag	A or P
LCS	Palladium	119.7 (85-115)	All water samples in SDG F8A290158	J+ (all detects)	Ρ

LCS ID	Analyte	%R (Limits)	Associated Samples	Flag	A or P
LCS	Platinum	124.4 (80-120)	All soil samples in SDG F8A290158	J+ (all detects)	P

## VIII. Internal Standards (ICP-MS)

Raw data were not reviewed for this SDG.

## 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-HR-05-10'L	Manganese Strontíum	10.1 (≤10) 10.6 (≤10)	All soil samples in SDG F8A290158	J (all detects) J (all detects)	A

## XI. Sample Result Verification

Raw data were not reviewed for this SDG.

## XII. Overall Assessment of Data

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

## XIII. Field Duplicates

Samples TSB-HR-06-0' and TSB-HR-06-0'-FD were identified as field duplicates. No metals were detected in any of the samples with the following exceptions:

	Concentr	ation (mg/Kg)				
Analyte	RPD TSB-HR-06-0' TSB-HR-06-0'-FD (Limits)			Difference (Limits)	Flag	A or P
Aluminum	7800	7880	1 (≤50)	-	•	-
Antimony	0.16	0.15	-	0.01 (≤1.4)	-	-
Arsenic	2.1	1.7	-	0.4 (≤2.7)	-	-

	Concentr	ation (mg/Kg)		D://			
Analyte	TSB-HR-06-0'	TSB-HR-06-0'-FD	RPD (Limits)	Difference (Limits)	Flag	A or P	
Barium	161	110	38 (≤50)	-	-	-	
Beryllium	0.49	0.56	-	0.07 (≤0.27)	-	-	
Boron	4.6	3.9	-	0.7 (≤27.0)	-	-	
Cadmium	0.14	0.094	-	0.046 (≤0.14)	-	-	
Calcium	10800	17100	45 (≤50)	-	-	-	
Chromium	8.9	13.3	40 (≤50)	•	-	-	
Cobałt	7.6	8.2	8 (≤50)	-	-	-	
Copper	14.5	14.3	1 (≤50)	-	-	-	
Iron	13100	12400	5 (≤50)	•	-	-	
Lead	9.4	7.6	21 (≤50)	-	-	-	
Magnesium	9570	9060	5 (≤50)	-	-	-	
Manganese	390	296	27 (≤50)	-	-	-	
Molybdenum	0.58	0.36	-	0.22 (≤1.4)	-	•	
Nickel	15.6	17.3	10 (≤50)	-	-	-	
Niobium	2.1	2.0U	-	0.1 (≤6.8)	-	-	
Palladium	0.22	0.21	-	0.01 (≤0.54)	-	-	
Phosphorus	1600	1250	25 (≤50)	-	-	-	
Potassium	1970	1960	1 (≤50)	-	-	-	
Silicon	194	83.7	-	110.3 (≤67.5)	J (all detects)	A	
Silver	0.090	0.094	-	0.004 (≤0.54)	-	-	

	Concentr	ation (mg/Kg)		D'#		
Analyte	TSB-HR-06-0'	TSB-HR-06-0'-FD	RPD (Limits)	Difference (Limits)	Flag	A or P
Sodium	232	184	-	48 (≤54.0)	-	-
Strontium	111	115	4 (≤50)	-	-	-
Tin	0.55	0.46	•	0.09 (≤0.54)	-	-
Titanium	623	488	24 (≤50)	-	-	-
Tungsten	0.36	0.27U	-	0.09 (≤1.4)	-	-
Uranium	0.72	0.66	-	0.06 (≤0.27)	-	-
Vanadium	34.2	34.4	1 (≤50)	-	-	-
Zinc	34.8	34.5	1 (≤50)	-	-	-
Zirconium	20.9	16.3	-	4.6 (≤27.0)	-	-
Lithium	5.7	3.2	-	2.5 (≤10.8)	-	-

Analyte	Concentration (ug/Kg)			D.14		
	TSB-HR-06-0'	TSB-HR-06-0'-FD	RPD (Limits)	Difference (Limits)	Flag	A or P
Mercury	7.0U	9.5	-	2.5 (≤36.0)	-	-

## BRC Tronox Parcel H Metals - Data Qualification Summary - SDG F8A290158

SDG	Sample	Analyte	Flag	A or P	Reason
F8A290158	RINSATE-2	Boron Niobium Silver	J+ (all detects) J+ (all detects) J+ (all detects)	Р	Calibration (%R)
F8A290158	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HJ-08-10' TSB-HR-05-10'	Silver	J+ (all detects)	Ρ	Calibration (%R)
F8A290158	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HJ-08-10' TSB-HR-05-0'	Palladium	J+ (all detects)	Ρ	Calibration (%R)
F8A290158	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HJ-08-10' TSB-HR-05-10'	Antimony	J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8A290158	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10'	Barium	J- (all detects) R (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8A290158	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10'	Niobium Palladium Magnesium	J+ (all detects) J+ (all detects) J+ (all detects)	A	Matrix spike/Matrix spike duplicates (%R)

SDG	Sample	Analyte	Flag	A or P	Reason
F8A290158	RINSATE-2	Palladium	J+ (all detects)	Ρ	Laboratory control samples (%R)
F8A290158	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10'	Platinum	J+ (all detects)	Ρ	Laboratory control samples (%R)
F8A290158	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10'	Manganese Strontium	J (all detects) J (all detects)	A	ICP serial dilution (%D)
F8A290158	TSB-HR-06-0' TSB-HR-06-0'-FD	Silicon	J (all detects)	A	Field duplicates (Difference)

## BRC Tronox Parcel H Metals - Laboratory Blank Data Qualification Summary - SDG F8A290158

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8A290158	RINSATE-2	Cadmium Niobium Sodium Tin Titanium Tungsten	0.50U ug/L 25.0U ug/L 50.0U ug/L 2.0U ug/L 2.0U ug/L 5.0U ug/L	A
F8A290158	TSB-HJ-10-0'	Boron Cadmium Silver Tungsten	26.5U mg/Kg 0.13U mg/Kg 0.53U mg/Kg 1.3U mg/Kg	A
F8A290158	TSB-HJ-10-10'	Boron Cadmium Niobium Silver Tin Tungsten	26.2U mg/Kg 0.13U mg/Kg 6.6U mg/Kg 0.52U mg/Kg 0.52U mg/Kg 1.3U mg/Kg	A

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8A290158	TSB-HR-06-0'	Boron Niobium Silver Tungsten	26.1U mg/Kg 6.5U mg/Kg 0.52U mg/Kg 1.3U mg/Kg	A
F8A290158	TSB-HR-06-0'-FD	Boron Cadmium Silver Tin	27.0U mg/Kg 0.14U mg/Kg 0.54U mg/Kg 0.54U mg/Kg	A
F8A290158	TSB-HR-06-10'	Boron Cadmium Silver Tin Tungsten	26.6U mg/Kg 0.13U mg/Kg 0.53U mg/Kg 0.53U mg/Kg 1.3U mg/Kg	A
F8A290158	TSB-HJ-08-0'	Boron Cadmium Silver Tin Tungsten	27.0U mg/Kg 0.14U mg/Kg 0.54U mg/Kg 0.54U mg/Kg 1.4U mg/Kg	A
F8A290158	TSB-HJ-08-10'	Boron Cadmium Silver Tin Tungsten	27.0U mg/Kg 0.14U mg/Kg 0.54U mg/Kg 0.54U mg/Kg 1.4U mg/Kg	A
F8A290158	TSB-HR-05-0'	Cadmium Sodium	0.54U mg/Kg 218U mg/Kg	A
F8A290158	TSB-HR-05-10'	Boron Cadmium Silver Tin Tungsten	26.8U mg/Kg 0.13U mg/Kg 0.54U mg/Kg 0.54U mg/Kg 1.3U mg/Kg	A

## BRC Tronox Parcel H Metals - Field Blank Data Qualification Summary - SDG F8A290158

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8A290158	TSB-HJ-10-0'	Cadmium Tungsten	0.13U mg/Kg 1.3U mg/Kg	A
F8A290158	TSB-HJ-10-10'	Cadmium Niobium Tin Tungsten	0.13U mg/Kg 6.6U mg/Kg 0.52U mg/Kg 1.3U mg/Kg	A

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8A290158	TSB-HR-06-0'	Niobium Tungsten	6.5U mg/Kg 1.3U mg/Kg	A
F8A290158	TSB-HR-06-0'-FD	Cadmium Tin	0.14U mg/Kg 0.54U mg/Kg	A
F8A290158	TSB-HR-06-10'	Cadmium Tin Tungsten	0.13U mg/Kg 0.53U mg/Kg 1.3U mg/Kg	A
F8A290158	TSB-HJ-08-0'	Cadmium Tin Tungsten	0.14U mg/Kg 0.54U mg/Kg 1.4U mg/Kg	A
F8A290158	TSB-HJ-08-10'	Cadmium Tin Tungsten	0.14U mg/Kg 0.54U mg/Kg 1.4U mg/Kg	A
F8A290158	TSB-HR-05-0'	Cadmium Sodium	0.54U mg/Kg 218U mg/Kg	A
F8A290158	TSB-HR-05-10'	Cadmium Tin Tungsten	0.13U mg/Kg 0.54U mg/Kg 1.3U mg/Kg	A

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## VALIDATION COMPLETENESS WORKSHEET Level III

SDG #: F8A290158 Laboratory: Test America

LDC #: 18386B4

### Date Page: $\cap$ Reviewer: 2nd Reviewer:

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
١.	Technical holding times	A	Sampling dates: 1/28/08
١١.	Calibration	4nd	
111.	Blanks	sw	
IV.	ICP Interference Check Sample (ICS) Analysis	A	
V.	Matrix Spike Analysis	5W	> MS/MSD
VI.	Duplicate Sample Analysis	N	· · · ·
VII.	Laboratory Control Samples (LCS)	5~	Lus
VIII.	Internal Standard (ICP-MS)	2	Nit veriened
IX.	Furnace Atomic Absorption QC	N	ipit verienced pit utilized
Х.	ICP Serial Dilution	SW	0
XI.	Sample Result Verification	N	
XII.	Overall Assessment of Data	A	
XIII.	Field Duplicates	SW	(3,4)
XIV.	Field Blanks	31	R=10

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:

Valida	ted Samples:	ere	nt # 10, 13, 14	As	
1	TSB-HJ-10-0'	11	TSB-HR-05-10'MS	21	31
2	TSB-HJ-10-10'	12	TSB-HR-05-10'MSD	22	32
3	TSB-HR-06-0'	13	RINSATE-2MS	23	33
4	TSB-HR-06-0'-FD	14	RINSATE-2MSD	24	34
5	TSB-HR-06-10'	15	PB	25	35
6	TSB-HJ-08-0'	16		26	36
7	TSB-HJ-08-10'	17		27	37
8	TSB-HR-05-0'	18		28	38
9	TSB-HR-05-10'	19		29	39
10	RINSATE-2	20		30	40

Notes:

LDC #: 18386134 SDG #: See con

F

## VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

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

All circled elements are applicable to each sample.

Sample ID	Matrix	Target Analyte List (TAL)
1-10	Si /m	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,
m-11,12	Sor	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,
113.14	A2	AI, Sb. As. Ba. Be. Cd. Ca. Cr. Co. Cu. Fe. Pb. Mg. MD. Hg. NI, K. Se, Ag, Na, TI, V. Zn, Mo, B, Sh
		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,
Ho	soil/m	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
n 11.12	Soil	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
1.13.14	M	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		(is)
ICP-MS		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Sj
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, TL V, Zn, Mo, B, Si, CN

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

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LDC #:	SDG #:

VALIDATION FINDINGS WORKSHEET **Calibration** 

Reviewer: 144 2nd Reviewer: 7n4 ð Page:

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

Plasse see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". <u>Y N N/A</u> Were all instruments calibrated daily, each set-up time, and were the proper number of standards used? <u>Y N N/A</u> Were all initial and continuing calibration verification percent recoveries (%R) within the control limits of 90-110% for all analytes except mercury (80-120%) and cyanide (85-115%)?

EVEL IV ONLY: Y N M/A

γ N N/A

Was a midrange cyanide standard distilled?

Are all correlation coefficients >0.995?

Were recalculated results acceptable? See Level IV Initial and Continuing Calibration Recalculation Worksheet for recalculations.

			Amelida	2%	Associated Samples	Qualification of Data
	1 2/2/0C	Calibration 10 / こく(しん)	Allaryle De,	ナミ	PBW	シキャン
1			<b>P</b>			
4	21815	UeV (>153)	ત	51211	A11 An	
1			ЧN	111.8		
<b></b>			Ac A	91211	<i>∧</i>	Y
1			R			
14	2 2 6/08	CeV (147)	Aq	C1211	1-1 PBS	J+ 47/10
<u>N</u>			ρ			
E	E 2/6/08	CcV ( 357)	A2	112.4	8.9 11.12	φ
1		1	þ		7	
	211108	ced (1849)	рd	1126	1 - 8, <del>28 /</del> 785	d/ft te
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ŏ	Comments:					

CAL 4SW

LDC #: 18356A4
SDG #: See Cover
METHOD: Trace Metals (EPA SW 846 Method 6010B/6020/7000)
Sample Concentration units, unless otherwise noted: mg/Kg

# VALIDATION FINDINGS WORKSHEET <u>PB/ICB/CCB QUALIFIED SAMPLES</u> Soil preparation factor applied: <u>ICP:100X, ICP/MS:200X, Hg:166.7X</u>



Analyte									Sample Id	Sample Identification				
	Maximum PBª (mg/Kg)	Maximum PB <sup>a</sup> (uq/L)	Maximum ICB/CCB <sup>a</sup> (ug/l.)	Blank Action I imit	-	2	e	4	5	9	7	8	6	
P	1.9													
Ba	0.052													
B			10.6		10.4 / 26.5	6.7 / 26.2	4.6 / 26.1	3.9 / 27.0	5.2 / 26.6	4.6 / 27.0	5.3 / 27.0		4.9 / 26.8	
Cd			0.1		0.094 / 0.13	0.090 / 0.13		0.094 / 0.14	0.077 / 0.13	0.10 / 0.14	0.10 / 0.14	0.14 / 0.54	0.071 / 0.13	
స	0.15													
qN			6.1			4.7/6.6	2.1/6.5							
đ	1.4													
¥	1.5		7.3											
Ag	0.13	-			0.072 / 0.53	0.092 / 0.52	0.090 / 0.52	0.094 / 0.54	0.10/0.53	0.11 / 0.54	0.11 / 0.54		0.11 / 0.54	
Na	3.4											138 / 218		
F	0.073		0.5											
Sn	0.054		0.2			0.50 / 0.52		0.46 / 0.54	0.47 / 0.53	0.52 / 0.54	0.50 / 0.54		0.50 / 0.54	
F	0.077		0.9											
N			0.7		0.74 / 1.3	0.46 / 1.3	0.36 / 1.3		0.35 / 1.3	0.28 / 1.4	0.33 / 1.4		0.27 / 1.3	
n D														
Zn	1.3													
									_					

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.

18386AS.wpd

LDC #: 18386A4 SDG #: <u>See Cov</u>	LDC #: 18386A4 SDG #: <u>See Cover</u>		HICH SKON				VALIDATION FINDINGS WORKSHEET <u>PB/ICB/ICCB QUALIFIED SAMPLES</u>	SHEET IPLES			Pa Revier
Sample (	METHOD: I race Metals (EFA SW 646 Metriod of 105/002) Sample Concentration units, unless otherwise noted: <u>ug/L</u>	als (EPA SV <u>1 units, unle</u>	v a4o Meun ss otherwis	e noted:	iguzur ruuu) ug/L	Associated Se	Soli preparation ractor appreu. Associated Samples: All AQ				
								Sample Ide	Sample Identification		
Analyte	Maximum PB <sup>a</sup> (mq/Kq)	Maximum PB <sup>a</sup> (10/1.)	Maximum ICB/CCB <sup>a</sup> (uq/l)	Blank Action I imit	9						
Sb			0.2								
В		22.4									
Cd		0.029	0.1		0.027 / 0.50						
Mo		0.37	0.2								
qN		20.1	6.1		6.3 / 25.0						
Na		5.5			21.0 / 50.0						
μ		1.4									
Sn		0.72			0.51 / 2.0						
ij		0.80	1.2		1.0 / 2.0						
3		1.9	0.6		0.67 / 5.0						

18386AW.wpd

## Page: of 2 Reviewer:

Samples with analyte concentrations within five times the associated ICB, CCB or PB concentration are listed above with the identifications from the Validation Completeness Worksheet. These sample results were qualified as not detected, "U". Note: a - The listed analyte concentration is the highest ICB, CCB, or PB detected in the analysis of each element.

LDC #: <u>18386A4</u> SDG #: <u>See Cover</u>

## VALIDATION FINDINGS WORKSHEET Field Blanks

2nd Reviewer: ر ور Page: (

METHOD: Trace Metals (EPA SW846 6010B/6020/7000)N N/AWere field blanks identified in this SDG?N N/AWere target analytes detected in the field blanks?N N/AWere target analytes detected in the field blanks?N N/ASolifactor appliedSampling date:1/28 /08Field blank type: (circle one) Field Blank / Rinsate / Other:

0.071 / 0.13 0.50 / 0.54 0.27 / 1.3 თ 0.14 / 0.54 138 / 218 ω 0.10/0.14 0.50 / 0.54 0.33 / 1.4 ~ 0.10/0.14 0.52 / 0.54 0.28 / 1.4 ဖ Associated Samples: All Soil Sample Identification 0.077 / 0.13 0.47 / 0.53 0.35 / 1.3 ŝ 0.094 / 0.14 0.46 / 0.54 4 0.36 / 1.3 2.1/6.5 ო 0.090 / 0.13 0.50 / 0.52 0.46 / 1.3 4.7./6.6 2 0.094 / 0.13 0.74 / 1.3 ~-Action Level Blank ID 0.027 72.3 32.9 21.0 0.67 0.67 0.51 9 9.2 6.3 1.0 Analyte Sa ğ g ร 8 Бе Na Š F ≥

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

N". Not applicable questions are identified as "V/A". X in this SDG? within the control limits of 75-125? If the sample concentration exceeded the spik differences (RPD) $\leq 20\%$ for water samples and $\leq 35\%$ for soil samples? differences (RPD) $\leq 20\%$ for water samples and $\leq 35\%$ for soil samples? I evel IV Recalculation Worksheet for recalculations. Bevel IV Recalculation Worksheet for recalculations. MS MS MS MS MS MS MS MS MS MS	METHOD: Trace metals (EPI SW 444 Method 6010/7000) Table analyzed for asserted Yr. Not applicable questions are identified as 'VA'. We an instrict spice analyzed for asserted Yr. This applicable questions blank virtual the SOF. YAN We all duplications asmithe relative percent differences (FPD) $\leq$ 20% for writer samples and $\leq$ 35% for soil samples? YAN We all duplicates asmithe relative percent differences (FPD) $\leq$ 20% for writer samples and $\leq$ 35% for soil samples? YAN We all duplicates asmithe relative percent differences (FPD) $\leq$ 20% for writer samples and $\leq$ 35% for soil samples? YAN We all duplicates asmithe relative percent differences (FPD) $\leq$ 20% for writer samples and $\leq$ 35% for soil samples? YAN We recalculated results acceptuale? See Lavel IV Fractional for the conclusted results acceptuale? We recalculated results asmither the form $\frac{1}{12}$								Reviewer: W
We can any set or the set of the	We native splete present eccent action by a factor with the source (RF) within the source (RF) within the source (RF) within the source (RF) of a monome (RF) $\leq 2\%$ for water samples and $\leq 35\%$ for soil sample of a monome (RF) $\leq 2\%$ for water samples and $\leq 35\%$ for soil sample of a monome (RF) $\leq 2\%$ for water samples and $\leq 35\%$ for soil samples (RF) within the source (RF) $\leq 2\%$ for water samples and $\leq 35\%$ for soil sample of a monome (RF) $\leq 2\%$ for water samples and $\leq 35\%$ for soil samples (RF) within the source (RF) $\leq 2\%$ for the sample relation worksheet for recalculation. We call cultures a monome (RF) $\leq 2\%$ for the recalculation worksheet for recalculation. We call cultures a monome (RF) $\leq 2\%$ for the recalculation of	ace metals (EPA lalifications belov Was a matriv v	SW 846 N w for all qu	lethod 601 lestions an	0/7000) swered "N". Not appli	icable questions ar	e identified as "N/A".		1
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	Were matrix sr of 4 or more, I Were all duplic ILY: Were recalcula	pike perce no action cate samp	it recoverié vas taken. le relative p acceptab	servent differences (F bercent differences (F le? See Level IV Rec	trol limits of 75-125 RPD) ≤ 20% for wa salculation Workshi	i? If the sample conc ter samples and <u>≤</u> 3! and for recelections	entration exceeded the 5% for soil samples?	e spike concentration by a fact
$ \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 $				SW	OSW			
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1 1 1 1 1 1 1 1 1 1 1 1 1 1	MD         (64,4         2/0,0         3/1         2/1         3/1           Mg         12/1,7         13/1         13/1         44,5200         No gas           MD         MD         13/1         13/1         44,5200         No gas           MD         MD         13/1         21,4         13/1         No gas           MD         MD         24         21,4         0         No gas           MD         MD         24         21,4         0         0           MD         MD         24         21,4         0         0           MD         MD         24         21,4         0         0           MD         MD         24         24         0         0           MD         MD         24         24         0         0			Ba	412	4.8		1 1	
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	to be client some							2	2
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	to be a wor client some		_						
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Page: <u> </u> o <sup>l</sup>   Reviewer: <u>에서</u> 2nd Reviewer: <i>에</i> 신台	nin laboratory established control limits.		Gualffications
VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)	Nease see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". N N/A Was a laboratory control sample (LCS) analyzed for each matrix in this SDG? Y N/A Were all aqueous LCS percent recoveries (%R) within the control limits of 80-120% and all soil LCS %R within laboratory established control limits. EVEL IV ONLY: X N/A Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations.	Associated Samnlee	
(000/	lifications below for all questions answered "N". Not applicable questions are id Was a laboratory control sample (LCS) analyzed for each matrix in this SDG? Were all aqueous LCS percent recoveries (%R) within the control limits of 80-1 Y: Were recalculated results acceptable? See Level IV Recalculation Worksheet	AR (limits)	11100001
د PA SW 846 Method 6	below for all questions operators operatory control sample aqueous LCS percent requeed results accept	Matrix Analyte	
LDC #: <u>  8 3 8 6 8 イ</u> SDG #: <u>5 6 9 co</u> ve METHOD: Trace Metals (EPA SW 846 Method 6010)	Please see qualifications t <u>Y M NA</u> Was a lab <u>Y M NA</u> Were all s LEFEL IV ONLY: <u>Y N NM</u> Were rec	# LCS ID	27

			( 120 - 120 ) A11 501 )												
		119.7(St-115) A11													
	Analyte	bd	¥			 _					-			-	
	Matrlx	₽ ₽	20r )												
· · · · · · · · · · · · · · · · · · ·	# LCS ID	13	27 202												Comments:

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See court LDC #: (8386 BY SDG #:

## VALIDATION FINDINGS WORKSHEET **ICP Serial Dilution**

ang j Reviewer:\_\_\_ Page: 2nd Reviewer:

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

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

If analyte concentrations were > 50X the MDL (ICP) ,or >100X the MDL (ICP/MS), was a serial dilution analyzed?

Were ICP serial dilution percent differences (%D) ≤10%? Is there evidence of negative interference? If yes, professional judgement will be used to qualify the data. M N/A Y N/N/A Y N/N/A

≻Ľ	K N/A	Were recalculated resul	ts acceptable?	See Level IV	Recalculation Wor	were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations.		
#	Date	Diluted Sample ID	Mạtrix	Analyte	%D (L imits)	Associated Samnles	Qualifications	-
		6	5° T )	٩'n	[p. ]	Ar 502	747	_
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ō	Comments:	Cu, the 1 (00 × 140 L	-Lahi					

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LDC#: 18386B4 SDG#: See Cover

## VALIDATION FINDINGS WORKSHEET Field Duplicates

Page: Reviewer: 2nd Reviewer: m

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

(<u>) n na</u> <u>Dn na</u>

Were field duplicate pairs identified in this SDG? Were target analytes detected in the field duplicate pairs?

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Compound	3	4	RPD	Difference	Limits	(Parent Only)
Aluminum	7800	7880	1			
Antimony	0.16	0.15		0.01	( ≤1.4)	
Arsenic	2.1	1.7		0.4	( ≤2.7)	
Barium	161	110	38			
Beryllium	0.49	0.56		0.07	( ≤0.27)	
Boron	4.6	3.9		0.7	( ≤27.0)	
Cadmium	0.14	0.094		0.046	( ≤0.14)	
Calcium	10800	17100	45			
Chromium	8.9	13.3	40			
Cobalt	7.6	8.2	8			
Copper	14.5	14.3	1			
Iron	13100	12400	5			
Lead	9.4	7.6	21			
Magnesium	9570	9060	5			
Manganese	390	296	27			
Molybdenum	0.58	0.36		0.22	( ≤1.4)	
Nickel	15.6	17.3	10		<u> </u>	
Niobium	2.1	2.0U		0.1	( ≤6.8)	
Palladium	0.22	0.21		0.01	( ≤0.54)	

## VALIDATION FINDINGS WORKSHEET Field Duplicates

Page: 2 of Reviewer: 2nd Reviewer: ME

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

<u>(P)n na</u> N NA Were field duplicate pairs identified in this SDG? Were target analytes detected in the field duplicate pairs?

	Concentration (mg/kg)		(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Compound	3	4	RPD	Difference	Limits	(Parent Only)
Phosphorus	1600	1250	25			
Potassium	1970	1960	1			
Silicon	194	83.7		110.3	( ≤67.5)	J det / A
Silver	0.090	0.094		0.004	( ≤0.54)	
Sodium	232	184		48	( ≤54.0)	
Strontium	111	115	4			
Tin	0.55	0.46		0.09	( ≤0.54)	
Titanium	623	488	24			
Tungsten	0.36	0.27U		0.09	( ≤1.4)	
Uranium	0.72	0.66		0.06	( ≤0.27)	
Vanadium	34.2	34.4	1			
Zinc	34.8	34.5	1			
Zirconium	20.9	16.3		4.6	( ≤27.0)	
Lithium	5.7	3.2		2.5	( ≤10.8)	
Mercury (ug/Kg)	7.0U	9.5		2.5	( ≤36.0)	

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## BRC Tronox Parcel H Data Validation Reports LDC# 18386

Wet Chemistry



## LDC Report# 18386A6

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	BRC Tronox Parcel H
Collection Date:	January 24, 2008
LDC Report Date:	March 7, 2008
Matrix:	Soil
Parameters:	Wet Chemistry
Validation Level:	EPA Level III & IV
Laboratory:	TestAmerica, Inc.

## Sample Delivery Group (SDG): F8A250221

## Sample Identification

TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HJ-04-0' TSB-HR-04-0'\*\* TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'\*\* TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'\*\* TSB-HJ-07-0'-FD TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-10' TSB-HJ-05-10'MS TSB-HJ-05-10'MSD TSB-HJ-05-10'DUP TSB-HR-08-0'MS TSB-HR-08-0'DUP

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 20 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, EPA Method 314.0 for Perchlorate, 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.

No field blanks were identified in this SDG.

## IV. Matrix Spike/Matrix Spike Duplicates

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

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
TSB-HR-08-0'MS (All samples in SDG F8A250221)	Oil & grease	71 (75-125)	-	-	J- (all detects) UJ (all non-detects)	А

## V. Duplicates

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

## VI. Laboratory Control Samples

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

## VII. Sample Result Verification

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

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

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

## IX. Field Duplicates

Samples TSB-HJ-07-0'\*\* and TSB-HJ-07-0'-FD were identified as field duplicates. No contaminant concentrations were detected in any of the samples with the following exceptions:

	Concentra	tion (mg/Kg)				
Analyte	TSB-HJ-07-0'**	TSB-HJ-07-0'-FD	RPD (Limits)	Difference (Limits)	Flag	A or P
Chloride	18.2	7.7	-	10.5 (≤2.2)	J (all detects)	А
Chlorine	36.4	15.3	-	21.1 (≤4.3)	J (all detects)	A
Fluoride	0.58 0.27U		-	0.31 (≤1.1)	-	-
Nitrate as N	0.66	0.77	-	0.11 (≤0.22)	-	-
Sulfate	8.9	6.8	-	2.1 (≤5.4)		-

	Concentra	ition (ug/Kg)				
Analyte	TSB-HJ-07-0'**	TSB-HJ-07-0'-FD	RPD (Limits)	Difference (Limits)	Flag	A or P
Perchlorate	8.6	12.8	-	4.2 (≤10.8)	-	-

## BRC Tronox Parcel H Wet Chemistry - Data Qualification Summary - SDG F8A250221

SDG	Sample	Analyte	Flag	A or P	Reason
F8A250221	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HR-04-0' TSB-HR-04-0'** TSB-HR-07-0' TSB-HR-07-10'** TSB-HR-06-10' TSB-HR-06-10' TSB-HJ-07-0'** TSB-HJ-07-0'-FD TSB-HJ-07-10' TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-10'	Oil & grease	J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8A250221	TSB-HJ-07-0'** TSB-HJ-07-0'-FD	Chloride Chlorine	J (all detects) J (all detects)	A	Field duplicates (Difference)

## BRC Tronox Parcel H

Wet Chemistry - Laboratory Blank Data Qualification Summary - SDG F8A250221

No Sample Data Qualified in this SDG

## BRC Tronox Parcel H Wet Chemistry - Field Blank Data Qualification Summary - SDG F8A250221

No Sample Data Qualified in this SDG

LDC #:18386A6	VALIDATION COMPLETENESS WORKSHEET	Date: <u>3/5/</u> •8
SDG #:		Page:
Laboratory: Test America		Reviewer:
		2nd Reviewer: MK

	TN	17	1	
METHOD: (Analyte) Bromide, Bromine, Chlorate, Chloride, Chorine, Fluoride,	Nitrate,	Nitrite, C	Orthophosphate-P	Sulfate (EPA
Method 300.0), Perchlorate (EPA Method 314.0), O & G (EPA SW846 Metho	od 7,091E	37		
	· · ·			

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: 1/24/08
lla.	Initial calibration	A	
llb.	Calibration verification	A	
Ш.	Blanks	4	
IV	Matrix Spike/Matrix Spike Duplicates	SW	> my/msp/pup
v	Duplicates	A	
VI.	Laboratory control samples	A	uspiso
VII.	Sample result verification	A	Not reviewed for Level III validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	52	C11,12)
x	Field blanks	N	

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

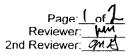
Validated Samples: \*\* Indicates sample underwent Level IV validation

1	, TSB-HJ-05-10'	11	TSB-HJ-07-0'**	21	MB	31	
2	TSB-HJ-05-0'	12	TSB-HJ-07-0'-FD	22		32	
3	TSB-HR-04-10'	13	TSB-HJ-07-10'	23		33	
4	TSB-HJ-04-0'	14	TSB-HR-08-0'	24		34	
5	TSB-HR-04-0'**	15	TSB-HR-08-10'	25	<u>.</u>	35	
6	TSB-HJ-04-10'	16	TSB-HJ-05-10'MS	26		36	
7	TSB-HR-07-0'	17	TSB-HJ-05-10'MSD	27		37	
8	TSB-HR-07-10'**	18	TSB-HJ-05-10'DUP	28		38	
9	TSB-HR-06-0'	19	TSB-HR-08-0'MS	29		39	
10	TSB-HR-06-10'	20	TSB-HR-08-0'DUP	30		40	

Notes:

Note:

### VALIDATION FINDINGS CHECKLIST



Method: Inorganics (EPA Method fee with				
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times			r Ás a	. TALER OF A DECK STRATE
All technical holding times were met.	~			
Coolor tomporaturo critoria was met.				
livealization	<b>*</b> #*	精整	he.	
Were all instruments calibrated daily, each set-up time?	1			
Were the proper number of standards used?	~			
Were all initial calibration correlation coefficients <a> 0.995?</a>	/			
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?	/			
Were titrant checks performed as required? (Level IV only)			/	
Were balance checks performed as required? (Level IV only)	arepsilon			
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.		/		
Withatos spike/Malmispike tuplicates and Duplicates (	677			。 新聞的 和 和 和 和 和 和 和 和 和 和 和 和 和
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.	1			
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.	K	$\checkmark$		
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.	/			
V Laboratory Control samples				
Was an LCS anaylzed for this SDG?				
Was an LCS analyzed per extraction batch?	1			
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 Odality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?			/	
Were the performance evaluation (PF) samples within the acceptance limits?				

### LDC # 18386 Ab SDG # <u>Sel cover</u>

### VALIDATION FINDINGS CHECKLIST

Page: Yof Y Reviewer: M9 2nd Reviewer: MA

Validation Area	Yes	No	NA	Findings/Comments
VII. Sample Result Ventication		ų at sr	ě. L	
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	~			
Were detection limits < RL?				
YIII SPAPAN CONTRACTOR STATES AND				ALLER LIE GALES IN
Overall assessment of data was found to be acceptable.	V			
Field duplicate pairs were identified in this SDG.	$\checkmark$			
Target analytes were detected in the field duplicates.	1			
Field blanks were identified in this SDG.		~	,	
Target analytes were detected in the field blanks.				

LDC #: 18386 Ab SDG # see over

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### VALIDATION FINDINGS WORKSHEET Sample Specific Analysis Reference

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All circled methods are applicable to each sample.

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Sample ID	Matrix	Parameter
1-15	507)	Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
u lb-18	Soi)	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>2</sub> O+G/TPH
19,00		Br) Bromine (C) Chlorine (B NO3 NO2 (SQ 0-PQ2 Chlorate CIO4 Q+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate 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 ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate 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 ClO <sub>4</sub> O+G/TPH

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

LDC #: 18386 Mb See cover SDG #:

VALIDATION FINDINGS WORKSHEET Matrix Spike Analysis

Reviewer: www. 2nd Reviewer: 9nd ا ح Page:

Lee Coner METHOD: Inorganics, Method Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A"

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

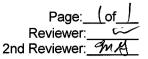
Were matrix spike percent recoveries (%R) within the control limits of 75-125 (85-115% for Method 300.0)? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken. LEVEL IV ONLY: Ϋ́

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

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*	Matrix Spike ID	Matrix	Analyte	%R	Associated Samples	Qualificatione
-	T5B-4J-02-101	502	504	<u></u>	<b>A</b> 11	No and
-						
T						rat = tox h th
T						with control 24
4	0	502	5+0	14	A1	T-/uT /4
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T						
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### VALIDATION FINDINGS WORKSHEET Field Duplicates



Inorganics, Method: See Cover

(<u>YN NA</u>

Were field duplicate pairs identified in this SDG? Were target analytes detected in the field duplicate pairs?

	Concentrat	ion (mg/Kg)				Qualification
Analyte	11	12	RPD (≤50)	Difference	Limits	(Parent only)
Chloride	18.2	7.7		10.5	(≤2.2)	J det / A
Chlorine	36.4	15.3		21.1	(≤4.3)	J det / A
Fluoride	0.58	0.27U		0.31	(≤1.1)	
Nitrate as N	0.66	0.77		0.11	(≤0.22)	
Perchlorate (ug/Kg)	8.6	12.8		4.2	(≤10.8)	
Sulfate	8.9	6.8		2.1	(≤5.4)	

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Validatin Findings Worksheet Initial and Continuing Calibration Calculation Verification

Reviewer: ਮਿ.ਕ 2nd Reviewer: ਆਟੀ Page: \_\_\_\_\_ of \_\_\_

 The correlation coefficient (r) for the calibration of  $\frac{a^{\delta}y}{\sqrt{2}}$  was recalculated.Calibration date:  $\frac{1}{\sqrt{2}}\sqrt{2}$ 

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

%R = Found X 100

True

Where,

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 or r²	r or r²	(VIN)
Initial calibration		۶۱	Ł	0.00316		-	
	CI04	s2	2.5	0.00918	0.9999372	0.999867	
		s3	Q	0.01935			7
		s4	10	0.04027			
		s5	20	0.07784			
		s6	40	0.15704			
رمی Calibration verification	clot	50	28, O		93.3	NR	X X
Calibration verification	cleart	4 000	3590.6		19.8	MP	
ددی Calibration verification	βγ	Orac	2019.0		101.45	(0).45 101,45	$\rightarrow$

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.

METHOD: Inorganics, Method       Set       Control         Percent recoveries (%A) for a laboratory control sample and a matix spike sample were recalculated using the following formula:         %A = Found =       concentration of each analyte in the analysis of the sample. For the matrix spike calculaton, Fund =         %A = Found =       concentration of each analyte in the source.         %A = Found =       concentration of each analyte in the source.         A sample and duplicate relative percent difference (RPD) was recalculated using the following termula:         A sample and duplicate relative percent difference (RPD) was recalculated using the following termula:         A sample and duplicate relative percent difference (RPD) was recalculated using the following termula:         A sample and duplicate relative percent difference (RPD) was recalculated using the following termula:         A sample and duplicate relative percent difference (RPD) was recalculated using the following termula:         A sample and duplicate relative percent difference (RPD)         A sample and duplicate rentation         RPD = [S,D]_{Z}         D unotative percent and the concentration         (B+D)]Z         D unotative control analysis         A sample up         A sample up         D unotative analysis         A sample         A sample up         A sample         A sample     <	LDC #: <u>0 100 (100)</u> SDG #: <u>500 co</u>	ser www		VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet	ugs workshe ation Workshee		2nd F	Page: of Reviewer: www 2nd Reviewer: CM/C
aboratory control sample Found = True = True = Percent difference (RPD S = S = D = Analysis Element Intol sample N 03-N ample of 4	METHOD: Inorga	nics, Method	e Cart					
Found = True = True = S = S = D = Analysis Element ntrol sample 0 1 4	Percent recoverie	s (%R) for a laboratory co	ntrol sample and	a metrix spike sampl	le were recalculate	d using the following	formula:	
$\begin{array}{l c c c c c c c c c c c c c c c c c c c$	%R = <u>Found</u> x 1 <sup>1</sup> True	Where	" II	sentration of each an nd = SSR (spiked sa sentration of each an	alyte <u>measured</u> in mple result) - SR (; alyte in the source.	the analysis of the s sample result).	ample. For the matri	x spike calculation
S = Original sample concentration       D = Duplicate sample concentration       Analysis     Element     Found / S     True / D       Analysis     Element     Found / S     True / D     Reactculeted     Reported       Analysis     Element     Found / S     True / D     Reactculeted     Reported       Analysis     Element     Found / S     True / D     Reactculeted     Reported       Analysis     Found / S     True / D     V     Y     P       Analysis     Found / S     True / D     Reactculeted     Reported       Analysis     Found / S     True / D     S     Y     P       Analysis     O 1 (1)     V     V     T     T       Analysis     O 1 (2)     V     T     T       Analysis     T     T     T     T       Analysis     T     T     T     T	A sample and dup	olicate relative percent diff	erence (RPD) wa	s recalculated using	the following form	la:		
Type of Analysis         Element         Found / S         Tue / D         Receivated         Receivated         Reported           Lubbratory control sample         N 05-N         4,05b         4,00         (units)         xsr, RPD         xsr, RPD           Matrix splike sample         N 05-N         4,05b         4,00         (2)         1.21           Duplicate sample         0.49         (.068         1.470         7)         7/           Duplicate sample         0.49         (.068         4.02         7)         7/	RPD = <u>!S-D!</u> × (S+D)/2			inal sample concentr licate sample concen	ation tration			
Type of AnalysisFigure 1Found (S (units)Tue (D (units)KR/ RPDKR / RPDLaboratory control sampleN $0_3$ -N $4$ , $c$ § 5 $4$ , $c$ ° ( $7$ ) $1 - 1$ Matrix spike sample0 + 4 $(c + 5)$ $4$ , $c$ ° ( $7$ ) $1 - 1$ Duplicate sample0 + 4 $(c - 8)$ $1 + 70$ $7$ ) $7$ Duplicate sampleC $4$ , $c - 2$						Receiculated	Reported	
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	Sample ID	Type of Analysis	Element	Found / S (units)	True / D (units)	%R / RPD	%R / RPD	Acceptable (Y/N)
S $Matrix epike sample N_{03-N}$ 4,056 4,00 (2) Matrix epike sample 014 (SSR-SR) (SSR-SR) (1470 7) Duplicate sample C 4,02 4,08 (.5		Laboratory control sample						-
Matrix spike sample of (ssR-sR) Duplicate sample C A A A A A A A A A A A A A A A A A A	Ľ		N 03-K	4,050	4,00	(c)	0	2
Duplicate sample Ce froz frog 7)		Matrix spike sample		(SSR-SR)				
2.7 J.			otg	& e°)	orth	17	/ 4	
		Duplicate sample		-	- -		51	
	d		Z	4.7	4, 08	7	X	>

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LDC #: 18386A6 SDG #: <u>Cee</u> co

VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	of
Reviewer:	МИ.
2nd reviewer:	MPS

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

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

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

Concentration =

**Recalculation:** 

G 4 - 0. 638× 40ml 0.174× 4g×0.919 = 53-65 mg/y

Soy = Aken X Ind John AXXX0.114 X Tuit. I WXX solid

#	Sample ID	Analyte	Reported Concentration ( Wy /y)	Calculated Concentration ( \\y\y\y)	Acceptable (Y/N)
1	5	chloute Q U2	6.3	6,3	Ý
		æ	130	(3)	
		l2	261	261	
		F	0.62	0-6/	
		NO3-N	27,2	27.1	
		F NOS-N Clay (Uglug) Gay	22200 53. y	22400	
		Gat'	53.9	53.7.	1
		<u>.</u>	/	,	
	7				
	······		····		
			<u> </u>		
	<u> </u>	<u> </u>	<u> </u>	<u> </u>	<u> </u>

Note:

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### LDC Report# 18386B6

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

Project/Site Name:	BRC Tronox Parcel H
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Collection Date: January 28, 2008

LDC Report Date: March 7, 2008

Matrix: Soil/Water

Parameters: Wet Chemistry

Validation Level: EPA Level III

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8A290158

### Sample Identification

TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10' **RINSATE-2** TSB-HJ-10-0'MS TSB-HJ-10-0'MSD TSB-HJ-10-0'DUP TSB-HJ-08-10'MS TSB-HJ-08-10'DUP TSB-HR-05-10'MS TSB-HR-05-10'DUP **RINSATE-2MS RINSATE-2MSD RINSATE-2DUP** 

### Introduction

This data review covers 16 soil samples and 4 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 300.0 for Bromide, Bromine, Chlorate, Chloride, Chorine, Fluoride, Nitrate as Nitrogen, Nitrite as Nitrogen, Orthophosphate as Phosphorus, and Sulfate, EPA Method 314.0 for Perchlorate, and EPA SW 846 Method 9071B and EPA Method 1664A 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.

Raw data were not reviewed for this SDG. The review was 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.

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

Rinsate ID	Sampling Date	Analyte	Concentration	Associated Samples
RINSATE-2	1/28/08	Sulfate	0.10 mg/L	All soil samples in SDG F8A250221

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

Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-HR-06-0'	Sulfate	4.8 mg/Kg	5.2U mg/Kg

### IV. Matrix Spike/Matrix Spike Duplicates

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

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Affected Analyte	Flag	A or P
TSB-HJ-08-10'MS (TSB-HJ-08-10' TSB-HR-05-0')	Chloride	57 (85-115)	-	-	Chloride Chlorine	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	A
TSB-HR-05-10'MS (All soil samples in SDG F8A290158)	Oil & grease	68 (75-125)	-	-	Oil and grease	J- (all detects) UJ (all non-detects)	A

### V. Duplicates

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

### VI. Laboratory Control Samples

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

### VII. Sample Result Verification

Raw data were not reviewed for this SDG.

### VIII. Overall Assessment of Data

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

### **IX. Field Duplicates**

Samples TSB-HR-06-0' and TSB-HR-06-0'-FD were identified as field duplicates. No contaminant concentrations were detected in any of the samples with the following exceptions:

	Concentra	tion (mg/Kg)				
Analyte	TSB-HR-06-0'	TSB-HR-06-0'-FD	RPD (Limits)	Difference (Limits)	Flag	A or P
Chloride	0.40	0.81	-	0.41 (≤2.2)	-	-
Chlorine	0.79	1.6	-	0.81 (≤4.3)	-	-
Nitrate as N	0.26	0.68	-	0.42 (≤0.22)	J (all detects)	A
Sulfate	4.8	15.4	-	10.6 (≤5.4)	J (all detects)	A

Analyte	Concentra	ation (ug/Kg)				
	TSB-HR-06-0'	TSB-HR-06-0'-FD	RPD (Limits)	Difference (Limits)	Flag	A or P
Perchiorate	1.9U	2.4	-	0.5 (≤10.8)	-	-

J.

### BRC Tronox Parcel H Wet Chemistry - Data Qualification Summary - SDG F8A290158

SDG	Sample	Analyte	Flag	A or P	Reason				
F8A290158	TSB-HJ-08-10' TSB-HR-05-0'	Chloride Chlorine	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)				
F8A290158	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HJ-08-10' TSB-HR-05-10'	Oil and grease	J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)				
F8A290158	TSB-HR-06-0' TSB-HR-06-0'-FD	Nitrate as N Sulfate	J (all detects) J (all detects)	A	Field duplicates (Difference)				

### **BRC Tronox Parcel H**

Wet Chemistry - Laboratory Blank Data Qualification Summary - SDG F8A290158

### No Sample Data Qualified in this SDG

### BRC Tronox Parcel H Wet Chemistry - Field Blank Data Qualification Summary - SDG F8A290158

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8A290158	TSB-HR-06-0'	Sulfate	5.2U mg/Kg	А

LDC #: 18386B6	VALIDATION COMPLETENESS WORKSHEET	Date: 3/5/08
SDG #:	Level III	Page:/_of/
Laboratory: Test America		Reviewer:
	-N 1	2nd Reviewer: MR

METHOD: (Analyte) Bromide, Bromine, Chlorate, Chloride, Chorine, Fluoride, Nitrate, Nitrite, Orthophosphate-P, Sulfate (EPA Method 300.0), Perchlorate (EPA Method 314.0), O & G (EPA SW846 Method 7091B)

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	Á	Sampling dates: 1/22/08
lla.	Initial calibration	A	
llb.	Calibration verification	A	
- 111.	Blanks	A	
١V	Matrix Spike/Matrix Spike Duplicates	SW	2 M5/M50/000
v	Duplicates	A	
VI.	Laboratory control samples	A	Les/LesD
VII.	Sample result verification	N	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	ŚW	(3,4)
x	Field blanks	SW	R=10

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: And Sin exert # 10, 18-20 A2

1	TSB-HJ-10-0'	11	TSB-HJ-10-0'MS	21	Mrs	31	
2 1	TSB-HJ-10-10'	12	TSB-HJ-10-0'MSD	22		32	
31	TSB-HR-06-0'	13	TSB-HJ-10-0'DUP	23		33	
4 1	TSB-HR-06-0'-FD	14	TSB-HJ-08-10'MS	24		34	
5	TSB-HR-06-10'	15	TSB-HJ-08-10'DUP	25		35	
61	TSB-HJ-08-0'	16	TSB-HR-05-10'MS	26		36	
$_{7}\checkmark$	TSB-HJ-08-10'	17	TSB-HR-05-10'DUP	27		37	
87	TSB-HR-05-0'	18	RINSATE-2MS	28		38	
<sub>9</sub> 1	TSB-HR-05-10'	19	RINSATE-2MSD	29		39	
10	RINSATE-2	20	RINSATE-2DUP	30		40	

Notes:\_

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Reviewer:	'w
2nd reviewer:	MA

All circled methods are applicable to each sample.

	Sample ID		Parameter
	1-10	Soil/AD	Br Bromine CI Chlorine F NO3 NO2 SO4 O-PO4 Chlorate CIO4 O+GTPH
			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
$\sim$	18:20	Ar	(Br) Bromine CI) Chlorine F/NO3 (NO2 SO4 0-PO4 Chlorate CIO4 0+G/TPH
	14-17	501	Br Bromine C) Chlorine (F) NO, SO Q-PO Chlorate CIO4 O+G/TPH
	16,17	1	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
	4-13	S	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> +G/TPH
	(8 * 19	AL	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> D+G/TPH
	· · ·		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
			Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
			Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
l			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
ļ			Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
			Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
			Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
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			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
ļ_			Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
			Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
			Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
			Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
			Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
			Br Bromine 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
L			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
F			
L			

Comments:

Page:	2nd Reviewer:														
ΈT		ation							Mication						a results were available on the day
VALIDATION FINDINGS WORKSHEET <u>Field Blanks</u>	Associated Samulae	Sample Identification						Associated Samples:	Sample identification					S NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMEN	outripies with analyte concentrations within five times the associated field blank concentration are listed above these semple restricted and the semple restricted activity of
VALIDATIO	anics, EPA Method <u>Xel</u> Could Were field blanks identified in this SDG? Were target analytes detected in the field blanks? <u>Market Associated sample units: writed</u> e: (circle one) Field Blank / Rinsate / Other: <u>R</u>			tr.				imple units: Soll factor applied k / Rinsate / Other:						I SULTS NOT CIRCLED WERE QUA	he associated field blank concentr
e la	anics, EPA Method <u>کور</u> Were field blanks identified in this SDG? Were target analytes detected in the fiel May ( Assoclated sample units: v Soil factor applie e: (circle one) Field Blank / Pinsate / Oth	Blank		14.8/5				Associated sample units; Soil factor a le one) Field Blank / Rinsate /	Blank	Limit				ון וסו מטאנודובם, אנו אבצ	strations within five times th
LDC #: 1838/18/6 SDG #: 722 LANU	METHOD: Inorganics, EPA Method <u>V N NA</u> Were field blanks identified in this SDG? <u>V N NA</u> Were target analytes detected in the field t Blank units: <u>where</u> Associated sample units: <u>where</u> Sampling date: <u>1, 2, 10, 5</u> Soil factor applied Field blank type: (circle one) Field Blank / Rinsate / Other.	Analyte Blank ID	10	010 705			dank mita.	Sampling date:Associated sample units Sampling date:Soil factor ε Field blank type: (circle one) Field Blank / Rinsate.	Analyte Blank ID					<u>dricted results were not qualified. All hesult</u>	MINDIES WILL ENERGIES CONCER

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LDC #: 18386 Bb SDG #: 500 WW

VALIDATION FINDINGS WORKSHEET Matrix Spike Analysis

Reviewer: w-Ъ Page: 2nd Reviewer:

METHOD: Inorganics, Method See Con

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

Was a matrix spike analyzed for each matrix in this SDG? Y D NA

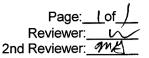
Were matrix spike percent recoveries (%R) within the control limits of 75-125 (85-115% for Method 300.0)? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.

LEVEL IV ONLY:

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

Matter         Analytic	alifications	4 ( que cet les		t,												
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$\begin{array}{c c} & \text{Analyte} & \\ \hline & & CC & \\ \hline \hline & CC & $	Associated Sa	1,8	A4 Co.									-				
	жп.	27	<u> </u>	800										-		
													-			
	# Matrix Spike ID		<b>x</b> :			 _	 							_	Comments:	

### VALIDATION FINDINGS WORKSHEET Field Duplicates



Inorganics, Method: See Cover



Were field duplicate pairs identified in this SDG? Were target analytes detected in the field duplicate pairs?

	Concentrati	on (mg/Kg)				Qualification
Analyte	3	. 4	RPD (≤50)	Difference	Limits	(Parent only)
Chloride	0.40	0.81		0.41	(≤2.2)	
Chlorine	0.79	1.6		0.81	(≤4.3)	
Nitrate as N	0.26	0.68		0.42	(≤0.22)	J det / A
Perchlorate (ug/Kg)	1.9U	2.4		0.5	(≤10.8)	
Sulfate	4.8	15.4		10.6	(≤5.4)	J det / A

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### BRC Tronox Parcel H Data Validation Reports LDC# 18386

Gasoline Range Organics



### LDC Report# 18386A7

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

Project/Site Name:	BRC Tronox Parcel H
Collection Date:	January 24, 2008
LDC Report Date:	March 11, 2008
Matrix:	Soil
Parameters:	Gasoline Range Organics
Validation Level:	EPA Level III & IV
Laboratory:	TestAmerica, Inc.

Sample Delivery Group (SDG): F8A250221

### Sample Identification

TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HJ-04-0' TSB-HR-04-0'\*\* TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'\*\* TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'\*\* TSB-HJ-07-0'-FD TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-10' TSB-HJ-05-10'MS TSB-HJ-05-10'MSD TSB-HR-08-0'MS TSB-HR-08-0'MSD

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 19 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8015 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.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. Calibration

### a. Initial Calibration

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

The percent relative standard deviations (%RSD) of calibration factors for all compounds were less than or equal to 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 difference (%D) of the second source calibration standard were less than or equal to 15.0% for all compounds.

### III. Blanks

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

No field blanks were identified in this SDG.

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

### a. Surrogate Recovery

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

Sample	Surrogate	%R (Limits)	Compound	Flag	A or P
TSB-HJ-07-0'-FD	a,a,a,-Trifluorotoluene	8.6 (21-146)	Gasoline range organics	J- (all detects) R (all non-detects)	A

### b. Matrix Spike/Matrix Spike Duplicates

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

### 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

Samples TSB-HJ-07-0'\*\* and TSB-HJ-07-0'-FD were identified as field duplicates. No gasoline range organics were detected in any of the samples.

### BRC Tronox Parcel H Gasoline Range Organics - Data Qualification Summary - SDG F8A250221

SDG	Sample	Compound	Flag	A or P	Reason
F8A250221	TSB-HJ-07-0'-FD	Gasoline range organics	J- (all detects) R (all non-detects)	A	Surrogate recovery (%R)

**BRC Tronox Parcel H** 

Gasoline Range Organics - Laboratory Blank Data Qualification Summary - SDG F8A250221

No Sample Data Qualified in this SDG

BRC Tronox Parcel H

Gasoline Range Organics - Field Blank Data Qualification Summary - SDG F8A250221

No Sample Data Qualified in this SDG

### VALIDATION COMPLETENESS WORKSHEET

LDC #: <u>18386A7</u> SDG #: <u>F8A250221</u> Laboratory: <u>Test America</u>

### Level III/IV

Date: 3/10/08 Page: \_\_\_\_\_\_\_ Reviewer: 2nd Reviewer

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METHOD: GC Gasoline Range Organics (EPA SW 846 Method 8015)

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	Ą	Sampling dates: 1200
lla.	Initial calibration	4	
llb.	Calibration verification/ICV	A	ICV = 15
.	Blanks	ND	
IVa.	Surrogate recovery	SW	
IVb.	Matrix spike/Matrix spike duplicates	SW	
IVc.	Laboratory control samples	А	LCS
V.	Target compound identification	Δ	Not reviewed for Level III validation.
VI.	Compound Quantitation and CRQLs	Δ	Not reviewed for Level III validation.
VII.	System Performance	Δ	Not reviewed for Level III validation.
VIII.	Overall assessment of data	4	
IX.	Field duplicates	ND	P= 11+12
Х.	Field blanks	N	

Note:

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

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

	Gol						
13	TSB-HJ-05-10'	11]	TSB-HJ-07-0'** -	21	8031129-BIK	31	\ اها،
2 <b>7</b>	TSB-HJ-05-0'	122	TSB-HJ-07-0'-FD	22 <sup>į</sup>	8030151-BIK	32	1/30
3 <b>7</b>	TSB-HR-04-10'	13 2	TSB-HJ-07-10'	23 <b>2</b>	8030149-BLK	33	1/29
43	TSB-HJ-04-0'	14P	TSB-HR-08-0'	24		34	· · · · · · · · · · · · · · · · · · ·
5 Z	TSB-HR-04-0'**	152	TSB-HR-08-10'	25		35	
6 2	TSB-HJ-04-10'	16 <b>3</b>	TSB-HJ-05-10'MS	26		36	
72	TSB-HR-07-0'	17 <b>3</b>	TSB-HJ-05-10'MSD	27		37	
82	TSB-HR-07-10'**	18 <b>7</b>	TSB-HR-08-0'MS	28		38	
92	TSB-HR-06-0'	19 <b>2</b>	TSB-HR-08-0'MSD	29		39	
10 V	TSB-HR-06-10'	20		30		40	

Notes:

### VALIDATION FINDINGS CHECKLIST

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

Method:GCHPLC			_	
Validation Area	Ye	s No	NA	Findings/Comments
1. Technical holding times	, sty			
All technical holding times were met.		-		
Cooler temperature criteria was met.	/	-		·
11. 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?	//	1		
IV Continuing 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	1		
Were all the retention times within the acceptance windows?	/			
V-Blanks			net iv	
Was a method blank associated with every sample in this SDG?	-	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.			-	
VI Surrogate spikes				
Were all surrogate %R within the QC limits?		/		
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?	/			
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?			/	
VII. Maltix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			
Was a MS/MSD analyzed every 20 samples of each matrix?				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?		7		
All. Laboratory control samples				
Nas an LCS analyzed for this SDG?	7	-	Ľ	
Vas an LCS analyzed per extraction batch?	7	-		
Vere the LCS percent recoveries (%R) and relative percent difference (RPD) vithin the QC limits?	/			

LDC #: 18386A7 SDG #: 41 Coner

### VALIDATION FINDINGS CHECKLIST

Page: 2 of 2 Reviewer: <u>P7</u> 2nd Reviewer: <u>1</u>

	T	T	T	r
Validation Area	Yes	No	NA	Findings/Comments
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	12			
Were the retention times of reported detects within the RT windows?		1.94.1.51		
		1. S.		
XI Compound duantitation/CRQLs				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?		-		
XII System performance				
System performance was found to be acceptable.			Ī	
XIII Overallassessment of data				
Overall assessment of data was found to be acceptable.				
XIV Field duplicates				
Field duplicate pairs were identified in this SDG.		T		
Farget compounds were detected in the field duplicates.		オ		
OV Field blanks				
ield blanks were identified in this SDG.		7		
arget compounds were detected in the field blanks.			イ	

LDC #: 18386A7 Cer SDG #: 11

## VALIDATION FINDINDS WORKSHEET Surrogate Recovery

∕of Reviewer: Page:

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

or No

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". Y N N/A. Were surrogates spiked into all samples and blanks?

	Qualifications																			Y Tetrachloro-m- vulene				
		-146 1 1-1R		(	(		) (	) (	(	) (	(		(	) [	(	) (	(	) (	Surrogate Compound	1-Chloro-3-Nitrobenzene	3.4-Dinitrotoluene	Tripentvltin	Trl-n-propyltin	Tributyl Phosphate
	(	7																		s	+		>	3
	%R (Limits)	8.6		)								~	<b>`</b>	<u></u>		)	~		Surrogate Compound	Benzo(e)Pyrene	Terphenyl-D14	Decachlorobiphenyl (DCB)	1-methylnaphthalene	Dichlorophenyl Acetic Acid (DCAA)
ns : nits?						+					+					_	 			۷	z	0	۵.	0
Did all surrogate recoveries (%R) meet the QC limits?	Surrogate Compound	iten b	V																Surrogate Compound	Octacosane	Ortho-Terphenyl	Fluorobenzene (FBZ)	n-Triacontane	Hexacosane
ecoveries	Detector/ Column		-																_	U	T			¥ .
ogate re		ton																	pu		BFB)		╉	
N/A Did all surr	Sample ID	21																	Surrogate Compound	Chlorobenzene (CBZ)	4-Bromofluorobenzene (BFB)	a,a.a-Trifluorotoluene	Bromochlorobenene	1.4-Dichlorobutane
	) #																			٩	8	U U		ш и

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

Page: \_\_of\_ £ Reviewer:

METHOD: GC HPLC Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A" Y M N/A Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? Y N/A Was an MS/MSD analyzed every 20 samples for each matrix or whenever a sample extraction was performed?

╢									and a second		ſ
)*		Compound	MS %R (Limits)	nits)	MSD %R (Limits)		RPD (Limits)		Associated Samples	Qualifications	
	1X 4 19	Bange Olganics		(1/2-02)	,		140 (30)	ر ن ا	14	w out	
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					)	$\widehat{}$	<u> </u>				
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LDC #: 18 3 80 47 9 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

		7 8.667									
Benord	098%	8.667				1					
Recalculated	Average CF (Initial)	15699364			•						
Reported	Average CF (initial)	156 op34									
Recalculated	CF (1.0 std)	62068131					·				
Reported	CF (1 - Ü std)	15 19902 3									
		gerour Hange Olopic									Commenter District 1 to 1 and 1 an
:	Calibration Date										
	Standard ID	1	·				. <u> </u>				ante: Dafa
	# -	-		2			m	Γ	 +	-	, amor

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LDC #: 18 356A7 Care 22 SDG #:

**Continuing Calibration Results Verification** VALIDATION FINDINGS WORKSHEET

Jol Page: 2nd Reviewer: Reviewer:

HPLC METHOD: GC\_

The percent differefice (%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

ave. CF = initial calibration average CF

Where:

- CF = continuing calibration CF A = Area of compound C = Concentration of compound

#Standard IDCalibration DateCompoundAverage CF(Ically CCV conc.1 $\mathcal{L}$ $1/3 \sigma   \mathcal{S}$ $\mathcal{G} \mathcal{R} \bigcup$ $1 \cdot O$ 2 $\mathcal{C} \vee$ $1/3 \sigma   \mathcal{S}$ $\mathcal{L}$ $1 \cdot O$ 3 $1 \psi \cdot S U$ $1/3 \sigma   \mathcal{S}$ $\mathcal{V}$ $1 \cdot O$ 3 $1 \psi \cdot S U$ $1/3 \sigma   \mathcal{S}$ $\mathcal{V}$ $1 \cdot O$	Reported			
standard ID     Calibration     Compound       CCV     1/30/8     GRU     Compound       Lbr32@     1/30/8     Lbr32@     L       Lbr32@     1/30/8     L     L		Recalculated	Reported	Recalculated
cu umase cev 1/30/8 GRU Cev 1/30/8 V Umase 14:51 Umase	Ige CF(Ical)/ CF/Conc.	CF/Conc.	0%	۵%
11-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1	╢	╢		
ccv 1/20/8 1/	0.9486	0.0486	S.)	
Cev 1/320/8 2				,
Cev 1/30/8 1/1 14:54				
Ceve 1/30/201		· ·		
	129120		1 1	c/ ,
╆╍╢╌╍╁╸┽╶╢┈		8 511-0	هرو	\$`\$
		•		
			-	
4				

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. Comments:

LDC #: 18386A) SDG #: I'V CON

## VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

ć of 2nd reviewer: Page: Reviewer:\_\_\_

METHOD: CC HPLC

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Sample ID: 22-5

Spiked	Column/Detector Spiked
	. 1
922200	
-	
	[

Sample ID:

	Reported		Difference
		Recalculated	
		-	

Sample ID:

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

SURRCALCNew.wpd

642	coner
18380A	3
#	#
LDC	SDG

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

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

g **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 HPLC

the following calculation: %Recovery = 100 \* (SSC - SC)/SA

Where

RPD =(({SSCMS - SSCMSD} + 2) / (SSCMS + SSCMSD))+100 18119 MS/MSD samples:

l

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

SC = Sample concentration

MSD = Matrix spike duplicate

		Spike	Sample	Spike	Spike Sample	Matrix	Matrix spike	Matrix Solke Dunlicate	e Dunlicate	MeMen	60
Compound	\$ ~	Depony	Conc.	Conce	ntration					E O E	20
		4			7 21	Percent	Percent Recovery	Percent Recovery	Recovery	RPD	0
	WS	MSD		WS	MSD	Reported	Recalc.	Reported	Recalc	Denoted	R
Gasoline (8015)	90.1	10.1	0	0.115 2440	0. 9 94	ŗ	ŗ		lander.	Dellodev	Kecalc.
Diesel (8015)					-	-	-	+	7	22	0 X 1
Benzene (8021B)											
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)											
Anthracene (8310)											
HMX (8330)											
2,4,6-Trinitrotoluene (8330)	6										
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%.	x Spike/Matr	rix Spike Dur	<u>ilicates finding</u>	s worksheet f	or list of gualifi	ications and a	ssociated sam	ples when rep	) Dorted results	do not agree	within 10.0%
AT HIS LOSSISHINGED LESUIS										22.82.22.2	

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	Labo
LDC #: 18386 A7	SDG #: In comen

DC HPLC

METHOD:

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



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

LPD =(({ssclcs - ssclcsd} \* 2) / (ssclcs + ssclcsd))\*100

LCS/LCSD samples: 8030 5 1 - し い

LCSD = Laboratory Control Sample duplicate percent recovery

	Spike	é	Sample	Spike S	ample	LCS LCS	ŝ	rcsD	0	LCS/LCSD	SD
Compound	( my		Conc.	Concentration	tration Ky	Percent Recovery	tecovery	Percent Recovery	ecovery	RPD	
	LCS	LCSD	> • •	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)	1.0	ΑM	0	126.0	47	26	46	NA -			
Diesel (8015)											
Benzene (8021B)											
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)											
Anthracene (8310)											
HMX (8330)											
2,4,6-Trinitrotoluene (8330)											
Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicate findings worksheet for list of qualifications and associated samples when reported	ory Control	Sample/L	aboratory Co	ntrol Sample	Duplicate fine	dings workshe	set for list of c	qualifications a	and associate	ed samples wt	en reported

LCSCLCNew.wpd

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

SAMPCALew.wpd

### **LDC Report#** 18386B7

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

Project/Site Name:	BRC Tronox Parcel H
Collection Date:	January 28, 2008
LDC Report Date:	March 11, 2008
Matrix:	Soil/Water
Parameters:	Gasoline Range Organics
Validation Level:	EPA Level III
Laboratory:	TestAmerica, Inc.

Sample Delivery Group (SDG): F8A290158

### Sample Identification

TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0'-FD TSB-HR-06-0'-FD TSB-HJ-08-0' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10' RINSATE-2 TSB-HR-05-10'MS TSB-HR-05-10'MSD RINSATE-2MS RINSATE-2MSD

### Introduction

This data review covers 11 soil samples and 3 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8015 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.

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. Calibration

### a. Initial Calibration

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

The percent relative standard deviations (%RSD) of calibration factors for all compounds were less than or equal to 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 difference (%D) of the second source calibration standard were less than or equal to 15.0% for all compounds.

### III. Blanks

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

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

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

### a. Surrogate Recovery

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

Sample	Surrogate	%R (Limits)	Compound	Flag	A or P
RINSATE-2	a,a,a,-Trifluorotoluene	153 (66-150)	Gasoline range organics	J+ (all detects)	Р

### 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

Raw data were not reviewed for this SDG.

### VI. Compound Quantitation and CRQLs

Raw data were not reviewed for this SDG.

### VII. System Performance

Raw data were not reviewed for this SDG.

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

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

### **IX. Field Duplicates**

Samples TSB-HR-06-0' and TSB-HR-06-0'-FD were identified as field duplicates. No gasoline range organics were detected in any of the samples.

### BRC Tronox Parcel H Gasoline Range Organics - Data Qualification Summary - SDG F8A290158

SDG	Sample	Compound	Flag	A or P	Reason
F8A290158	RINSATE-2	Gasoline range organics	J+ (all detects)	Р	Surrogate recovery (%R)

**BRC Tronox Parcel H** 

Gasoline Range Organics - Laboratory Blank Data Qualification Summary - SDG F8A290158

No Sample Data Qualified in this SDG

BRC Tronox Parcel H

Gasoline Range Organics - Field Blank Data Qualification Summary - SDG F8A290158

No Sample Data Qualified in this SDG

### VALIDATION COMPLETENESS WORKSHEET Level III

LDC #: 18386B7 SDG #: F8A290158

### Laboratory: Test America

Date: <u>3/6/08</u> Page: <u>/of /</u> Reviewer: <u>1</u> 2nd Reviewer: \_\_\_\_\_

METHOD: GC Gasoline Range Organics (EPA SW 846 Method 8015)

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

	Validation Area		Comments
· · ·	Technical holding times	A	Sampling dates: 1/18/09
lla.	Initial calibration	4	
llb.	Calibration verification/ICV	A	$101 \leq 15$
111.	Blanks A	-571	
IVa.	Surrogate recovery	SW	
IVb.	Matrix spike/Matrix spike duplicates	Δ	
IVc.	Laboratory control samples	A	103
V.	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	Ŋ	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	ND	p = 3 + 4
<b>X</b> .	Field blanks	ND	R = 0

Note:

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

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ND = No compounds detected R = Rinsate FB = Field blank D = Duplicate TB = Trip blank EB = Equipment blank

Validated	Samp	les	:	
		-	~	

	<u>coil t v</u>	Sau	-				
1	TSB-HJ-10-0'	11	TSB-HR-05-10'MS	21	8037078	31	
2	TSB-HJ-10-10'	12	TSB-HR-05-10'MSD	22	8037174	32	
3	TSB-HR-06-0'	13	RINSATE-2MS	23	80 390 35	33	
4	TSB-HR-06-0'-FD	14	RINSATE-2MSD W	24		34	
5	TSB-HR-06-10'	15		25		35	
6	TSB-HJ-08-0'	16		26		36	
7	TSB-HJ-08-10'	17		27		37	
8	TSB-HR-05-0'	18		28		38	
9	TSB-HR-05-10'	19		29		39	
10	RINSATE-2 W	20		30		40	

Notes:

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DC #: /8	SDG #: 1

## VALIDATION FINDINDS WORKSHEET Surrogate Recovery



₹ GC

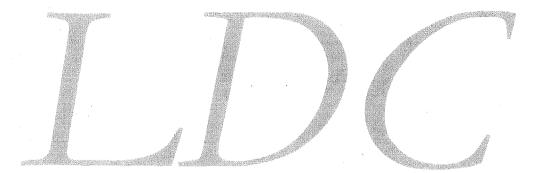
METHOD: //GC \_\_\_\_\_HPLC Are surrogates required by the method? Yes\_\_\_\_\_or No\_\_\_\_\_. Physe see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Tetrachloro-m- xylene Qualifications ≻ Surrogate Compound 1-Chloro-3-Nitrobenzene Triphenvl Phosphate Tributyl Phosphate 3,4-Dinitrotoluene Tri-n-propyltin Tripentyltin 951-97 S ≥  $\supset$ F > %R (Limits) Dichlorophenył Acetic Acid (DCAA) Surrogate Compound Decachlorobiphenyl (DCB) 1-methvinaphthalene. Benzo(e)Pyrene Terphenyl-D14 4-Nitropheno ŝ 5 σ Σ z 0 ٩ œ Were surrogates spiked into all samples and blanks? Did all surrogate recoveries (%R) meet the QC limits? Surrogate Compound Surrogate Compound Fluorobenzene (FBZ c)Ortho-Terphenyl Bromobenzene n-Triacontane Hexacosane Octacosane 2 10700 Detector/ Column ഗ I ¥ 104 4-Bromofluorobenzene (BFB) Surrogate Compound .4-Difluorobenzene (DFB) Chlorobenzene (CBZ) a,a,a-Trifluorotoluene Bromochlorobenene 1,4-Dichlorobutane Sample <u>0</u> ٥ Y N N/A # മ q υ w

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### BRC Tronox Parcel H Data Validation Reports LDC# 18386

Diesel Range Organics



### LDC Report# 18386A8

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

Project/Site Name:	BRC Tronox Parcel H
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Collection Date: January 24, 2008

LDC Report Date: March 11, 2008

Matrix:

Parameters: Diesel Range Organics

Soil

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8A250221

### Sample Identification

TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HJ-04-0' TSB-HR-04-0'\*\* TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'\*\* TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'\*\* TSB-HJ-07-0'-FD TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-10' TSB-HR-08-0'MS TSB-HR-08-0'MSD

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 17 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8015 for Diesel Range Organics.

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

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. Calibration

### a. Initial Calibration

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

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

### **b.** Calibration Verification

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

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

### III. Blanks

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

No field blanks were identified in this SDG.

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

### a. Surrogate Recovery

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

Sample	Surrogate	%R (Limits)	Compound	Flag	A or P
TSB-HR-06-10'	ortho-Terphenyl	65 (73-150)	Diesel range organics	J- (all detects) UJ (all non-detects)	A
TSB-HR-08-10'	ortho-Terphenyl	66 (73-150)	Diesel range organics	J- (all detects) UJ (all non-detects)	A

### 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

Samples TSB-HJ-07-0'\*\* and TSB-HJ-07-0'-FD were identified as field duplicates. No diesel range organics were detected in any of the samples.

### BRC Tronox Parcel H Diesel Range Organics - Data Qualification Summary - SDG F8A250221

SDG	Sample	Compound	Flag	A or P	Reason
F8A250221	TSB-HR-06-10' TSB-HR-08-10'	Diesel range organics	J- (all detects) UJ (all non-detects)	A	Surrogate spikes (%R)

BRC Tronox Parcel H

Diesel Range Organics - Laboratory Blank Data Qualification Summary - SDG F8A250221

No Sample Data Qualified in this SDG

BRC Tronox Parcel H

Diesel Range Organics - Field Blank Data Qualification Summary - SDG F8A250221

No Sample Data Qualified in this SDG

VALIDATION COMPLETENESS WORKSHEET
Level III/IV

Date:	<u>3/7</u> /08
Page: 7	of <u>7</u> ′
Reviewer:	P
2nd Reviewer:	

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Laboratory: Test America

LDC #: 18386A8

SDG #: F8A250221

METHOD: GC Diesel Range Organics (EPA SW 846 Method 8015)

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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: 12409
lla.	Initial calibration	Δ	
IIb.	Calibration verification/ICV	Δ	101 5
III.	Blanks	4	\$
IVa.	Surrogate recovery	لمري	
IVb.	Matrix spike/Matrix spike duplicates	Δ	
IVc.	Laboratory control samples	А	LCS
V.	Target compound identification	<u> </u>	Not reviewed for Level III validation.
VI.	Compound Quantitation and CRQLs	$\land$	Not reviewed for Level III validation.
VII.	System Performance	4	Not reviewed for Level III validation.
VIII.	Overall assessment of data		
IX.	Field duplicates	NP	p = 11 + 12
<b>X</b> .	Field blanks	N	

Note:

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

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

-	SOL						
1	TSB-HJ-05-10'	11	тѕв-нј-07-0'** 👔	21	8029395	31	
2	TSB-HJ-05-0'	12	тѕв-нј-07-0'-FD р	22		32	
3	TSB-HR-04-10'	13	TSB-HJ-07-10'	23		33	
₩ <b>*</b> 4	TSB-HJ-04-0'	14	TSB-HR-08-0'	24		34	
5	TSB-HR-04-0'**	15	TSB-HR-08-10'	25		35	
1 <sub>6</sub>	TSB-HJ-04-10'	16	TSB-HR-08-0'MS	26		36	
<b>†</b> 7	TSB-HR-07-0'	17	TSB-HR-08-0'MSD	27		37	
8	TSB-HR-07-10'**	18		28		38	
g	TSB-HR-06-0'	19		29		39	
10	TSB-HR-06-10' 🗸	20		30		40	

Notes:

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### VALIDATION FINDINGS CHECKLIST

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

Method:GCHPLC				
Validation Area	Yes	No	NA	Findings/Comments
1. Technical holding times				
All technical holding times were met.	/			
Cooler temperature criteria was met.	-			<u>.</u>
11. Initial calibration:	$\tilde{T}_{ij}$			
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?		v	/	
Were the RT windows properly established?		- 1		
IV: Continuing calibration			4.59	
What type of continuing calibration calculation was performed?%D or%R	1	-		
Was a continuing calibration analyzed daily?	1	-		
Were all percent differences (%D) $\leq$ 15%.0 or percent recoveries 85-115%?				
Were all the retention times within the acceptance windows?		-		
V. Blanks				
Was a method blank associated with every sample in this SDG?	1		Τ	
Was a method blank analyzed for each matrix and concentration?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
VI Surrogate spikes		ça İ	140	
Were all surrogate %R within the QC limits?			Τ	
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	-
VII. Mattix 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?	7			
Nere the MS/MSD percent recoveries (%R) and the relative percent differences RPD) within the QC limits?	7			
/III. Laboratory control samples		4		
Vas an LCS analyzed for this SDG?	F	Γ		
Vas an LCS analyzed per extraction batch?	T			
Vere the LCS percent recoveries (%R) and relative percent difference (RPD) ithin the QC limits?	7			

LDC #: 18 SDG #: \_\_\_\_

### VALIDATION FINDINGS CHECKLIST

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

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Validation Area	Yes	No	NA	Findings/Comments
IX. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				F
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 and dry weight factors applicable to level IV validation?	$\Box$	-		
XIII System performance				and the part of the second second
System performance was found to be acceptable.	T	- 1	T	
XIII Overallessessment of data				
Overall assessment of data was found to be acceptable.		Ī		
XIV Field duplicates			Ci ( I	
Field duplicate pairs were identified in this SDG.	$\Box$		Τ	
Target compounds were detected in the field duplicates.		7	-+-	
QV. Field planks				
ield blanks were identified in this SDG.				
arget compounds were detected in the field blanks.			オ	

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LDO	SDG

## VALIDATION FINDINDS WORKSHEET Surrogate Recovery



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METHOD: GC HPLC Are surrogates required by the method? Yes or No Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

N/A Did all surro	gate rec	overies (%R)	Did all surrogate recoveries (%R) meet the QC limits?	ts?				
Sample ID		Detector/ Column	Surrogate Compound		%R (Limits)			Qualifications
0	tou	brifians	Н		65	73-150	12n/-1 1	'A
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40		Xe	- Araly	S Z	rector wear	/	(	
			~				)	
							)	
Surrogate Compound	pu	Surrog	Surrogate Compound		Surrogate Compound		Surrogate Compound	
Chlorobenzene (CBZ)		ŏ о	Octacosane	Σ	Benzo(e)Pyrene	s	1-Chloro-3-Nitrobenzene	Y Tetrachloro-m- xylene
4-Bromofluorobenzene (BFB)		т	Ortho-Terphenyl	z	Terphenyl-D14	 	3,4-Dinitrotoluene	
a,a,a-Trifluorotoluene		Fluorc	Fluorobenzene (FBZ)	0	Decachlorobiphenyl (DCB)	Э	Tripentyltin	
<b>Bromochlorobenene</b>			n-Triacontane	٩	1-methvinaohthalene	>	Trt-n-oroovitin	
1.4-Dichlorobutane	╀	Ĭ	Hexacosane	σ	Dichlorophenyi Acetic Acid (DCAA)	3	Tributyl Phosphate	
1.4-Difluorobenzene (DFB)	(B)	Brc	Bromobenzene	œ	4-Nitrophenol	×	Tricheny Bhoschoto	

1 X 386 MS ろうし 3 SDG #: LDC #:

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

ر 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

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

		Calibration		Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Date	Compound	( SU <sup>std</sup> )	cF (SV Gtd)	Average CF (initial)	Average CF		
-	7	antich	Lund	16699	16699	17265	17265	Uch Cl	KRSD KO
	-	-						~	6212
~									
1							· ·		
m		•							
4	<u></u>								
T									
in the second									
esults.	erits: <u>Kererto I</u>	nitial Calibratio	Comments: Refer to Initial Calibration findings worksheet for list of qualifications and associated samples when reported results do not arme within 10 nm of the section o	ins and associa	ated samples wi	<u>hen reported res</u>	ults do not aorea	within 10 00/	
					I		00 KG (010 010 010 010 010 010 010 010 010 01	WIRTH 10.0 %	<u>ul lite recalcula</u>

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LDC # 18 380 RY en care SDG #:

**Continuing Calibration Results Verification** VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer: 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

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

Where:

A = Area of compound C = Concentration of compound

**Recalculated** 0% 0 Ľ 5.2 1 0 0,7 ý Reported いべ 0% 1006.5768 Recalculated 271-965 9 80.13.81 CF/Conc. CCV Role. STLX 871.965 280.1386 CF/Conc. CCV Reported Average CF(Ical)/ CCV Conc. 1000.00 000 Compound 2 101 08 2) 01 08 Calibration Date 70/12/1 Standard ID ECALSOS 819 1 33 \* 2 ო

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

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## VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

, of Page: 2nd reviewer: 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 Percent Difference

0

Recalculated Percent Recovery 5 Percent Recovery Reported 77 19.3267 Surrogate Found Surrogate Spiked 2 Column/Detector Z hot speci Surrogate terpheny ه\ ά Sample ID:

### Sample ID:

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

Sample ID:

Percent Difference				
Percent Recovery	Recalculated			
Percent Recovery	Reported			-
Surrogate Found				
Surrogate Spiked		 •		
<b>Column/Detector</b>				
Surrogate				
			<u> </u>	

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2	, z
#	#
20	SDG

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

Page: / of Z Reviewer: 2nd Reviewer:

ပ္လ **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 HPLC

the following calculation: %Recovery = 100 \* (SSC - SC)/SA

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

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MS/MSD samples:

SC = Sample concentration

MSD = Matrix spike duplicate

		Ű	Chiko	-io								
•		AdA	Added	Conc.	Spike:	Spike Sample Concentration	Matri	Matrix spike	Matrix Spike Duplicate	e Duplicate	DSM/SM	ISD
Compound	pund	1	Kel )	1 mrs les	um)	(ker)	Percent	Percent Recovery	Percent Recovery	Recovery	U A B D	
		WS	MSD		WS	MSD	Reported	Recalc	Renorted	Basels		8
Gasoline	(8015)								netiodati	Vecalc.	релодея	Kecaic.
Diesel	(8015)	86.7	1. XS	0	7.4	כאיין	0				c	5
Benzene	(8021B)				0			2	14	75	50	è a
Methane	(RSK-175)	2.5	120									
2,4-D	(8151)	i series de la companya de la comp										
Dinoseb	(8151)											
Naphthalene	(8310)											
Anthracene	(8310)	a ter										
HMX	(8330)											
2,4,6-Trinitrotoluene (8330)	uene (8330)	-										
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.	<u>er to Matrix Spik</u> ed results.	<u>e/Matrix S</u>	Spike Dupli	cates finding	s worksheet f	or list of qualif	cations and a	ssociated sam	ples when rep	ll <u>ported results</u>	l do not agree	within 10.09

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METHOD:	g	НРСС							HPLC		2nd F	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:	s (%R) a below us	ind relativ	'e percent illowing ci	t differences ( alculation:	RPD) of the	laboratory co	ntrol sample	and laborator	y control sam	ple duplicate	were recalcula	ated for the
%Recovery = 100 * (SSC - SC)/SA	C)/SA		Where	SSC = Spiked concentration SA = Spike added	icentration		SC = Sample concentration	ncentration				
RPD =(((SSCLCS - SSCLCSD) * 2) / (SSCLCS + SSCLCSD))*100 LCS/LCSD samples:	5039'2)/(ssci	ssclcs + s	s + ssclcsD))*	n (* 1000) Na station Station (* 1000) Station (* 1000)	LCS = Labora	ttory Control Sam	S = Laboratory Control Sample percent recovery		LCSD = Laboratory Control Sample duplicate percent recovery	Control Sample	duplicate percent r	ecovery
		Spike	ke	Sample	Spike	Spike Sample		LCS	rcsD	Ģ	rcs/rcsD	SD
Compound		Add Add	E C	Conc.	Concent ( w-X	Concentration	Percent I	Percent Recovery	Percent Recovery	ecovery	CPD	
		LCS	LCSD	, , ,	, rcs		Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)		) (1) (1) (1) (1) (1) (1) (1) (1) (1) (1										
Diesel (8015)		83.3	NA	0	٦ <i>6.</i> ८	42	40	d d	NA			
Benzene (8021B)		алы.										
Methane (RSK-175)	75)											
2,4-D (8151)												
Dinoseb (8151)		ar an										
Naphthalene (8310)		1. M										
Anthracene (8310)												
HMX (8330)										×		
2,4,6-Trinitrotoluene (8330)	833 <b>0</b> )											
		1										
Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicate findings worksheet for list of qualifications and associated samples when reported	aborator	V Control	Sample/I	Laboratory Cc	ntrol Sample	Duplicate fin	<u>dings worksh</u>	eet for list of c	<u>jualifications a</u>	ind associate	ed samples wh	en reported

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VALIDATION FINDINGS WORKSHEET	2nd		ulated and verified for all level IV samples?	Example:	Sample ID. Compound Name		Concentration =			compound Reported Recalculated Results Concentrations Concentrations Concentrations Qualifications						
VALIDATION FINDIN Sample Calculati			Were all reported results recalculated and verified for all level IV samples? Were all recalculated results for detected farmet communicals within 10% of							Compound Concent						
LDC #: 18 3 86 AS SDG #: 24 Coner		METHOD: CC HPLC	Y N/N/A Were all reported re Y N/N/A Were all recalculate	Concentration= (A)(Fv)(Df) (RF)(Vs or WsV%S1400)		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 Vs= Initial volume of the sample Ws= Percent Solid		# Sample ID			*		Comments:	

SAMPCALew.wpd

### LDC Report# 18386B8

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

Collection Date: January 28, 2008

LDC Report Date: March 11, 2008

Matrix: Soil/Water

Parameters: Diesel Range Organics

Validation Level: EPA Level III

Laboratory: TestAmerica, Inc.

### Sample Delivery Group (SDG): F8A290158

### Sample Identification

TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HJ-08-10' TSB-HR-05-10' TSB-HR-05-10'RE RINSATE-2 TSB-HJ-10-0'MS TSB-HJ-10-0'MSD TSB-HR-05-10'MSD

### Introduction

This data review covers 14 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 8015 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.

Raw data were not reviewed for this SDG. The review was 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-2 was identified as a rinsate. No diesel range organic contaminants were found in this blank.

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

### a. Surrogate Recovery

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

Sample	Surrogate	%R (Limits)	Compound	Flag	A or P
TSB-HR-05-10'	ortho-Terphenyl	71 (73-150)	Diesel range organics	J- (all detects) UJ (all non-detects)	A
8039219-Blank	ortho-Terphenyl	62 (73-150)	Diesel range organics	J- (all detects) UJ (all non-detects)	Р

### b. Matrix Spike/Matrix Spike Duplicates

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

### c. Laboratory Control Samples

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

### V. Target Compound Identification

Raw data were not reviewed for this SDG.

### VI. Compound Quantitation and CRQLs

Raw data were not reviewed for this SDG.

### VII. System Performance

Raw data were not reviewed for this SDG.

### VIII. Overall Assessment of Data

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

### IX. Field Duplicates

Samples TSB-HR-06-0' and TSB-HR-06-0'-FD were identified as field duplicates. No diesel range organics were detected in any of the samples.

### BRC Tronox Parcel H Diesel Range Organics - Data Qualification Summary - SDG F8A290158

SDG	Sample	Compound	Flag	A or P	Reason
F8A290158	TSB-HR-05-10'	Diesel range organics	J- (all detects) UJ (all non-detects)	A	Surrogate spikes (%R)

BRC Tronox Parcel H

Diesel Range Organics - Laboratory Blank Data Qualification Summary - SDG F8A290158

No Sample Data Qualified in this SDG

**BRC Tronox Parcel H** 

Diesel Range Organics - Field Blank Data Qualification Summary - SDG F8A290158

No Sample Data Qualified in this SDG

### VALIDATION COMPLETENESS WORKSHEET

LDC #: 18386B8 SDG #: F8A290158

### Level III

Date: 3/6/08 Page: ( of Reviewer: 2nd Reviewer:

Laboratory: Test America

METHOD: GC Diesel Range Organics (EPA SW 846 Method 8015)

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: 1 28 08
lla.	Initial calibration		
llb.	Calibration verification/ICV	Δ	$ u  =  \overline{1} $
111.	Blanks	A	
IVa.	Surrogate recovery	SW	
IVb.	Matrix spike/Matrix spike duplicates	Δ	
IVc.	Laboratory control samples	A	LCSIP
V.	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	N	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	ND	D = 3, 4
Х.	Field blanks	ND	R = 11

Note:

A = Acceptable N = Not provided/applicableSW = See worksheet

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

D = Duplicate TB = Trip blank EB = Equipment blank

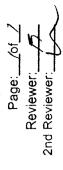
Validated Samples:

valida	ted Samples:	Va	ur			
1	TSB-HJ-10-0'	112	RINSATE-2	21	8031303	31
2	TSB-HJ-10-10'	12	TSB-HJ-10-0'MS	22 V	8635123	32
3	TSB-HR-06-0' ĵ	13	TSB-HJ-10-0'MSD	23 <b>3</b>	8039219	33
4	TSB-HR-06-0'-FD	14 1	TSB-HR-05-10'MS	24		34
5	TSB-HR-06-10'	15 1	TSB-HR-05-10'MSD	25		35
6	TSB-HJ-08-0'	16		26		36
7	TSB-HJ-08-10'	17		27		37
8	TSB-HR-05-0'	18		28		38
91	TSB-HR-05-10'	19		29		39
10	TSB-HR-05-10'RE	20		30		40

Notes:

LDC #: 1 × 386 8 ×

## VALIDATION FINDINDS WORKSHEET Surrogate Recovery



METHOD: GC HPLC Are surrogates required by the method? Yes or No Ptease see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A" Y N/A Were surrogates spiked into all samples and blanks?

A/N/N/A		ogate re	Liavoja	( <u>10/) c</u> 2	Did all surrogate recoveries (%K) meet the QC limits?	2						
#	Sample ID		Detector/ Column	r./ n	Surrogate Compound		%R (Limits)			đ	Qualifications	
	0	ton	- NG	s' i cel	Н		) 1L	73 - 1	150)	J-/ m/ /A		
			-	4		+	}					
	2039219-Blank		7		-		29	Ì		1-/u//p		
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	Surrogate Compound	g		Surrog	Surrogate Compound		Surrogate Compound		Surrogate Compound	compound		
٩	Chlorobenzene (CBZ)		U	ŏ	Octacosane	Σ	Benzo(e)Pyrene	s	1-Chloro-3-Nitrobenzene	itrobenzene Y	Tetrachloro-m- xylene	1
æ	4-Bromofluorobenzene (BFB)	3FB)	T	νO	Ortho-Terphenyl	z	Terphenyl-D14		3,4-Dinitrotoluene	stoluene		
U	a,a,a-Trifluorotoluene		-	Fluord	Fluorobenzene (FBZ)	0	Decachlorobiphenyl (DCB)	n	Tripentyltin	tyltin		
6	Bromochlorobenene	1	+	5	n-Triacontane	۵.	1-methvinaphthalene	>	Tri-n-propyltin	povltin		$\square$
ш	1,4-Dichlorobutane		¥	T	Hexacosane	σ	Dichlorophenyl Acetic Acid (DCAA)	3	Tributyl Phosphate	iosphate		
Ш	L 1.4-Difluorobenzene (DFB)	B)		, Brc	Bromobenzene	ы	4-Nitrophenol	L X L	Triphenvl Phosohate	hosohate		Π

### BRC Tronox Parcel H Data Validation Reports LDC# 18386

Dioxins/Dibenzofurans



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

Project/Site Name:	BRC Tronox Parcel H
Collection Date:	January 24, 2008
LDC Report Date:	March 12, 2008
Matrix:	Soil
Parameters:	Dioxins/Dibenzofurans
Validation Level:	EPA Level III & IV
Laboratory:	TestAmerica, Inc.

Sample Delivery Group (SDG): F8A250221

### Sample Identification

TSB-HJ-05-10'\*\* TSB-HJ-05-0'\*\* TSB-HR-04-10' TSB-HJ-04-0' TSB-HR-04-0'\*\* TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'\*\* TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'\*\* TSB-HJ-07-0'-FD TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-10' TSB-HR-06-0'MS TSB-HR-06-0'MSD

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 17 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 with the following exceptions:

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
8043106-Blank	2/12/08	1,2,3,7,8-PeCDD OCDD OCDF	0.098 pg/g 0.33 pg/g 0.15 pg/g	All samples in SDG F8A250221

Sample concentrations were compared to concentrations detected in the method blanks. The sample concentrations were either not detected or were significantly greater (>5X blank contaminants) than the concentrations found in the associated method blanks with the following exceptions:

Sample	Compound	Reported Concentration	Modified Final Concentration
TSB-HJ-05-10'**	OCDD OCDF	0.30 pg/g 0.12 pg/g	11U pg/g 11U pg/g
TSB-HR-04-10'	OCDD OCDF	0.24 pg/g 0.17 pg/g	11U pg/g 11U pg/g
TSB-HJ-04-0'	OCDD	0.30 pg/g	11U pg/g
TSB-HR-04-0'**	OCDD	0.19 pg/g	10U pg/g
TSB-HJ-04-10'	OCDD	0.20 pg/g	11U pg/g
TSB-HR-07-0'	OCDD	0.21 pg/g	11U pg/g
TSB-HR-07-10'**	OCDD	0.34 pg/g	11U pg/g
TSB-HR-06-10'	OCDD	0.26 pg/g	11U pg/g
TSB-HJ-07-0'**	OCDD OCDF	0.80 pg/g 0.21 pg/g	11U pg/g 11U pg/g
TSB-HJ-07-0'-FD	OCDD	0.30 pg/g	11U pg/g
TSB-HJ-07-10'	1,2,3,7,8-PeCDD OCDD	0.071 pg/g 0.23 pg/g	5.4U pg/g 11U pg/g
TSB-HR-08-0'	1,2,3,7,8-PeCDD OCDD	0.12 pg/g 0.22 pg/g	5.3U pg/g 11U pg/g
TSB-HR-08-10'	OCDD	0.39 pg/g	11U pg/g

No field blanks were identified in this SDG.

### VI. 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.

### VII. Laboratory Control Samples (LCS)

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

### VIII. Regional Quality Assurance and Quality Control

Not applicable.

### IX. Internal Standards

All internal standard recoveries were within QC limits.

### X. Target Compound Identifications

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

### XI. Compound Quantitation and CRQLs

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

Sample	Compound	Finding	Criteria	Flag	A or P
TSB-HR-06-0'	2,3,7,8-TCDF (from DB-225)	Confirmation was not performed for this compound.	All compounds must be confirmed on the 2nd column per the QAPP.	None	Р

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

### XII. System Performance

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

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

Samples TSB-HJ-07-0'\*\* and TSB-HJ-07-0'-FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

	Concentr	ation (pg/g)		D:#		
Compound	TSB-HJ-07-0'**	TSB-HJ-07-0'-FD	RPD (Limits)	Difference (Limits)	Flag	A or P
OCDD	0.80	0.30	-	0.50 (≤11)	-	-
1,2,3,7,8,9-HxCDF	0.070	0.14	-	0.07 (≤5.4)	-	-
1,2,3,4,6,7,8-HpCDF	0.064	5.3U	-	5.24 (≤5.4)	_	-
OCDF	0.21	11U	-	10.79 (≤11)	-	-

### BRC Tronox Parcel H Dioxins/Dibenzofurans - Data Qualification Summary - SDG F8A250221

SDG	Sample	Compound	Flag	A or P	Reason
F8A250221	TSB-HR-06-0'	2,3,7,8-TCDF (from DB-225)	None	Ρ	Compound quantitation and CRQLs

### BRC Tronox Parcel H Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG F8A250221

SDG	Sample	Compound	Modified Final Concentration	A or P
F8A250221	TSB-HJ-05-10'**	OCDD OCDF	11U pg/g 11U pg/g	A
F8A250221	TSB-HR-04-10'	OCDD OCDF	11U pg/g 11U pg/g	A
F8A250221	TSB-HJ-04-0'	OCDD	11U pg/g	A
F8A250221	TSB-HR-04-0'**	OCDD	10U pg/g	A
F8A250221	TSB-HJ-04-10'	OCDD	11U pg/g	A
F8A250221	TSB-HR-07-0'	OCDD	11U pg/g	A
F8A250221	TSB-HR-07-10'**	OCDD	11U pg/g	A
F8A250221	TSB-HR-06-10'	OCDD	11U pg/g	A
F8A250221	TSB-HJ-07-0'**	OCDD OCDF	11U pg/g 11U pg/g	A
F8A250221	TSB-HJ-07-0'-FD	OCDD	11U pg/g	A
F8A250221	TSB-HJ-07-10'	1,2,3,7,8-PeCDD OCDD	5.4U pg/g 11U pg/g	A
F8A250221	TSB-HR-08-0'	1,2,3,7,8-PeCDD OCDD	5.3U pg/g 11U pg/g	A
F8A250221	TSB-HR-08-10'	OCDD	11U pg/g	A

### BRC Tronox Parcel H Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG F8A250221

No Sample Data Qualified in this SDG

### VALIDATION COMPLETENESS WORKSHEET

LDC #:<u>18386A21</u> SDG #:<u>F8A250221</u>

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### Laboratory: Test America

Level III/IV

Date: 3/11/08 Page: / of Reviewer: 2nd Reviewer:

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

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

	Validation Area		Comments
	Technical holding times	A	Sampling dates: 1 24 08
П.	GC/MS Instrument performance check	Δ	
111.	Initial calibration	$\Delta$	
IV.	Routine calibration	Δ	
V.	Blanks	ىرى	
VI.	Matrix spike/Matrix spike duplicates	A	
VII.	Laboratory control samples	A	LC5
VIII.	Regional quality assurance and quality control	" N	
IX.	Internal standards	A	
Х.	Target compound identifications	<u> </u>	Not reviewed for Level III validation.
XI.	Compound quantitation and CRQLs	sω	Not reviewed for Level III validation.
XII.	System performance	Δ	Not reviewed for Level III validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SU	D= 11+12
XV.	Field blanks	N	

Note:

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

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

	SOLL	·					
11	TSB-HJ-05-10'**	117	TSB-HJ-07-0'**	21	8043106 - Blank	31	
2+1	TSB-HJ-05-0'**	12 3	TSB-HJ-07-0'-FD	22		32	
3	TSB-HR-04-10'	133	TSB-HJ-07-10'	23		33	
4	TSB-HJ-04-0'	14 3	TSB-HR-08-0'	24		34	
51	TSB-HR-04-0'**	152	TSB-HR-08-10'	25		35	
<sub>6</sub>	TSB-HJ-04-10'	16	TSB-HR-06-0'MS	26	<u></u>	36	
7	TSB-HR-07-0'	17	TSB-HR-06-0'MSD	27		37	
*7	TSB-HR-07-10'**	18		28		38	
9 <b>-1</b>	TSB-HR-06-0'	19		29		39	
10		20		30		40	

Notes:

LDC #: 18386A2) SDG #: <u>fre cone</u>

Page: / of <u>3</u> Reviewer: <u>7</u> 2nd Reviewer: <u>1</u>

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	/			
Cooler temperature criteria was met.	/	ł		
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?	/			
Were the retention time windows established for all homologues?	1			
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				
Was a routine calibration performed at the beginning and end of each 12 hour period?	-			
Were all percent differences (%D) $\leq$ 20% for unlabeled standards and $\leq$ 30% for labeled standards?	_			
Did all routine calibration standards meet the Ion Abundance Ratio criteria?				
V. Blanks				
Was a method blank associated with every sample in this SDG?	_			
Was a method blank performed for each matrix and concentration?	-			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?	/			
VI. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	~			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VII. Laboratory control samples	,		<u> </u>	
Was an LCS analyzed for this SDG?	/			

LDC #: 18386A2) SDG #: pre coner

### VALIDATION FINDINGS CHECKLIST

Page: 2 of	3
Reviewer: 5	· · · · · · · · · · · · · · · · · · ·
2nd Reviewer:	
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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?			$\square$	
Were the performance evaluation (PE) samples within the acceptance limits?				
IX. Internal standards				
Were internal standard recoveries within the 40-135% criteria?	/			
Was the minimum S/N ratio of all internal standard peaks $\geq$ 10?	/			
X. Target compound identification				
For 2,3,7,8 substituted congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard?	/			
For 2,3,7,8 substituted congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration?	/			
For non-2,3,7,8 substituted congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution?	/			
Did compound spectra contain all characteristic ions listed in the table attached?	$\angle$			
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	/			
Was the signal to noise ratio for each target compound and labeled standard $\geq$ 2.5?				
Does the maximum intensity of each specified characteristic ion coincide within $\pm$ 2 seconds (includes labeled standards)?	/			
For PCDF identification, was any signal (S/N $\geq$ 2.5, at $\pm$ seconds RT) detected in the corresponding PCDPE channel?	/			
Was an acceptable lock mass recorded and monitored?		-		
XI. Compound quantitation/CRCLs				
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		1		
Overall assessment of data was found to be acceptable.	$\square$			
XIV. Field duplicates			1	
Field duplicate pairs were identified in this SDG.	$\square$			

VALIDATION FINDINGS CHECKLIST

LDC #: 18386A2/ SDG #: <u>fue cone</u>

Page:<u>3</u>of<u>3</u> Reviewer:<u>5</u> 2nd Reviewer:

Validation Area	Yes	No	NA	Findings/Comments
Target compounds were detected in the field duplicates.	$\overline{\left\langle \cdot \right\rangle}$			
XV. Field blanks	<b>.</b>			
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. 2,3,7,8-1CDD	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-HYCOD				
	1. 1, 2, 3, 7, 9-F BCUF	N. 1,2,3,7,8,9-HxCDF	S. Total PeCDD	X. Total HxCDF
F 1 2 3 7 A 9-HVCDD				
CONVIL-Sist Hotels	4. 2,3,4,7,0-PBCUF	0.1,2,3,4,6,7,8-HpCDF	T. Total HxCDD	Y. Total HDCDF

Notes:

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LDC #: [Y	SDG #:

### VALIDATION FINDINGS WORKSHEET Blanks

Page: <u>/</u>of 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".

V N/A Were all samples associated with a method blank?

Was a method blank performed for each matrix and whenever a sample extraction was performed? N N/A

Associated samples: A||

Compound	Blank ID				S.	Sample Identification	tion			
	rot 3106	-	7	3	4	<b>ئ</b> /	د	٢	Ş	8
В	0. 098	L	1	J	1	1	ł	1	,	L
G	0.33	N11/0E.0	(b.4)	0. 24/11N	0.30/114	no1/61.0	0.20/11N	0.21/111	0. 34/114	(2.0)
প	0.15	0.12 /11M	(91)			-			7	(a. r)
		•	,							,
	Soutstope Blank		10	11	12	13	14	15		
8	හ.පොල		-		nt. 5/ et. 0 hts/110 0 th. 5/1 000	vh:s/120 .01	0.13 /5.3 U	•		
ط	0.73		0.26/11 N	n11/000	0.30/11U	N11/ 61.0 N11/0E.0	0.22/114	MIY Lec.a		
8	0.15		-	0. 21 /14		- 1	-	<b>`</b> ı		
				-						

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

V:\Validation Worksheets\Dioxin90\BLANKS90.21

X3 She AV	Le court
	SDG #:

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".

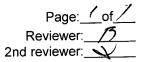
Were the correct internal standard (IS), quantitation ions and relative response factors (RRF) used to quantitate the compound? Compound quantitation and CRQLs were adjusted to reflect all sample dilutions and dry weight factors (if necessary). <u>Y N N/A</u> V N/A

Qualifications	me / P									
Associated Samples										
Finding	no DB-225 conjimation	0								
- <del>S</del> ampite ID										
Date										
#										

Comments: See sample calculation verification worksheet for recalculations

LDC #: 18 3 86 A21 SDG #: 18 Coner

### VALIDATION FINDINGS WORKSHEET Field Duplicates



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

Y N N/A Y N N/A

Were field duplicate pairs identified in this SDG. Were target compounds detected in the field duplicate pairs?

	Concentrat	$\frac{1}{2} \left( \frac{p_{2}}{q} \right)$	Diffirence
Compound	11	10, 0	
4	0.80	0.30	0.50 (- 5,4)
N	0.070	0.14	0.07 (-4,1) (=5.4)
Ø	0.064	5.34	5.24 (5.4)
Q	0.21	11 1	10.79 (± 11)

	Concentration ()	
Compound		RPD

	Concentration ( )	_
Compound		RPD

	Concentration ()	=
Compound		RPD

LDC #: 18 386 A2/ fue could SDG #:

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

ò 5 Page: Reviewer: 2nd Reviewer:

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

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

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

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

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

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Average RRF (initial)	RRF (Cらろ std)	RRF (ፈረን std)	%RSD	%RSD
-	Ical	2/12/08	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.045	1. 0(5	1-040	1.040	0.0 0	9.9
		,	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	H8_2t0.1	1.084	1.075	Sho-1	11.0	1.0
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.053	1.053	0401	040.1	4.O	0.1
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	0.976	0.97L	1110	116-0	₽.Ú	04
			OCDF ("C-OCDD)	1.102	101-1	911-1	911-1	5.0	8.1)
2	ICA	20/s1/2	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	+ 119	1.19	200 1	1 10	4.0	L 0.2
	28-225	-	23.7.8.1CDD (12 2.3.7.8.7CBD)	1.081	1.80.1	1.1255	241-	L-7	6.9
			1,2,3,6,7,8-HxCDD (*C-1,2,3,6,7,8-HxCDD)					X	
			1,2,3,4,6,7,6-HpCDD (1-C-1,2,4,6,7,8,-HpCDD)						
			COF (*e-ceau)						
ო			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)						
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)						
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)						
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)						
			OCDF (13C-OCDD)						
								<b></b>	

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

LDC #:	SDG #:
	S

## VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

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

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF = (A<sub>x</sub>)(C<sub>is</sub>)/(A<sub>is</sub>)(C<sub>x</sub>)

Where: ave. RRF = initial calibration average RRF RRF = continuing calibration RRF  $A_x = A_{tee}$  of compound,  $A_x = C_x = C_x$  = Concentration of compound,  $C_z$ 

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

**Recalculated** ۵% 4.4 Reported ふし %۵ **Recalculated** RRF (CC) 7701 Reported 1.04J RRF (CC) Average RRF (initial) 1.125 1,2,3,4,6,7,8-HpCDD (<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) 1,2,3,4,6,7,8-HpCDD (<sup>13</sup>C-1,2,4,6,7,8,-HpCDD) Compound (Reference Internal Standard) 1,2,3,6,7,8-HxCDD (<sup>13</sup>C-1,2,3,6,7,8-HxCDD) 1,2,3,6,7,8-HxCDD (<sup>13</sup>C-1,2,3,6,7,8-HxCDD) 1,2,3,6,7,8-HxCDD (<sup>13</sup>C-1,2,3,6,7,8-HxCDD) 2,3,7,8-TCDD (<sup>13</sup>C-2,3,7,8-TCDD) 2,3,7,8-TCDD (<sup>13</sup>C-2,3,7,8-TCDD) 2,3,7,8-TCDD (<sup>13</sup>C-2,3,7,8-TCDD) 2,3,7,8-TCDF (<sup>13</sup>C-2,3,7,8-TCDF) 2,3,7,8-TCDF (<sup>13</sup>C-2,3,7,8-TCDF) 2,3,7,8-TCDF (<sup>13</sup>C-2,3,7,8-TCDF) OCDF (<sup>13</sup>C-OCDD) OCDF (13C-OCDD) OCDF (13C-OCDD) Calibration Date 2/2/08 000000055 Standard ID ო # 2

Comments: Refer to Routine Calibration findings worksheet for list of gualifications and associated samples when reported results do not agree within 10.0% of the recalculated results. LDC # 123 50 AV SDG #: The course

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



METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method TO-9A)

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)

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:

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

Standard ID Date Calibration						Deremenau
Cev 2/2000 12:34	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	<b>D</b> %	<b>D</b> %
46:21 720260804	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.045	0.784	0.984	5.9	sq
hc	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.084	1.021	1.021	5.a	5.9
	1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.053	1.001	1.001	e: 2	۲. ب
	1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	0,916	1.030	1.030	5.5	ŝ
Alalos		1.102	1.276	1-276	15.8	15.8
	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)		0.996	966.0	4.7	4.7
A08022 23:53 23:53 2	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)		1.035	1.035	4- <i>S</i>	4.S
-	1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)		1. 00 D	1.023	2.8	82
-	1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)		1.021	(40.1	4-2	4-5
	OCDF ( <sup>13</sup> C-OCDD)	$\downarrow$	1-171	1-17-1	د . ک	د. م
3 cev 2/2/08 2	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)		0.989	0-989	S.4	S.4
	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)		1.055	<u>-035</u>	a.1	2.7
- - 1	1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)		1.019	610.1	3.2	3.7
	1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)		1.032	1.032	s.7	S.7
	OCDF (13C-OCDD)		1.130	i-170	6.1	( م)

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

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



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

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

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

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

% Recovery = 100 \* (SSR - SR)/SA

Where:

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

MS/MSD samples: 16 4 17

	ŝ	Spike	Sample	Spiked Sample	Sample	Matrix Spike	Spike	Matrix Spik	Matrix Spike Duplicate	Reported	Recalculated
Compound	Added (Pa)a	lded  a_)	Concentration	Concentration ( <b>DA A</b> )	tration A )	Percent F	Percent Recovery	Percent Recovery	Recovery		RPD
	Disw	U MSD		n f MS	( MSD	Reported	Recalc	Renorted	Ranaln		
2,3,7,8-TCDD	22.2	24.7	Q N	Lun	h.2.6	102	101	101	101	برا -	<u>ز</u> ې
1,2,3,7,8-PeCDD	111	111	d v N	211	111	501	Eal	105	los	6.1	6.1
1,2,3,4,7,8-HxCDD	III	1	QN	211	011	106	Jal	99	99	8.9	b.8
1,2,3,4,7,8,9-HpCDF	111		0.45	117	111	101	اەكر	001	0.01	6.4	6.7
OCDF	111	222	۲.۲	ex Bx	250	ااک	ااکر	112	112	3.	3,1
											and the second se

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

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## VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Results Verification

Page: / of / Reviewer:

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: 8043106 - 103

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

LCSD = Laboratory control sample duplicate percent recovery

	ŝ	oike	Spiked S	Sample		y S	I CSD	D		csn
Сотроина	PA PA	Added (pa. /a.)	Concentration	tration	Percent Recovery	ecoverv	Percent Recovery	coverv	Cda	
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						Recall	Kelporeo	Recal	Керопед	Kecalculated
2,3,7,8-1 CDD	0.97	ЪД	20,	<b>₹</b> 2	n ol	100				
1,2,3,7,8-PeCDD	001	-	lo S	-	501	10)				
1,2,3,4,7,8-HxCDD	7		104		hoi	hai				
1,2,3,4,7,8,9-HpCDF	· →		9 0l		101	106		$\left  \right $		
OCDF	200		مامه		120	2	42			

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.

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### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	1_of/
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METHO	D: HI	RGC/HRMS Dioxins/Dibenzofurans (EPA SW 8	46 Method 8290)
	<u>/A</u> /A	Were all reported results recalculated and v Were all recalculated results for detected ta	erified for all level IV samples? rget compounds agree within 10.0% of the reported results?
- Concer	tration	= <u>(A,)(I,)(DF)</u> (A, <sub>is</sub> )(RRF)(V <sub>o</sub> )(%S)	Example:
A <sub>x</sub>	=	Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D. <u># 2 ,                                   </u>
$A_{is}$	=	Area of the characteristic ion (EICP) for the specific internal standard	
l <sub>s</sub>	=	Amount of internal standard added in nanograms (ng)	Conc. = (52145) (2000) () () (0.976) (0.976) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975) (0.975)
V <sub>o</sub>	=	Volume or weight of sample extract in milliliters (ml) or grams (g).	
RRF	=	Relative Response Factor (average) from the initial calibration	= 6.44 pg/g
Df	=	Dilution Factor.	
%S	=	Percent solids, applicable to soil and solid matrices only.	

#	Sample ID	Compound	Reported Concentration ()	Calculated Concentration (  )	Qualification
		#2 H conjirmation			
	<b></b>				
		= 8462 (1000)			
		= <u>8462 (1000)</u> 388684 (1.081)(	10) (0.953)		
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		= 2.1 pg/g			
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Elemental Composition	C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7ClO C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7ClO C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O <sup>13</sup> C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O <sup>13</sup> C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7ClO C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O <sup>13</sup> C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O <sup>13</sup> C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O	C <sub>12</sub> <sup>36</sup> Cl <sub>3</sub> <sup>37</sup> ClO C <sub>12</sub> <sup>32</sup> Cl <sub>3</sub> <sup>37</sup> ClO C <sub>12</sub> <sup>32</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> <sup>32</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> <sup>32</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub> 13C <sub>12</sub> <sup>32</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>10</sub> F <sub>17</sub> C <sub>10</sub> F <sub>17</sub>	
DI NO	Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z	M M M M M M M M M M M M M M M M M M M	
Accurate Mass <sup>(a)</sup>	407.7818 409.77818 417.8250 417.8250 419.8220 419.8220 423.7737 423.7737 423.7737 423.7737 435.8169 437.8140 437.8140 437.8140 437.8140 437.8140 437.9728]	441.7428 443.7399 457.737 459.7348 459.7780 471.7750 513.6775 [422.9278]	
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Analyte	TCDF TCDF (S) TCDD (S) TCDD (S) TCDD (S) TCDD (S) TCDD (S)	PeCDF PeCDF (S) PeCDF (S) PeCDD (S) PeCDD (S) PeCDD (S) PFK PFK	HXCDF HXCDF HXCDF (S) HXCDD (S) HXCDD (S) HXCDD (S) OCDPE OCDPE (S)
Elemental Composition	C12H 3*C(O C12H 3*C(3) C12H 3*C(3) C12H 3*C(4) 1*C12H 3*C(4) C12H 3*C(4) C12H 3*C(4) C12H 3*C(4) 1*C(4) 1*C(4) 1*C(4) 1*C(5) 1*C(5) C12H 3*C(0) C12H 3*C(0) C12	C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>0</sub> C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>0</sub>	C,H,*CI,*CIO C,H,*CI,*CIO C,H,*CI,*CIO <sup>13</sup> C,H,*CI,0 C,H,*SCI,*CIO C,H,*SCI,*CIO C,H,*CI,*CIO C,H,*CI,*TCI_0 C,H,*CI,*TCI_0 C,H,*CI,*TCI_0 C,F,*CI,*TCI_0 C,F,*CI,*TCI_0
lon ID	CCK 2 2 CCK 2 2 CCK 2 3 CCK 2 4 CCK 2 5 CCK 2	M M M M M M M M M M M M M M M M M M M	M M M M M M M M M M M M M M M M M M M
Accurate mass <sup>(a)</sup>	303.9016 305.8987 315.9419 317.5989 319.8985 321.8836 331.9688 331.9688 333.9338 375.8364 [354.9792]	339.8597 341.8567 351.9000 353.8970 355.8546 357.8516 357.8516 357.8949 369.8919 409.7974 (354.9792]	373.8208 375.8178 383.86178 385.8610 385.8610 389.8156 391.8127 401.8559 403.8529 403.8529 403.8529 403.8529 403.8529 403.8529 403.8529 403.8529 403.8529
Descriptor	-	N	ς

The following nuclidic masses were used:

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

O = 15.994915 <sup>33</sup>Cl = 34,968853 <sup>37</sup>Cl = 36.965903

S = internal/recovery standard

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

### LDC Report# 18386B21

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

Dioxins/Dibenzofurans

Project/Site Name:	BRC Tronox Parcel H
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Collection Date: January 28, 2008

LDC Report Date: March 12, 2008

Matrix: Soil/Water

Parameters:

Validation Level: EPA Level III

Laboratory: TestAmerica, Inc.

### Sample Delivery Group (SDG): F8A290158

### Sample Identification

TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10' RINSATE-2 TSB-HR-06-0'MS TSB-HR-06-0'MSD

### Introduction

This data review covers 11 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was 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).

### 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.

### 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 with the following exceptions:

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
8045178-BLK	2/14/08	OCDD	0.19 pg/g	All soil samples in SDG F8A290158
8042278-BLK	2/11/08	1,2,3,7,8-PeCDD 1,2,3,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,4,7,8-PeCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF 0CDF	1.1 pg/L 0.84 pg/L 1.2 pg/L 1.7 pg/L 6.6 pg/L 1.1 pg/L 1.1 pg/L 0.72 pg/L 1.2 pg/L 1.2 pg/L 1.6 pg/L 3.8 pg/L	All water samples in SDG F8A290158

Sample concentrations were compared to concentrations detected in the method blanks. The sample concentrations were either not detected or were significantly greater (>5X blank contaminants) than the concentrations found in the associated method blanks with the following exceptions:

Sample	Compound	Reported Concentration	Modified Final Concentration
TSB-HJ-10-10'	OCDD	0.22 pg/g	10U pg/g
TSB-HR-06-0'-FD	OCDD	0.70 pg/g	11U pg/g
TSB-HR-06-10'	OCDD	0.24 pg/g	11U pg/g
TSB-HJ-08-10'	OCDD	0.26 pg/g	11U pg/g
TSB-HR-05-10'	OCDD	0.40 pg/g	11U pg/g
RINSATE-2	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF OCDF	0.84 pg/L 3.9 pg/L 0.35 pg/L 0.56 pg/L 1.7 pg/L	50U pg/L 100U pg/L 50U pg/L 50U pg/L 100U pg/L

Sample "RINSATE-2" was identified as a rinsate. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Rinsate ID	Sampling Date	Compound	Concentration	Associated Samples
RINSATE-2	1/28/08	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF OCDF	0.84 pg/L 3.9 pg/L 0.35 pg/L 0.56 pg/L 1.7 pg/L	All soil samples in SDG F8A290158

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

Sample	Compound	Reported Concentration	Modified Final Concentration
TSB-HJ-10-0'	1,2,3,7,8,9-HxCDF	0.51 pg/g	5.3U pg/g
TSB-HJ-10-10'	OCDD	0.22 pg/g	10U pg/g
TSB-HR-06-0'	1,2,3,4,6,7,8-HpCDD OCDD OCDF	0.16 pg/g 1.5 pg/g 0.62 pg/g	5.2U pg/g 10U pg/g 10U pg/g
TSB-HR-06-0'-FD	OCDD OCDF	0.70 pg/g 0.49 pg/g	11U pg/g 11U pg/g
TSB-HR-06-10'	OCDD	0.24 pg/g	11U pg/g
TSB-HJ-08-0'	OCDD 1,2,3,6,7,8-HxCDF OCDF	1.2 pg/g 0.26 pg/g 1.3 pg/g	11U pg/g 5.4U pg/g 11U pg/g
TSB-HJ-08-10'	OCDD	0.26 pg/g	11U pg/g
TSB-HR-05-0'	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,6,7,8-HxCDF OCDF	1.6 pg/g 9.2 pg/g 0.16 pg/g 1.7 pg/g	5.4U pg/g 11U pg/g 5.4U pg/g 11U pg/g
TSB-HR-05-10'	OCDD	0.40 pg/g	11U pg/g

### VI. Matrix Spike/Matrix Spike Duplicates

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

### VII. Laboratory Control Samples (LCS)

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

### VIII. Regional Quality Assurance and Quality Control

Not applicable.

### **IX. Internal Standards**

All internal standard recoveries were within QC limits.

### X. Target Compound Identifications

Raw data were not reviewed for this SDG.

### XI. Compound Quantitation and CRQLs

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

Sample	Compound	Finding	Criteria	Flag	A or P
TSB-HJ-10-10'	2,3,7,8-TCDF (from DB-225)	Confirmation was not performed for this compound.	All compounds must be confirmed on the 2nd column per the QAPP.	None	Ρ

Raw data were not reviewed for this SDG.

### XII. System Performance

Raw data were not reviewed for this SDG.

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

Samples TSB-HR-06-0' and TSB-HR-06-0'-FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

	Concentr	ation (pg/g)	RPD	Difference		
Compound	TSB-HR-06-0'	TSB-HR-06-0'-FD	(Limits)	(Limits)	Flag	A or P
1,2,3,4,6,7,8-HpCDD	0.16	5.4U	-	5.24 (≤5.4)	-	-
OCDD	1.5	0.70	-	0.8 (≤11)	-	-
1,2,3,4,6,7,8-HpCDF	0.19	0.26	-	0.07 (≤5.4)	-	-
OCDF	0.62	0.46	-	0.13 (≤11)	-	-

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### BRC Tronox Parcel H Dioxins/Dibenzofurans - Data Qualification Summary - SDG F8A290158

SDG	Sample	Compound	Flag	A or P	Reason
F8A290158	TSB-HJ-10-10'	2,3,7,8-TCDF (from DB-225)	None	Р	Compound quantitation and CRQLs

### BRC Tronox Parcel H Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG F8A290158

SDG	Sample	Compound	Modified Final Concentration	A or P
F8A290158	TSB-HJ-10-10'	OCDD	10U pg/g	A
F8A290158	TSB-HR-06-0'-FD	OCDD	11U pg/g	A
F8A290158	TSB-HR-06-10'	OCDD	11U pg/g	A
F8A290158	TSB-HJ-08-10'	OCDD	11U pg/g	A
F8A290158	TSB-HR-05-10'	OCDD	11U pg/g	A
F8A290158	RINSATE-2	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF OCDF	50U pg/L 100U pg/L 50U pg/L 50U pg/L 100U pg/L	A

### BRC Tronox Parcel H Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG F8A290158

SDG	Sample	Compound	Modified Final Concentration	A or P
F8A290158	TSB-HJ-10-0'	1,2,3,7,8,9-HxCDF	5.3U pg/g	Α
F8A290158	TSB-HJ-10-10'	OCDD	10U pg/g	A
F8A290158	TSB-HR-06-0'	1,2,3,4,6,7,8-HpCDD OCDD OCDF	5.2U pg/g 10U pg/g 10U pg/g	A

SDG	Sample	Compound	Modified Final Concentration	A or P
F8A290158	TSB-HR-06-0'-FD	OCDD OCDF	11U pg/g 11U pg/g	A
F8A290158	TSB-HR-06-10'	OCDD	11U pg/g	A
F8A290158	TSB-HJ-08-0'	OCDD 1,2,3,6,7,8-HxCDF OCDF	11U pg/g 5.4U pg/g 11U pg/g	A
F8A290158	TSB-HJ-08-10'	OCDD	11U pg/g	A
F8A290158	TSB-HR-05-0'	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,6,7,8-HxCDF OCDF	5.4U pg/g 11U pg/g 5.4U pg/g 11U pg/g	A
F8A290158	TSB-HR-05-10'	OCDD	11U pg/g	A

### VALIDATION COMPLETENESS WORKSHEET Level III

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to cos constructions -

LDC #: <u>18386B21</u> SDG #: <u>F8A290158</u> Laboratory: <u>Test America</u>

### Date: **3**/7/08 Page: \_\_\_\_\_\_ Reviewer: \_\_\_\_\_\_ 2nd Reviewer: \_\_\_\_\_\_

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

STELLER DATE SMOLLER MUSHING MENNEN SOM SOM

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	X	Sampling dates: 1 28 08
11.	GC/MS Instrument performance check	A	
- 111.	Initial calibration	А	
IV.	Routine calibration	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	LISID
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
Х.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	SW	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	D = 3 + 4
XV.	Field blanks	SW	R = 10

Note:

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

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ND = No compounds detected R = Rinsate FB = Field blank D = Duplicate TB = Trip blank EB = Equipment blank

Validated Samples:

	501L 7 W	a	-				
1 J	TSB-HJ-10-0'	11	TSB-HR-06-0'MS	21	8042278-BIK	31	
2 2	ТЅВ-НЈ-10-10' <i>Х</i>	12	TSB-HR-06-0'MSD	22 Z	8045178-BLK	32	
31	TSB-HR-06-0'	13		23		33	
4 2	TSB-HR-06-0'-FD	14		24		34	
5 1	TSB-HR-06-10'	15	·	25		35	
<b>7</b> 6	TSB-HJ-08-0'	16		26		36	
<sub>7</sub> ✓	TSB-HJ-08-10'	17		27		37	
8 <sup>γ</sup>	TSB-HR-05-0'	18		28		38	
8 <sup>Y</sup> 9 Y	TSB-HR-05-10'	19		29		39	
10 )	RINSATE-2	20		30		40	

Notes:

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:

LDC # / X 2 X 282/			VALIDAT	<b>LION FINDI</b>	VALIDATION FINDINGS WORKSHEET	KSHEET			Page: / of 2
SDG #: pre cover	,			Bla	<u>Blanks</u>				2nd Reviewer:
: HRG e qua	lioxins/Dibenz( ⊌ow for all qu€	SC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290) lifications below for all questions answered "N". Not applicable questions are identified as "N/A".	SW 846 Meth ed "N". Not a	1od 8290) applicable que	stions are ide	⊧ntified as "N/A	=		
ANN NA	hod blank peri	Were all samples associated with a method blank? Was a method blank performed for each matrix and whenever a sample extraction was performed?	thod blank?	whenever a	sample extrac	tion was perfo	rmed?		
. Ei	lank c	ontaminated? Blank analysis date: ַ	1	2 23 08		Associated	Associated samples:	All 801/5	\$
Conc. units: ba / a Composind	Blank ID				ŭ	Samole Identification	uo		
	-44-811 Sho X	6	4	2	L	d			
ত	0.19	0.7.	0.70/11N	N11/hz.0	0.26/111	0.40/11M			
			-	1					
		sund	イリト						
CIRCLED RESULTS WERE NOT OLIALIEIED ALL RESULTS NOT CIRCLED WERE OLIALIEIED BY THE FOLLOWING STATEMENT.		I RESULTS NOT	- CIRCLED WEI	RE OLIALIFIED F	3V THF FOLLOV	VING STATEMEN	L L		

All contaminants within five times the method blank concentration were qualified as not detected, "U".

V:\Validation Worksheets\Dioxin90\BLANKS90.21

2DG # 18 386 BV		VALIDATION	VALIDATION FINDINGS WORKSHEET <u>Blanks</u>	EET	C	Page: 76 Reviewer: 77 2nd Reviewer: 7
ETHOD: HRG ease see qual <u>N N/A</u>	ioxins/Dibenzofurans flow for all questions mples associated wi nod blank performed	(EPA SW 846 Method 8 answered "N". Not applic th a method blank? for each matrix and whe	ic/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290) ifications below for all questions answered "N". Not applicable questions are identified as "N/A". Were all samples associated with a method blank? Was a method blank performed for each matrix and whenever a sample extraction was performed?	l as "N/A". ∕as performed?	V	
Y/N N/A Was the me Blank extraction date: <u></u> Conc. units: <u>pa</u> / <u>1</u>	Was the method blank contaminated? n date: <u>과내o</u> S Blank analys 24.1L	ontaminated? Blank analysis date: 2/15/00		Associated samples:	All water	
6	Blank ID		Sample	Sample Identification		
	204 2213-614	01				
<del>4</del>	1.1	1				
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ji.	1.1	1				
ι⊥	-	a. 84 /sou				
b	6.6	3.9 /1001				
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	1.1	Nasy 55.0				
٤	21.0	•				
z		10.56/son				
2						
d	٥.1					
প	3.8	1.7/1004				
		-				
			ED WEDE OF MILLERED BY THE FOLLOWING STATEMENT	STATEMENT <sup>.</sup>		

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

ere love LDC #: 1 8 3 % 82 SDG #:

## VALIDATION FINDINGS WORKSHEET

**Field Blanks** 

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

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

All Soils

Associated Samples:

¢

M11/0H ĺ б o 15.4 M 0.16 /5.44 ر ۲ 1.7/114  $\mathcal{V}$ <u>,</u> 100 0-26/114 1 ٢ 0.26/544 1.2/114 1.3/11v ٢ Sample Identification 5 1 0.24 MIV of o 0.49/11M Í -62 /10u 0.16 K.24 115/10M 3 ø N01/22.0 1 0 ( 1 <u> 2.3 / 5.2 / 5.0 </u> ( ł ۱ Blank ID 2 0.35 48.0 0.56 6.2 Compound Ø Ш Z ত CRQL

## Associated sample units: Blank units:

Sampling date:

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

Sample Identification Associated Samples: Blank ID Compound CRQL

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

5380 821	ere vover
LDC #: /	SDG #:

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

Ġ Page: 2nd Reviewer: Reviewer:

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

Pease see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". N N/A Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.

Was a MS/MSD analyzed every 20 samples of each matrix? Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

<b>ン</b> と	Y N N/A	Was a MS/MSD analy Were the MS/MSD pe	/zed every 20 Prcent recove	) samples of ries (%R) an	each mat id the rela	rix? tive percent difference	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?	: limits?	
-1									
#	Date	di dsw/sw	Compound	MS %R (Limits)	nits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
	┢	21 511	4	130 71	( Crel-017	021-0L) h21	(	k	no out lesin
			5		(hel-hL)	(133 ( 14-124)	( )	· -	
			<u>ч</u>		( 1-1-1-	~	( )		
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18380 82/	fee coner	ر
LDC #	SDG #:_	

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

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

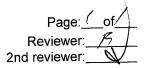
Were the correct internal standard (IS), quantitation ions and relative response factors (RRF) used to quantitate the compound?	Compound quantitation and CRQLs were adjusted to reflect all sample dilutions and dry weight factors (if necessary).	
N N/A	N N/A	<u> </u>

Qualifications	more /p									
Associated Samples	4									
Finding	no conjernation on	DE - 22-								
dr no do mon Barantes										
# Date										

Comments: See sample calculation verification worksheet for recalculations

LDC #: 18386B 2/ SDG #: free coner

### VALIDATION FINDINGS WORKSHEET Field Duplicates



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

Y N N/A Y N N/A Were field duplicate pairs identified in this SDG. Were target compounds detected in the field duplicate pairs?

		199/2, 4 situ	Di perence RPD
Compound		7 / //	
F	0.16	HER SHU	10-54 5.24 (= 5.4)
G	1.5	0.70	0.8 (411)
Ø	0.19	0.26	$0.07 ( \pm 5.4 )$
Q	0.62	0.49	$0.13 ( \le 11 )$

	Concentration ( )	
Compound		RPD

	Concentration ()	
Compound		RPD

	Concentration ()	
Compound		RPD