

### LABORATORY DATA CONSULTANTS, INC.

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

**ERM** 

March 19, 2008

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#	<u>Fraction</u>
F8A250221, F8A290158	Volatiles, Semivolatiles, Chlorinated Pesticides, Polychlorinated Biphenyls, Metals, Gasoline Range Organics, Diesel Range 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,

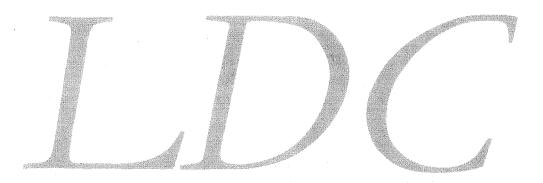
Erlinda T. Rauto

**Operations Manager/Senior Chemist** 

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

Volatiles



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

Project/Site Name:

**BRC Tronox Parcel H** 

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.

TSB-HR-08-10'MS

TSB-HR-08-10'MSD

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

<sup>\*\*</sup>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)	Α
1/30/08	Ethanol	0.00855 (≥0.05)	All water samples in SDG F8A250221	J (all detects) UJ (all non-detects)	Α

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-07-0' TSB-HR-07-10'** TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-10'** TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-0' TSB-HR-08-0' TSB-HR-08-0' TSB-HR-08-0'MSD 8038049-Blank	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	А
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)	А

### 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)	А

Date	Compound	%D	Associated Samples	Flag	A or P
2/5/08 (09:12)	1,1-Dichloroethane lodomethane 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-HJ-04-0' TSB-HR-04-0'** TSB-HR-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-HJ-08-0' TSB-HR-08-0' TSB-HR-08-0' 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-HJ-07-10'** TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-0' TSB-HR-08-0' TSB-HR-08-0'MSD 8038049-Blank	J+ (all detects)	А
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)	Α
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)	а

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

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
1/30/08	Dibromomethane	0.04735 (≥0.05)	All water samples in SDG F8A250221	J (all detects) UJ (all non-detects)	А
2/5/08 (09:12)	Acetonitrile	0.01921 (≥0.05)	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-HJ-07-10'** TSB-HS-06-10' TSB-HS-08-0' TSB-HS-08-0' TSB-HS-08-0' TSB-HR-08-0' TSB-HR-08-0'	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-HR-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-HS-06-10' TSB-HS-08-0' TSB-HS-08-0' TSB-HS-08-0' TSB-HS-08-0' TSB-HS-08-0'	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)	А

### VII. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Although the MS/MSD percent recoveries 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:

	Concentration (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-HR-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-HJ-07-10'	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)	Α	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-HR-04-0' TSB-HR-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' 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-04-10' 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-HJ-07-10' TSB-HJ-07-10' TSB-HJ-07-10' TSB-HJ-07-10'	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)	Α	Continuing calibration (ICV %D)
F8A250221	TSB-HJ-07-0'-FD TSB-HR-08-10'	Acetonitrile	J- (all detects) UJ (all non-detects)	Α	Continuing calibration (ICV %D)
F8A250221	TSB-TB-3 TSB-TB-2 TSB-TB-1	Dibromomethane	J (all detects) UJ (all non-detects)	Α	Continuing calibration (RRF)
F8A250221	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HR-04-0'** TSB-HR-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)	А	Surrogate recovery (%R)

### 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	А
F8A250221	TSB-TB-2	Dichloromethane	1.0U ug/L	Α
F8A250221	TSB-TB-1	Dichloromethane	1.0U ug/L	Α

LDC #: 18386A1 SDG #: F8A250221	VALIDATION COMPLETENESS WORKSHEET Level III/IV	Date: 3/10/08/ Page: / of /
Laboratory: Test America		•
METHOD: GC/MS Volatiles (E	PA SW 846 Method 8260B)	Reviewer:2nd Reviewer:

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

	Validation Area		Comments
l.	Technical holding times	Δ	Sampling dates: 1/24/08
II.	GC/MS Instrument performance check	Α	( 1)
III.	Initial calibration	SW	% PSD, 12
IV.	Continuing calibration/ICV	SW	1W=25
V.	Blanks	SW	
VI.	Surrogate spikes	5W	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	SW	Les D
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	<b>△</b>	
XI.	Target compound identification		Not reviewed for Level III validation.
XII.	Compound quantitation/CRQLs	<u> </u>	Not reviewed for Level III validation.
XIII.	Tentatively identified compounds (TICs)	<u></u>	Not reviewed for Level III validation.
XIV.	System performance	A	Not reviewed for Level III validation.
XV.	Overall assessment of data	A	
XVI.	Field duplicates	للاي	D = 11+ 12
XVII.	Field blanks	SW	TB= 16, 14, 18

Note:

A = Acceptable

ND = No compounds detected

D = Duplicate

N = Not provided/applicable SW = See worksheet

R = Rinsate

TB = Trip blank

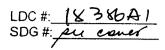
FB = Field blank

EB = Equipment blank

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

valida	coll + Water	sam	oie und	derwent Level IV valida	tion						
13	TSB-HJ-05-10'	1	113	TSB-HJ-07-0'**		3	214	TSB-HR-08-10'MS	31	803/135-Blank	
2 3	TSB-HJ-05-0'	1	124	TSB-HJ-07-0'-FD		3	224	TSB-HR-08-10'MSD	32 <b>2</b>	8036136 - Blank	2/4
3 <b>3</b>	TSB-HR-04-10'	1	133	TSB-HJ-07-10'		3	23		- 33 <b>3</b>	803849-Blank	2/5/
4 3	TSB-HJ-04-0'	1	143	TSB-HR-08-0'	·	3	24		٠.	8038277-Blank	7/c/
5 ን	TSB-HR-04-0'**	1	15 4	TSB-HR-08-10'		3	25		3 <del>5 S</del>	8031135 -BANK	1/30
6 3	TSB-HJ-04-10'	Ì	16	TSB-TB-3	V	1	26		36		(
7 <b>3</b>	TSB-HR-07-0'	2	17	<b>1∕</b> TSB-TB-2		2	27		37		
है 3	TSB-HR-07-10'**	2	18 I	√ TSB-TB-1		3	28		38		
9 <b>3</b> 7	TSB-HR-06-0'	2	193	TSB-HR-08-0'MS		•	29		39		
103	TSB-HR-06-10'	1	20 <b>3</b>	TSB-HR-08-0'MSD			30		40		

Batch #4 analyzed after ICAL

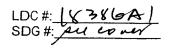


### **VALIDATION FINDINGS CHECKLIST**

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

Method: Volatiles (EPA SW 846 Method 8260B)

Method: Volatiles (EPA SW 846 Method 8260B)				
Validation Area	Yes	No	NA	Findings/Comments
Il Tochnica bolding times a service of the control				
All technical holding times were met.	/	1		
Cooler temperature criteria was met.		1		
II. GOMS havument periormance check				
Were the BFB performance results reviewed and found to be within the specified criteria?				
Were all samples analyzed within the 12 hour clock criteria?				
Did the laboratory perform a 5 point calibration prior to sample analysis?				
Were all percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?				
Was a curve fit used for evaluation?				
Did the initial calibration meet the curve fit acceptance criteria of > 0.990?				
Were all percent relative standard deviations (%RSD) $\leq$ 30% and relative response factors (RRF) $\geq$ 0.05?				
IIV Sommande financit kilose sa kalendaria				
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?	س			
Were all percent differences (%D) $\leq$ 25% and relative response factors (RRF) $\geq$ 0.05?		1		
Valenta na la companya da la companya da la companya da la companya da la companya da la companya da la companya				
Was a method blank associated with every sample in this SDG?				
Was a method blank analyzed at least once every 12 hours for each matrix and concentration?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
Memoralespices, its control of the property of the				
Were all surrogate %R within QC limits?				
If the percent recovery (%R) for one or more surrogates was out of QC limits, was a reanalysis performed to confirm samples with %R outside of criteria?		1		
Alta Nationa stake i Maria kan di Alta Maria Maria Maria Maria Maria Maria Maria Maria Maria Maria Maria Maria				
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?	AA	1	1	
Ли trabotatory.combol.sambles a				
Vas an LCS analyzed for this SDG?	1			



### VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
Reviewer: P7
2nd Reviewer: V

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per analytical batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX Regional Guality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
Xabilanal standards (**) 3. 25. 25. 25. 25. 25. 25. 25. 25. 25. 25				The Paris Harry Area with
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?	/			
Metaloga estructura de time adoit :				
Were relative retention times (RRT's) within + 0.06 RRT units of the standard?		-		
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	4			
Were chromatogram peaks verified and accounted for?				
Ale conjection quality is not selected by the				
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 a enemon a librario escomponas (108); a 2007, como escomponas (108); a 2007, como escomponas (108); a 2007,				
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?	1			
Were relative intensities of the major ions within $\pm$ 20% between the sample and the eference spectra?	-	-		
Did the raw data indicate that the laboratory performed a library search for all equired peaks in the chromatograms (samples and blanks)?	1	/		·
system performance was found to be acceptable.	7			
V. Coalillassa suomanantin				
verall assessment of data was found to be acceptable.			Microscope.	
V), pentupie ie spa				
eld duplicate pairs were identified in this SDG.		-	Name of the con-	
arget compounds were detected in the field duplicates.	1		1	
Vio Flad Danks (Fig. 1) 14 (Fig. 1)				
eld blanks were identified in this SDG.				
rget compounds were detected in the field blanks.		$\dashv$	$\dashv$	
	<u></u> l			

## TARGET COMPOUND WORKSHEET

METHOD: VOA (EPA SW 846 Method 8260B)

A. Chloromethane*	U. 1,1,2-Trichloroethane	OO. 2,2-Dichloropropane	III. n-Butylbenzene	CCCC.1-Chlorohexane
B. Bromomethane	V. Benzene	PP. Bromochloromethane	JJJ. 1,2-Dichlorobenzene	DDDD. Isopropyl alcohol
C. Vinyl choride**	W. trans-1,3-Dichloropropene	QQ. 1,1-Dichloropropene	KKK. 1,2,4-Trichlorobenzene	EEEE. Acetonitrile
D. Chloroethane	X. Bromoform*	RR. Dibromomethane	LLL. Hexachlorobutadiene	FFFF. Acrolein
E. Methylene chloride	Y. 4-Methyl-2-pentanone	SS. 1,3-Dichloropropane	MMM. Naphthalene	GGGG. Acrylonitrile
F. Acetone	Z. 2-Hexanone	TT. 1,2-Dibromoethane	NNN. 1,2,3-Trichlorobenzene	HHHH. 1,4-Dioxane
G. Carbon disulfide	AA. Tetrachloroethene	UU. 1,1,1,2-Tetrachloroethane	000. 1,3,5-Trichlorobenzene	IIII. Isobutyl alcohol
H. 1,1-Dichloroethene**	BB. 1,1,2,2-Tetrachloroethane*	VV. Isopropylbenzene	PPP. trans-1,2-Dichloroethene	JJJJ. Methacrylonitrile
1. 1,1-Dichloroethane*	CC. Toluene**	WW. Bromobenzene	QQQ. cls-1,2-Dichloroethene	KKKK. Propionitrile
J. 1,2-Dichloroethene, total	DD. Chlorobenzene*	XX. 1,2,3-Trichloropropane	RRR. m,p-Xylenes	LLLL. Ethyl ether
K. Chloroform**	EE. Ethylbenzene**	YY. n-Propylbenzene	SSS. o-Xylene	MMMM. Benzyl chloride
L. 1,2-Dichloroethane	FF. Styrene	22. 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	рррр.
O. Carbon tetrachloride	II. 2-Chloroethylvinyl ether	CCC. tert-Butylbenzene	WWW. Ethanol	מססס.
P. Bromodichloromethane	JJ. Dichlorodifluoromethane	DDD. 1,2,4-Trimethylbenzene	XXX. Di-isopropyl ether	RRRR.
Q. 1,2-Dichloropropane**	KK. Trichlorofluoromethane	EEE. sec-Butylbenzene	YYY. tert-Butanol	SSSS.
R. cis-1,3-Dichloropropene	LL. Methyl-tert-butyl ether	FFF. 1,3-Dichlorobenzene	ZZZ. tert-Butyl alcohol	TITT.
S. Trichloroethene	MM. 1,2-Dibromo-3-chloropropane	GGG. p-Isopropyltoluene	AAAA. Ethyl tert-butyl ether	uuuu.
T. Dibromochloromethane	NN. Methyl ethyl ketone	HHH. 1,4-Dichlorobenzene	BBBB. tert-Amyl methyl ether	VVVV.

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

8380A)	the count
LDC #: 1	SDG #:

## VALIDATION FINDINGS WORKSHEET

2nd Reviewer:

Page: Reviewer:\_

**Initial Calibration** 

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

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

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

A/A

N/A

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? Were all %RSDs and RRFs within the validation criteria of ≤30 %RSD and ≥0.05 RRF? Did the initial calibration meet the acceptance criteria? N/A

																T
Qualifications	1/43/A		-		1/25/2			1/m/c						and the second s		A POLICE AND A POL
Associated Samples	5031135-Blan 1C	All water	->	100 20 20 20 20 20 20 20 20 20 20 20 20 2		1		8038277-13/ant,	12, 15, 21, 22				-			
Finding RRF (Limit: >0.05)	0.04510	0.00977	0.00855	0.00.29	0.01869			0.00366								
Finding %RSD (Limit: <30.0%)					TEEF)										0.000	
Compound	pp	-ل <del>صاسات</del>	$\omega \omega \omega$	Man war	او			$\omega$								
Standard ID	1421		ICAL	ICAL				1047								
# Date	80 17/1	-	30/06/1	2/4/08				3/6/08	-							

LDC #: 18 38 64 ) SDG #: pre coner

### VALIDATION FINDINGS WORKSHEET Continuing Calibration

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".

Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?
Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's?
Were all %D and RRFs within the validation criteria of <25 %D and <a href="#color: blue;">20.05 RRF</a>?

<u></u>	7	<del></del>	_	-	 T	1 1	-		_	<del></del>	T	<del></del>	_			r -			<del>,</del>	7	_	_	 <del></del>	T	
Qualifications	14/A det							24/A dat	1/43/A					1+/A dut			}	17/4 あれ	A/1/n/-1 2					The state of the s	
Associated Samples	80 3 1135-8 Ank	+ all water							->					803849-Blank,	1-01.19.20			8038277-Blank,	12, 15, 21, 28						
Finding RRF (Limit: >0.05)									0.04735																
Finding %D (Limit: <25.0%)	33.60319	31.00877						48.37597						12.08435				34.53645	27. 6027U						
Compound	Indomethane	エエ					\$	2	RR					ę-l			a	ð	EBEE						
Standard ID	101							) )						ICV			1	HCV							
# Date	4 1/21/08	-					00/22/2	1,40,00	14:32				ПΠ	4 02			2011	200	•						

LDC #: X 3804/ SDG #:

### VALIDATION FINDINGS WORKSHEET Continuing Calibration

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

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

A/N X/A

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

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

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

																				T
		1+/v 4. <del>+</del>	)			1/43/4	4	V/ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	1											
	Associated Samples	403849 - Blank	71 61 11 4-	19,20				->												
	Finding RRF (Limit: >0.05)					0.01921		0.0000												
	Finding %D (Limit: <25.0%)	89. 46153	32. 31122	74.82024	33.4997			1000m							,					
	Compound	Н	Iodomethane	ф	2. Nitropropane	EFE 1		ر الا يا												
	Standard ID	مهم						eev												
	Date	2/5/B	9:17					3/2/08	71.01											
<u> </u>	#	+	+	+	4								L							

LDC #: | 83% 人/ SDG #:

## VALIDATION FINDINGS WORKSHEET

Page: Reviewer: 2nd Reviewer:

> Rease see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". METHOD: GC/MS VOA (EPA SW 846 Method 8260B) N N N N N N N N N

Was a method blank analyzed at least once every 12 hours for each matrix and concentration? 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.

Blank analysis date: Conc. units:

Associated Samples:

Compound	Blank ID									
					Ĉ	Sample Identification	llon	•	•	
	8031135-8ANK	an K	ي		7					
Wethylene chleride	0.16		1.00	7 7 7	0.0/0.0					
A CONTRACTOR OF THE CONTRACTOR			R	0.17/1.04	30.10			-	_	
arman)	è									
									-	
CROL										
li	2									
Blank analysis date: 1000 Conc. units: 49 Kg	0		Ası	Associated Samples:		12, 15				
0	Cl June			•						
	G VIIBIG				Š	Sample Identification	tlon			
Wildering the se	3038277-8an	700	12	5						
Methylana chlorida	2.8		6.1/M	4.2 /5.5W		-				
Asetone										
CROL										

All results were qualified using the criteria stated below except those circled.

Note: Common contaminants such as Mathylene 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, "U".

1# 1× 2×2 #1	G#: per coner
LDC #	SDG#

### VALIDATION FINDINGS WORKSHEET Field Blanks

7 4 Page: 2nd Reviewer: Reviewer:

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

Were target compounds detected in the field blanks? Were field blanks identified in this SDG? Brank units: wa Y N N/A

Brank units: wg | Associated sample units: wg |を Field blank type/ (circle one) Field Blank / Rinsate / アrip Blank)/ Other:

Associated Samples:

x2 + QN ) 0) 4-1 Sample Identification 214 士 BIN S ف 12 M Blank 10 Blank ID 6 24 100 0.20 4.3 Oi chlorome thane. Nethylene chloride Compound Ghlereform Acetone CRQL

XSZ FON ) CI 4-L Associated Samples: Blank units: wall Associated sample units: walked | Field blank type: (circle one) Field Blank / Rinsate/Trip Blank / Other: Blank units: ug

Sample Identification Blank ID Blank ID 17 124 108 0.14 Dichlorome thank Compound Chloroform Acetone SRO

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

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LDC#:	SDG#

### VALIDATION FINDINGS WORKSHEET Field Blanks

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

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

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

Blank units: wall Associated sample units. Yelf Blank / Other. Field blank type: (circle one) Field Blank / Rinsate / Trip Blank / Other.

8 + ON) 31 4-\_\_ Sample Identification Associated Samples: /2|N L T 7 Q Blank ID Blank ID 18 9,18 द्र <u>ح</u> - Lover Compound **Chioroform** Acetone CROL

Associated sample units: Blank units:

Field blank type: (circle one) Field Blank / Rinsate / Trio Blank / Other

Associated Samples:

Compound	Blank ID	Blank ID		Sample identification	ntification		
The second secon							
Methylene chloride							
Acetone							
Chloroform							
CRQL				ź			

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

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*	#
FDC	SDG

### VALIDATION FINDINGS WORKSHEET Surrogate Spikes

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".

N NA 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?

<del></del>	,	,			b											 				
Qualifications	3+/P dut			1t/Adut awas nonanal only																
nits)	(63-132-)	( 511-97)	( )	(66/15)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	<u> </u>	( )	( )	( )	
%Recovery (Limits)	77.			120											,					
Surrogate	BFB			BFB																
Sample ID	8036136 - BRNK			ગ																
Date																				
*																				

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

QC Limits (Water) 86-115 80-120 86-118 88-110

LDC #: (8386A SDG # Sec wer

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

Page: Reviewer: 2nd Reviewer:

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

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

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?

ZC IIITIIIS :	
any o porcent differences (NPD) within the c	

I											
#	Date	MS/MSD ID	Compound	 3%	MS %R (Limits)	MSD %R (Limits)	D mits)	RPD (1 imits)	nite)	Accordance of the second	1000
		M420	F	198	(021-93)					Associated Samples	۲
T			4	2	1201 66		7	7	000	<i>y</i>	m out les in
$\exists$			EFEE	243	(251-06) 672	<u> </u>		75	200		
1			HH		( )			-	3		
$\neg$					· ·		^	_	<u> </u>		A
$\exists$							_				
1					(		(	,	_		
		242	AA	152	051-30	)  51	(	)	· ·	51	MO DUA). Les
$\exists$			EFEE		(			2	3	_	
$\top$		Toda	Todomethane		( )		(		3		
1			HH		( )	)	(	_	~ <sub>○</sub> な	<b>\</b>	
十					(	)	_	<b>-</b>			
1							(	J			
$\neg \dagger$					( )	)	(	)	-		
_					)	`	^		^		
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$\dashv$					)	)	(		^		
_					( )		(	)			
					(	<u> </u>	)	)	(		
		Compound	pun		QC Limits (Soll)	its (Soil)		RPD (Soil)		QC Limits (Water)	RPD (Water)
	I	1,1-Dichloroethene			59-1	59-172%		< 22%		61-145%	< 14%
	S.	Trichloroethene			62-1	62-137%		< 24%		71-120%	< 14%
	>	Benzene			66-1	66-142%		< 21%		76-127%	< 11%
	.00	Toluene			59-1	59-139%		< 21%		76-125%	< 13%
	DD.	Chlorobenzene			60-133%	33%		< 21%		75-130%	< 13%

(8380 A) SDG #: LDC #:

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

Page: Reviewer: 9

KS/

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".

N/A	A/N	
<b>~</b> ₹	Z	
~	X	

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

	Qualification	JAMO ON																							
	Associated Samples	80 3849-Blank,	71 8 +1 41	1,, 1																					
	RPD (Limits)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )			( )	( )	(	( )	)	( )	)
CSD	%R (Limits)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(	( )	( )	( )	( )	( )	( )	( )	( )	
SOT	%R (Limits)				(		( )	( )	(	( )	( )	( )	)	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
	Compound	3-Nitrographence	f (																						
	CCS/FCSD ID	507-6h08608	ł																						
	Date																								

SDG#: Sec Cover

### LDC#: 18386 A) VALIDATION FINDINGS WORKSHEET **Field Duplicates**

Page:_	of	
Reviewer:_	مو	7
nd reviewer:	10	

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

1	$\frown$		
1	Υ'	N	N/A
	Y	N	N/A
١	_		

Were field duplicate pairs identified in this SDG2

	Concentra	tion ( ug kg	Dilago	ene_
Compound		12	VI PR	rene_ PD
dichloromethane	5.8	6.1	0.3	(£ 5.4)
DOD	0.4	5.34	4.89	(= 5.4
			1	
	Concentral	tion (		
Compound			RPD	
	<del></del>			

	Concentration (	=
Compound		RPD
		·
·		
		·

	Concentration ( )	
Compound		RPD

LDC#: 1838bA | SDG#: 244 caner

# VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: 1 of 7 Reviewer: 2nd Reviewer: 1

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

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

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

 $A_{k}$  = Area of compound,  $A_{k}$  = Area of as  $C_{x}$  = Concentration of compound,  $C_{k}$  = Concentra S = Standard deviation of the RRFs X = Mean of the RRFs

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

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	RRF ( 22 std)	RRF (72) std)	Average RRF (initial)	Average RRF	%RSD	usa%
<u>-1</u> 2	ICAL	3/4/08	Vingl chloriolC (1st internal standard)	0.77522	0.718v2	0.73013	043013	0.161.0	2
			(2nd internal standard)	1.79313	1.79313	1.80952	(3P28-1	18175.4	
$\parallel$			( ス~ かん) (3rd internal standard)	1.53477	1-53477	1.5456	<i>188⁻</i> 1	3.54081	3,5408
7			しょう (1st internal standard)	0,00342	0.00347	0.0039	16200.0	S\$015.11	Ħ
T			Vince that (2nd internal standard)	6.50639	6.5063.0	0.54575	St 245.0	13.72254	
1			(3rd internal standard)	0.58190	0-88190	0.8997	(1798x-0	9.47511	9.47511
8			(1st internal standard)						1
T			(2nd internal standard)						
			(3rd internal standard)						
4			(1st internal standard)						
T			(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

1838BA SDG# 12 sore LDC#:

### Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

ó Page: 2nd Reviewer: Reviewer:

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

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

% Difference = 100  $^*$  (ave. RRF - RRF)/ave. RRF RRF =  $(A_x)(C_s)/(A_s)(C_x)$ 

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

Where: ave. RRF = initial calibration average RRF RRF = continuing calibration RRF  $A_k$  = Area of compound,  $A_k$  = Area of  $C_k$  = Concentration of compound,  $C_k$  = Concer

_								
				·	Reported	Recalculated	Renorted	Recalculated
#	Standard ID	Calibration Date	Compound (Reference internal Standard)	Average RRF	RRF	RRF	<b>α</b> %	<b>Q</b> %
-	Sev.	80/she	Ving Chlande (1st internal standard)	0.13013	0.829%	(cc)	7 7 2	2
		4:15	Ethyl Benz C. (2nd Ditemal standard)	1.809.22	1-87 688	1.8768	3.7227	15,57,7
			い。ユーンCB Tard internal standard)	1.545७।	1.531 80	08/85/	0.89307	0.89307
7	col	2 200	して、 (1st internal standard)	0.0029	0.00359	0.00.0	11.12218	1 -
		h1:01	Vine that Visal National Standard)	27542.0	69 785.0	0.58219	571-11-9	
╝			1, 5, 5- Mendernal standard	1.07630	holbo.1	1.08104	126984	126954 1.26954
က			(1st internal standard)				12/-	2197
			(2nd internal standard)					
			(3rd internal standard)					
4			(1st internal standard)					
			(2nd internal standard)					
			(3rd internal standard)					

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results. LDC #: 18 386A)

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

Page:_	_/	_of_	
Reviewer:		p.	7
2nd reviewer:			

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found

SS = Surrogate Spiked

S

am	ple	ID:_	#	5

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8	50	54.6314	109	109	6
Bromofluorobenzene		52.9992	106	106	1
1,2-Dichloroethane-d4		48.7043	97	97	
Dibromofluoromethane		48.6525	97	97	

Sample ID:

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

Sample ID:

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

Sample ID:

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

Sample ID:

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

LDC #: 18 3 8 4 2002 SDG #:\_

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

Page: Reviewer:

2nd 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

SSC = Spiked sample concentration Where:

SC = Sample concentration

SA = Spike added

MSDC = Matrix spike duplicate percent recovery

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

MSC = Matrix spike percent recovery

19+ 20 MS/MSD sample:

	σ.	Spike	Sample	Spiked Sample	ample	Matrix Spike	Spike	Matrix Spike Duplicate	Duplicate	MS	MS/MSD
Compound	(vey	2 (kg)	(we ke)	Concentration ( )	ration  X.	Percent Recovery	ecovery	Percent Recovery	ecovery		RPD
	MS	U'' /	?	MS	O MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
1,1-Dichloroethene	\$7.7g	52.9	ηd	58.3	1-65	011	Ol 1	13	113	۲. بر	2.4
Trichloroethene		- 		80.4	55.1	٥	90	104	hal	8.8	3
Benzene				56.0	<u>क</u> रें	٥	9	102	701	n o	2.6
Toluene				2.12	£.3	103	103	201	201	6.1	2.0
Chlorobenzene	<b>-&gt;</b>	<b>~</b>	->	53.0	54.2	50	Jø!	60	70.2	7.7	

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

18 386 A SDG #: LDC #:

### Laboratory Control Sample Results Verification VALIDATION FINDINGS WORKSHEET

ō \ 2nd Reviewer: Reviewer:\_ Page:

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

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

% Recovery = 100 \* SSC/SA

Where: SSC = Spiked sample concentration SA = Spike added

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

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

507-6408Cas LCS ID:

	USD 1801	, Cox		Recalc Reported Recalculated									-		
	1,	Percent		Reported					42						
	CS	Recovery		Recato	99	100	26	97	96						
		Ĭ		Percent Recovery		Reported	66	44	9	9.7	90				
	ample	ample ration	K	7 Icsn	\$ 2				->						
	Spiked S	Spiked Sample Concentration (\sqrt{\sq}}}}}}}}}}}}}} \end{\sqrt{\sqrt{\sqrt{\sqrt{\sqrt{\sqrt{\sqrt{\sqrt{\sqrt{\sqrt{\sqrt{\sqrt{\sqrt{\sqrt{\sqrt{\sqrt{\sqrt{\sqrt{\sq}}}}}}}}}}}} \end{\sqnt{\sqnt{\sqrt{\sq}}}}}}}}} \end{\sqnt{\sqnt{\sq}}}}}}}}} \end{\sqnt{\sqnt{\sq}}}}}}}} \sqnt{\sqnt{		108	۶. <del>ما</del>	47.	4. Vh	48.4	48.2						
	ike	Added	0	1 CSD	AC.	_				ı					
	ds:	Ad ov		108	50.0				>,						
		Compound	(2) 日本のでは、これでは、大きなないできた。		1,1-Dichloroethene	Trichloroethene	Benzene	Toluene	Chlorobenzene						

Comments: Refer to Laboratory Control Sample findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results. LDC #: 18 386 A 1 SDG #: pu coner

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

Reviewer: 2nd reviewer:

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

Y N N/A Y N N/A

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

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

Concentration =

 $(A_x)(I_s)(DF)$ 

 $\overline{(A_{is})(RRF)(V_o)(\%S)}$ 

Area of the characteristic ion (EICP) for the A, compound to be measured

Area of the characteristic ion (EICP) for the specific A<sub>is</sub> internal standard

Amount of internal standard added in nanograms l<sub>s</sub>

RRF Relative response factor of the calibration standard. V<sub>o</sub>

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

Df Dilution factor.

%S Percent solids, applicable to soils and solid matrices Example:

Sample I.D. #5 Aufore

	only.				
#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		x = (10043 \/ 1	-0.13125		
		$\frac{\times}{50} = \frac{10043}{619429} = \frac{1}{0.06192}$			
		X = 6-529			
				····	
		Lind = 6.529 = 6.	& ug/ks/		
		0.959	1 0 1 3		
		·			
					***
			1		

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

Project/Site Name:

BRC Tronox Parcel H

**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)	А
1/30/08	Ethanol	0.00855 (≥0.05)	All water samples in SDG F8A290158	J (all detects) UJ (all non-detects)	Α

### IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

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

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

Date	Compound	%D	Associated Samples	Flag	A or P
1/30/08	Bromomethane	48.37592	All water samples in SDG F8A290158	J+ (all detects)	А
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)	. 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 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)	А

### 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)					
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)	-	-

### 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)	Α	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-05-10'	Ethanol 2-Methylhexane 3-Ethylpentane n-Heptane	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	Α	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)	Α	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-08-10' TSB-HJ-08-10' TSB-HR-05-0'	Acetonitrile 4-Methyl-2-pentanone 4-Chlorotoluene	J+ (all detects) J+ (all detects) J+ (all detects)	А	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)	А	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	А
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	Α

### 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 .	А
F8A290158	TSB-HR-06-0'	Dichloromethane	7.4U ug/Kg	Α
F8A290158	TSB-HR-06-0'-FD	Dichloromethane	5.4U ug/Kg	Α
F8A290158	TSB-HR-06-10'	Dichloromethane	7.3U ug/Kg	Α
F8A290158	TSB-HJ-08-0'	Dichloromethane	8.8U ug/Kg	Α
F8A290158	TSB-HJ-08-10'	Dichloromethane	5.8U ug/Kg	Α
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	Α
F8A290158	RINSATE-2	Acetone	8.0U ug/L	Α

LDC #: 18386B1	_ VALIDATION COMPLETENESS WORKSHEET	
SDG #: F8A290158	Level III	
Laboratory: Test America	<u> </u>	
-		2nc

Reviewer: 2nd Reviewer:

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

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

	Validation Area		Comments
1.	Technical holding times	Δ	Sampling dates: \28/08
II.	GC/MS Instrument performance check	$\triangle$	1 '
III.	Initial calibration	لىنى	% BD (2 Zo.990
IV.	Continuing calibration/ICV	لىپى	INEX
V.	Blanks	SW	
VI.	Surrogate spikes	3 ح	
VII.	Matrix spike/Matrix spike duplicates	SV	
VIII.	Laboratory control samples	Δ	LCS/D
IX.	Regional Quality Assurance and Quality Control	N	
X.	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	SW	D = 3,4
XVII.	Field blanks	5W	R = 12 TB = 10 TB=

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:	water	~								
1 3	TSB-HJ-10-0'		1	11	7- TSB-TB-2-1/28/08	W	2 21 1	8031135	31		1/30
2 3	TSB-HJ-10-10'		١	12	RINSATE-2		3 22 Z	80 36 136	32	nonunal only	2/4
3 2	TSB-HR-06-0'	0	1	13	ン TSB-TB-03-1/28/08	1	23 3	8043263	33	· ·	2/11
4 3	TSB-HR-06-0'-FD	b	1	14	TSB-HR-05-10'MS		24	8.	34	,	
5 <sup>3</sup> 7	TSB-HR-06-10'		١	15	TSB-HR-05-10'MSD		25		35		
6 3	TSB-HJ-08-0' ·		*	16			26		36		
73	TSB-HJ-08-10'.		~	17			27		37		
8 3	TSB-HR-05-0'		2	18			28		38		
9 Y	TSB-HR-05-10'	~~~	γ	19			29		39		
10	TSB-TB-1-1/28/08	W	Ţ	20			30		40		

## TARGET COMPOUND WORKSHEET

METHOD: VOA (EPA SW 846 Method 8260B)

A. Chloromethane*	U. 1,1,2-Trichloroethane	OO. 2,2-Dichloropropane	III. n-Butylbenzene	CCCC.1-Chlorohexane
B. Bromomethane	V. Benzene	PP. Bromochloromethane	JJJ. 1,2-Dichlorobenzene	DDDD. Isopropyl alcohol
C. Vinyl choride**	W. trans-1,3-Dichloropropene	QQ. 1,1-Dichloropropene	KKK. 1,2,4-Trichlorobenzene	EEEE. Acetonitrile
D. Chloroethane	X. Bromoform*	RR. Dibromomethane	LLL. Hexachlorobutadiene	FFFF. Acrolein
E. Methylene chloride	Y, 4-Methyl-2-pentanone	SS. 1,3-Dichloropropane	MMM. Naphthalene	GGGG. Acrylonitrile
F. Acetone	Z. 2-Hexanone	TT. 1,2-Dibromoethane	NNN. 1,2,3-Trichlorobenzene	HHHH. 1,4-Dioxane
G. Carbon disulfide	AA. Tetrachloroethene	UU. 1,1,1,2-Tetrachloroethane	000. 1,3,5-Trichlorobenzene	IIII. Isobutyl alcohol
H. 1,1-Dichloroethene**	BB. 1,1,2,2-Tetrachloroethane*	VV. Isopropylbenzene	PPP. trans-1,2-Dichloroethene	JJJJ. Methacrylonitrile
I. 1,1-Dichloroethane*	CC. Toluene**	WW. Bromobenzene	QQQ. cis-1,2-Dichloroethene	KKKK. Propionitrile
J. 1,2-Dichloroethene, total	DD. Chlorobenzene*	XX. 1,2,3-Trichloropropane	RRR. m,p-Xylenes	LLLL. Ethyl ether
K. Chloroform**	EE. Ethylbenzene**	YY. n-Propyibenzene	SSS. o-Xylene	MMMM. Benzyl chloride
L. 1,2-Dichloroethane	FF. Styrene	ZZ. 2-Chlorotoluene	TTT. 1,1,2-Trichloro-1,2,2-trifluoroethane	NNNN.
M. 2-Butanone	GG. Xylenes, total	AAA. 1,3,5-Trimethylbenzene	UUU. 1,2-Dichlorotetrafluoroethane	0000.
N. 1,1,1-Trichloroethane	HH. Vinyl acetate	BBB. 4-Chlorotoluene	VVV. 4-Ethyltoluene	pppp.
O. Carbon tetrachloride	II. 2-Chloroethylvinyl ether	CCC. tert-Butylbenzene	WWW. Ethanol	<b>a</b> aaa.
P. Bromodichloromethane	JJ. Dichlorodifluoromethane	DDD. 1,2,4-Trimethylbenzene	XXX. Di-isopropyl ether	RRRR.
Q. 1,2-Dichloropropane**	KK. Trichlorofluoromethane	EEE. sec-Butylbenzene	YYY. tert-Butanol	SSSS.
R. cis-1,3-Dichloropropene	LL. Methyl-tert-butyl ether	FFF. 1,3-Dichlorobenzene	ZZZ. tert-Butyl alcohol	ттт.
S. Trichloroethene	MM. 1,2-Dibromo-3-chloropropane	GGG. p-Isopropyltoluene	AAAA. Ethyl tert-butyl ether	uuuu.
T. Dibromochloromethane	NN. Methyl ethyl ketone	HHH. 1,4-Dichlorobenzene	BBBB. tert-Amyl methyl ether	vvvv.

4

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

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LDC #: T	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". NNNN

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?

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

Qualifications	18/4	7	7											
Associated Samples	\$031135-Blank	All water	<b>→</b>											
Finding RRF (Limit: >0.05)	0.240.0	<del>0.00977</del>	0.00855											
Finding %RSD (Limit: <30.0%)														
Compound	RR	بمصمم	2000											
Finding %RSD Finding RRF  Standard ID Compound (Limit: <0.05)	IGIL		ICAL											
# Date	89 141	•	1/30/00											

LDC # [8368] SDG # ser com

## VALIDATION FINDINGS WORKSHEET Continuing Calibration

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". YN N/A

Was a continuing calibration standard analyzed at least once every 12 hours for each instrument? Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's ? Were all %D and RRFs within the validation criteria of ≤25 %D and ≥0.05 RRF ?

N N/A

Qualifications	17/A da								J+/A dut	1/W1/A			1/W/A	1+/A dut			->		11/A dut	-		
Associated Samples	8031135-Blank	All water								<b>→</b>			304 3763 18 ank	41 50:15			M					
Finding RRF (Limit: >0.05)										0.04735			0.006491									
Finding %D (Limit: <25.0%)	33.60319	31.00872			•				48.37592					28.63874	27. 01574	25.56456	त्र6.94019		27.39844	27.930SS	00896.SE	
Compound	1006methane	H#							В	RR			$\omega \omega \omega$	3 3 3	2 + Melhy / hexanc	3 + Ethylpen bane	. heptana	2	EFFE	7	BBB	
Standard ID	101								56 <				cen		8	3.	u	+	4 par 100			
Date	1 21 08	-							1 30 08	14:37		,	30 11/2	71:81				,	2 11 00	Us:50		
) #	+	+				$\prod$		<u> </u>	+					+	+	+	+		+	+	+	

SDG #: 100 2002 LDC #: 18386

## VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Reviewer:

> Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". METHOD: GC/MS VOA (EPA SW 846 Method 8260B) V N/N

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.

130/08 Plank analysis date: Conc. units: Y/N N/A

Y N N/A

ad vate sample 0 4 0 Associated Samples:

Sample Identification 0.72/104 3 2 5 no.1/61.0 0-51/15.0 J 2 8031135-Blank Blank ID ٥.اد Vichloromethane Methylene chloride Compound Acetone CRO

Blank analysis date: Conc. units:

Associated Samples:

Methylene chloride Acetone	Blank ID
Methylene chloride Acetone	Sample Identification
Methylene chloride Acetone	
Acetone	
<u>CRai.</u>	

All results were qualified using the criteria stated below except those 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 also qualified as not detected, "U". Other contaminants within five times the method blank concentration were also qualified as not detected, "U".

LDC #: 1X3806 SDG #:

### VALIDATION FINDINGS WORKSHEET Field Blanks

41/ soils

Associated Samples:

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

> Were field blanks identified in this SDG? METHOD: GC/MS VOA (EPA SW 846 Method 8260B) Y N/N/A

Were target compounds detected in the field blanks? X/N N/A

Blank units: walk Associated sample units: walke | Kalker | Field blank type: (circle one) Field Blank / Rinsate) Trip Blank / Other

15/4 5.8/7 **≤** 8.8 Q 7.3/W Sample Identification 3,0/5,44 7.4/2 3.915.24 6.1/W Blank ID Blank ID 12 1208 Se O 9 Dichlow ethane Compound Methylene chloride Chloreform Acetone CRQL

Associated Samples: Blank units: we hasociated sample units: which the Field blank type: (circle one) Field Blank / Rinsate / Trip Blank / Other:

< 1102 11 ×

Compound	Blank ID 📝 Blank ID	Blank ID			Sample Identification	ntification		
	1 20 20/2		0					
Pichlorone than Mathylana chlorida	1,		h/5-9					
Acetone	6.0		14.5 LA					
Shlereform								
				-				
CROL								

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

12 50 E LDC #: 18 28013 SDG#:

### VALIDATION FINDINGS WORKSHEET Field Blanks

Page: 2nd Reviewer: Reviewer:

DN + XDIA 54 Sample Identification Associated Samples: H Y/N N/A Were target compounds detected in the jielu biains :

Blank units: vo / Associated sample units: vo / kg
Field blank type. (circle one) Field Blank / Rinsate / Trip Blank) Other: [ D Were field blanks identified in this SDG? METHOD: GC/MS VOA (EPA SW 846 Method 8260B) Blank ID Blank ID [0 1/20/02 0.7 4.0 sichloro metham Compound Methylene chloride Y N/N/A **Ghloroforn** Acetone

DN + XOIN 0 4 ف Sample Identification Associated Samples: Field blank type: (circle one) Field Blank / Rinsate/Trip Blank / Other: 15/31 Ø Associated sample units: Blank ID Blank ID 30/22/1 o A schlorome than Blank units: vey (レ Compound Methylene chloride Chleroform Acetone

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

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LDC #:	SDG #:

### VALIDATION FINDINGS WORKSHEET Field Blanks

Page: / ot/ 2nd Reviewer: Reviewer:

18260B)	C C C C C C C C C C C C C C C C C C C
3 Method	
SW 846	
OA (EPA	-
SC/MS V(	•
<u> È<b>тнор</b>:</u> GC/MS VOA (ЕРА SW 846 Methoc	× : : : : 4

7

Sample Identification Associated Samples: 3 8,0 B 6.27 Dirchlar nethan Chleroform Acetone CRaL

Associated sample units: Blank units:

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

Associated Samples:

Compound	Blank ID	Blank ID		Sample ide	Sample identification		
	11 1						
Methylene chloride							
Acetone							
Chloroform							

CRQL

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

LDC #: 1838b) SDG #:

## VALIDATION FINDINGS WORKSHEET Surrogate Spikes

2nd Reviewer: Reviewer:

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

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

-Y N/N/A

Were all surrogate %R within QC limits?

Y N/N/A

If the percent recovery (%R) for one or more surrogates was out of QC limits, was a reanalysis performed to confirm samples with %R out of outside of criteria?

Qualifications	1+/pax						1														
its)	( 63-133)	(511-77)	(	( )	( )	( )	( )	( )	(	( )	( )	( )	( )	^	(	( )	^	( )	(	(	(
%Recovery (Limits)	428 124	(511-33)																			
Surrogate	BFB				-																
Sample ID	8036136 - Blank																				
Date												-									
*																					

SMC4 (DFM) = Dibromofluoromethane 80-120

QC Limits (We	88-110	86-115	00100
র র			

LDC#: 1838081 SDG#: Sec cover

# VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Page: / of Reviewer: / C7

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/N/N/

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?

# Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
	14+15	Dichlerometh	Dichleromethane 157 (47-145)	(	(	p	Mo court. 1 . C. C.
		ΔA	( OS1-SC ) E91	(160 (25-150)	)		-
		НН	)	li	× (22)	->	
			( )	( )	( )		A
			( )	( )	)		
			)		( )		
			( )	( )	( )		
			)	( )			
					( )		
			( )	( )	( )		
				( )			
			( )		( )		
			)	( )			
			( )	( )	( )		
			( )	( )	( )		
			· ·	( )	( )		
			( )	( )	( )		
				( )	( )		
	Con	Compound	QC Limits (Soil)	ts (Soil)	RPD (Soil)	QC Limits (Water)	RPD (Water)
Ï	1,1-Dichloroethene		59-172%	72%	< 22%	61-145%	< 14%
Š	Trichloroethene		62-137%	37%	< 24%	71-120%	< 14%
>	Benzene		66-142%	42%	< 21%	76-127%	< 11%
CC	Toluene		59-139%	%68	< 21%	76-125%	< 13%
a O	Chlorobenzene		60-133%	33%	970	, do 0, 1	

LDC#: 18380Bl SDG#: Sec Cover

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

Page:/_of	_
Reviewer:	7
2nd reviewer:	

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

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?

	Concentral	tion ()	Difference
Compound	3	4	\ \ RPD
Dichloro methane	7.4	3,0	4.4 ( 5.4)
Toluene	0.54	5,44	4.86 (5.4)
			•

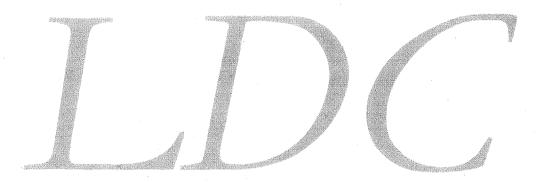
	Concentration (	
Compound		RPD

	Concentration (	
Compound		RPD

	Concentration (	
Compound		RPD

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

Semivolatiles



### 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

<sup>\*\*</sup>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	Pentachiorophenol	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 EPÁ 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-07-10'** TSB-HR-07-10'** TSB-HR-06-0' TSB-HR-06-0' TSB-HR-07-10'+* TSB-HR-07-10' TSB-HR-07-0'-FD TSB-HR-08-0' TSB-HR-08-0' TSB-HR-08-10'	Pentachlorophenol	None	Р	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	А
F8A250221	TSB-HJ-05-0'	Unknown aldol condensate (4.2526)	7600U ug/Kg	Α
F8A250221	TSB-HR-04-10'	Unknown aldol condensate (4.2534)	7800U ug/Kg	Α
F8A250221	TSB-HJ-04-0'	Unknown aldol condensate (4.2519)	9400U ug/Kg	Α
F8A250221	TSB-HR-04-0'**	Unknown aldol condensate (4.2572)	7700U ug/Kg	Α
F8A250221	TSB-HJ-04-10'	Unknown aldol condensate (4.2632)	8300U ug/Kg	Α
F8A250221	TSB-HR-07-0'	Unknown aldol condensate (4.2583)	8000U ug/Kg	Α
F8A250221	TSB-HR-07-10'**	Unknown aldol condensate (4.2472)	9300U ug/Kg	Α
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	А
F8A250221	TSB-HJ-07-0'**	Unknown aldol condensate (4.2678)	9600U ug/Kg	А

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	Α
F8A250221	TSB-HJ-07-10'	Unknown aldol condensate (4.2555)	9200U ug/Kg	Α
F8A250221	TSB-HR-08-0'	Unknown aldol condensate (4.2578)	9300U ug/Kg	А
F8A250221	TSB-HR-08-10'	Unknown aldol condensate (4.2595)	9100U ug/Kg	Α

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

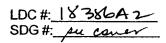
No Sample Data Qualified in this SDG

SDG Labor	#: 18386A2 #: F8A250221 ratory: Test America		_	Le	evel III/I		WORKSHEE	Т	Date: 3/// Page: _/of / Reviewer:
The s		e revi				validati	on areas. Valida	tion find	dings are noted in attached
	Validation	Area					Com	ments	
1.	Technical holding times			Δ	Sampling	dates:	1/24/09		
II.	GC/MS Instrument performa	ance c	heck	Δ			7-4		
III.	Initial calibration			Δ	% PSS	۲, ۲	2 ZO-990		
IV.	Continuing calibration/ICV			3	l.	52			
V.	Blanks			3					
VI.	Surrogate spikes			Δ					
VII.	Matrix spike/Matrix spike du	plicate	es	کیک					
VIII.	Laboratory control samples			A	LCS	>			
IX.	Regional Quality Assurance	and (	Quality Control	N			•		
X.	Internal standards		4	^					
XI.	Target compound identificat	ion		^1	Not revie	wed for I	_evel III validation.		
XII.	Compound quantitation/CR	QLs		Δ	Not revie	wed for I	_evel III validation.		
XIII.	Tentatively identified compo	unds	(TICs)	Δ	Not revie	wed for I	_evel III validation.		
XIV.	System performance			٨	Not revie	wed for I	_evel III validation.		
XV.	Overall assessment of data			A					
XVI.	Field duplicates			NP	0=	11+	12		
XVII.	Field blanks			Ŋ					
Note:	A = Acceptable N = Not provided/applicable SW = See worksheet ed Samples: ** Indicates sam		R = Rins FB = Fie	o compounds sate eld blank	s detected		D = Duplicate TB = Trip blank EB = Equipment bla	ank	
1	TSB-HJ-05-10'	11	TSB-HJ-07-0'	**	<u>\$</u> 1	802	9394-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	
<b>-</b> 5	TSB-HR-04-0'**	15	TSB-HR-08-1	D'	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	

TSB-HR-07-10'\*\*

TSB-HR-06-0'

TSB-HR-06-10'

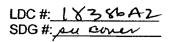


### **VALIDATION FINDINGS CHECKLIST**

Page: / of 2
Reviewer: \_ F7
2nd Reviewer: \_ \_\_\_\_

Method: Semivolatiles (EPA SW 846 Method 8270C)

Metrod. Serrivolatiles (EFA SW 640 Metrod 6270C)	T.,	T	T	
Validation Area	Yes	No	NA	Findings/Comments
All technical holding times were met.				
Cooler temperature criteria was met.				
D SWAS ASTRONOMIC STRANCISC SCC25				
Were the DFTPP performance results reviewed and found to be within the specified criteria?				
Were all samples analyzed within the 12 hour clock criteria?		GE 0777.05.05	22 17 20 20 20	
Dispritore distribution			and the second second	
Did the laboratory perform a 5 point calibration prior to sample analysis?				11 Mart 1974 2 1 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2
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 <a> 0.990?</a>	/			
Were all percent relative standard deviations (%RSD) $\leq$ 30% and relative response factors (RRF) $\geq$ 0.05?				
TV. Comin Ungrealite Styri				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?				
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?	/			
Were all percent differences (%D) ≤ 25% and relative response factors (RRF) ≥ 0.05?		_		
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.				
vy somostike				
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?				-
M. Zaro-sciezwiatowania-kinjipanes				
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?				
yju bank alksyystyji olesinijas 1821. Lista 1924 ili sa 1925. Lista 1925.				
Was an LCS analyzed for this SDG?	1			



### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 7 Reviewer: 7 2nd Reviewer: \( \subseteq \)

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?			٠	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?			-	
Ko Register Prairie/essuegovani i Carilly Ostato				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?			_	
X interval statesticis				
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?				
exi elge (computationimic)			4-11-11	
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?				
ZUI SEI DE SUITE BEI BEI BEI BEI BEI BEI BEI BEI BEI BE				
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?		_		
Min fizeriaditæt faktalitiste applystigda tillega				
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)?				
an sveter experiments				
System performance was found to be acceptable.			A.F. C. S.	
yearn periodical to too too too too too too too too too				
Ar y sell assessificateds				
Overall assessment of data was found to be acceptable.				
Market elepages				
Field duplicate pairs were identified in this SDG.	-			
Target compounds were detected in the field duplicates.		1		
Wisipenies				
rield blanks were identified in this SDG.		1		
arget compounds were detected in the field blanks.			1	

## VALIDATION FINDINGS WORKSHEET

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

A. Phenol**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenol**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	VV. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	OO. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	111.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	חחח
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	VVV.
O. 2,4-Dimethyiphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	www.

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

VALIDATION FINDINGS WORKSHEET

LDC # 18386A7

Continuing Calibration

Page: 7 of

Plaqse see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". SDG #: Are COMM BNA (EPA SW 846 Method 8270C)

A/N

Was a continuing calibration standard analyzed at least once every 12 hours of sample analysis for each instrument? Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's? Were all %D and RRFs within the validation criteria of ≤25 %D and ≥0.05 RRF?

Qualifications	d/ own	7													
Associated Samples	All+ Blk														
Finding RRF (Limit: >0.05)															
Finding %D (Limit: <25.0%)	2253156														
Compound	77 (00)														
Standard ID	ccv														
Date	80/7/2	30:45													
#		-													

7	}
1386	2
<u>4</u>	#:
9	SDC

## VALIDATION FINDINGS WORKSHEET

<u>Blanks</u>

Page:o 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".

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

Was a method blank analyzed for each concentration preparation level?

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

Y M N/A Was the blank contaminated? If yes, please see qualification below. Blank analysis date: フレック

1.20.21 4.2472) 9300 4. 2833 4.2632) 2006.2 Sample Identification 4.2572) 1305 7 0.5% 9400 Associated Samples: 2800 1. xx 4 800 840D 14.2676 2 8029394 - Blank (ख्रिंग) Blank ID 20098 contration Conc. units: Malka Compound **MAKADUS** A 100 7

Associated Samples: Blank extraction date: 1/2/08 Blank analysis date: 2/6/08 Conc. units: 교

Compound

9100 14.2595 Sample Identification 9300 4.2555 42000 4.269) 7 4. 267B ŏ Blank ID 4656298 Blank 12x 1

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".

LDC #: 18386 A.S. SDG #: 450 COLL

## Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

Page: Reviewer:

2nd Reviewer:\_

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

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

Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits? Was a MS/MSD analyzed every 20 samples of each matrix?

N M/A A N/A

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

25.52 Qualifications no out Associated Samples <del>ار</del> ح RPD (Limits) 687 10-9 %R (Limits) Q 16-99 6-01) MS %R (Limits) 7 Compound 开 MS/MSD ID 12 × 17 Date

	Compound	QC Limits (Soli)	RPD (Soil)	QC Limits (Water)	RPD (Water)		Compound	QC Limits (Soil)	RPD (Soll)	QC Limits (Water)	RPD (Water)
Α̈́	Phenol	26-90%	≥ 35%	12-110%	< 42%	GG.	Acenaphthene	31-137%	< 19%	46-118%	< 31%
σ	2-Chlorophenol	25-102%	%05 ⋝	27-123%	< 40%	=	4-Nitrophenol	11-114%	< 50%	10-80%	> 50%
ш	1,4-Dichlorobenzene	28-104%	< 27%	36-97%	< 28%	축	2,4-Dinitrotoluene	28-89%	< 47%	24-96%	≥ 38%
٦	N-Nitroso-di-n-propylamine	41-126%	≪8€ ⋝	41-116%	~38%	Ë	Pentachlorophenol	17-109%	< 47%	9-103%	× 20%
æ	1,2,4-Trichlorobenzene	38-107%	%62 ⋝	39-98%	≥ 28%	77.	Pyrene	35-142%	× 36%	26-127%	< 31%
>	4-Chloro-3-methylphenol	26-103%	%£ ≥	23-97%	< 42%						

LDC # 183847 SDG #: Au com

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

/of / 2nd Reviewer: Page: Reviewer:

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

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

 $RRF = (A_x)(C_y)/(A_m)(C_x)$  average RRF = sum of the RRFs/number of standards %RSD = 100 \* (S/X)

$$\begin{split} A_x &= A \text{rea of compound,} \\ C_x &= Concentration of compound,} \\ S &= Standard deviation of the RRFs, \end{split}$$

 $A_{\rm h}$  = Area of associated internal standard  $C_{\rm h}$  = Concentration of internal standard X = Mean of the RRFs

L									
				Reported	Recalculated	Reported	Recalculated	Renorted	Detellipted
					<u> </u>				
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	RRF (50 std)	RRF ( \$\mathcal{S})	Average RRF (initial)	Average RRF (initial)	%RSD	%RSD
	1CAL	80/60/	Phenol (1st internal standard)	2. 6625Y	4	14167.2	7.6914	6. 25.349	6,25349
		•	Naphthalene (2nd internal standard)	1.10277	1.10277	1. 09527	Lespo .1	103992	10.39092
		<b></b>	Fluorene (3rd internal standard)	1.36978	1.36978	1.34878	1.34878	(14-5745S)	14. Sy450
			Pentachlorophenol (4th internal standard)	15002 O	0.15002	0.15050	তেথ্য-০	11.84795	1
			Bis(2-ethylhexyl)phthatate (5th internal standard)	0.80769	696000	0.81450		7.06979	1
			Benzo(a)pyrene (6th internal standard)	1.21357	1.21257	1.18939	1.18939	3.6028	2.6086
7		-1	Phenol (1st internal standard)						
		·····,	Naphthalene (2nd internal standard)						
		,	Fluorene (3rd internal standard)						
		· · · · · · · · · · · · · · · · · · ·	Pentachiorophenol (4th internal standard)						
			Bis(2-ethylhexyl)phthalate (5th internal standard)						
			Renzo(a)pyrene (6th internal standard)						
3			Phenol (1st internal standard)						
		<del></del> +	Naphthalene (2nd internal standard)						
		····	Fluorene (3rd internal standard)						
		<del></del>	Pentachlorophenol (4th 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 qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 1838AV SDG #: su coner

### Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

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 =  $(A_{\star})(C_{\star})/(A_{\star})(C_{\star})$ 

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

					Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q%	<b>G</b> %
	KCAL4350	2/1/08	Phenol (1st internal standard)	2.6974)	2.64086	2.64086	2.09649	2.09649
$\underline{I}$			Naphthalene (2nd internal standard)	1.09527	1.09989	1.09989	0.42132	25124.0
			Fluorene (3rd internal standard)	1.34878	1. 34529	1-34529	0 2866	0.25.26.0
			Pentachlorophenol (4th internal standard)	asas1.0	0.18441	0.1844)	25185-22	218312
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.814SB	0.79520	05797.0	2. 30440	4.26.4
			Benzo(a)pyrene (6th internal standard)	1.18939	1.19972	1.19972	C 2898 D	0-22.823
7			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)					
٣			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results. LDC#: 18386A2 SDG #: per coner

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

Page:_	of <u>/</u>
Reviewer:	[7]
2nd reviewer:	1

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. U	36.4457	13	13	0
2-Fluorobiphenyl		37.8084	710	16	
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 #: 18380A7 SDG #: per court

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

Page: \_\_of/ 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

SSC ≈ Spiked sample concentration SA ≈ Spike added Where:

SC = Sample concentation

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

MSD = Matrix spike duplicate percent recovery

MS/MSD samples:

MS = Matrix spike percent recovery

و

Compound	Spi	ike	Sample	Spiked Sample	Sample	Matrix Spike	Spike	Matrix Spike Duplicate	Duplicate	MS/MSD	SD
	(VA)	Added (Ma)	Concentration (ve)	Concentration (ver)	tration	Percent Recovery	есочегу	Percent Recovery	ecovery	RPD	
	MS	MSD	)	MS	U MSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenol 35	क्षेड ६	3470	Øn		2400	72	12	69	69	5.5	5,5
N-Nitroso-di-n-propylamine	-			2440	a7so	/8	18	10	9	4.8	87
4-Chloro-3-methylphenol				oile	assa	17	11	73	13	0.9	09
Acenaphthene				2530	2340	13	EL.	ጾባ	Хq	7.9	7.9
Pentachiorophenol				188	199	16	٦	19	19	13	(3
Pyrene			,	C281	asm	R	. B	٦	16	6,6	6.6
-											

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% of the recalculated results.

LDC #: 18 380A2 SDG #: as comer

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

Reviewer:\_

2nd Reviewer: 1

Page:

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

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

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

8029394-165 LCS/LCSD samples: \_

	Š	oike	as S	ķe	-	CS	I CSD	g	1 CS/I	CS/I CSD
Compound	Ad (V.º	Added ( M.S. K.)	Concer (WG)	Concentration (we)	Percent Recovery	Recovery	Percent Recovery	ecovery	R	RPD
	1.08	1 CSD	SUL	) I CSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
	3330	ΑN	7.300	ΑM	وه	69				
N-Nitroso-di-n-propylamine			2480	-	7	7	•			
4-Chloro-3-methylphenol			usn		76	- 1				
Acenaphthene			01/2		71	71				
Pentachlorophenol			2692		8	18				
		_ <del>`</del>	טנאב	<b>→</b>	6	62	1 42			
	-									

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results. LDC#: 18386A2 SDG#: \_w eoner

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

Page:_	/of/_
Reviewer:_	<u></u>
2nd reviewer:_	L

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

Percent solids, applicable to soil and solid matrices only.

Factor of 2 to account for GPC cleanup

Y	N	N/A
Y	N	N/A

%S

2.0

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

Concer	itration	$= \frac{(A_{x})(I_{x})(V_{1})(DF)(2.0)}{(A_{1x})(RRF)(V_{0})(V_{1})(\%S)}$	Example:
A <sub>*</sub>	=	Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D.
$A_{is}$	=	Area of the characteristic ion (EICP) for the specific internal standard	
l <sub>s</sub>	=	Amount of internal standard added in nanograms (ng)	Conc. = ( )( )( )( )( )( )
V <sub>o</sub>	=	Volume or weight of sample extract in milliliters (ml) or grams (g).	M / I/
V <sub>i</sub>	=	Volume of extract injected in microliters (ul)	= ////
$V_{\iota}$	=	Volume of the concentrated extract in microliters (ul)	
Df	=	Dilution Factor.	

Calculated Reported Concentration Concentration Qualification Compound Sample ID #

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

Project/Site 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	Pentachiorophenoi	22.52510	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-08-10' TSB-HJ-08-10' TSB-HJ-08-10' TSB-HR-05-10' TSB-HR-05-10' RINSATE-2 TSB-HR-05-10'MS TSB-HR-05-10'MSD 8031299-BIK	None	Р
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-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HS-05-10' 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	Pentachlorophenol	None	P.	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-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10' RINSATE-2	N-Hydroxymethylphthalimide	J+ (all detects)	А	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	А
F8A290158	TSB-HJ-10-10'	Unknown aldol condensate (4.2597) Unknown aldol condensate (4.7565)	21000U ug/Kg 350U ug/Kg	А
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	А
F8A290158	TSB-HR-06-10'	Unknown aldol condensate (4.2583) Unknown aldol condensate (4.7497)	23000U ug/Kg 370U ug/Kg	А
F8A290158	TSB-HJ-08-0'	Unknown aldol condensate (4.2624) Unknown aldol condensate (4.7486)	23000U ug/Kg 380U ug/Kg	А

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	А

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

No Sample Data Qualified in this SDG

DG#	: 18386B2 :: F8A290158 atory: Test America	_ VA 	LIDATIOI -		LETENE _evel III	ESS W	ORKSHEET		Date: 3/6/0 Page: 1 of 1 Reviewer: 2nd Reviewer:
	OD: GC/MS Semivolatile	-							
	imples listed below were ion findings worksheets.		ewed for eac	ch of the fo	ollowing va	alidation	areas. Validatior	n find	lings are noted in attached
	Validation	Area					Comme	ents_	
<b>I</b> .	Technical holding times			Δ	Sampling d	ates:	1/2/08		
11.	GC/MS Instrument performa	ance ch	neck	厶			(		
III.	Initial calibration			Д	% PSD	· ( 2 -	20.990		
IV.	Continuing calibration/ICV			ىسى		\ \ \ \ \ \			
V.	Blanks			SW					
VI.	Surrogate spikes			Δ					
VII.	Matrix spike/Matrix spike du	plicate	s	SWA					
VIII.	Laboratory control samples			SW)					
IX.	Regional Quality Assurance	and Q	uality Control	N					
Χ.	Internal standards			Δ					
XI.	Target compound identificat	tion		N					
XII.	Compound quantitation/CR0	QLs		N			10000		
XIII.	Tentatively identified compo	ounds (	TICs)	N			· · · · · · · · · · · · · · · · · · ·		
XIV.	System performance			N					
XV.	Overall assessment of data			4					
XVI.	Field duplicates			NO	D	2 3 <del>1</del>	4		
	Field blanks		- Name and a	ND	R	= 10			
Note:	A = Acceptable N = Not provided/applicable SW = See worksheet	<b>e</b>	R = Rin	o compound		D TI	= Duplicate 3 = Trip blank 3 = Equipment blank	(	
/alidate	ed Samples: ** Indicates sam ეს ჯ ააბ	ple und	derwent Level	IV validation				1	
1	TSB-HJ-10-0'	11	TSB-HR-05-1	10'MS	21	8029	233-Blank	31	
2	TSB-HJ-10-10'	12	TSB-HR-05-	I0'MSD	22	8031	299-13/an/	32	
3	TSB-HR-06-0'	13		·	23			33	
4	TSB-HR-06-0'-FD	14			24			34	

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

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

VALIDATION FINDINGS WORKSHEET

LDC # 1838 B2

Page: Lof

Reviewer:

Continuing Calibration

SDG # Lange Composition (EPA SW 846 Method 8270C)

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

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

Nere 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?

	1	<u> </u>			T	T	T	T	П	<del></del>	Г	Г	П	<u> </u>	T	ī	Π		<del></del>	T	<u></u>	<del></del>	Ī	T	Τ	T
Qualifications	oran	~		- 1	1./A du		01/ ann																			
Associated Samples	5031299-BK,	+ All samples,	11, 12		>		7(日) 8526408																			
Finding RRF (Limit: ≥0.05)																										
Finding %D (Limit: <25.0%)	01525.22			7 - 1 - 1 - 1	21248.47		22.53156																			
Compound	TT (ccc)	,		N- (Hydroxymethy)	phthalimide		TT (ccc)																			
Standard ID	KCAL 4400				0127 1 271		KCA Ly350																			
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*				1	-	Ш																				

LDC #: [8 386 82 gras sa SDG#:

### VALIDATION FINDINGS WORKSHEET Blanks

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".

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

Y N N/A

Was a method blank analyzed for each concentration preparation level?

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

Was the blank contaminated? If yes, please see qualification below. Y/N N/A

凶ank extraction date: ハラハック Blank analysis date: 2 00 00

(4.247) 4.7438 00024 δ 4.2682) 11921.H \*7443 4.288 4.7486) 1.262 00062 e ١ Sample Identification 37000 (4.7497) (4. X9t) H-7509 2000 4 Associated Samples: (3656.4 000 62 (4.75) 980 3 (4.756S (4.25.97) 21000 4 ١ 350 (4.7401) (4.2487) 22000 100000 (4.85.4) - 6621208 (3.8408) (4749 Blank ID Blank condensale condens at Conc. units: walke Compound unknown unknown unknown aldo

. J

Blank analysis date: Blank extraction date:

Conc. units:

Associated Samples:

Compound	Blank ID	Sar	Sample Identification
a supplied to			
			-

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".

SDG #: Ace could LDC #: / X > 80 B 7

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

Page: Reviewer: 2nd Reviewer:

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

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

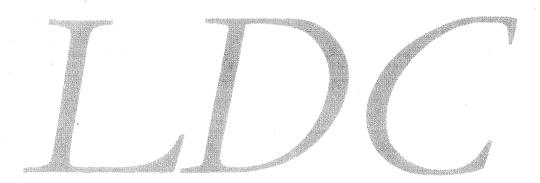
| N | N/A | Was a LCS required? | Y/N | N/A | Were the LCS/LCSD percent recoveries (%R) and the relative percent differences (RPD) within the telephones (RPD) within the relative percent differences (RPD) within the telephones (RPD) within the telepho

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

*	Date	TCS/TCSD ID	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		8029-58-108	44	(16-0E) SC	( )	( )	8029233-Blant,	NO ONAL MSDIN
				1	( )	)	0 #	
				( )	(			
				( )	( )	)		
				( )	( )	( )		
				(	( )	)		
				( )	( )			
				( )	( )	)		
				)	(	( )		
					( )	( )		
				( )	( )			
$\dashv$				( )	( )	( )		
$\dashv$				(	( )	( )		
$\exists$				(	( )	( )		
				(	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
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				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	(		
				( )	( )	( )		
				( )	( )	( )		
$\dashv$				)	)	( )		

### 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

<sup>\*\*</sup>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)	А
2/8/08	KCAL587	RTX-CLP2	Toxaphene	27.4	TSB-HJ-07-0'-FD	J+ (all detects)	А
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)	А

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-07-10' TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-0' TSB-HR-08-0'MSD	J+ (all detects)	Α
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)	А
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)	Α

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)	Р

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)	222	D:#		
Compound	TSB-HJ-07-0'**	TSB-HJ-07-0'-FD	RPD (Limits)	Difference (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-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'	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) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	A	Continuing calibration (%D)
F8A250221	TSB-HJ-05-0' TSB-HR-08-10'	All TCL compounds	J+ (all detects)	Р	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

	#: <u>18386A3a</u> #: <u>F8A2502<b>3</b>1</u>	VA	LIDATIO		PLETE evel III		ss wo	RKSHEE <sup>1</sup>	Г	Date: 3 /10/08 Page: 1 of 1
Labo	ratory: Test America			<b>—</b>		/ I V				Reviewer: 15
MET	HOD: GC Chlorinated Pe		•				<b>1-1</b> :	<i>\\-\</i> :-	: e:	2nd Reviewer:
valida	ation findings worksheets		ewed for ea	ch of the f	ollowing	valid	ation ar	eas. Validat	ion Tinai	ings are noted in attached
	Validation	Area						Comi	nents	
l.	Technical holding times			Δ	Samplin	g date	s:	124/08		
11.	GC/ECD Instrument Perform	nance	Check	Δ				1 .1		
111.	Initial calibration			Δ						
IV.	Continuing calibration/ICV			يبي						
V.	Blanks			Δ						
VI.	Surrogate spikes			SW						
VII.	Matrix spike/Matrix spike du	plicate	s	Δ						
VIII	Laboratory control samples			Δ	Le	5				
IX.	Regional quality assurance	and qu	uality control	N						
Xa.	Florisil cartridge check			N						
Xb.	GPC Calibration			N						
XI.	Target compound identification	tion		$\Delta$	Not revi	ewed	for Level I	II validation.		
XII.	Compound quantitation and	l repor	ted CRQLs	Δ	Not revi	ewed	for Level I	II validation.		
XIII.	Overall assessment of data			Δ						
XIV	Field duplicates			ςω	D =	11	<i>4</i> /	2		
XV.	Field blanks			N		,				
Note:	A = Acceptable N = Not provided/applicable SW = See worksheet	€	R = Rin	o compound sate eld blank	s detected	d	TB =	Duplicate Trip blank Equipment bla	ınk	4
Valida	ted Samples: ** Indicates sam らいし	ple un	derwent Level	IV validation						
1	TSB-HJ-05-10'	11	TSB-HJ-07-0	**	21	q	0293	97-Blan	31 ے	
2	TSB-HJ-05-0' /	† 12	TSB-HJ-07-0	-FD	22	:			32	
3	TSB-HR-04-10'	13	TSB-HJ-07-1	0'	23				33	
4	TSB-HJ-04-0'	14	TSB-HR-08-0	)'	24				34	
<b>5</b>	TSB-HR-04-0'**	15	TSB-HR-08-1	0' /	25				35	
6	TSB-HJ-04-10'	16	TSB-HR-08-0	'MS	26				36	

TSB-HR-08-0'MSD

TSB-HR-07-0'

TSB-HR-06-0'

TSB-HR-06-10'

TSB-HR-07-10'\*\*

LDC #: 18386 + 39

VALIDATION FINDINGS CHECKLIST

SDG #: pu cones

Page: 1 of 2
Reviewer: F7
2nd Reviewer: V

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

Method: Pesticides/PCBs (EPA SW 846 Method 8081/80	1	ī	T	T
Validation Area	Yes	No	NA	Findings/Comments
I. 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) ≤ 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?				

LDC #: L8386A3a SDG #: pu cones

### VALIDATION FINDINGS CHECKLIST

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

Validation Area	Yes	No	NA	Findings/Comments
VII: Matrix spike/Matrix spike duplicates	1	1	1	1 managa comments
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				
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	<del>/                                    </del>	,		
Overall assessment of data was found to be acceptable.				
KIV. Field duplicates				
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.	7			
CV: Field:blanks		<u>,                                      </u>		
Field blanks were identified in this SDG.				
arget compounds were detected in the field blanks.				

## VALIDATION FINDINGS WORKSHEET

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

A state A				
A alpiid-bnc	I. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	.00
B. beta-BHC	1 44"-DDE			
		K. Endrin aldehyde	Z. Aroclor-1248	HH.
C. delta-BHC	7. Till 7. Til			
		S. alpha-Chlordane	AA. Aroclor-1254	<b>:</b>
D. gamma-BHC				
	L. Endosulian II	T. gamma-Chlordane	BB. Aroclor-1260	JJ.
E. Hentachlor				
	m. 4,4 -DDD	U. Toxaphene	CC. DB 608	KK.
E. Aldrin				
	N. Emosuran surate	V. Aroclor-1016	DD. DB 1701	LL.
G. Hentachlor enovide				
	100-4,4	W. Aroclor-1221	EE.	MM.
H Endocate				
	P. Methoxychlor	X. Aroclor-1232	FF.	NN.
				_

C:\docs\Work\Pesticides\COMPLST-3S.wpd

Notes:

LDC# 18380 R32 SDG#: Les coves BC HPLC

METHOD:

### VALIDATION FINDINGS WORKSHEET Continuing Calibration

10t/ Page: Reviewer:

2nd Reviewer:

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

What type of continuing calibration calculation was performed? — %D or \_\_\_RPD

Were continuing calibration standards analyzed at the required frequencies?

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

Y N NA N NA Level IV Only

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

Y N/N/A

11		Detector		000/0/			
	Standard ID	Column	Compound	%27 RPD (Limit ≤ 15.0)	RT (limit)	Associated Samples	Qualifications
KCA	KCA1587	RTX-apl	٨	39.5	(	12	)+/A dut
		RIX-CLPZ	7	₽.F¢	(	<b>ラ</b>	7
					( )		
					(		
KeA 1	4661	RTX-CLP1	12×1	カ'火	(	8029397-BANK,	1-1/A dit
			-		( )	1.23.4,5 (1-05)	
						\ =	
K	KCA1677	RTX-41P)	5	31.6	( )	16,17	1+/A det
					(	13,14	
					( )		
					(		•
N S	KCA1 689	19 tx -a1 P1	2	8.91	( )	31	1+/A dut
				0.9	(		
			ø	15.2	(		
			8	9.51	(		
			Z	0-11	( )		
			8	19.4	( )	7	7
					( )		

LDC #: 18 386A3A SDG# Les cover

### VALIDATION FINDINGS WORKSHEET Continuing Calibration

/ot/ Page: 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

YNAN/A

Were continuing calibration standards analyzed at the required frequencies?

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

Y N N/A Level IV Only

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

						П																7	
	Qualifications	1+/A ext	,				1+/A dit	<i>\</i>															
	Associated Samples	5				51	17																
	RT (limit)	( )	)	( )	( )	( )	(	( )	( )	(	(	(	( )	( )	( )	( )	( )	)	)	( )	( )	( )	( )
	%D / RPD (Limit ≤ 15.0)	32.28				اه. ا	16.6								-								
	Compound	Ϋ́				2,4'- PDE	000-14.E																
	Detector/ Column	RTX-erp				RTX-aLPI																	
	Standard ID	*CALC9				KCAL692																	
		2/9/08	-			30/6/2																	
١I	*		<u> </u>					<u> </u>		L	L		<u> </u>	L	<u></u>	L	<u></u>	<u> </u>		<u> </u>	L_		

LDC#: 18380A3-SDG #: 1

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METHOD:

## VALIDATION FINDINDS WORKSHEET

Surrogate Recovery

2nd Reviewer: Reviewer Page:

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". HPLC

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

	I		Γ	Ī	Γ		ľ	Ī	Ι	Γ	T	T	T	T	Ī		Γ						T	T	T	T		Γ
Qualifications																							Tetrachloro-m- xylene					
σ	ts to			128																			>					
	7			/ + / (		(				(		(		(			(	(	) (	(	(	Surrogate Compound	1-Chloro-3-Nitrobenzene	3,4-Dinitrotoluene	Tripentyltin	Tri-n-propyltin	Tributyl Phosphate	Triphopyl Phosphate
	63-117			$\rightarrow$																			S	-	n	>	≥	×
%R (Limits)	11	)		) ६८।	)	)	)	)	)	)		)				)			<u>`</u>		)	Surrogate Compound	Benzo(e)Pyrene	Terphenyl-D14	Decachlorobiphenyl (DCB)	1-methvinaohthalene	Dichlorophenyl Acetic Acid (DCAA)	4-Nitroohenol
																	-			-	-		Σ	z	0	۵	0	α
Surrogate Compound	DcB			7																		Surrogate Compound	Octacosane	Ortho-Terphenyl	Fluorobenzene (FBZ)	n-Triacontane	Hexacosane	Bromobenzene
or/	أدوا																					Surr			F			
Detector/ Column	Savited	-		<i>→</i>																			O	I	-	1	¥	-
	Lou																					pur	(2)	BFB)				ÉBI
Sample ID	7			12																		Surrogate Compound	Chlorobenzene (CBZ)	4-Bromofluorobenzene (BFB)	a,a,a-Trifluorotoluene	Bromochlorobenene	1,4-Dichlorobutane	1.4-Difluorobenzene (DFB)
<b>)</b> #																							٨	8	U	9	ш	4

LDC #: 18 386 434 SDG #: 44 const

## VALIDATION FINDINGS WORKSHEET Field Duplicates

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

Y N N/A

Reviewer:

Parent only / All Samples Qualification %RPD Dillo Limit Limit 7 る Concentration ( ug | | } × × × Compound 8

Compound	Concentration (	•	%RPD	Qualification
				Parent only / All Samples

LDC#: (8 386A34 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

average CF = sum of the CF/number of standards %RSD = 100  $^{\star}$  (S/X) CF = A/C

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

				Reported	Recalculated	Reported	Recalculated	Paported	Dooplandad
#	Standard ID	Calibration Date	Compound	CF (0.62/std)	CF (ach std)	Average CF (initial)	Average CF	Cod%	000%
	<b>1</b> ₩	3/1/08	endosulgan) RtxuP)	Ses 745040	ohashlans Ohashlans	57238634	5	i.	4L7.9
Т				Q769153 arc	Oashsour Oashsour		अभरवार्ड १० २५२ वास्त्र भ	6.78.7	181.9
1			RTXCNPZ	264274520	264234520 264234520	26 5 bs 9 47G	offesosan offesosan	585.5	S.S.S
2			-	Ookaloses	C0196825	55734490	35 334490	4.736	4.727
T							1		\ \ !
ᅦ									
_									
6									
Т									
1									
4					ē				
$\neg \Gamma$									

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

LDC# (830643a SDG#: Ru coun

### Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Page: Reviewer: 2nd Reviewer:

> HPLC. METHOD: GC\_

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

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

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

<del></del>					Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc. CCV	Ω%	Q%
1	Kalesa	2/9/08	endosultan Ptx-cup)	0،0عم	0.0262	2010.0	4.9	6.4
	_	 -	methory chief D	->	6000	0.0%9	4.7	ブレ
$\dashv$	KCALL 15	2000	S, RIX-crp-1		2120.0	21.00.0	0.01	0.0
7		- -	7	->	41600	hLe0.0	9.٤	م ای
ю								
4								

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

### LDC #: 18 386 A30 VALIDATION FINDINGS WORKSHEET **Surrogate Results Verification**

Page:	/_of/
Reviewer:_	
2nd reviewer:	1 ~

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found

SS = Surrogate Spiked

Sample ID: 5

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene						
Tetrachloro-m-xylene	Rex-cipl	0.07	0.02117	106	106	106
Decachlorobiphenyl	\ \ \		0.02272	114	114	lid
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	
Tetrachioro-m-xylene						
Tetrachloro-m-xylene						
Decachlorobiphenyl				A		
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						

Notes:	•		
		1,000,000,000,000	 

LDC#: 18 280 A34 SDG#: au com

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

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

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

SSC = Spiked sample concentration SA = Spike added Where:

SC = Concentration

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

MS = Matrix spike percent recovery

MSD = Matrix spike duplicate percent recovery

MS/MSD samples:\_

	S	pike	Sample		Spiked Sample	Matri	Matrix Spike	Matrix Spil	Matrix Spike Duplicate	W	MS/MSD
Compound	₹ % (~~)	(mg) (sq)	Concentration (ベカーな)		ntration	Percent	Percent Recovery	Percent	Percent Recovery		RPD
100 mg	MS	MSD	) } '	Ž	OMSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
gamma-BHC	9.[1	11.3	9	18.6	1.41	901	901	96	96	8.9	8.9
4,4'-DDT	->	17.3	0	2).4	19.1	121	12)	110	011		
			-								

Comments: Refer ot Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results. LDC#: 1838443

# VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicate Results Verification

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

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

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

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

SSC = Spiked sample concentration SA = Spike added Where:

SC = Concentration

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

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples:\_

	SF	oike	Spiked	Sample	רי	SOT	FC	CCSD	SOT	rcs/rcsd
Compound	γ <sub>ν</sub> )	( 43 Fa	AM)		Percent	Percent Recovery	Percent	Percent Recovery	<u>α</u>	RPD
	CCS	CCSD	SOT	CSD	Reported	Recalc.	Reported	Recalc	Perorted	2000
gamma-BHC	۲.	2	13.2	2	601	10.2			nanodavi	Necalc.
4,4'-DDT	1	->	18.7	7	51	7:	4			
				<b> </b>		. 1 -	< 2			
							\			

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 #:_	pu	con	W

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

- · · · · · · · · · · · · · · · · · · ·
7

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

<u>Y</u>	<u>N</u>	N/A
Y	N	N/A

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

Example:	
Sample I.D.	
Conc. = (	
=	

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
	4				
		7 15 150 · · · · · · · · · · · · · · · · · · ·			

Note:	

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

**Project/Site Name:** 

BRC Tronox Parcel H

**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

### 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)	А
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)	А

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)	Р
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)	P
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)	Р	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

LDC #: 18386B3a	VALIDATION COMPLETENESS WORKSHEET
SDG #: F8A290158	Level III
Laboratory: Test America	

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	Д	Sampling dates: 1/22 00
II.	GC/ECD Instrument Performance Check	A	1 1
III.	Initial calibration	/4	
IV.	Continuing calibration/ICV	Sale	t <sub>V</sub> √
V.	Blanks	Δ	
VI.	Surrogate spikes	كيدك	
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	N_	
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

Valida	ted Samples:	7					
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' <b>√</b>	13		23		33	
4	TSB-HR-06-0'-FD <i>(</i> )	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: Pesticide/PCBs (EPASW 846 Method 8081/8082)

A sinha.Buc				
	I. Dieldrin	Q. Endrin ketone	Y. Araclar-1242	
B. beta-BHC				
	J. 4,4DDE	R. Endrin aldehyde	Z. Aroclor-1248	
C. delta-BHC	X 115.55			
		S. alpha-Chlordane	AA. Aroclor-1254	=
D. gamma-BHC	L. Endosufan II			=
		T. gamma-Chlordane	BB. Aroclor-1260	17
E. Heptachlor	M 44-Don			
		U. Toxaphene	CC. DB 608	777
F. Aldrin				
	n. Endosullan sulfate	V. Aroclor-1016	DD, DB 1701	
G. Heptachlor enovide				1
	O. 4,4. DDT	W. Aroclor-1221	EE.	1944
H. Endosulfan i				MM.
	methoxychior	X. Aroclor-1232	FF.	
				Ž

C:\docs\Work\Pesticides\COMPLST-3S.wpd

Notes:

LDC#: 18786 B3C ku cover SDG#: GC HPLC

METHOD:

### VALIDATION FINDINGS WORKSHEET Continuing Calibration

Page: /of / Reviewer:

2nd Reviewer:

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

What type of continuing calibration calculation was performed? — \*\*D or \_\_ RPD

Were continuing calibration standards analyzed at the required frequencies?

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

N N/A N N/A Level IV Only Y N MA

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

		٦	ī	1	П		7	٦										7			7	$\neg$
1 1	JA GUT				17/A &CT																	
Associated Samples	18-2905 COS (#	-			711115																	
RT (limit)	(	)	(	( )	( )	( )	( )	) (	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	
%D / RPD (Limit ≤ 15.0)	21.7	-			トコス																	
Compound	5				<i>&gt;</i>																	
Detector/ Column	RTX-0091				1	•																
Standard ID	kca LC17				KCAL63																	
Date	148/08	-			80 8 2	1																
#	+				+				<u> </u>	<u> </u>					<u> </u>							

LDC #: 18386 832 SDG #: Les Conner

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

2nd Reviewer: 1 Reviewer: Page:

**МЕТНОD**: <u>/</u> GC \_\_ HPLC Are surrogates required by the method? Yes\_\_

X N N X

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

) #	Sample ID	Detector/ Column	or/ nn	Surrogate Compound		%R (Limits)				Qualifications
	l n	not specified	illed	Ļ		)	S11 -SS	ر ا	3+/P dut	
		•	<b>^</b>	P		126	63-117	7	-3	
	7	7		Ф		) <b>9</b> 7)	63-117	17 )	1+/Part	
	ج	→		Ъ		) 171	7	- (	1+/PdvT	
	ħ			¥		(N	52-115	` `	1./Pact	
		<b>→</b>		Ð		127	11-89	1		
								(		
	\s					)   121		<u> </u>		
		~		<del>^</del>		129	<i>&gt;</i>		<b>→</b>	
	<b>a</b>							(		
	و					) 921		(	1+/Par	
		$\rightarrow$		7		132	~	(		
					_			,		
						)	_	(	1+/Paul	
		^		~>		123 (	<i>&gt;</i>	(	,	
						)		(		
	8	1				124		)	1+/Par	
		->		<i>→</i>		ا ( ا	>			
	Surrogate Compound		Surrog	Surrogate Compound		Surrogate Compound		Surrogate Compound	punodwo	
٨	Chlorobenzene (CBZ)	9	Ō	Octacosane	Σ	Benzo(e)Pyrene	S	1-Chloro-3-Nitrobenzene		Y Tetrachloro-m- xylene
В	4-Bromofluorobenzene (BFB)	В) Н	νО	Ortho-Terphenyl	Z	Terphenyl-D14	<b>F</b> ~	3,4-Dinitrotoluene	toluene	
С	a,a,a-Trifluorotoluene	-	Fluor	Fluorobenzene (FBZ)	0	Decachlorobiphenyl (DCB)	)	Tripentyltin	yltin	
۵	Bromochlorobenene	7	.d	n-Triacontane	۵	1-methyinaohthalene	>	Tri-n-propyllin	povitin	
E	1,4-Dichlorobutane	¥	Τ.	Hexacosane	O	Dichlorophenyl Acetic Acid (DCAA)	3	Tributyl Phosphate	osphate	
ŭ.	1.4-Diffuorobenzene (DFB)	_	, Br	Bromobenzene	m	4-Nitrophenol	×	Triphenyl Phosohate	hosphate	

LDC# 1838 6830 SDG #: 1

METHOD:

## VALIDATION FINDINDS WORKSHEET

Reviewer: 2nd Reviewer:

Page:

Surrogate Recovery

METHOD: \_\_\_GG \_\_ HPLC
Are surrogates required by the method? Yes\_\_\_\_ or No\_\_ 25/

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". Did all surrogate recoveries (%R) meet the QC limits? Were surrogates spiked into all samples and blanks? Y N N/A

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					)		)	
Surrogate Compound		Surrogate (	Surrogate Compound		Surrogate Compound	S	Surrogate Compound	pur
A Chlorobenzene (CBZ)	ŋ	Octacosane	sane	Σ	Benzo(e)Pyrene	S	1-Chloro-3-Nitrobenzene	ene Y Tetrachloro-m- xylene
B 4-Bromofluorobenzene (BFB)	В) н	Ortho-Te	Ortho-Terphenyl	z	Terphenyl-D14	Τ	3,4-Dinitrotoluene	
C a,a,a-Trifluorotoluene		Fluorobenzene (FBZ)	zene (FBZ)	0	Decachlorobiphenyl (DCB)	)	Tripentyltin	
D Bromochlorobenene	7	n-Triacontane	ontane	а	1-methylnaohthalene	>	Tri-n-propyltin	
E 1,4-Dichlorobutane	¥	Hexacosane	osane	0	Dichlorophenyl Acetic Acid (DCAA)	3	Tributyl Phosphate	
F 1.4-Difluorobenzene (DFB)		, Bromobenzene	Jenzene	Ж	4-Nitrophenol	X	Triphenyi Phosphate	O.

### 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

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

<sup>\*\*</sup>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 Level III/IV SDG #: F8A250221 Reviewer: Laboratory: Test America 2nd Reviewer:

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

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

	Validation Area		Comments
I.	Technical holding times	٨	Sampling dates: 기계 0명
II.	GC/ECD Instrument Performance Check	NV	'
III.	Initial calibration	۵	
IV.	Continuing calibration/ICV	٨	1cv = 15
V.	Blanks	٨	
VI.	Surrogate spikes	ム	
VII.	Matrix spike/Matrix spike duplicates	۵	
VIII.	Laboratory control samples	ىسى	LCS
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	
Xb.	GPC Calibration	N	
XI.	Target compound identification	٨	Not reviewed for Level III validation.
XII.	Compound quantitation and reported CRQLs	<u> </u>	Not reviewed for Level III validation.
XIII.	Overall assessment of data	<u>A</u>	
XIV.	Field duplicates	ND	D= 11+12
XV.	Field blanks	$\bigvee$	

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

D = Duplicate

R = Rinsate FB = Field blank TB = Trip blank

EB = Equipment blank

Valida	ated Samples: ** Indicates	sample un	derwent Level IV validation				
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	

LDC #: 18386 A3b SDG #: peu coner

### VALIDATION FINDINGS CHECKLIST

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

Method: GC \_\_\_\_HPLC

Method: / GCHPLC				
Validation Area	Yes	No	NA	Findings/Comments
1. Technical holding times	**,			
All technical holding times were met.	/			
Cooler temperature criteria was met.	/			:
III initial calibration 1 1 2 Exs. Called March 1997				
Did the laboratory perform a 5 point calibration prior to sample analysis?	/			
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) ≤ 20%?	/			
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?		_		
Did the initial calibration meet the curve fit acceptance criteria?			•	
Were the RT windows properly established?				
IV: Continuing calibration				
What type of continuing calibration calculation was performed?%D or%R	/			
Was a continuing calibration analyzed daily?				
Were all percent differences (%D) ≤ 15%.0 or percent recoveries 85-115%?				
Were all the retention times within the acceptance windows?	/			
V. Blanks			12.71	
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.		/		
M Surrogate spikes。		17.5		
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?			1	
VII. Mainx 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.				e de la companya de la companya de la companya de la companya de la companya de la companya de la companya de La companya de la co
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?	$/ \overline{ }$		T	
VIII. Laboratory control samples				
Was an LCS analyzed for this SDG?	$\Box$		<u> </u> _	
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				

LDC#: [8386A36 SDG#: Leu Coner

### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 2 Reviewer: 7 2nd Reviewer: \_\_\_\_\_

			<del>,</del>	
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 11 per 1				
Were the retention times of reported detects within the RT windows?				
M. Compound quantitation/CRQLs		Villa)		
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII, So, stein petromance: The street of the				
System performance was found to be acceptable.				
XIII Overalliessessment 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	i e		****	
Field blanks were identified in this SDG.				3.70 Em 2.00 (3.00) (3.00)
Target compounds were detected in the field blanks.				

LDC # 1838 6A3b SDG #: As cover

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

Page: 2nd Reviewer: Reviewer:

METHOD: CGC HPLC

Level IV/D Only

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

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Nr D (Ellillis)

LDC # 18386436 Carl SDG#:

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

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 =  $100 \cdot (S/X)$ 

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

				Reported	Recalculated	Reported	Receimeted	7 7 7 7 6	
*	Standard ID	Calibration Date	Compound	CF (Sto std)	CF.	Average CF	Average CF	<u> </u>	Kecalcillated
-	1001	46/07	1200-	Rix enpl 12182	12182	181	(mining)	AKSD ANSO	%RSD
	-		Arochor 1260-1 RIX-CLP2 11265	11205	11265	HOII	HON	8. 799	1.587 1991.8
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7									
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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

LDC#: 18386A3b Cere SDG#:

### Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Page: Reviewer: 2nd Reviewer:

> HPLC METHOD: GC\_

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

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

ave. CF = initial calibration average CF Where:

CF ≈ continuing calibration CF

A = Area of compound C = Concentration of compound

					Ж	Reported	Recalculated	Reported	Recalculated
**	Standard ID	Calibration Date		Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc. CCV	Q%	Q%
-	本厂	19/18/21	Arochov	Arocloy 1260-1 RTX cup)	1000.000	1006.6919	1006.69	6.7	6.7
	CC\	80/18/1	Aroclor	Aroclor 120-1 RTXCVP2	1000.00	1098.2936		8. p	8.8
$\ $						-			
2				w + 37 %.					
Γ									
6						`			
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		,							
		<del>.</del>							

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

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

> GC HPLC METHOD:

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

サク Sample ID:

Surrorate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
- A - A - A - A - A - A - A - A - A - A				Reported	Recalculated	
DCR	ATA CAPI	07	19.7627	lolo	٠٥٥	0
		•		-		

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

Sample ID:

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

LDC # 18386 A36 coner SDG#:

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

Page: Lof Z Reviewer:\_ 2nd Reviewer:

HPLC

METHOD:

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

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

SC = Sample concentration

RPD =(((SSCMS - SSCMSD) \* 2) / (SSCMS + SSCMSD))\*100 و MS/MSD samples:\_

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

MSD = Matrix spike duplicate

MS MSD (c)  MS MSD (c)  17 (c) 17 (d)  Spike/Matrix Spike Duplical			S	ike	Sample	Spike	Sample						
Casoline         (8015)         MS         MSD         Reported         Reported         Reported         Reported         Reported           Casoline         (8015)         MSD         MSD         Reported         Reported         Reported         Reported         Reported           Diesel         (8015)         Methane         (8051)         MSD	i de co	7	PA .	ded	Conc	Conce	January Tration	Matrix	spike	Matrix Spik	e Duplicate	W/SW	ISD
Casoline         (8015)         MSD         —         MSD         Reported         Reported         Reported         Reported           Diesel         (8015)         —         MSD         —         MSD         —         Reported         Repo		Name of the second	3	7 82	(82 km)	χ, ,	Kar L	Percent F	ecovery	Percent	Recovery	RP	۵
Gasoline         (8015)         Percentage         Percentage <th></th> <th></th> <th>MS</th> <th>MSD</th> <th>1</th> <th>MS</th> <th>۱</th> <th>Reported</th> <th>Recalc.</th> <th>Reported</th> <th>Recalc</th> <th>Potrough</th> <th>H</th>			MS	MSD	1	MS	۱	Reported	Recalc.	Reported	Recalc	Potrough	H
Diesel (8015)   Diesel (80219)   Diesel (80219)   Dinoseb (8151)   Dinos	Gasoline	(8015)	F	:	1 3							nerindexi	Vecalic.
Benzene (8021B)         Methane (RSK-175)	Diesel	(8015)											
Methane       (RSK-175)       Methane       (R151)       Methane       (R151)       Methane	Benzene	(8021B)											
2.4-D       (8151)       (8151)         Dinoseb       (8151)       (8151)         Naphthalene       (8310)       (8310)         Anthracene       (8310)       (8330)         HMX       (8330)       (8330)         Arecket       12.C       174         Arecket       12.C       12.1         Arecket       12.C	Methane	(RSK-175)											
Dinoseb (8151)         <	2,4-D	(8151)											
Naphthalene (3310)         Anthracene (3310)         Anthracene (3310)         Anthracene (3310)         Anthracene (3310)         Anthracene (3310)         Ancelor (1200)         Anthracene (3310)         Ancelor (1200)         Ancelor (1200) </td <td>Dinoseb</td> <td>(8151)</td> <td></td>	Dinoseb	(8151)											
Anthracene (8310)         Another (8330)         Ano	Naphthalene	(8310)											
Aroclof 126 176 114 3 312 311 12 121 131 0.9 이 이 이 이 이 이 이 이 이 이 이 이 이 이 이 이 이 이 이	Anthracene	(8310)											
Arockof 12CO 17C 11나 3 212 211 12U 12U 12I 12I 12I 0.9U	НМХ	(8330)											
Areclot         12C         17C         11d         0         312         311         12C         12I         12I         12I         0.9C           omments: Refer to Matrix Spike/Matrix Spike Duplicates findings workshoot for list of an arrising since publicates findings workshoot for list of an arrising since publicates findings workshoot for list of an arrising since publicates findings workshoot for list of an arrising since publicates findings workshoot for list of an arrising since publicates findings workshoot for list of an arrival since publicates findings workshoot for list of an arrival since publicates findings workshoot for list of an arrival since publicates findings workshoot for list of a since publicate findings workshoot for list of a since publicate findings workshoot for list of a since publicate finding workshoot fi	2,4,6-Trinitroto	luene (8330)											
Omments: Refer to Matrix Spike/Matrix Spike Duplicates findings workshoot for list of an arrival and a spike Duplicates findings workshoot for list of an arrival and a spike Duplicates findings workshoot for list of an arrival and a spike Duplicates findings workshoot for list of an arrival and a spike Duplicates findings workshoot for list of an arrival and a spike Duplicates findings workshoot for list of an arrival and a spike Duplicates findings workshoot for list of a spike Duplicates findings workshoot findings workshoot findings workshoot for list of a spike Duplicates findings workshoot findings workshoot findings workshoot findings workshoot findings workshoot findings workshoot findings workshoot findings workshoot findings workshoot findings workshoot findings workshoot find		021	116	디디	C	212		(, 2	0.57			( )	
omments: Refer to Matrix Spike/Matrix Spike Duplicates findings workshoot for list of an arising strength of the contract of t						-	1	7	0.71	12	14	0.70	70
omments: Refer to Matrix Spike/Matrix Spike Duplicates findings workshoot for list of an arise.													
omments: Refer to Matrix Spike/Matrix Spike Duplicates findings workshoot for list of all sites at the contract of the contrac													
omments: Refer to Matrix Spike/Matrix Spike Dunlicates findings worksheet for list of an alicensis.													
of the control of the	comments: Ref	er to Matrix Spil	e/Matrix	Spike Dupl	icates findings	worksheetf	or list of qualif	ications and as	sociated sam	nles when rer	orted results		10,000

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# VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

GC\_HPLC METHOD:

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

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

SSC = Spiked concentration SA = Spike added

SC = Sample concentration

Where

LCS = Laboratory Control Sample percent recovery

LCSD = Laboratory Control Sample duplicate percent recovery

80293916-1165 LCS/LCSD samples:

RPD =(((SSCLCS - SSCLCSD) \* 2) / (SSCLCS + SSCLCSD))\*100

	Spik	و به	Sample	Spike Sample	ample	SOT	Ş	rcsD	٥	rcs/rcsd	csD
Compound	X 54 X	Ŋ,	( 12   15/2)	Concern ( VX	(ration (-ex)	Percent Recovery	lecovery	Percent Recovery	scovery	RPD	Q
	LCS	CSD	o , !	SOT	Ccsp	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)	. 1										
Diesel (8015)	et.	. :									
Benzene (8021B)							,				
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)											
Anthracene (8310)											
HMX (8330)	`										
2,4,6-Trinitrotoluene (8330)											
Arocles 1260	167	7	S	179	マス	107	107	4 2			

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

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\$ #	**
201	SDG

### SHEET ion

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VALIDATION FINDINGS	nole Calcı		
VALID	Sar		
	4		

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

Example:

Sample ID.

(RF)(Vs or Ws)(%S/100)

(A)(Fv)(Df)

Concentration=

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

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

Concentration =

Compound Name

ns						
Qualifications						
Recalculated Results Concentrations						
Reported Concentrations						
Compound						
Sample ID						
#			$\dagger$	1		

### 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:

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'

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 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	Arocior-1016 Arocior-1221 Arocior-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

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

LDC #: 18386B3b	VALIDATION COMPLETENESS WORKSHEET	Date: <u>3 / 2 /</u> 0
SDG #: F8A290158	Level III	Page:of/
Laboratory: Test America		Reviewer:
		0

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

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

	Validation Area		Comments
I.	Technical holding times	Δ	Sampling dates: リルクと
II.	GC/ECD Instrument Performance Check	W-A	
III.	Initial calibration	Δ	
IV.	Continuing calibration/ICV	~SW	1W = 15
V.	Blanks	\$W	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	ςw	10>
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

Valida	ted Samples:						
î	TSB-HJ-10-0' .	11	TSB-HR-05-10'MS	21 /	8029316-BIK	31	
2	TSB-HJ-10-10',	12	TSB-HR-05-10'MSD	22 7	8031455-BI/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 sloha-Ruc				
	i. Dielgin	Q. Endrin ketone	Y. Aroclor-1242	.99
B. beta-BHC	.1.44'-DNF			
		K. Endrin aldehyde	Z. Aroclor-1248	HH.
C. delta.BHC				
	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	H.
D. camma- BHC				
	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	JJ.
Hentech				
	M. 4,4*-DDD	U. Toxaphene	CC. DB 608	KK.
in Alaka	1			
	N. Endosulfan sulfate	V. Aroclor-1016	DD. DB 1701	Lt.
G. neptachior epoxide	O. 4,4'-DDT	W. Aroclor-1221	Ä	MM.
	P. Methoxychior	X. Aroclor-1232	F.	NN.

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Notes:

LDC#: 18386835 SDG#: 14 cover

## VALIDATION FINDINGS WORKSHEET Continuing Calibration

Page: /of / Reviewer: /

2nd Reviewer: /

METHOD: \_\_\_\_ GC \_\_\_ HPLC
Please see qualifications below for all questions answered "N" Not applicable question

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 <15.0%?

Y (N) N/A Level IV Only Y N M/A

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

					 									 ,				,	==	
Qualifications	1 + /A det	DUAL V, W, X	/ 1 1											:						
	<del>/</del> 4-/																			
RT (limit)	)	( )	(	(		( )	( )	)	)	( )	( )	( )	( )	( )	(	( )	( )	( )	( )	( )
%D / RPD (Limit ≤ 15.0)	16-8																			
Compound	^																			
Detector/ Column	Atx-cup1																			
	PCAL SY/																			
Date	80/1/2	, ,																		
#	+																			

LDC #: 18386836 SDG #: 4 Comm

## VALIDATION FINDINDS WORKSHEET Surrogate Recovery

Reviewer:\_\_ Page:

2nd Reviewer:

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

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

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

Column	olumr		Compound		%R (Limits)	- 11	-	3	danneauons
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Surrogate Compound	Surrogate Co	ate C	punoduc		Surrogate Compound		Surrogate Compound	punodi	
Octacosane	Octacos	tacos	ane	Σ	Benzo(e)Pyrene	S	1-Chloro-3-Nitrobenzene	Jenzene Y	Tetrachloro-m- xylene
Ortho-Terphenyl	Ortho-Te	5 T-0	srphenyí	z	Terphenyl-D14	⊦	3,4-Dinitrotoluene	lene	
Fluoroben	Fluoroben	pe Pe	Fluorobenzene (FBZ)	0	Decachlorobiphenyl (DCB)	Э	Tripentyltin	_	
o-Inia	n-Trie	Ë	n-Triacontane	۵	1-methylnaohthalene	>	Tri-n-propyltin	tín	
Hexa	Hexa	ě	Hexacosane	σ	Dichlorophenyl Acetic Acid (DCAA)	3	Tributyl Phosphate	hate	
, Bromobenzene				c	) A Nitrophone	>	Total Control		

LDC # 1838 6336 SDG#: au cover

## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Page: 2nd Reviewer: Reviewer:\_

> GC HPLC METHOD:

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

Y/N N/A

Were a laboratory control samples (LCS) and laboratory control sample duplicate (LCSD) analyzed for each matrix in this SDG?

Y/N N/A

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

Level IVID Only Y N NIA Was an LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed?

	_	<del>-</del>	<del></del>		I		=	<u> </u>	<del></del> T		1	Ŧ	T	T	- 1		<del>-</del>	Т	_		T	1	1	7
Qualifications	Jum ou	ni dism																					·	
Associated Samples	802/455 - B/K	1/2 / 4 ×																						
RPD (Limits)	,			(	( )	( )	( )		( )	( )	( )	( )	( )	( )	( )		( )	( )	( )	( )	( )	( )	( )	
LCSD %R (1 imits)	(2)				( )	( )	( )		( )		( )	( )	( )	( )	( )		( )	( )	( )	( )	( )	( )	( ) .	( )
LCS %B (1 imits)	\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\				( )	( )	( )		( )		)	( )	( )		( )	( )	( )	( )		( )	( )	( )	( )	
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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

<sup>\*\*</sup>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	Analyte	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

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)	225.0 (75-125) 160.2 (75-125)	-	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	А

### VI. Duplicate Sample Analysis

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

### VII. Laboratory Control Samples (LCS)

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

LCS ID	Analyte	%R (Limits)	Associated Samples	Flag	A or P
LCS	Palladium 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)	DDD	Difference		
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)	А

	Concentra	ation (mg/Kg)		D:#*		
Analyte	TSB-HJ-07-0'**	TSB-HJ-07-0'-FD	RPD (Limits)	Difference (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)	А
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)	-	-	<u>-</u>

	Concentr	ation (mg/Kg)	222	D:#			
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)	555	D.W		
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

	Ī			<u> </u>	
SDG	Sample	Analyte	Flag	A or P	Reason
F8A250221	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-07-0'** TSB-HJ-07-0'-FD TSB-HJ-07-10' 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-HR-04-10' TSB-HR-04-0' TSB-HR-04-0'** TSB-HR-07-10' TSB-HR-07-10'** TSB-HR-06-0' TSB-HR-06-10' TSB-HR-07-0'-** TSB-HR-08-10' TSB-HJ-07-10' TSB-HJ-07-10' TSB-HR-08-10' TSB-HR-08-10'	Barium Calcium Chromium Lead Magnesium Niobium Silicon Strontium Vanadium Zinc	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) 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-04-10' TSB-HR-07-0' TSB-HR-07-10'** TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'-FD TSB-HJ-07-10' TSB-HJ-07-10' TSB-HJ-07-10' TSB-HJ-08-0' TSB-HR-08-0' TSB-HR-08-10'	Palladium Platinum	J+ (all detects) J+ (all detects)	Р	Laboratory control samples (%R)
F8A250221	TSB-HJ-07-0'** TSB-HJ-07-0'-FD	Calcium	J (all detects)	А	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	Α
F8A250221	TSB-HJ-05-0'	Cadmium Lithium	0.26U mg/Kg 21.0U mg/Kg	Α
F8A250221	TSB-HR-04-10'	Niobium	6.6U mg/Kg	Α
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	А
F8A250221	TSB-HR-07-10'**	Niobium	6.7U mg/Kg	Α
F8A250221	TSB-HR-06-0'	Cadmium Niobium Lithium	0.28U mg/Kg 5.5U mg/Kg 22.2U mg/Kg	A
F8A250221	TSB-HR-06-10'	Cadmium Niobium	0.55U mg/Kg 5.5U mg/Kg	Α
F8A250221	TSB-HJ-07-0'**	Niobium Lithium	5.4U mg/Kg 21.7U mg/Kg	Α
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	Α
F8A250221	TSB-HR-08-10'	Cadmium Niobium	0.55U mg/Kg 5.5U mg/Kg	А

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

No Sample Data Qualified in this SDG

SDG #	t: 18386A4 #: F8A250221 atory: Test America	<b>VA</b>	LIDATIOI _		PLETE evel II		ESS WORK	SHEET	Date: 356 Page:of Reviewer: 2nd Reviewer:
METH	IOD: Metals (EPA SW 84	46 Me	ethod 6020/	6010B/70	00)				2nd Reviewer.
	amples listed below were tion findings worksheets.		ewed for ead	ch of the f	followin	g va	alidation areas	. Validation findi	ngs are noted in attached
					T			Comments	
1	Validation  Tochnical holding times	Alta		A	Sampli	20 d	ates: 1/24/0	Comments_ ∤	
<u>I.</u> II.	Technical holding times  Calibration			A	Sampli	ng u	ales.	<u> </u>	
     .	Blanks			5~					
IV.	ICP Interference Check San	nple (I	CS) Analysis	A					
V.	Matrix Spike Analysis	· · · · · · · ·		SW		ı lu	5 /45D		
VI.	Duplicate Sample Analysis			N		71-	<del>/ / / / / / / / / / / / / / / / / / / </del>		
VII.	Laboratory Control Samples	(LCS)	<b>)</b>	5W	1	cz			
VIII.	Internal Standard (ICP-MS)			A	Vo	<u> </u>	berseved f	m len 3	
IX.	Furnace Atomic Absorption	QC		N	M.4	r V	tilies '		
Χ.	ICP Serial Dilution			A			U		
XI.	Sample Result Verification			A	Not re	view	ed for Level III val	idation.	
XII.	Overall Assessment of Data	1		A					
XIII.	Field Duplicates			324	(I	1,	12)		
XIV.	Field Blanks		UIME .	۲					
Note: /alidat	A = Acceptable N = Not provided/applicable SW = See worksheet ed Samples: ** Indicates sam		R = Rin FB = Fid	eld blank		ed	D = Duplio TB = Trip EB = Equ		
	501	1	I					····	
1	TSB-HJ-05-10'	11	TSB-HJ-07-0	**	2	21		31	
2	TSB-HJ-05-0'	12	TSB-HJ-07-0	'-FD	2	22		32	
3	TSB-HR-04-10'	13	TSB-HJ-07-1	0'	2	23		33	
4	TSB-HJ-04-0'	14	TSB-HR-08-0	)1	2	24		34	
5	TSB-HR-04-0'**	15	TSB-HR-08-1	0'	2	25		35	
6	TSB-HJ-04-10'	16	TSB-HR-08-0	MS	2	26		36	
7	TSB-HR-07-0'	17	TSB-HR-08-0	'MSD		27		37	
8	TSB-HR-07-10'**	18	prs		2	28		38	
9	TSB-HR-06-0'	19			2	29		39	

Notes:		

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TSB-HR-06-10'

### **VALIDATION FINDINGS CHECKLIST**

Page: of A Reviewer: uuu 2nd Reviewer: W.H

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

Method:Metals (EPA SW 846 Method 6010/7000/6020)	Т	1	_	1
Validation Area	Yes	No	NA	Findings/Comments
i. Technical fiolding times	a . 41			
All technical holding times were met.	<u> </u>			
Cooler temperature criteria was met.	1			
II.:Calibration				
Were all instruments calibrated daily, each set-up time?	1			
Were the proper number of standards used?	1			
Were all initial and continuing calibration verification %Rs within the 90-110% (80- 120% for mercury and 85-115% for cyanide) QC limits?	1			
Were all initial calibration correlation coefficients > 0.995? (Level IV only)		- 34 9 384 B-10	250722	
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.	8	575 din 1000		
IV. IGR Interference Check Sample				
Were ICP interference check samples performed daily?	1			
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?				
V: Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	~			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.		1		
Were the MS/MSD or duplicate relative percent differences (RPD) ≤ 20% for waters and ≤ 35% for soil samples? A control limit of +/- RL(+/-2X RL for soil) was used for samples that were ≤ 5X the RL, including when only one of the duplicate sample values were ≤ 5X the RL.		/		
V Laboratory control samples		147	œ.	
Was an LCS anaylzed for this SDG?	\			
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?		/		
VI, Furnace Atomic Absorption QC				And the second s
If MSA was performed, was the correlation coefficients > 0.995?			\( \)	
Do all applicable analysies have duplicate injections? (Level IV only)				
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)			/	
Were analytical spike recoveries within the 85-115% QC limits?			/	

LDC #: \ 8386 A4 SDG #: See we

### **VALIDATION FINDINGS CHECKLIST**

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Validation Area	Yes	No	NA	Findings/Comments
VII. ICR Serial Dilution	ere i Lan			
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the IDL?	1			7101 x MON for Tepher
Were all percent differences (%Ds) < 10%?				
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.			/	
VIII Internat Standards (EPA/SW 845/Method 8020)		24.4		
Were all the percent recoveries (%R) within the 30-120% of the intensity of the internal standard in the associated initial calibration?	/			
If the %Rs were outside the criteria, was a reanalysis performed?			/	
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 Verification: See Sec. 1992				Extra Control of the
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XI Overall assessment of data				
Overall assessment of data was found to be acceptable.				
XII Field duolicates				
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.			ノ	

LDC #: <u>1838644</u> SDG #: <u>See cover</u>

### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

All circled elements are applicable to each sample.

Sample ID	Matrix	Target Analyte List (TAL)
1-15	>01	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,
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,
	l	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,
12/6.17	50,~)	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U. Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Analysis Method
ICP		Li, S,
ICP-MS		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na. Tl, V, Zn, Mo, B, Si,
ICP-MS		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Zr,
GFAA		Al Sh. As. Ba. Be. Cd. Ca. Cr. Co. Cu. Fe. Ph. Mg. Mn. Hg. Ni, K. Se. Ag. Na, Tl. V. Zn. Mo. B. Si, CN <sup>-</sup>

Comments:	Mercury by CV	AA if performed	2											
Nb: Niobium,	Pd: Palladium,	P: Phosphorus,	Pt: F	Platinum,	S:	Sulfur,	W:	Tungsten,	U:	<u>Uranium,</u>	Zr:	Zirconium		

VALIDATION FINDINGS WORKSHEET

PB/ICB/CCB QUALIFIED SAMPLES

Soil preparation factor applied: ICP:100X, ICP/MS:200X, Hg:166.7X

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

SDG #: See Cover

LDC #: 18356A4

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Associated Samples: 6/2/8~ Sample Concentration units, unless otherwise noted: mg/Kg

0.068 / 0.55 3.4 / 5.5 9 0.11/0.28 12.0 / 22.2 3.8 / 5.5 6 4.4 / 6.7 ω 0.069 / 0.27 17.3 / 21.4 4.0 / 5.4 / 0.064 / 0.27 15.3 / 53.2 5.7 / 6.7 Sample Identification ဖ 0.076 / 0.13 14.3 / 20.9 0.29 / 1.3 3.3 / 5.2 2 10.7 / 21.8 0.10/0.554.8 / 5.5 4 5.5 / 6.6 ო 0.063 / 0.54 0.099 / 0.26 14.7 / 21.0 N Blank Action Maximum ICB/CCB<sup>a</sup> 0.036 0.5 6.0 7.6 0.5 6.8 9.0 0.5 0.4 Maximum PBª Maximum mg/Kg) 0.067 0.33 14.0 PBª <u>ج</u> 2.4 5.6 <u>4</u>. 3.0 3.9 8.2 Analyte Sa ၓ ပိ E. g ž ರ Z ≥

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.

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

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

Soil preparation factor applied: ICP:100X, ICP/MS:200X, Hg:166.7X

Associated Samples: 6-12

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Analyte	Maximum PBª	Σ			7-	12	13	4	15			
	3.1	(I/Biii)	(VBIII)	Imit								
	2.4											
Ca	14.0											
පි			0.036			0.081 / 0.27		0.099 / 0.27	0.076 / 0.55			
ပ်	0.33		0.5									
යි			0.5									
Fe	5.6											
Ξ			0.5									
qN	1.4		6.8		3.4 / 5.4	3.5 / 5.3	3.3 / 5.4	3.1 / 5.3	4.9 / 5.5			
	3.0											
	3.9											
Na	8.2											
E			0.4									
j	0.067		9:0									
M			6.0									
:=			9.7		10.2 / 21.7	12.0/21.2	20.6 / 21.5	9.4 / 21.2				

qualified as not defected, "U".

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

LDC #:\_

## VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

マスケ 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". Y N N/A Was a matrix spike analyzed for each matrix in this SDG?

Y N 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 Y NONA

of 4 or more, no action was taken.

Were all duplicate sample relative percent differences (RPD) ≤ 20% for water samples and ≤35% for soil samples? M N/A WE

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

N/A

*	MS/MSD ID	Matrix	Analyte	MS %Recovery	MSD %Recovery	RPD (Limits)	Associated Samples	Qualifications
=	16/11	\$0;	5b	9.09	547		A.	7/1/7
1	-		Ba	123.6	9,051		-	41.4
<u> </u>			ઙ	335.9	41.7			T
			Cr	( ) 6 10 1	134. 7			
			dd	ا کم ٔ لم				
			Ma	160.4				
			Nb 0	190,9	186.9			
			Ф	31.3	5-29			T-1,-T/2
			۲ >	9.82	ったなっ			2 1
			ر ک	9,691	4,00			1
			۸	6.971	139.0			
			7 h	ナー~1				
			, Δ			139 (420)		
							,	1 2 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
					-			
Comr	Comments:	, <u>X</u>						

TDC #:

## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Reviewer: 4H Page: of

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

M N/A Was Y N/A N/A Wer EVEL IV ONLY:

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

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

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.

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

							T		Ī	T											Ī		
	Qualifications	d/ (1) to	<b>\</b>																				
Associated States	Associated samples		,																				
%R (limits)	(ex/08/) sec/	17 8 141																		-			
Analyte	Pa	72	}			·																	
Matrbx	(1,105																						
di son	103												-									Comments:	
*					1	1							Ė	1	<u> </u>	<u> </u>	<u> </u>	上	$\bot$	1	┨.	Com	

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

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

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

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

YN NA YN NA 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	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#:\_18386A4 SDG#: See Cover

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

Page: Vof X Reviewer: V 2nd Reviewer: MX

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

YN NA YN NA 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	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)	

V:\FIELD DUPLICATES\FD\_inorganic\18386A4.wpd

LDC# 1838/AH SDG# SEX WE

# VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

Page: Of A Reviewer: Wry 2nd Reviewer: MA

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

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

%R = <u>Found</u> x 100 WI True

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

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
750/	ICP (Initial calibration)	5	<u>०१</u> ६५	40000	1.99	1-65	7
	GFAA (Initial calibration)						
7·0	CVAA (Initial calibration)	4	1.5)	2-5	اهه، لم	4.00)	>
cw	ICP (Continuing calibration)	٦,	3868	4000	999	546	7
	GFAA (Continuing calibration)						
cal	CVAA (Continuing calibration)	Ha	Jo6	0-5	10/2	7. 0	γ
In	ICP/MS (Inittal calibration)	5N	SE) or	200	1,00)	(, ec)	
col	ICP/MS (Continuing calibation)	Ca	216.73	002	(٥٤٠٦	1.801	

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

LDC #: (8386 Art SDG# Let Con

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

Reviewer: And Page: \_\_of\_

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

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

%R = Found x 100 True

Where, Found = Concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation, Found = SSR (spiked sample result) - SR (sample result).

True = Concentration of each analyte in the source.

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

RPD = <u>IS-DL</u> × 100 (S+D)/2

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

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

%D = 1-SDR x 100

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

					Recalculated	Renorted	
Sample ID	Type of Analysis	Element	Found / S / I (units)	True / D / SDR (units)	%R/RPD/%D	%R/RPD/%D	Acceptable (Y/N)
ILSAB	ICP interference check	95	49 93	00)	676	9,19	À
169	Laboratory control sample	Ľ	からへ	0 %	(°)(°)	(.69.7	
27	Matrix spike	o H	9 24 4 1 (us-uss)	53.019	p.01)	110.3	
16/19	Duplicate	( <del>-</del> <del>-</del> <del>-</del> <del>-</del> <del>-</del> <del>-</del> <del>-</del> <del>-</del> <del>-</del> <del>-</del> <del>-</del>	781	25)	٠٠٠	6,1	
<del>)</del> )	ICP sertal dilution	<b>4</b>	Etilla	4tst.of	4.0	40	<b>\</b>

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

LDC #: 18386 A4 SDG #: Cel Cour

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	_( of 2
Reviewer:_	my
2nd reviewer:_	gne

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

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

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

Are results within the calibrated range of the instruments and within the linear range of the ICP?

N N/A Are all detection limits below the CRDL?

	ed analy ig equat	te results forion:	····	were recalculated	and verified using the	
Concentration =		(RD)(FV)(Dil) (In. Vol.)(%S)	Recalcula			
RD FV In. Vol. Dil %S	= = = =	Raw data concentration Final volume (ml) Initial volume (ml) or weight (G) Dilution factor Decimal percent solids	Mn=	861.2mlx	0.959	= 359,2 mg/mg

Sample ID	Analyte	Reported Concentration ( Wg/kg/	Calculated Concentration ( Mg/kg )	Acceptable (Y/N)
<u> </u>	Lì	14.3	14.3	У
	Al	1860	1860	
	As	1,3	1,3	
	Ba	145	145	
	Be	0.53	0.55	
	Cd	0.076	0.076	
	Ca	12600	12600	
	<u>cr</u>	10,4	10,4	
	Co	6-5	6-5	
	Cy	16.4	lbig	
	Fe	12700	2700	
	Pb	10	10	
	Mg	7460	1460	
	lyn	359	359	
	140	0,4]	240	
	<u>Vr</u>	17.3	17.3	
	<u>M</u> b,	3,3	3.3	
	Pd	0.75	0,75	
	P	1010	(000	
	K	1840	(840	
	Si	112	112	
	Va	٤٩	203	J

LDC #:	18386AV
SDG #:	believe

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	2 or 2
Reviewer:	MH
2nd reviewer:	me

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

MEITH	JD: IIa	ce Metals (EPA SW 846 Meth	od 6010/7000)		
Please Y N O N Y N	see qua N/A N/A N/A	Have results been reported	and calculated correctly ated range of the instrur	applicable questions are identifie y? ments and within the linear range	
	ed analy ig equat	te results forion:	5	were recalculat	ted and verified using the
Concent	ration =	(RD)(FV)(Dil) (In. Vol.)(%S)	Recalculation:		
RD FV In. Vol. Dil %S	== == ==	Raw data concentration Final volume (ml) Initial volume (ml) or weight (G) Dilution factor Decimal percent solids	√= <u>8</u>	0-59 x 0-959	- = 35.34 mg/ng

Sample ID	Analyte	Reported Concontration ( W.J.y. )	Calculated Concentration ( MA K )	Acceptable (Y/N)
5	5 <sub>V</sub>	122	122	Y
	5 N	0.084	0.084	1
	<u> 7</u> i	なり	526	
	·W	0,29	0.29	
	U	0.90	0-95	
	V	74.}	35.3	
	2n	31.7	31.7	/
	y walny)	13-6'	13-6	Λ
	0			
		-		
				· · · · · · · · · · · · · · · · · · ·
				· · · · · · · · · · · · · · · · · · ·

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

**BRC Tronox Parcel H** 

**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'

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

**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)	P
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)	Р
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)	Р
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)	Р
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)	Р

### III. Blanks

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

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
PB (prep blank)	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 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 3.4 mg/Kg 0.073 mg/Kg 0.054 mg/Kg 0.077 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

Sample	Analyte	Reported Concentration	Modified Final 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
	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	0.47 mg/Kg	0.53U mg/Kg
	Tungsten	0.35 mg/Kg	1.3U 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	5.3 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	0.50 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
TSB-HR-05-10'	Boron	4.9 mg/Kg	26.8U mg/Kg
	Cadmium	0.071 mg/Kg	0.13U mg/Kg
	Silver	0.11 mg/Kg	0.54U mg/Kg
	Tin	0.50 mg/Kg	0.54U mg/Kg
	Tungsten	0.27 mg/Kg	1.3U mg/Kg

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

Rinsate ID	Sampling Date	Analyte	Concentration	Associated Samples
RINSATE-2	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 1.0 ug/L 1.0 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 ( >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)	А

### VI. Duplicate Sample Analysis

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

### VII. Laboratory Control Samples (LCS)

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

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

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)	Р

### 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 Strontium	10.1 (≤10) 10.6 (≤10)	All soil samples in SDG F8A290158	J (all detects) J (all detects)	Α

### 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	TSB-HR-06-0'	RPD 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)	RPD	D.44	"	
Analyte	TSB-HR-06-0'	TSB-HR-06-0'-FD	(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)	-	-	-
Cobalt	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)	А
Silver	0.090	0.094	-	0.004 (≤0.54)	-	-

	Concentr	ation (mg/Kg)				
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)	-	-

	Concentration (ug/Kg)		` ~ ~'			
Analyte	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

600	O a maral s	A 1 4 -	Flore	A === D	B
<b>SDG</b> F8A290158	Sample RINSATE-2	Boron Niobium Silver	Flag  J+ (all detects)  J+ (all detects)  J+ (all detects)	A or P	Reason  Calibration (%R)
F8A290158	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-10' TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HJ-08-10' TSB-HR-05-10'	Silver	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-05-0'	Palladium	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-HR-05-0' 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-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-08-10' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10'	Niobium Palladium Magnesium	J+ (all detects) J+ (all detects) J+ (all detects)	А	Matrix spike/Matrix spike duplicates (%R)

SDG	Sample	Analyte	Flag	A or P	Reason
F8A290158	RINSATE-2	Palladium	J+ (all detects)	Р	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)	Р	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-HR-05-0' TSB-HR-05-0'	Manganese Strontium	J (all detects) J (all detects)	А	ICP serial dilution (%D)
F8A290158	TSB-HR-06-0' TSB-HR-06-0'-FD	Silicon	J (all detects)	А	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	А
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	А
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	А

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	А
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	А
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	Α
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	Α
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	А
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	А
F8A290158	TSB-HJ-08-0'	Cadmium Tin Tungsten	0.14U mg/Kg 0.54U mg/Kg 1.4U mg/Kg	А
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	А

SDG :	#:18386B4 #:F8A290158 atory:_Test America	VA -	LIDATION		LETI _evel		ESS WORKS	HEET	Date: 3/t/oz Page: lof / Reviewer: ~ 2nd Reviewer: MA
METH	IOD: Metals (EPA SW 84	46 Me	ethod 6020/6	6010B/700	00)				Zild Neviewer.
	amples listed below were tion findings worksheets.		ewed for eac	ch of the fo	ollowin	ng va	alidation areas. \	√alidation findi	ngs are noted in attached
	Validation	Area						Comments	
l.	Technical holding times			A	Sampl	ing da	ates: 1/28/08		
11.	Calibration			4W			· · · · · · · · · · · · · · · · · · ·		
III.	Blanks			sW					
IV.	ICP Interference Check San	nple (I	CS) Analysis	A				SS	
V.	Matrix Spike Analysis			5W		М	5 /usp		
VI.	Duplicate Sample Analysis			N	)		,		
VII.	Laboratory Control Samples	(LCS)		5W	Lo	5			
VIII.	Internal Standard (ICP-MS)			h	:h	<u>/;+</u>	veriewed	***************************************	
IX.	Furnace Atomic Absorption	QC		N	h:+		utilizes	****	
X.	ICP Serial Dilution			SW			V		
XI.	Sample Result Verification			N					
XII.	Overall Assessment of Data	1		A					
XIII.	Field Duplicates			5W	(	٤, د	t )		
XIV.	Field Blanks			>w/	R.	>   C	)		
Note:	A = Acceptable N = Not provided/applicable SW = See worksheet	<b>;</b>	R = Rin	o compounds sate eld blank	s detec	ted	D = Duplica TB = Trip b EB = Equip	lank	
Validat	ed Samples: 세 🤫 🕌	exe	+ + 1	0, 13, 1	14 1	42			
1	TSB-HJ-10-0'	11	TSB-HR-05-1	0'MS		21	-	31	
2	TSB-HJ-10-10'	12	TSB-HR-05-1	0'MSD		22		32	
3 ,	TSB-HR-06-0'	13	RINSATE-2M	s Ar		23		33	
4	TSB-HR-06-0'-FD	14	RINSATE-2M	sd 1/		24		34	***************************************
5	TSB-HR-06-10'	15	PB			25	<b>**</b>	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 #: 1838684 SDG #: <u>See an</u>

### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page: of / Reviewer: 2nd reviewer: 9My

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,
m11,12	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,
13.14	A2	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mr, Hg, Wi, K, Se, Ag, Na, Tl, 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, Śi,
		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,
Mo	501/82	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,
1213,14	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		<u>(19</u>
ICP-MS		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si
ICP-MS		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Zr,
GEAA		Al Sh As Ba Be Cd Ca Cr Co Cu Fe Ph Mg Mn Hg Ni K Se Ag Na Tl V Zn Mo B Si CN

Comments: Mercury by CVAA if performed

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

1878684

# VALIDATION FINDINGS WORKSHEET Calibration

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Reviewer: Jud

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

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

Y N N/A

Were all initial and continuing calibration verification percent recoveries (%R) within the control limits of 90-110% for all analytes except mercury (80-120%) and cyanide (85-115%)?

EVEL IV ONLY:

Was a midrange cyanide standard distilled?

Are all correlation coefficients >0.995? Y N MA

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

	_			3	According Semples	Qualification of Data
*	2/Clos		Analyte	ナミ	₽BW	1/-T1 +T
<u> </u>	0		0			
٧	3/8/2	(51 (x) /27)	2	(1213	A11 Mg	
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<u></u>	80191	(44)	Aq	<b>لایدا</b> ا	1-1, PBS	T+ 11/10
<u>H</u>			ρ	,		
17	2/6/08	(1324)	49	ナバス	8,9 11,12	ø
<u>†</u>	4_	1	D			
<u>                                     </u>	2/1/28	(p) (1819)	γd	9 \( \)	Sad <del>Mad</del> 8 -	J+176
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SDG #: See Cover LDC #: 18356A4

VALIDATION FINDINGS WORKSHEET **METHOD:** Trace Metals (EPA SW 846 Method 6010B/6020/7000)

Soil preparation factor applied: ICP:100X, ICP/MS:200X, Hg:166.7X Associated Samples: All Soil PB/ICB/CCB QUALIFIED SAMPLES

Page: Vof Y Reviewer: 4NZ

Analyte		The Control of the Co							Sample Id	Sample Identification	The state of the second second	ACCORDING AND THE PROPERTY OF THE PARTY OF T		
	Maximum PB <sup>a</sup> (mα/Kα)	Maximum PB <sup>a</sup>	Maximum ICB/CCB <sup>a</sup>	Blank Action I imit	<i>F</i>	2		4	5	9	7	8	6	
₹	1.9													
Ва	0.052													
В			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	
ပ္ပ			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	
ప	0.15													
QN.			6.1			4.7 / 6.6	2.1 / 6.5							
Q.	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	
ï	0.077		6:0											
8			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	
ם														-
Zn	1.3													

qualified as not defected, "U".

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

SDG #: See Cover LDC #: 18386A4

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

Sample Concentration units, unless otherwise noted: ug/l

VALIDATION FINDINGS WORKSHEET PB/ICB/CCB QUALIFIED SAMPLES

Soil preparation factor applied: All AQ Associated Samples:

Page: (of ≯ Reviewer:

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Sample Identification																				
Sa																				
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								:												
				0.50		5.0	0.0		2.0	5.0	5.0	·								
	10			0.027 / 0.50		6.3 / 25.0	21.0 / 50.0		0.51 / 2.0	1.0 / 2.0	0.67 / 5.0									
	Blank Action Limit																			
									-											
	Maximum ICB/CCB <sup>a</sup>	0.2		0.1	0.2	6.1				1.2	9.0									
	Maximum PB <sup>a</sup> (110/1)		22.4	0.029	0.37	20.1	5.5	1.4	0.72	08.0	1.9									
				Ö	J	7				_										
	Maximum PB <sup>a</sup> (mq/Kq)																			
							·													
	Analyte	Sb	В	පු	Мо	Q Q	Na	F	Sn	F	3									

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.

# **VALIDATION FINDINGS WORKSHEET**

Field Blanks

Reviewer:

Page:

SDG #: See Cover

LDC #: 18386A4

Were target analytes detected in the field blanks?

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

No. N/A

Were field blanks identified in this SDG?

No. N/A

Were target analytes detected in the field blank units: ug/L

Associated sample units: mg/Kg

200X Soil factor applied \_ Field blank type: (circle one) Field Blank / Rinsate / Other. 1 /28 /08 Sampling date:\_\_

Associated Samples: All Soil

													 		_		_
				-													
	6	0.071 / 0.13							0.50 / 0.54		0.27 / 1.3						
	8	0.14 / 0.54					138 / 218										
	7	0.10 / 0.14							0.50 / 0.54		0.33 / 1.4			100			
tion	9	0.10 / 0.14							0.52 / 0.54		0.28 / 1.4						
Sample Identification	5	0.077 / 0.13							0.47 / 0.53		0.35 / 1.3						
Š	4	0.094 / 0.14							0.46 / 0.54								
	င					2.1/6.5					0.36 / 1.3						
	2	0.090 / 0.13				4.7.16.6			0.50 / 0.52		0.46 / 1.3						
	-	0.094 / 0.13									0.74 / 1.3						
	Action Level																
Blank ID	10	0.027	72.3	32.9	9.2	6.3	21.0	0.67	0.51	1.0	29.0						
Analyte		ΡΌ	Ca	Fe	Мg	qN	Na	JS	uS	!L	Μ						

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".

see cost M9888) LDC #:

# Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

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".

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

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

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

of 4 or more, no action was taken.

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

LEVEL IV ONLY:

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

54,5 41,2 41,8 41,2 41,8 41,9 128,3 128,3 128,3 128,3 128,3 128,3 128,3 128,4 128,5 128,4 128,5 128,4 128,5 128,4 128,5 128,6 128,7 128,6 128,7 128,6 128,7 128,6 128,7 128,6 128,7 128,6 128,7 128,6 128,7 128,7 128,6 128,7	Me/Men in	Martin	l	SW	MSD			
56   54.5   57.9   All 2.   2-18.4     Map   129.7   128.2   3-18.4     Map   129.7   128.3   444.2 (520.)   14.4     Map   129.7   129.1   129.4     Map   129.7   129.1   129.4     Map   129.7   129.7   129.4     Map   129.7   120.7   120.7   120.7     Map   120.7   120.7   120.7     Map   120.7   120.7   120.7     Map   120.7   120.7   120.7     Map   120.7   120.7   120.7     Map   120.7   120.7   120.7   120.7   120.7     Map   120.7   120.7   120.7   120.7   120.7     Map   120.7   120.7   120.7   120.7   120.7   120.7     Map   120.7   120.7   120.7   120.7   120.7   120.7   120.7   120.7   120.7   120.7   120.7   120.7     Map   120.7		alra		*Recovery	%Recovery	RPD (Limits)	Associated Samples	Qualifications
Sec   41,2   4,8   4,9   4,8   4,0		701		54.5	57.9		(41 Soir)	J-/"Z/A
Mb (69,4 2,0,0 Mg (28,3) 128,3 Mh (44,2(520) 1 No and (2 Nh (44,2(520) 1 No and (2 Nh (44,2(520) 1 No and (2 No and (2) Mh (44,2(520) 1 No and (2) No and (				412	4.8			T-/0/A
Mg 129,7 138,3 444,2 (220) 6 604 (1) ND 1,4 54 A 44 0 wm client sourple.				169.4	2000			7+1+1
My No and 131 445 (520) & No and 12 54 54 54 54 54 54 54 54 54 54 54 54 54				129,7	5 '871			
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A99888 LDC #:

# VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Reviewer: | MH 2nd Reviewer: 3ル名 Page: Reviewer:

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

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

| N N/A | Was a laboratory control sample (LCS) analyzed for each matrix in this SDG?
| N N/A | Were all aqueous LCS percent recoveries (%R) within the control limits of 80-120% and all soil LCS %R within laboratory established control limits. Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations. Y N KI/A

See contr LDC#: (8386B4 SDG#:

# VALIDATION FINDINGS WORKSHEET **ICP Serial Dilution**

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.

(↑) N N/A If are Y (N) N/A We Y N N/A Is the LEVEL IV ONLY: Y N N/A We

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

Thate Diluted Sample ID Matrix Analyse % Of Timits) Associated Samples (%) Associated Sampl		Qualifications	ナナナ												
Diluted Sample ID  As T  Analyte  Analy	Asheet for recalculations.	Associated Samples	10° 10°												
Diluted Sample ID Matrix Analyte So T   Mn Sv	I Secarculation VV OI	%D (Limits)	ا م)	(0.6											
Diluted Sample ID Matrix	000 000	Analyte	Д,	5r											
Diluted Sample ID	- acceptable:	Matrix	50T)												
		Diluted Sample ID	6												
# ## ## ## ## ## ## ## ## ## ## ## ## ## ## ##															

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Comments:

LDC#:\_\_18386B4 SDG#:\_\_See Cover

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

Page: of Pag

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

YN NA YN NA 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			
Niobium	2.1	2.0U		0.1	( ≤6.8)	
Palladium	0.22	0.21		0.01	( ≤0.54)	

LDC#: <u>18386B4</u> SDG#: <u>See Cover</u>

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

Page: Vof V Reviewer: 2nd Reviewer: 9NH

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

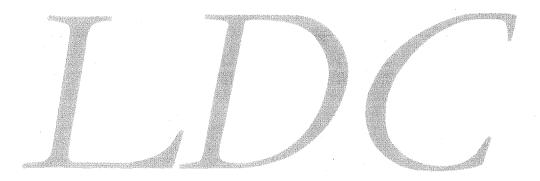
YN NA YN NA Were field duplicate pairs identified in this SDG? Were target analytes detected in the field duplicate pairs?

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Compound	3	4	RPD	Difference	Limits	(Parent Only)
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)	

V:\FIELD DUPLICATES\FD\_inorganic\18386B4.wpd

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

Wet Chemistry



## 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

<sup>\*\*</sup>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, 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)				A or P
Analyte	TSB-HJ-07-0'**	TSB-HJ-07-0'-FD	RPD (Limits)	Difference (Limits)	Flag	
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)	А
Fluoride	0.58	0.27Ù	-	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)				A or P	
Analyte	TSB-HJ-07-0'**	TSB-HJ-07-0'-FD	RPD (Limits)	Difference (Limits)	Flag		
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-04-10' 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-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)	А	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 SDG #: F8A250221 Laboratory: Test America					PLETEN evel III/I	Date: 3/5/s Page: of Page: of Page: work  Reviewer: work  2nd Reviewer: MK					
Metho	od 300.0), Perchlorate (E	<u>PA N</u>	lethod 314.0	)), O & G (	EPA SW	846 Method	7091B	ophosphate-P, Sulfate (EPA			
	amples listed below wer tion findings worksheets		ewed for ea	ch of the f	ollowing v	alidation are	as. Validation find	dings are noted in attache			
	Validation	Area					Comments				
I.	Technical holding times			A	Sampling	dates: ¹√γ√	108				
lla.	Initial calibration			À							
IIb.	Calibration verification			A							
111.	Blanks			Δ							
IV	Matrix Spike/Matrix Spike D	Duplicat	es	5W	) IM	1450/Du	<del></del> Р				
				A	7	,,,,,,,					
VI.				A	ushusp						
VII.				A	Not reviewed for Level III validation.						
VIII.				A							
IX.	Field duplicates		5W	CII, 12)							
x	Field blanks		N								
Note:	A = Acceptable N = Not provided/applicabl SW = See worksheet		R = Rin: FB = Fie	eld blank			plicate rip blank quipment blank	•			
validate	ed Samples: ** Indicates sam	ipie un	derwent Level	v 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	D'	23		33				
4	TSB-HJ-04-0'	14	TSB-HR-08-0	,	24		34				
5	TSB-HR-04-0'**	15	TSB-HR-08-1	0'	25		35				
6	TSB-HJ-04-10'	16	TSB-HJ-05-10	O'MS	26		36				
7	TSB-HR-07-0'	17 TSB-HJ-05-10		O'MSD	27		37				
8	TSB-HR-07-10'**	18	TSB-HJ-05-10	D'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:\_\_\_\_

### **VALIDATION FINDINGS CHECKLIST**

Page: 1 of 1 Reviewer: 1 MM 2nd Reviewer: 9M &

Method:Inorganics (EPA Method Sel Will)

Method:Inorganics (EPA Method )			7	
Validation Area	Yes	No	NA	
I. Technical holding times.			, A. A	
All technical holding times were met.	1			
Coolor temperature criteria was met.	/			
Licabration .	145			
Were all instruments calibrated daily, each set-up time?	1			
Were the proper number of standards used?	1			
Were all initial calibration correlation coefficients > 0.995?	_			
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?	/			
Were titrant checks performed as required? (Level IV only)			_	
Were balance checks performed as required? (Level IV only)				
Was a method blank associated with every sample in this SDG?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/		
IV Manux spike Mainx spike kluplicates and Duplicates 43 - 43 - 44 - 44 - 44 - 44 - 44	k)			
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.	K	<b>√</b>		
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 services				
Was an LCS anaytzed for this SDG?				
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% (85-115% for Method 300.0) QC limits?		41)W 50.W.		
VI. Regional Cidality Assurance and Quality Control				
		-		
Were performance evaluation (PE) samples performed?		1		

LDC#: \ 838646 SDG#: Sel cover

### VALIDATION FINDINGS CHECKLIST

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

Validation Area	Yes	No	NA	Findings/Comments
VII_Sample Result Verification		fila ka		
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	V			
Were detection limits < RL?	/			
AN TABLEMASS SOUTH OF TAILS 18 18 18 18 18 18 18 18 18 18 18 18 18		Mil		
Overall assessment of data was found to be acceptable.	V	**************************************		
Complicate of the second secon				
Field duplicate pairs were identified in this SDG.	$\checkmark$			
Target analytes were detected in the field duplicates.	1	-		
Complete and the complete of t				
Field blanks were identified in this SDG.		~		
Target analytes were detected in the field blanks.			1	

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### VALIDATION FINDINGS WORKSHEET Sample Specific Analysis Reference

Page: of Reviewer: 2nd reviewer: ML

All circled methods are applicable to each sample.

T		
Sample ID	Matrix	Parameter
1-15	50:	Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
u lb-18	Soi)	Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
19,00		Br) Bromine (C) Chlorine (B) NO3 (NO2 (SO2 O-PO2 (Chlorate CIO4 Q+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine 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

Comments:	

SDG #: 18386 Mb

## VALIDATION FINDINGS WORKSHEET Matrix Spike Analysis

Page: of Reviewer: 2nd Reviewer: 3nd

METHOD: Inorganics, Method

See cover

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

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

*	Matrix Spike ID	Matrix	Analyte	%R	Associated Samples	Outlibushase
	128-43-02-101	1508	\$0¢	カカ	Ī	No and
- ]						#10 00 - 469
						With control the
4	0	50,2	5+0	14	1	1/4T /4
		-				
·						
		,				
	,					
Com	Comments:					

LDC#: <u>18386A6</u> SDG#: <u>See Cover</u>

### VALIDATION FINDINGS WORKSHEET

Field Duplicates

Page:_	(of	
Reviewer:	رتت	_
2nd Reviewer:	MA	

Inorganics, Method: See Cover

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

	Concentrat	ion (mg/Kg)				0
Analyte	11	12	RPD (≤50)	Difference	Limits	Qualification (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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SDG #: (\$386 M6)

# Validatin Findings Worksheet Initial and Continuing Calibration Calculation Verification

Page: \_\_\_\_of \_\_\_ Reviewer: \_\_\_µ\_\_\_ 2nd Reviewer: ¬¾ ∠

Method: Inorganics, Method\_

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

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

%R = Found X 100

Where,

Found = concentration of each analyte measured in the analysis of the ICV or CCV solution

True = concentration of each analyte in the ICV or CCV source

					Recalculated	Reported	Acceptable
Type of analysis	Analyte	Standard	Conc. (ug/L)	Area	r or r²	r or r²	(Y/N)
Initial calibration		s1	-	0.00316			
	CIO4	s2	2.5	0.00918	0.9999372	0.999867	
		s3	ည	0.01935			7
		s4	10	0.04027			_
		s5	20	0.07784			
		9S	40	0.15704			
لمي Calibration verification	clos	oξ	28,0		93.3	NR	/ X
$\mathcal{L}_{\mathcal{L}}$ Calibration verification	ellat	2307	3690.6		19.8	MR	
$c\omega$	βY	2000	o.frez		(0).45	(01.45 101.45	+

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

LDC #: 18386 MG

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

Reviewer: WA 2nd Reviewer:

METHOD: Inorganics, Method \_

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

Where %R = Found x 100

Found =

True =

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

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

RPD =  $\frac{|S-D|}{(S+D)/2}$  x 100 Where,

11 11 SO

Original sample concentration Duplicate sample concentration

i i					Recalculated	Reported	
Sample ID	Type of Analysis	Element	Found / S (units)	True / D (units)	%R / RPD	%R / RPD	Acceptable (Y/N)
	Laboratory control sample						-
.Z		N 03-N	4.056	4,00	(0)	0	<del>-</del>
	Matrix spike sample		(SSR-SR)			-	
		016	<i>⊗</i> ®°)	04)	11	7 /	-
9	Duplicate sample	0 ,	۲, ۶	4 o K	\	w,	
<i>}</i>		3	2	)	1,	\ \ \ \ \	•

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

LDC #: 18786A6 SDG #: <u>Cee</u> cover	VALIDATION FINDINGS WORKSHEET Sample Calculation Verification
METHOD: Inorganics, Method	See couer
Please see qualifications below to	or all questions answered "N". Not applicable question

Page:_	of	
Reviewer:_	Ми	
2nd reviewer:_	mps	

				Ziid leviewe		
METH	IOD: Inorganics, Metho	d See cover				
Please Y N Ø N	N/A Have results N/A Are results w	ow for all questions answered "N". Not been reported and calculated correctl ithin the calibrated range of the instrur tion limits below the CRQL?	y?	are identified as "i	N/A*.	
	ound (analyte) results f ulated and verified usin	or 5	repo	rted with a positiv	e detect were	<u> </u>
Concer	Sof = Krun	Recalculation:  X Final Volume	G 042 -0.1	6381 40ml 24x 4g 16	2959 = 5;	3-65-mg/
#	Sample ID	Analyte	Reported Concentration ( W / / / /	Calculated Concentration ( wylu, )	Acceptable (Y/N)	
	5	choute ce	6.3	6.3	У	
H		Q	130	131	, '	

#	Sample ID	Analyte	Reported Concentration ( W / W)	Calculated Concentration ( \wf/\(\gamma\)	Acceptable (Y/N)
	5	Analyte  Chlorate  Cl  (l2  F  NOS-N  Cloy (vyfy)  404	(mg/y) 6.3	6.3	У
		æ	130	13/	,
		Q2	261	261	
		F	0.62	0-6	
		NO3-N	21,2 22200 53,9	27-1	
		clay (ugly)	22200	22400	
		404'	53.7	53.7	1
			,	,	
	7				

Note:		 		 		

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

**Project/Site Name:** 

**BRC Tronox Parcel H** 

**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)	Α
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)	Α

### V. Duplicates

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

### VI. Laboratory Control Samples

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

### VII. Sample Result Verification

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)	А
Sulfate	4.8	15.4	-	10.6 (≤5.4)	J (all detects)	А

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

### 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-HR-05-0' 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

	#: <u>18386B6</u> #: <u>F8A290158</u>					ESS	WORKSH	EET	Date: <u>3/1/-</u> 8 Page: <u></u> _of
Laboi	ratory: Test America							./ ,	Reviewer: W. 2nd Reviewer: M. K.
METI Metho	HOD: (Analyte) <u>Bromide</u> od 300.0), Perchlorate (B	Brom	nine, Chlorat lethod 314.0	e, Chloride )), O & G	e, Chorine (EPA SW8	Fluo 346 M		~	nophosphate-P, Sulfate (EPA
The s valida	amples listed below wer ation findings worksheets	e revi s.	ewed for ead	ch of the f	ollowing v	alidat	ion areas. Va	lidation fin	idings are noted in attached
	Validation	Area					C	omments	
1.	Technical holding times			Á	Sampling d	lates:	1/28/08	<u> </u>	
lla.	Initial calibration			A					
IIb.	Calibration verification			A					
III.	Blanks			A					
IV	Matrix Spike/Matrix Spike [	Ouplica	tes	SW	2 14	/ M4	,0/pup		
V	Duplicates			A		<u>'</u>	, <u>, , , , , , , , , , , , , , , , , , </u>		
VI.	Laboratory control samples	<u> </u>		A	Les/L	25D			
VII.	Sample result verification			N					
VIII.	Overall assessment of data	1		A					
IX.	Field duplicates			SW	(3,4				
L <sub>X</sub>	Field blanks			SW	R=	10			
Note:	A = Acceptable N = Not provided/applicable SW = See worksheet	e	R = Rins	o compound sate eld blank	s detected		D = Duplicate TB = Trip blant EB = Equipme		
/alidat	red Samples:	ent	# 10,1	8-20 7	12				
1	TSB-HJ-10-0'	11	TSB-HJ-10-0'	MS	21	Mß		31	
2 1	TSB-HJ-10-10'	12	TSB-HJ-10-0'	MSD	22			32	
3 1	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	D'DUP	25			35	
6 1	TSB-HJ-08-0'	16	TSB-HR-05-1	0'MS	26			36	
<sub>7</sub> <b>√</b>	TSB-HJ-08-10'	17	TSB-HR-05-1	0'DUP	27			37	
8 7	TSB-HR-05-0'	18	RINSATE-2M	s Az	28			38	
91	TSB-HR-05-10'	19	RINSATE-2M	SD	29			39	
10	RINSATE-2	20	RINSATE-2DI	UP Y	30			40	
Votes	s:								<u>.</u>

LDC #:_	(8 386 Bb
SDG #:	See com

### VALIDATION FINDINGS WORKSHEET Sample Specific Analysis Reference

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

All circled methods are applicable to each sample.

Sample ID	Matrix	Parameter Parameter
1/10	501/A	Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
18,20	Ar	(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
14-17	501	Br) Bromine (C) Chlorine (F) NO) NO) SO) Q-PO) (Chlorate) CIO <sub>4</sub> O+G/TPH
16,17	1	Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+O/TPH
4-13	J	Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> +G/TPH
18 1 19	A2	Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
/ \		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH

Comments:	 <del></del>		

LDC #: 1838/BB SDG #: 508

## VALIDATION FINDINGS WORKSHEET

Field Blanks

Page: of / Reviewer:

> Associated Samples:\_ Were target analytes detected in the field blanks? S Blank units: water Associated sample units: water Sampling date: 1/28/08 Soil factor applied Field Blank / Rinsate / Other: Were field blanks identified in this SDG? METHOD: Inorganics, EPA Method ON N/A

Analyte	Blank ID	Blank	Sample Identification	
	_	Action		
	2)		~	
78	0)`0		4.8/5.2	
Blank units:	3:	Associated sampl	ted sample units:	
Sampling d	late:		Soll factor applied	
Field blank	Field blank type: (circle one) Field Blank / F	one) Field	d Blank / Rinsate / Other: Associated Samples:	

Analyte	Blank ID	Blank	Samula Idantification
		Action	
		Limit	
CHCLED RESI	OLIS WERE NO	T QUALIFIED.	OHICLED RESULTS WERE NOT ONALIFIED. ALL RESULTS NOT CHOLED WERE QUALIFIED BY THE FOLLOWING STATEMENT.
Serripies with a	Samples with analyte concentrations within five times the	ations within fi	ive times the associated field blank concentration are listed above semilar partitions.
			The second was a second of the

LDC #: (838/86 bb

### VALIDATION FINDINGS WORKSHEET Matrix Spike Analysis

Page: of Reviewer:

METHOD: Inorganics, Method

See core

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?

YAN NA

Were matrix spike percent recoveries (%R) within the contr

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:
Y N (V) Wer

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

*	Matrix Solke ID	Mathy	Amalytic	r,		
	4	\$0.7	Language	5.7	Associated Samples	Qualifications Co. 1 Co. 1 0
ļ Ī					0.47	1 + C + C + C + C + C + C + C + C + C +
4	9)	So.;	5+0	68	A1 501	T-/n2/4
			`			
- 1			-			
	,					
Ē	Comments:					
١						

LDC#:_	<u>18386B6</u>
SDG#	See Cover

### VALIDATION FINDINGS WORKSHEET Field Duplicates

Page:_	of	
Reviewer:	ν	_
2nd Reviewer:	ank)	

Inorganics,	Method:	See	Cover	

YN NA YN NA 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

V:\FIELD DUPLICATES\FD\_inorganic\18386B6.wpd

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

Gasoline Range Organics



### 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

TOD LID OF SUMA

TSB-HR-08-0'MS

TSB-HR-08-0'MSD

<sup>\*\*</sup>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

SDG Labo	#:18386A7 #:F8A250221 ratory:_Test America		LIDATIO	Le	evel III/	V	ORKS	HEET		Date: 3 //2 Page: _/of _/ Reviewer:/2 2nd Reviewer:/	0/08
The s	HOD: GC Gasoline Rang amples listed below were tion findings worksheets.	revie				·	areas.	Validatio	on fin	dings are noted in attached	
	Validation	Area						Comm	nents		
1.	Technical holding times			Ą	Sampling	dates:	1/24	108			
lla.	Initial calibration			4			ĺ	t			
IIb.	Calibration verification/ICV			A	lev	2 15	•				
III.	Blanks			NO							
IVa.	Surrogate recovery			SW							
IVb.	Matrix spike/Matrix spike du	plicate	s	SW							
IVc.	Laboratory control samples			А	LCS						
V.	Target compound identificat	ion		٨	Not revie	wed for Lev	el III valid	lation.			
VI.	Compound Quantitation and	I CRQI	Ls	Δ	Not reviewed for Level III validation.						
VII.	System Performance			Δ	Not reviewed for Level III validation.						
VIII.	Overall assessment of data			٨							
IX.	Field duplicates			ND	P	11+	12				
X.	Field blanks			N							
Note: Valida	A = Acceptable N = Not provided/applicable SW = See worksheet ed Samples: ** Indicates sam		R = Rin FB = Fie	eld blank	s detected	Т	= Duplica B = Trip b B = Equip		ık		
1 3	TSB-HJ-05-10'	111	TSB-HJ-07-0'	** /	21	18031	129-1	1K	31	1/31	
2 3	TSB-HJ-05-0'	12 2	TSB-HJ-07-0'	-FD	22	8030	151-B	اإد	32	1/30	
3 <b>3</b>	TSB-HR-04-10'	13 2	TSB-HJ-07-10	0'	23	3 80 30	149 -8	ILK	33	1/29	
4 3	TSB-HJ-04-0'	147	TSB-HR-08-0	ı	24				34		
5 <b>Z</b>	TSB-HR-04-0'**	152	TSB-HR-08-1	0'	25				35		
6 r	TSB-HJ-04-10'	16 3	TSB-HJ-05-10	D'MS	26				36		
72	TSB-HR-07-0'	173	TSB-HJ-05-10	D'MSD	27				37		
82	TSB-HR-07-10'**	18 2	TSB-HR-08-0	'MS	28				38		
9 <b>r</b>	TSB-HR-06-0'	192	TSB-HR-08-0	'MSD	29				39		
102	TSB-HR-06-10'	20		-	30				40		

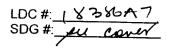
Notes:	 	

LDC #: 18386A7 SDG #: pu coner

### **VALIDATION FINDINGS CHECKLIST**

Page: /of 1
Reviewer: //
2nd Reviewer: //

Method: GCHPLC		<del></del>	<del>-</del> 1	
Validation Area	Ye:	s N	o N	IA Findings/Comments
It Technical holding times	T I			
All technical holding times were met.	+-	+	+	
Cooler temperature criteria was met.				
11. Initial calibration:		T T		
Did the laboratory perform a 5 point calibration prior to sample analysis?	-	4	+	
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?	-	-		
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?			-	
Did the initial calibration meet the curve fit acceptance criteria?			-	
Were the RT windows properly established?	/			
IV:Continuing calibration				
What type of continuing calibration calculation was performed?%D or %R				
Was a continuing calibration analyzed daily?				
Were all percent differences (%D) ≤ 15%.0 or percent recoveries 85-115%?				
Were all the retention times within the acceptance windows?	/			
V:Blanks			ive.	
Was a method blank associated with every sample in this SDG?	1-	f		
Was a method blank analyzed for each matrix and concentration?	1			
Nas there contamination in the method blanks? If yes, please see the Blanks alidation completeness worksheet.			1	
// Surrogate spikes				
Vere all surrogate %R within the QC limits?			-	
f the percent recovery ( $\%$ R) of one or more surrogates was outside QC limits, was reanalysis performed to confirm $\%$ R?				
any %R was less than 10 percent, was a reanalysis performed to confirm %R?			/	
II. Mainx spike/Matrix spike duplicates				
Vere a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each natrix in this SDG? If no, indicate which matrix does not have an associated IS/MSD. Soil / Water.				
/as a MS/MSD analyzed every 20 samples of each matrix?				
/ere the MS/MSD percent recoveries (%R) and the relative percent differences RPD) within the QC limits?		_	-	
III. Laboratory control samples				
as an LCS analyzed for this SDG?	-			
as an LCS analyzed per extraction batch?		-		
ere the LCS percent recoveries (%R) and relative percent difference (RPD) thin the QC limits?				



### **VALIDATION FINDINGS CHECKLIST**

Page: 20f 2
Reviewer: 7
2nd Reviewer: 1

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?				
XII S) Stern performance				
System performance was found to be acceptable.	J			
XIII Soverallessessmen of data				
Overall assessment of data was found to be acceptable.	$\supset$			
XIV-Field duplicates: The state of the state				
Field duplicate pairs were identified in this SDG.			T	
Target compounds were detected in the field duplicates.				
CV-Field blanks				
ield blanks were identified in this SDG.		7	- T	
arget compounds were detected in the field blanks.			1	

LDC #: 1838647 SDG #:

## VALIDATION FINDINDS WORKSHEET

Reviewer:

Page:

Surrogate Recovery

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

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

Were surrogates spiked into all samples and blanks?

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

) #	Sample ID	Detector/ Column	otor/ Imn	Surrogate Compound		%R (Limits)				6	
	۲۱	not ,	Parifixas	ગ			21-	146	1-/R		Guannications
			7						1		
						)					
						)					
					$\dashv$	)		(			
					-	)		) (			
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	Surrogate Compound		Surrogat	Surrogate Compound		Surrogate Compound		Surrogate Compound	punoamo		
∢	Chlorobenzene (CBZ)	ပ	Octa	Octacosane	Σ	Benzo(e)Pyrene	S	1-Chloro-3-Nitrobenzene	robenzene	>	Tetrachloro, m. cachoo
a	4-Bromofluorobenzene (BFB)	I	Ortho	Ortho-Terphenyl	z	Terphenyl-D14	-	3,4-Dinitrotoluene	luene	1	
U	a,a,a-Trifluorotoluene	-	Fluorobe	Fluorobenzene (FBZ)	0	Decachlorobiphenyl (DCB)	D	Tripentyltin	Fin		
9	Bromochlorobenene	1	p-Tri	n-Triacontane	a	1-methylnaphthalene	>	Trl-n-propyltin	vitin		
ш	1,4-Dichlorobutane	¥	Hex	Нехасоѕапе	0	Dichlorophenyl Acetic Acid (DCAA)	3	Tributy! Phosphate	sphate		
-	1.4-Difluorobenzene (DFB)		Brom	Bromobenzene	В	4-Nitrophenol	X	Triphenyl Phosphate	sohate		

LDC #: 18 386A7 SDG#: Lu count

25

METHOD:

N/A Y M N/A

V Z

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

Page: /of/

Reviewer:\_\_ 2nd Reviewer:

Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

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

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

)*	MS/MSD ID	Compound	MS %R (Limits)	ıits)	MSD %R (Limits)		RPD (Limits)	(\$	Associated Samples	Qualifications
H		Bange Organics	П	(ml-02)	)	$\overline{}$	e) OH	3O )	눼	m out
		o •	)	(	)	. (	)	•		m dsm
1			)		)		)	)		
			)	)	}	)	)	)		
			)	(	)	)	)	^		
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			)	<u> </u>	)	\(\cappa_1\)	)	)		
			)	(	)	^	)	(		
			)	(	)	(	)	(		
			)	(	)	)	)	(		
			)	(	)	^	<u> </u>	^		
			)	(	<u> </u>		J	î		
			)	( )	)	,	)	(		
			)	(	)	^	)	)		
			)	(	)	(	)	(		
			)	( )	`)	(	)	(		
				( )	)	)	)	)		
			)	(	<u> </u>			^		
			)	( )	)	(	)	(		
			)	( )	)	)	)	)		
			)	( )	)	(	)	(		
			)	( )	)	)	)	)		
							$\checkmark$	^		
				<u></u>	`	^	)	^		
			J	T ()	)	7	)	(		

LDC #: 18 3 8047 SDG #:

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer: Reviewer:

METHOD: GC\_

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

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

A = Area of compound,

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

	ated			T	T			T	T	T		T	=
	Recalculated	088%	8.667	2									
	Reported	%RSD	8.667										
, in land	Della maria	Average CF (Initial)	15699364										
Renorted		Average CF (initial)	156 op34										
Recalculated	ä	(1.0 std)								·			
Reported	<u>.</u>	(1.7 std)	K189023										
		Compound	garoun Hange Organica										
	Calibration	19/08	·										
	Standard ID	1991	1										
	#	-				7	T	6			4	T	_

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

LDC#: 18 35647 SDG#:

### Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

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

					Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc.	Q%	۵%
-	32	1/30/8	GRU	0.1	0.9486	0.04×	()	
T	28.22							
7	٥٥٧	9/04/1	<b>→</b>	0.1	929128	7 216.0	7 /	\ \'\>
1	14:57					0011	0	
е								
T								
$\parallel$								
4								
7		ı						
7		•						
								_

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

~~	HPLC
186A)	GC
7 3	, METHOD: ∠
LDC	MET

## VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

of Reviewer: Page:

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

% Recovery: SF/SS \* 100

Sample ID: 12 5

Where: SF = Surrogate Found SS = Surrogate Spiked

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
777	mily spe ton	Po.0	811600	56	15	1
	_					
	-					

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

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

LDC #: 18380A7

SDG #:

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

2nd Reviewer: Reviewer:\_

HPLC

METHOD:

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

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

Where

SC = Sample concentration

MS/MSD samples:\_\_

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

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

MSD = Matrix spike duplicate

		S	pike	Sample	Spike	Spike Sample	Matrix	Matrix spike	Matrix Spike	Dunlicate	Men	
Gasoline         (8015)         1-01/6         1-01/	Compound	¥ 34	70860	Conc.	Conce	ntration   X	Percent 6	Pecovery	0,000		OCE/CE	OS.
Gasoline (8015)         (8015)         1・0 p         1・0 p         0・1 ms p         MsD p         Reported p         Recalc.         Reported poore           Diesel (8015)         (8015)         1・0 p         0・1 ms p         1・1 p         1 p         1 p         1 p         1 p         1 p         1 p         1 p         1 p         1 p         1 p         1 p         1 p         1 p <th></th> <th>WS.</th> <th>USM</th> <th></th> <th>، ا</th> <th>11</th> <th></th> <th>(12.22)</th> <th>Leicelli</th> <th>Gecovery</th> <th>RPD</th> <th>0</th>		WS.	USM		، ا	11		(12.22)	Leicelli	Gecovery	RPD	0
Gasoline (8015)   1-06   1-0				` )I	M30	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Diesel (8015)		1.06	110	35.72	14. d	0.994		_	<del>ا</del> ت 0	3	Cri	C15,
Benzene         (8021B)         Methane         (RSK-175)         Comments: Refer to Matrix Spike Duplicates findings workshear for its of marings         Comments: Refer to Matrix Spike Duplicates findings workshear for its of marings         Comments: Refer to Matrix Spike Duplicates findings workshear for its of marings         Comments: Refer to Matrix Spike Duplicates findings workshear for its of marings         Comments: Refer to Matrix Spike Duplicates findings workshear for its of marings         Comments: Refer to Matrix Spike Duplicates findings workshear for its of marings         Comments: Refer to Matrix Spike Duplicates findings workshear for its of marings         Comments: Refer to Matrix Spike Duplicates findings workshear for its of marings         Comments: Refer to Matrix Spike Duplicates findings		<del>z</del>										2
Methane         (RSK-175)         Methane         (RSK-175)         Methane												
2,4-D         (8151) </td <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>												
Dinoseb         (8151)         Maphthalene         (8310)         Mathracene         (8310)         Mathracene         Mathracene         (8310)         Mathracene         <												
Naphthalene (8310)         Anthracene (8310)         (8330)         Anthracene (8330)         Ant												
Anthracene (8310)         HMX (8330)         Comments: Refer to Matrix Spike Puplicates findings worksheet for list of musifications and second an												
HMX         (8330)         Carrintrotoluene (8330)         Carrintrot												
2,4,6-Trinitrotoluene (8330)         ————————————————————————————————————												
Comments: Refer to Matrix Spike Duplicates findings worksheet for list of qualifications and sold and	2,4,6-Trinitrotoluene (8330)											
Comments: Refer to Matrix Spike Duplicates findings worksheet for list of qualifications and services.												
Comments: Refer to Matrix Spike Duplicates findings worksheet for list of qualifications and services.												
Comments: Refer to Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and account of the state of the s												
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Comments: Refer to Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and accounts.												
Action of the state of the stat	Comments: Refer to Matrix S	spike/Matrix	Spike Dup	licates finding	s worksheet 1	or list of qualif	ications and as	sociated sam	noles when rer	JL Northard regulite	do not some	o of righting

LDC# (8286 A7 SDG #: La coner

## VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

Reviewer:

METHOD:

OC HPLC

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

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

SSC = Spiked concentration SA = Spike added Where

SC = Sample concentration

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

8030151-16

LCS/LCSD samples:

LCS = Laboratory Control Sample percent recovery

LCSD = Laboratory Control Sample duplicate percent recovery

	Spike	ke	Sample	Spike Sample	sample	רנ	SOT	rcsp	מנ	rcs/rcsD	SD
Compound	( W.	X.	(my /kx)		rication Z	Percent !	Percent Recovery	Percent Recovery	ecovery	RPD	
	rcs	LCSD	<b>)</b>	SOT		Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)	1.0	γV	0	126.0	<b>₹</b> 2	46	76	- AM			
Diesel (8015)											
Benzene (8021B)											
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)											
Anthracene (8310)											
HMX (8330)											
2,4,6-Trinitrotoluene (8330)											
					-						

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

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rage:	Reviewer:	2nd Reviewer:

VALIDATION FINDINGS WORKSHEET Sample Calculation Verification	GC HPLC	Were all reported results recalculated and verified for all level IV samples? Were all recalculated results for detected target compounds within 10% of the reported results?
SDG #: Le cons	метнор:	Y N N/A
1 ()	_	ノーノー

Compound Name\_ Concentration ≂\_ Sample ID. 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

Example:

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

	Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications
Comments:			٠		

## 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'

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

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

DG #	: 18386B7 t: F8A290158 atory: <u>Test America</u>				Level III	ESS WOR			Date: 3 / 6 Page:
ETH	OD: GC Gasoline Ran	ge Org	anics (EPA	SW 846 N	Method 80	015)			Č
ne sa	amples listed below we	re revie	ewed for ead	ch of the fo	ollowing v	alidation are	as. Validatio	n finc	lings are noted in attache
	ion findings worksheet								
						All Control of the Co			
	Validation	n Area		A			Comm	ents	
l.	Technical holding times				Sampling	dates:	1/18/09		
lla.	Initial calibration			<u> </u>	<del> </del>	W. A. 15			
IIb.	Calibration verification/ICV	<u>'</u>			10	14 1			
111.	Blanks	•	<u> </u>	-5VV SW					
IVa.	Surrogate recovery	lumliaat -		7					
IVb.	Matrix spike/Matrix spike o		5	A	LC	·5	***		
IVc.	Laboratory control sample  Target compound identific			N					
V.	Compound Quantitation a			N					
VI.									
VIII.				Å					
IX.					p =	3+4			
X.					<del>                                     </del>	= 10			
	T Total Statistics		, , ,				:		
ote:	A = Acceptable N = Not provided/applicab SW = See worksheet	ole	R = Rin	o compound sate eld blank	ls detected	TB = T	uplicate Trip blank Equipment blar	nk	
alidate	ed Samples:	valu	7						
T				OME	104	80370	7x	31	-
	TSB-HJ-10-0'	11	TSB-HR-05-1		21	80371		32	
	TSB-HJ-10-10'	12	TSB-HR-05-1		22	80390		33	
	TSB-HR-06-0'	13	RINSATE-2M		23	00 0,0	-	34	
	TSB-HR-06-0'-FD	14	KINSA I E-ZIV	ISD W	25			35	
	TSB-HR-06-10' TSB-HJ-08-0'	15 16		<u> </u>	26			36	
	TSB-HJ-08-10'	17			27			37	
	TSB-HR-05-0'	18			28			38	
	TSB-HR-05-10'	19			29			39	
<del>'                                    </del>	RINSATE-2	20	<del> </del>		30			40	

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

LDC #: (8 386.87

# VALIDATION FINDINDS WORKSHEET

Surrogate Recovery

2nd Reviewer: 1

Page: Reviewer:

метнор: // Gc \_\_ нРLC Are surrogates required by the method? Yes \_\_\_ or No \_\_\_. Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Were surrogates spiked into all samples and blanks?

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

#	Sample ID	် မြ	Detector/ Column	Surrogate Compound		%R (Limits)	ts)		Qualifications	ıtions
	0 01	not :	Specifical	J		(53	- 97	( 05/	3+1Pat	
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	Surrogate Compound		Surroga	Surrogate Compound		Surrogate Compound		Surrogate Compound	punodu	
∢	Chlorobenzene (CBZ)	9	ő	Octacosane	Σ	Benzo(e)Pyrene	S	1-Chloro-3-Nitrobenzene	\ \	Tetrachloro-m- xylene
8	4-Bromofluorobenzene (BFB)	I	Out	Ortho-Terphenyl	z	Terphenyl-D14	⊢	3,4-Dinitrotoluene		
ပ	a,a,a-Trifluorotoluene	-	Fluoro	Fluorobenzene (FBZ)	0	Decachlorobiphenyl (DCB)	U	Tripentyltin	ltin	
4	Bromochlorobenene	1	a	n-Triacontane	۵	1-methvinaphthalene	>	Tri-n-propyltin	vltin	
w	1,4-Dichlorobutane	×	ř	Hexacosane	σ	Dichlorophenyl Acetic Acid (DCAA)	۸ ۷	Tributyl Phosphate	sphate	
ш	1.4-Diffuorobenzene (DFB)	1	, Bro	Bromobenzene	В	4-Nitrophenol	×	Triphenyl Phosphate	sohate	

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

Diesel Range Organics



## 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:

Diesel 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-HR-08-0'MS

TSB-HR-08-0'MSD

<sup>\*\*</sup>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)	Α

### 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)	А	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

SDG#	: 18386A8 : F8A250221 atory: Test America	<b>V</b> A _ _	LIDATIO		PLETEN evel III/I\	ESS WORKSHE /	ET	Date: 3/7 Page: of / Reviewer: 2nd Reviewer:
METH	<b>OD:</b> GC Diesel Range (	Orgar	nics (EPA S\	N 846 <b>M</b> e	thod 8015	)		
	amples listed below were ion findings worksheets		ewed for ea	ch of the f	following v	alidation areas. Valid	lation findi	ngs are noted in attached
	Validation	Area				Co	mments	
I.	Technical holding times			A	Sampling o	lates: 12408		
IIa.	Initial calibration			٨				
IIb.	Calibration verification/ICV			Δ	1CV =	= 15		
III.	Blanks			٨	5			
IVa.	Surrogate recovery			المري				
IVb.	Matrix spike/Matrix spike du	plicate	es	Δ				
IVc.	Laboratory control samples			A	LCS			
V.	Target compound identification				Not review	ed for Level III validation.		
VI.	Compound Quantitation and	CRQ	Ls		Not review	ed for Level III validation		
VII.	System Performance			4	Not review	ed for Level III validation.		
VIII.	Overall assessment of data			<u> </u>	_			
IX.	Field duplicates			NP	P =	11+12	<del></del>	
X	Field blanks			N				
Note:	A = Acceptable N = Not provided/applicable SW = See worksheet		R = Rin FB = Fi	eld blank		D = Duplicate TB = Trip blank EB = Equipment	blank	
Validate	ed Samples: ** Indicates sam	ple un	derwent Level	IV validation			·	No.
1	TSB-HJ-05-10'	11	TSB-HJ-07-0	** 0	21	8029395	31	
2	TSB-HJ-05-0'	12	TSB-HJ-07-0	-FD F	22		32	
3	TSB-HR-04-10'	13	TSB-HJ-07-1	0'	23		33	
3 # 4	TSB-HJ-04-0'	14	TSB-HR-08-0	ı	24		34	
	TOD LID OA O!**	15	TCD UD 00 1	01	25		35	

<b>1</b> 0	TSB-HR-06-10' ✓	20	30	40
Note	s:			

TSB-HR-08-0'MS

TSB-HR-08-0'MSD

TSB-HJ-04-10'

TSB-HR-07-0' TSB-HR-07-10'\*\*

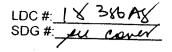
TSB-HR-06-0'

LDC #: 18386 18 SDG #: you come

### **VALIDATION FINDINGS CHECKLIST**

	,		
Method:		_GC _	HPLC

Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was 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  U. Saffiglies Spiks  Were all surrogate %R within the QC limits?  If any %R was less than 10 percent, was a reanalysis performed to confirm %R?  If any %R was less than 10 percent, was a reanalysis performed to confirm %R?  Was a MSMSD partyzed every 20 samples of each matrix?  Were the MSMSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits ?  Was an LCS analyzed for this SDG?  Was an LCS analyzed for this SDG?  Was an LCS analyzed for this SDG?	Method: / GC HPLC				
All technical holding times were met.  Cooler temperature criteria was met.  Liss all sharpers of the state o	Validation Area	Yes	No	NA	Findings/Comments
Cooler temperature criteria was met.  ### ### ### ### ### ### ### ### ### #	I. Technical holding times				
Links Labruration.  Did the laboratory perform a 5 point calibration prior to sample analysis?  Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?  Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?  Did the initial calibration meet the curve fit acceptance criteria?  Where the RT windows properly established?  What type of continuing calibration calculation was performed?%D or%R  Was a continuing calibration analyzed daily?  Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?  Were all the retention times within the acceptance windows?  Zhains  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.  Xi siming as spike.  Were all surrogate %R within the QC limits?  If any %R was less than 10 percent, was a reanalysis performed to confirm %R?  If any %R was less than 10 percent, was a reanalysis performed to confirm %R?  If any %R was less than 10 percent, was a reanalysis performed to confirm %R?  Were the MS/MSD percent recovere (%R) and the relative percent differences (%R) and the relative percent differences (%R) 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?  Was an LCS analyzed for this SDG?	All technical holding times were met.	-	1		
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 (KRSD) < 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?  What type of continuing calibration salculation was performed?  What type of continuing calibration analyzed daily?  What a continuing calibration analyzed daily?  Were all the retention times within the acceptance windows?  Was a method blank associated with every sample in this SDG?  Was a method blank analyzed for each matrix and concentration?  Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.  Wishtrigad spice.  Were all the recovery (KR) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm KR?  If any KR was less than 10 percent, was a reanalysis performed to confirm KR?  If any KR was less than 10 percent, was a reanalysis performed to confirm KR?  Was a MSMSD analyzed every 20 samples of each matrix?  Was a MSMSD analyzed every 20 samples of each matrix?  Were link MSMSD percent recoveries (KR) and the relative percent differences (KR) with the QC limits?  Were the MSMSD percent recoveries (KR) and the relative percent differences (KR) with the QC limits?  Were the MSMSD percent recoveries (KR) and the relative percent differences (KR) with the QC limits?	Cooler temperature criteria was met.				:
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?  Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?  Did the initial calibration meet the curve fit acceptance criteria?  Were the RT windows properly established?  If y Socitifuing zalibration  What type of continuing calibration calculation was performed? — %D or %R  Was a continuing calibration analyzed daily?  Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?  Were all the retention times within the acceptance windows?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  If any sample	II. Initial calibration:				
Was a continuing calibration analyzed daily?  Were the RT windows properly established?  Whas a continuing calibration analyzed daily?  Were all the retention times within the acceptance windows?  Was a method blank associated with every sample in this SDG?  Was a method blank associated with every sample in this SDG?  Was a method blank analyzed for each matrix and concentration?  Was there contamination in the method blanks? If yes, please see the Blanks  Was a free only spike (MS) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?  If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?  If any %R was less than 10 percent, was a reanalysis performed to confirm %R?  If any %R was less than 10 percent, was a reanalysis performed to confirm %R?  Were all surrogate %R within the QC limits yield explicates  Were a matrix spike (MS) and matrix spike duplicates  Was a MS/MSD analyzed every 20 samples of each matrix?  Was a MS/MSD analyzed every 20 samples of each matrix?  Was a MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	Did the laboratory perform a 5 point calibration prior to sample analysis?	/			
Did the initial calibration meet the curve fit acceptance criteria?  Were the RT windows properly established?  What type of continuing calibration calculation was performed? — %D or %R  Was a continuing calibration analyzed daily?  Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?  Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?  Were all the retention times within the acceptance windows?  W. Blanks  Was a method blank associated with every sample in this SDG?  Was a method blank analyzed for each matrix and concentration?  Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.  V. Sirrogale Spices  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?  VI. Matrix spice Matrix spice diplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated  MSMSD. 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 a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) ≤ 20%?	-	<u> </u>		
Were the RT windows properly established?  What type of continuing calibration calculation was performed?	Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?				
What type of continuing calibration calculation was performed?	Did the initial calibration meet the curve fit acceptance criteria?		·		
What type of continuing calibration calculation was performed?%D or	Were the RT windows properly established?				
Was a continuing calibration analyzed daily?  Were all percent differences (%D) ≤ 15% 0 or percent recoveries 85-115%?  Were all the retention times within the acceptance windows?  V.Blanks  Was a method blank associated with every sample in this SDG?  Was a method blank analyzed for each matrix and concentration?  Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.  VI. Surrogate spikes)  Were all surrogate %R within the QC limits?  If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?  If any %R was less than 10 percent, was a reanalysis performed to confirm %R?  VII. Matrix spikerMatrix 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?  Were the MS/MSD percent recoveries (%R) and the relative percent differences  VIII. Lationatory control samples  Was an LCS analyzed for this SDG?	IV: Continuing calibration			4.9	
Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?  Were all the retention times within the acceptance windows?  Withanks:  Was a method blank associated with every sample in this SDG?  Was a method blank analyzed for each matrix and concentration?  Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.  With 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?  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?  Will Laboratory control samples  Was an LCS analyzed for this SDG?	What type of continuing calibration calculation was performed?%D or%R				
Were all the retention times within the acceptance windows?  W:Blanks:  Was a method blank associated with every sample in this SDG?  Was a method blank analyzed for each matrix and concentration?  Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.  W: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. Maliox spike/Matrix spike duplicates  Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.  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?  Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	Was a continuing calibration analyzed daily?	/			
Was a method blank associated with every sample in this SDG?  Was a method blank analyzed for each matrix and concentration?  Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.  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. Maliox spike/Matrix spike duplicates  Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated  MS/MSD, Soil / Water.  Was a MS/MSD paralyzed 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?	Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?	/			
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Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?  Will: Laboratory control samples  Was an LCS analyzed for this SDG?	matrix in this SDG? If no, indicate which matrix does not have an associated				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?  Will: Laboratory control samples  Was an LCS analyzed for this SDG?	Was a MS/MSD analyzed every 20 samples of each matrix?	才	$\neg \dagger$	$\top$	
Was an LCS analyzed for this SDG?	Were the MS/MSD percent recoveries (%R) and the relative percent differences	7	-		
Was an LCS analyzed per extraction batch?	Was an LCS analyzed for this SDG?	7			
		7		丁	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	1			



### **VALIDATION FINDINGS CHECKLIST**

Page: 20f 2
Reviewer: P7
2nd Reviewer: 1

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?			-	ATTENDED TO THE PROPERTY OF TH
XI Compound quantitation/CRQLs				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII. System performance				
System performance was found to be acceptable.				
UF Overall essessment of data				
Overall assessment of data was found to be acceptable.				
RIV. Field duplicates				
ield duplicate pairs were identified in this SDG.		-	# 0 1975 	
arget compounds were detected in the field duplicates.		7	_	
V. Faeld blanks				
ield blanks were identified in this SDG.		Ť	Ī	
arget compounds were detected in the field blanks.			$\exists$	

SDG #: 1838918 SDG #: \*\*

# VALIDATION FINDINDS WORKSHEET

Surrogate Recovery

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

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

METHOD:

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

Were surrogates spiked into all samples and blanks?

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

	T	T	T	T	T	Τ	T	T	Π	Γ	Ī	Ī	Γ	T	T		Ī	T	T	Ī			ſ	T	Γ			
Ouslifications	W//\		7																			P	e Y Tetrachloro-m-xylene					
	<u> </u>																					Surrogate Compound	1-Chloro-3-Nitrobenzene	3,4-Dinitrotoluene	Tripentyltin	Tri-n-propyltin	Tributyl Phosphate	Triphenyl Phosphate
	13-150								(	(	(	(	(		^	(	)	)	(	(	(	Surrogate	1-Chloro-3	3,4-Dinit	Tripe	Td-n-c	Tributyl F	Triphenyl
	7.5																	2	/				S	1	כ	>	3	×
%R (Limits)	651		9	)			)	)	)	`	<u> </u>			)	)	<u> </u>		)	section may	`)	)	Surrogate Compound	Benzo(e)Pyrene	Terphenyl-D14	Decachlorobiphenyl (DCB)	1-methylnaohthalene	Dichlorophenyl Acetic Acid (DCAA)	4-Nitrophenol
									•										9			S	Σ	z	0	a	ODichk	В
Surrogate Compound	I		#																スコンター	•		Surrogate Compound	Octacosane	Ortho-Terphenyl	Fluorobenzene (FBZ)			Bromobenzene
) L	Proits	P																	K			Surroga	Oct	Orth	Fluorot	T-u	윈	Bror
Detector/ Column	14	ı	<b>-</b>																1				U	I	1	1	¥	1
	hou								-		1					1	-		7			puno	382)	ie (BFB)	iene	aua	ne	(DFB)
sample ID	<u>C</u> 1		<u>\S</u>																0			Surrogate Compound	Chlorobenzene (CBZ)	4-Bromofluorobenzene (BFB)	a,a,a-Trifluorotoluene	Bromochlorobenene	1,4-Dichlorobutane	1.4-Difluorobenzene (DFB)
) #																							∢	В	U	۵	w	<u>.</u>

1838BRS SDG#: LDC#:

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer: Reviewer:

> FIC 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

ſ	7		_	<u></u>	7		T	7	 T	=		7		 <del>_</del>	-	r	_	_	-
	Recalculated		%RSD	10 10 01	22.2														
	Reported	400	WKSU	()611 ()1	27.7														
	Recalculated	Average CF	, minday	17265															1
L	Reported	Average CF (initial)		17265															
	- re-acmared	CF (SP (Ztd)		16699															
Renorted		(Sector)		16699															
		Compound	Sur Sur Sur Sur Sur Sur Sur Sur Sur Sur										から きょうこう 直接						
	Calibration	Date	20/1 4/1																
		Standard ID	チアナ		-	-						<u> </u>					-		
		#	-					7			۳.		T	-	4		T	<del></del> ,-	

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

LDC #: 18 380 RS SDG#: Les cors

# Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer: Reviewer:

METHOD: GC\_

HPLC

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

% 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

					Reported	Recalculated	Reported	r sycling cool
#	Standard ID	Calibration Date	Compound	Average CF(Ical)/ CCV Conc.	CF/Conc.	CF/Conc.	q%	Q%
-	ECALSOS	70/12/	فسنك	1000.00	871.9651	27 9/F	Ş	7.55
						201		173
	819	2010/2	*	->	980.1286	25	3.5	
7	2%.7	20/10/2	~	(200)	MOGE, STLX	1001 CTLX	0.7	2 7 0
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Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the

LDC #: 18386 PKS

# VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page: of Reviewer:

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Sample ID:

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
0 - terphony	bestings ton	26	19.327	77	77	С
7	-					
		The second secon	A STATE OF THE PARTY OF THE PAR			

Sample ID:

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

ample ID:

Surrogate Column/Detector Spiked Found Recovery Recovery Difficent Percond Recovery Difficent Difficent Recovery Difficulty Difficul							
Reported	Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
					Reported	Recalculated	
			•				

LDC #: 18 385 AS

SDG#:7

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

Page: Lof Z Reviewer:

2nd Reviewer:

HPLC

METHOD:

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

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

SC = Sample concentration

RPD =(({SSCMS - SSCMSD} \* 2) / (SSCMS + SSCMSD))\*100 16 4 17 MS/MSD samples:\_

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

MSD = Matrix spike duplicate

Č		S PA	Spike Addęd	Sample Conç.	Spike	Spike Sample Concentration	Matrix	Matrix spike	Matrix Spik	Matrix Spike Duplicate	MS/MSD	ASD
Comp	Compound	38	1 24	1 was 24	km)	Kg)	Percent	Percent Recovery	Percent F	Percent Recovery	RPD	c.
		MS	MSD	) i	SW	OSW	Reported	Recalc	Benorted	Bessle		R
Gasoline	(8015)								Periodo	Necarc.	керопеа	Recaic.
Diesel	(8015)	1.98	2.78	0	7.5	L	9	0	-		2	9
Benzene	(8021B)				Q .	2.48		2	34	7	9.0	i ê
Methane	(RSK-175)	3.5	42.0									
2,4-D	(8151)	gi w		A Company								
Dinoseb	(8151)	1.60 1.00 1.00	1.1 15.1 18.0									
Naphthalene	(8310)											
Anthracene	(8310)	2 50										
НМХ	(8330)											
2,4,6-Trinitrotoluene (8330)	oluene (8330)											
omments: Ref	Comments: Refer to Matrix Spike/Matrix Spike Duplicates findings worksheet for list of analytications of the second secon	ce/Matrix	Spike Dupli	icates finding	s workshoot f	or liet of our life						
of the recalculated results.	ted results.			5	20110011001	or list of dualif	ications and a	ssociated san	ples when reg	ported results	do not agree	within 10.0

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SDG #: 10 coner

VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

Reviewer:

| GC | METHOD:

HPLC

compounds identified below using the following calculation:

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

SSC = Spiked concentration SA = Spike added Where

SC = Sample concentration

The percent recoveries (%R) and relative percent differences (RPD) of the laboratory control sample duplicate were recalculated for the

LCS = Laboratory Control Sample percent recovery

LCSD = Laboratory Control Sample duplicate percent recovery

RPD =(((SSCLCS - SSCLCSD) \* 2) / (SSCLCS + SSCLCSD))\*100 3029395-108 LCS/LCSD samples:

Recalc. LCS/LCSD Reported Recalc. Percent Recovery CSD Reported 42 Recalc. 7 Percent Recovery LCS Reported 4 となっ Concentration Spike Sample いらし rcs Cong. Sample Q CSD 42 Spike Added 3 rcs 89 2,4,6-Trinitrotoluene (8330) (RSK-175) (8021B) (8330)(8310) (8015) (8015) (8151) (8310) (8151) Compound Naphthalene Anthracene Gasoline Benzene Methane Dinoseb Diesel 2,4-D HMX

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

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LDC i	SDG

# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page: 2nd Reviewer: Reviewer:

HPLC
GC
//
10D:

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

)		
Soncentration≈	(A)(Evinal)	

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

Compound Name

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

Concentration =

#	Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications
Comments.	nts.				

## 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:

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'

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'RE

**RINSATE-2** 

TSB-HJ-10-0'MS

TSB-HJ-10-0'MSD

TSB-HR-05-10'MS

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)	А	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

SDG#	: 18386B8 : F8A290158 atory: Test America	<b>VA</b> - 	LIDATIOI		<b>PLET</b> _evel		ESS W	ORKSI	HEET		Date: 3/6/0 Page: 1 of / Reviewer: 7 2nd Reviewer:
Γhe sa	<b>OD:</b> GC Diesel Range Camples listed below were tion findings worksheets.	revie	•					areas. V	'alidatio	n find	lings are noted in attached
	Validation	Area							Comm	ents	
I.	Technical holding times			۵	Samp	ling da	ates:	1280	3		
lla.	Initial calibration			Δ				, ,			
IIb.	Calibration verification/ICV			Δ	LCV	_	15				
III.	Blanks			A							
IVa.	Surrogate recovery			SW							
IVb.	Matrix spike/Matrix spike dup	olicate	S	Δ			, , , , , , , , , , , , , , , , , , , ,				
IVc.	Laboratory control samples			A	1	ಡಿ	IP				
V.	Target compound identificati	on		N							
VI.	Compound Quantitation and	CRQ	_S	N							
VII.	System Performance			N				·			
VIII.	Overall assessment of data										
IX.	Field duplicates	.,,		ND	2	= '	3,4				
X.	Field blanks			MN	R	, = '	11				
	A = Acceptable N = Not provided/applicable SW = See worksheet ed Samples:		R = Rin: FB = Fie	o compounds sate eld blank	s detec	ted	TI	= Duplicat B = Trip bla B = Equipn	ank	Κ	
1	TSB-HJ-10-0'	112	RINSATE-2	W		21	803	1303		31	
	TSB-HJ-10-10'	12	TSB-HJ-10-0'	MS		22 Z		5123		32	
	TSB-HR-06-0' ()	13	TSB-HJ-10-0'			23 ኝ	802	2980	39219	33	
	TSB-HR-06-0'-FD /	14 1	TSB-HR-05-1	0'MS		24				34	
5	TSB-HR-06-10'	15 <sup>)</sup>	TSB-HR-05-1	0'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	
9 1	TSB-HR-05-10'	19				29				39	

30

10 TSB-HR-05-10'RE

Notes:\_

20

RDC #: 1838688 SDG #: 15 Com

## VALIDATION FINDINDS WORKSHEET Surrogate Recovery

2nd Reviewer:

Page: Reviewer:

METHOD: \_\_\_GC\_\_\_HPLC Are surrogates required by the method? Yes\_\_\_\_ or No\_\_

Progression of the state of the

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

Qualifications d/(n/-1 %R (Limits) Surrogate Compound Say it Detector/ Column 2 8039219-Blank Sample ID σ

				-	)				
	Surrogate Compound		Surrogate Compound		Surrogate Compound		Surrogate Compound		
A	Chlorobenzene (CBZ)	ပ	Octacosane	Σ	Benzo(e)Pyrene	S	1-Chloro-3-Nitrobenzene	<b>&gt;</b>	Tetrachloro-m- xylene
В	4-Bromofluorobenzene (BFB)	I	Ortho-Terphenyl	z	Terphenyl-D14	⊦	3,4-Dinitrotoluene		
U	a,a,a-Trifluorotoluene	_	Fluorobenzene (FBZ)	0	Decachlorobiphenyl (DCB)	٦	Tripentyltin		
٥	Bromochlorobenene	ſ	n-Triacontane	ο.	1-methylnaohthalene	>	Tologophia		
П	1,4-Dichlorobutane	¥	Нехасозапе	ø	Dichlorophenyl Acetic Acid (DCAA)	3	Tributyl Phosphate		
Ч	1.4-Difluorobenzene (DFB)	_	, Bromobenzene	α	lonahaon!k-4	×	Triphenyl Phosobate		

### 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

<sup>\*\*</sup>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)	222	p://		
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	21 TSB-HR-06-0' 2,3,7,8-TCDF (from DB-225)		None	P	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	Α
F8A250221	TSB-HR-04-10'	OCDD OCDF	11U pg/g 11U pg/g	А
F8A250221	TSB-HJ-04-0'	OCDD	11U pg/g	А
F8A250221	TSB-HR-04-0'**	OCDD	10U pg/g	Α
F8A250221	TSB-HJ-04-10'	OCDD	11U pg/g	А
F8A250221	TSB-HR-07-0'	OCDD	11U pg/g	А
F8A250221	TSB-HR-07-10'**	OCDD	11U pg/g	А
F8A250221	TSB-HR-06-10'	OCDD	11U pg/g	А
F8A250221	TSB-HJ-07-0'**	OCDD OCDF	11U pg/g 11U pg/g	А
F8A250221	TSB-HJ-07-0'-FD	OCDD	11U pg/g	Α
F8A250221	TSB-HJ-07-10'	1,2,3,7,8-PeCDD OCDD	5.4U pg/g 11U pg/g	Α
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	Α

### BRC Tronox Parcel H Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG F8A250221

No Sample Data Qualified in this SDG

### **VALIDATION COMPLETENESS WORKSHEET** LDC #: 18386A21 SDG #: F8A250221

Level III/IV

Date:	3/11/08
Page:_	<u>/_of</u>
Reviewer:	<u> </u>
2nd Reviewer:	_/0_

Laboratory: Test America

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: \  24 \ 0 B
II.	GC/MS Instrument performance check	Δ	' '
111.	Initial calibration	Δ	
IV.	Routine calibration	Δ	
V.	Blanks	سی	
VI.	Matrix spike/Matrix spike duplicates	A	
VII.	Laboratory control samples	Α	LCS
VIII.	Regional quality assurance and quality control	<sup>n</sup> N	
IX.	Internal standards	A	
Χ.	Target compound identifications	Δ	Not reviewed for Level III validation.
XI.	Compound quantitation and CRQLs	ŞW	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	SW	D= 11+12
XV.	Field blanks	LV	

Note:

A = Acceptable

SW = See worksheet

ND = No compounds detected N = Not provided/applicable

R = Rinsate

FB = Field blank

D = Duplicate TB = Trip blank

EB = Equipment blank

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

	SOLL						
1 1	TSB-HJ-05-10'**	113	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'	143	TSB-HR-08-0'	24		34	
51	TSB-HR-04-0'**	15 15	TSB-HR-08-10'	25		35	
6	TSB-HJ-04-10'	16	TSB-HR-06-0'MS	26		36	
7	TSB-HR-07-0'	17	TSB-HR-06-0'MSD	27		37	
\$7 8	TSB-HR-07-10'**	18		28		38	
97	TSB-HR-06-0'	19		29		39	
10	TSB-HR-06-10'	20		30		40	

Notes:			
			- 11. 1190 W

### LDC #: \\\ 386 AZ\\ SDG #: \text{fie cover}

### **VALIDATION FINDINGS CHECKLIST**

Page: /_of	<u>3</u>
Reviewer:	
2nd Reviewer:	

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

Validation Area	Yes	No	NA	Findings/Comments
Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.				
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?				
Were the retention time windows established for all homologues?	/			
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers $\leq$ 25% ?				
Is the static resolving power at least 10,000 (10% valley definition)?				
Was the mass resolution adequately check with PFK?	/			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?				
III. Initial calibration	<u></u>			
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	<b>,</b>			
Was a routine calibration performed at the beginning and end of each 12 hour period?				
Were all percent differences (%D) $\leq$ 20% for unlabeled standards and $\leq$ 30% for labeled standards?				
Did all routine calibration standards meet the Ion Abundance Ratio criteria?				
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				
Was an LCS analyzed for this SDG?		<u> </u>		

LDC #: [8386A2] SDG #: pre cover

### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 3
Reviewer: 5
2nd Reviewer:

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

LDC #: 18386A2/ SDG #: fre coner

### VALIDATION FINDINGS CHECKLIST

	Page:	<u>3</u> 0	3
	Reviewer:		2
2nd	Reviewer:	('	

Validation Area	Yes	No	NA	Findings/Comments
Target compounds were detected in the field duplicates.	-			
XV. Field blanks			1	
Field blanks were identified in this SDG.		_		
Target compounds were detected in the field blanks.			1	

### 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	8 OCD			
		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	1. 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	0. 1,2,3,4,6,7,8-HpCDF	T. Total HxCDD	Y Total Harone

Notes:

LDC#: [\(\frac{3\colon}{3\colon}\) SDG#: \(\frac{1\colon}{3\colon}\)

### VALIDATION FINDINGS WORKSHEET RIANKS

Page: Cof Reviewer: 7 2nd Reviewer: 4

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 all samples associated with a method blank?

Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y N N/A Was the method blank contaminated?

Y Z Z

Blank analysis date: 2/20/08 Blank extraction date: 21208

Associated samples: A

ſ						r	1	1	1	1	<u> </u>						-	1	_
		6	1	(5.0)															
		<b>2</b> 00	1	0. 34/114	~_1														
		7	,	0.21/11M							15	1	MIN PE.0	, ~					
	ion	٩	i								14	nt·5/ el·0	0.22/114	,					
	Sample Identification	ار/	-	0.24/114  0.30/114  0.19/104  0.20/114							13	0, OTI/5.4V	n11/22.0   n11/82.0   n11/08.0	1					
	Sa	4	1	0.30/114	, =						71	aog 1/5.44	0.30/114	1					
+		3	j	0. 24/11M	0.17 /11M						11		n11/00io	0.21/1M	1				
		7	1	(p.4)	(91)	,					10	-	0.26/11 W	_					
				0.30/11M	0.12 /11M		:												
<b>)</b>	Blank ID	8043106 - Blank	0.098		0.15					•	8043106 31ank	9:00	0.33	0.15		:			
Conc. units: og a			В	5	9							8	þ	Ø					

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".

LDC #: 18380 AV SDG#:\_\_

### Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

Page: Lof / 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".

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). Y N N/A

			 	<del></del> i	 	 	 	<del>-</del> T	<del>- 1</del>	Ť	Т	_	ĺ
Qualifications	Word /												
Associated Samples	4												
Finding	NO DB-725 confirmation	1 1	100										
-Sample ID													
Date													
#													

Comments: See sample calculation verification worksheet for recalculations

LDC #: 18386A21 SDG #: 412 Coned

### VALIDATION FINDINGS WORKSHEET Field Duplicates

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

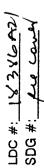
Ŷ	N N/A	Were field duplicate pairs identified in this SDG.
Y	N N/A	Were target compounds detected in the field duplicate pairs?

	Concentra	tion ( pg /g)	Diffirence
Compound	11	10,12	RPD
G	0.80	0.30	0.50 (- 5,4)
N	0.070	0.14	0.07 (451) (5.4)
Ð	0.064	5.34	5.24 (5.4)
Q	0.2	NN.	10.79 (± 11)

	Concentration (	
Compound		RPD

	Concentration (	
Compound		RPD

	Concentration (	
Compound		RPD



## VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

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

 $A_x$  = Area of compound,  $A_k$  = Area of associated internal standard  $C_x^*$  = Concentration of compound,  $C_k$  = Concentration of internal standard S = Standard deviation of the RRFs, X = Mean of the RRFs

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Average RRF (initial)	RRF (CS3 std)	RRF (453 std)	%RSD	%RSD
-	104)	80/s1/z	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.045	1.0(5	0.040	0,01	٥٠٥	9.6
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	H8540.1	1:081	1.075	1.075	1:0	0.0
_ ]			1,2,3,6,7,8-HXCDD (13C-1,2,3,6,7,8-HXCDD)	1.053	1.053	1.070	040.)	4.0	7.0
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	216.0	2776	1710	176-0	÷.	24
			ocpf ("c-ocpb)	1.102	1-102	1.118	1.118	8.0	8.1)
2	1CA ].	20/s1/e	2,3,7,8-TCDF (1°C-2,3,7,8-TCDF)	+119	1.179	1. 205	1.105	Q.4	4.0
	08-225		-23,7,81CDD (19C-2,3,7,8-TCBD)	1.081	1.08)	1.125	12	6.7	6.7
			1,2,3,6,7,8-HxCDD (**C-1,2,3,6,7,8-HxCDD)					9	
			1,2,3,4,6,7,6-HpCDD (13C-1,2,4,6,7,8,-HpCDD)						
			o <del>cer ("e.eca</del> a)						
ဗ			2,3,7,8-TCDF (13C-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 (13C-1,2,3,6,7,8-HxCDD)						
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)						
			OCDF (13C-OCDD)						

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

LDC #:_	SDG#.

## VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

	2	-
Page:	Reviewer:	and Reviewer:
		2nd

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 =  $(A_x)(C_g)/(A_g)(C_x)$ 

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

A<sub>x</sub> = Area of compound, C<sub>x</sub> = Concentration of compound,

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

					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q%	Q%
	55220803	2/xlog	2,3,7,8-TCDF (¹³C-2,3,7,8-TCDF)	1.125	1.044	1,044	7.7	7-4
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)					
			OCDF (13C-OCDD)					
2			2,3,7,8-TCDF (¹³C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD (¹³C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)					
			OCDF (¹3C-OCDD)					
٣.			2.3.7.8-TCDF (13C-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)					

Comments: Refer to Routine Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results. LDC# 143 56A2/ SDG# 446 const

## VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

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 =  $100 * (ave. RRF - RRF)/ave. RRF RRF = (A_{\lambda})(C_{ls})/(A_{ls})(C_{\lambda})$ 

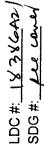
Where: ave. RRF = initial calibration average RRF RRF = continuing calibration RRF

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

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

		,			Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Ф.	Q%
-	\ 0	80/01/2	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.045	184	0.984	5.0	65
	408011084		2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.084	1.021	1.0al	5.9	5.9
1		70.7	1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.053	1.001	1.001	4.9	6;4
		r	1,2,3,4,6,7,8-HpCDD (¹3C-1,2,4,6,7,8,-HpCDD)	216,0	0801	0,00,1	5.5	S.
			OCDE (13C-OCDD)	1.102	1.2.76	1-276	15.8	8.51
2	(Page 1)	2/20/08	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)		0.996	966.0	4.7	4.7
	AOSU 22/52	23:53	2,3,7,8-TCDD (¹³C-2,3,7,8-TCDD)		1.035	1.035	4-5	4.5
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)		1.033	1.023	7.7	2.8
			1,2,3,4,6,7,8-HpCDD (¹3C-1,2,4,6,7,8,-HpCDD)		1.021	1.62	4.5	4-5
			OCDE (¹3C-OCDD)	7	1-17-1	1-17-1	6.3	6.3
3	٥٤٧	20/20/2	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	•	6860	6-98-0	ς.ψ	6.4
	MOSOURS	9:19	2,3,7,8-TCDD (¹³C-2,3,7,8-TCDD)		1.055	1-055	2.7	7.7
		•	1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)		1.019	1.019	3.7	3.5
		T	1,2,3,4,6,7,8-HpCDD (¹3C-1,2,4,6,7,8,-HpCDD)		1.03	1.032	5.7	5.7
		I	OCDF (13C-OCDD)		0.130	0.41.1	6,1	<u>`</u>

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 Comments:



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



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

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

% Recovery = 100 \* (SSR - SR)/SA

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

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

MS/MSD samples:

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

Concentration (pg/g)
MSD WSD MS
Livy ND 4.72
211 AN 111
XII ON III
[1] O.45  11
227 2.8 xB

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

LDC #: 18386 A2/ SDG #: ALR CONT

# 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 = | LCS - LCSD | \* 2/(LCS + LCSD)

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 8043106 - 103

	r	T-	1	Τ=	T	T	<del>-</del>	<del></del>	<del></del>	<del></del>	T	1	 <del>T</del>
csn	Qı	Pote live leaded											
I CS/I CSD	RPD	todag											
g	ecovery	Pacalc											
ICSD	Percent Recovery	Reported					\$ 2						
8	Recovery	Recalc	001	70]	hal	901	3						
SUL	Percent Recovery	Reported	0 01	501	hol	) ol	120						
ample	ration o	()	٩٧	_			>						
Spiked S	Concentration	OÎ LES	70.)	Sol	† 01	اه ل	ahr						
ike	ded [a_)	, U I CSD	VA	1									
ds .	Added (pg/pg/	01	20.02	100	<b>\</b>	•	2002						
	Compound		2,3,7,8-TCDD	1,2,3,7,8-PeCDD	1,2,3,4,7,8-HxCDD	1,2,3,4,7,8,9-HpCDF	OCDF						-

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

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SDG #:_	μ	could

### VALIDATION FINDINGS WORKSHEET

Sample Calculation Verification

Page:_	1 of
Reviewer:	15
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METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

Υ/	N	N/A
Y	N	N/A

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

Concentr	ation -	$(A_{is})(RRF)(V_o)(\%S)$
A <sub>x</sub>	=	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)
$V_o$	=	Volume or weight of sample extract in milliliters (ml) o grams (g).
RRF	=	Relative Response Factor (average) from the initial calibration
Df	=	Dilution Factor.
%S	=	Percent solids, applicable to soil and solid matrices

Example:		
Sample I.D. #2	<u> </u>	:
Conc. = ( <u>52145</u> )( 1739867)(0.6	2000 )( 176 )( 10	)(o.953
= 6.44 P	818	

	only.					
#	Sample ID	Compound		Reported Concentration ( )	Calculated Concentration ( )	Qualification
		#2 H conjirn	nation			
		= 8462 (100	20)			
	····	= 8462 (100 388684 /1.	0817/	10) (0.953)		
		7 00 00 7 (1.	001 /(	(1) (11)		
			19/			
		= 2.1 pg	17			
	-					
	1.44.4.4					
ļ						
-						

LDC #:_	
SDG #:	

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification (additional page)

Page:_	of
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# lons Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

		<del></del>	
Analyte	HPCDF HPCDF HPCDF HPCDD HPCDD HPCDD (S) NCDPE PFK	ocor ocor ocor ocor ocor ocor bcope PFK	
Elemental Composition	C <sub>12</sub> H <sup>26</sup> C <sub>11</sub> <sup>27</sup> ClO C <sub>12</sub> H <sup>26</sup> C <sub>11</sub> <sup>27</sup> ClO 13 C <sub>12</sub> H <sup>26</sup> C <sub>11</sub> O 13 C <sub>12</sub> H <sup>26</sup> C <sub>11</sub> <sup>27</sup> ClO C <sub>12</sub> H <sup>26</sup> C <sub>11</sub> <sup>27</sup> ClO C <sub>12</sub> H <sup>26</sup> C <sub>11</sub> <sup>27</sup> ClO 13 C <sub>12</sub> H <sup>26</sup> C <sub>11</sub> <sup>27</sup> ClO 13 C <sub>12</sub> H <sup>26</sup> C <sub>11</sub> <sup>27</sup> ClO C <sub>12</sub> H <sup>26</sup> C <sub>11</sub> <sup>27</sup> ClO C <sub>12</sub> H <sup>26</sup> C <sub>11</sub> <sup>27</sup> ClO C <sub>2</sub> F <sub>1</sub> <sup>27</sup> ClO	C, 26Cl, 37ClO C, 26Cl, 37ClO C, 26Cl, 37ClO C, 26Cl, 37ClO 13C, 26Cl, 37ClO 13C, 26Cl, 37ClO 13C, 26Cl, 37ClO C, 26Cl, 37ClO	
Ion ID	Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z	M M + 4 + 4 + 2 M M + 4 + 4 M M + 4 + 4 M M + 4 + 4 M M + 4 M + 4 M M + 4 M M + 4 M M M M	
Accurate Mass <sup>(*)</sup>	407.7818 409.7788 417.8250 419.8220 423.7767 425.7737 435.8169 437.8140 479.7165 [430.9728]	441.7428 443.7399 457.7377 459.7348 469.7780 471.7750 513.6775	
Descriptor	4	ശ	
Analyte	TCDF TCDF (8) TCDD (8) TCDD (8) TCDD (8) TCDD (8)	Pecde Pecde Pecde (S) Pecdo Pecdo Pecdo (S) Pecdo (S) Pecdo (S) Pecdo (S)	HXCDF HXCDF HXCDF (S) HXCDD HXCDD HXCDD (S) HXCDD (S) COCPE PFK
Elemental Composition	C12H, 45Cl, 0 C12H, 45Cl, 10 13C12H, 45Cl, 0 13C12H, 45Cl, 0 C12H, 45Cl, 0 C12H, 45Cl, 0 13C12H, 45Cl, 0 13C12H, 45Cl, 0 13C12H, 45Cl, 10 13C12H, 45Cl, 10 13C1	C <sub>12</sub> H <sub>3</sub> *C <sub>1</sub> O C <sub>12</sub> H <sub>3</sub> *C <sub>1</sub> O C <sub>12</sub> H <sub>3</sub> *C <sub>1</sub> O <sub>1</sub> O C <sub>12</sub> H <sub>3</sub> *C <sub>1</sub> O <sub>1</sub> O <sub>2</sub> C <sub>12</sub> H <sub>3</sub> *C <sub>1</sub> O <sub>1</sub> O <sub>2</sub> C <sub>12</sub> H <sub>3</sub> *C <sub>1</sub> O <sub>1</sub> O <sub>2</sub> C <sub>12</sub> H <sub>3</sub> *C <sub>1</sub> O <sub>2</sub> O <sub>2</sub> C <sub>12</sub> H <sub>3</sub> *C <sub>1</sub> O <sub>2</sub> O <sub>2</sub> C <sub>12</sub> H <sub>3</sub> *C <sub>1</sub> O <sub>2</sub> O <sub>2</sub> C <sub>12</sub> H <sub>3</sub> *C <sub>1</sub> O <sub>2</sub> O <sub>2</sub> C <sub>12</sub> H <sub>3</sub> *C <sub>1</sub> O <sub>2</sub> O <sub>2</sub> C <sub>12</sub> H <sub>3</sub> *C <sub>1</sub> O <sub>2</sub> O <sub>2</sub> C <sub>12</sub> H <sub>3</sub> *C <sub>1</sub> O <sub>2</sub> O <sub>2</sub> C <sub>12</sub> H <sub>3</sub> *C <sub>1</sub> O <sub>2</sub> O <sub>2</sub> C <sub>2</sub> F <sub>13</sub>	C <sub>1</sub> , H <sub>2</sub> acl <sub>3</sub> , TClO C <sub>1</sub> , H <sub>2</sub> acl <sub>3</sub> , TClO C <sub>1</sub> , H <sub>2</sub> acl <sub>3</sub> , TClO C <sub>1</sub> , H <sub>2</sub> acl <sub>3</sub> , TClO C <sub>1</sub> , H <sub>2</sub> acl <sub>3</sub> , TClO <sub>2</sub> C <sub>1</sub> , H <sub>2</sub> acl <sub>3</sub> , TClO <sub>2</sub> C <sub>2</sub> , H <sub>2</sub> acl <sub>3</sub> , TClO <sub>2</sub> C <sub>2</sub> , H <sub>2</sub> acl <sub>3</sub> , TCl <sub>2</sub> O <sub>2</sub> C <sub>2</sub> , H <sub>2</sub> acl <sub>3</sub> , TCl <sub>2</sub> O <sub>2</sub> C <sub>2</sub> , H <sub>2</sub> acl <sub>3</sub> , TCl <sub>2</sub> O <sub>2</sub> C <sub>2</sub> , F <sub>1</sub> ,
Ol nol	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	M + 2
Accurate mass <sup>(a)</sup>	303.9016 305.8987 315.9419 317.9389 319.8965 321.8965 331.3368 333.9338 375.8364 [354.9792]	339,8597 341,8567 351,9000 353,8970 355,8546 357,8516 367,8949 369,8919 409,7974 [354,9792]	373.8208 375.8178 383.8639 385.8610 389.8156 391.8127 401.8559 403.8529 445.7555
Descriptor	<b>v</b> ·	Ø	ဗ

(a) The following nuclidic masses were used:

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

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

S = internal/recovery standard

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

**Project/Site Name:** 

**BRC Tronox Parcel H** 

**Collection Date:** 

January 28, 2008

LDC Report Date:

March 12, 2008

Matrix:

Soil/Water

Parameters:

Dioxins/Dibenzofurans

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 1,2,3,6,7,8-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 1,2,3,4,6,7,8-HpCDF	1.1 pg/L 0.84 pg/L 1.2 pg/L 1.7 pg/L 6.6 pg/L 1.1 pg/L 0.72 pg/L 1.2 pg/L 1.1 pg/L 1.3 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	Р

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:

	Concentration (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)	-	-

### 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	А
F8A290158	TSB-HR-06-0'-FD	OCDD	11U pg/g	А
F8A290158	TSB-HR-06-10'	OCDD	11U pg/g	А
F8A290158	TSB-HJ-08-10'	OCDD	11U pg/g	А
F8A290158	TSB-HR-05-10'	OCDD	11U pg/g	Α
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	Α
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	Α

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	Α
F8A290158	TSB-HJ-08-0'	OCDD 1,2,3,6,7,8-HxCDF OCDF	11U pg/g 5.4U pg/g 11U pg/g	Α
F8A290158	TSB-HJ-08-10'	OCDD	11U pg/g	А
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	А

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 attache validation findings worksheets.    Validation Area	SDG Labor	#:_18386B21 #:_F8A290158 ratory:_Test America		ALIDATIO	1	Level			RKSHEE	Т	Date: 3/7/ Page: _/of _/ Reviewer: _/7/ 2nd Reviewer:
Validation Area										tion final	
1. Technical holding times				ewed for ear	cn or the r	OllOWII	ng va	alidation are	eas. Valloa	tion fina	ings are noted in attached
I.   Technical holding times		Validation.	A			<u> </u>		·	0		
II.   GC/MS Instrument performance check			Area	<u> </u>	, ,	Samn	ling d	ates:	1 1	ments	
III. Initial calibration			nce c	heck	<u>A</u>	Samp	illig u	ales.	17000		
IV.   Routine calibration   A   V.   Blanks   S.W     VI.   Matrix spike/Matrix spike duplicates   S.W     VII.   Laboratory control samples   A   L ← S.W     VIII.   Laboratory control samples   A   L ← S.W     VIII.   Regional quality assurance and quality control   N     IX.   Internal standards   A     X.   Target compound identifications   N     XI.   Compound quantitation and CRQLs   S.W     XIII.   System performance   N     XIII.   Overall assessment of data   A     XIV.   Field duplicates   S.W   R = 1.0     XIV.   Field blanks   S.W   R = 1.0     Note:   A = Acceptable   N   English   E	-			HOOK							
V.         Blanks         SW           VI.         Matrix spike/Matrix spike duplicates         5 W           VII.         Laboratory control samples         A         L C ⊆ ID           VIII.         Regional quality assurance and quality control         N           IX.         Internal standards         A           X.         Target compound identifications         N           XI.         Compound quantitation and CRQLs         SIM           XIII.         System performance         N           XIII.         Overall assessment of data         A           XIV.         Field duplicates         SW         R = 10           XV.         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:         SOLL         TSB-HJ-10-0°         11         TSB-HR-06-0°MS         21											
VI.         Matrix spike/Matrix spike duplicates         5 ₩           VII.         Laboratory control samples         A         L C ≤ ID           VIII.         Regional quality assurance and quality control         N           IX.         Internal standards         A           X.         Target compound identifications         N           XII.         Compound quantitation and CROLs         SIM           XIII.         Overall assessment of data         A           XIV.         Field duplicates         SW         R = IO           XV.         Field blanks         SW         R = IO           Note:         A = Acceptable 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:         SW	1			·							
VII.         Laboratory control samples         A         L C S I/O           VIII.         Regional quality assurance and quality control         N           IX.         Internal standards         A           X.         Target compound identifications         N           XII.         Compound quantitation and CRQLs         SIM           XIII.         Overall assessment of data         A           XIV.         Field duplicates         SW         R = TO           XV.         Field blanks         SW         R = TO           Note:         A = Acceptable Not provided/applicable SW = See worksheet         ND = No compounds detected R = Rensate FB = Field blank         TB = Trip blank EB = Equipment blank           Validated Samples:         SW         TSB-HR-06-0'MS         21			plicate	es					-		
VIII.         Regional quality assurance and quality control         N           IX.         Internal standards         A           X.         Target compound identifications         N           XII.         Compound quantitation and CRQLs         SIM           XIII.         Overall assessment of data         A           XIV.         Field duplicates         SW         R = To           XV.         Field blanks         SW         R = To           Note:         A = Acceptable N= Field blank         ND = No compounds detected R= Remaster Field blank         D = Duplicate Ties = Trip blank Eigen = Ties = Trip blank Eigen = Ties = Trip blank           Validated Samples:         SW = See worksheet         Field blank         Ties = Trip blank Eigen = Ties = Trip blank           2					A	L	<u>(</u>	10			
IX.   Internal standards			and qu	uality control							
XII.   System performance	IX.				Δ						
XII.   System performance	X.	Target compound identificat	ions		N						
XIII.   Overall assessment of data   XIV.   Field duplicates   SW   D = 3 + 4     XV.   Field blanks   SW   R = 10     XV.   Table blanks   SW   R = 10     XV.   Table blanks   SW   R = 10     XV.   Table blanks   SW   R = 10     XV.   Table blanks   SW   R = 10     XV.   Table blanks   SW   R = 10     XV.   Table blanks   SW   R = 10     XV.   Table blanks   SW   R = 10     X	XI.	Compound quantitation and	CRQI	_S	SW						
XIV   Field duplicates   SW   D = 3 + 4     XV   Field blanks   SW   R = 10     XV   Field blanks   SW   Field blank   EB = Fi	XII.	System performance			N						
XV.   Field blanks   SW   R = 10	XIII.	Overall assessment of data			A						
Note: A = Acceptable	XIV.	Field duplicates			SW	D	) =	= 34 '	1		
Note: A = Acceptable	XV.	Field blanks			SW	1	R :	= 10		•	
1 TSB-HJ-10-0'	Note:	N = Not provided/applicable SW = See worksheet		R = Rin FB = Fi	sate	ls detec	eted	TB =	Trip blank	ank	
2 TSB-HJ-10-10' X 12 TSB-HR-06-0'MSD 22 2 8 0 共 5 1 7 8 - B レ 32 33 33 33 4 TSB-HR-06-0'-FD 14 24 34 34 5 TSB-HR-06-10' 15 25 35 35 6 TSB-HJ-08-0' 16 26 36 7 TSB-HJ-08-10' 17 27 37 37 37 8 7 TSB-HR-05-0' 18 28 38 9 TSB-HR-05-10' 19 29 39	I 7	SOIL + W	للم	<u> </u>		· I				1 1	
2   ISB-HJ-10-10'   X   12   ISB-HR-06-0'MSD   22		TSB-HJ-10-0'	11	TSB-HR-06-0	'MS						
3 TSB-HR-06-0' 13 23 33 34 4 TSB-HR-06-0'-FD 14 24 34 34 5 TSB-HR-06-10' 15 25 35 35 6 TSB-HJ-08-0' 16 26 36 7 TSB-HJ-08-10' 17 27 37 37 8 TSB-HR-05-0' 18 28 38 38 9 TSB-HR-05-10' 19 29 39	2	TSB-HJ-10-10' χ	12	TSB-HR-06-0	MSD			80451	78-BLK		
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 39 TSB-HR-05-10' 19 29 39	3								<del> </del>		
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					<b>+</b>						
7 TSB-HJ-08-10' 17 27 37 8 TSB-HR-05-0' 18 28 38 39 TSB-HR-05-10' 19 29 39	7										
7 TSB-HJ-08-10' 17 27 37 37 8 Y TSB-HR-05-0' 18 28 38 39 39											
9 TSB-HR-05-10' 19 29 39	7										
	Y										
	10	RINSATE-2	19 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:

1858× FDC #: 7 SDG#:

### VALIDATION FINDINGS WORKSHEET

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

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

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

Were all samples associated with a method blank?

Was a method blank performed for each matrix and whenever a sample extraction was performed?

Was the method blank contaminated? N/A

N/A

Blank analysis date: 2 2 08

Brank extraction date: 2 1 나 0 음 Conc. units: 22 / a

Associated samples:\_

_
-
40/1111
M11/07 0   M11/200
1111/1100
10/11M
0.22 /10 u
0.0

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".

LDC # 1838687

### VALIDATION FINDINGS WORKSHEET

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

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

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

Were all samples associated with a method blank?

N/A Y N N/A ∀/N N/≻

Was a method blank performed for each matrix and whenever a sample extraction was performed?

Blank analysis date: 2/15/00 Was the method blank contaminated? Blank extraction date: 2/11/08

All walk Associated samples:\_

Sample Identification 3.9 /loou 1.7/1004 0.35 Jaw 0.56/spu mas/ hs 9 Q 8042218-B Blank ID <u>ڊ</u> ف 0.84 7 ج <u>-</u> 7 <u>\_</u> ÷ Conc. units: pa | 1 Compound ٤ Z Ø 9 4 D ш প্র Ω 4

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".

LDC#: /8 3882 SDG#:

### VALIDATION FINDINGS WORKSHEET Field Blanks

Page: of / Reviewer: 2nd Reviewer:

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

Associated Samples: Associated sample units: P3 | 9 Field blank type: (circle one) Field Blank / Rinsate / Other /Y N N/A Were field blanks identified in this SDG? Blank units: 24 /

Compound	Blank ID				Š	Sample Identification	ation			
	0	_	7	8	-1	5	J	7	8	b
IJ	₩8.0	(		0.16/5.2W			1	-	1.6/5.4V	1
9	68	ł	orr/10u	1.5/101	MIN/02.0	0.24 /In	1.2/11U	0.26/114	1,04   1.5/104   0.40/114   0.24/114   1.2/114   0.26/114   9.2/114   0.40/114	0. 40/11M
,1	54.0	١	,	- 1	- 1	<u>,                                     </u>	0.26/5.4u	1	0.16/5.44	_
Z	0.56	N53/15.01	(	1	١	١	-	(		-
Ø	1.7	) -	ı	10.62/10u 0.49/11M	0.49/11M	١	1.3/114	1	N1/L.1	
				,	1		. /		,	
CRQL										

Associated sample units: Blank units:

Sampling date:

Field blank type: (circle one) Field Blank / Rinsate / Other.

Associated Samples:

_					
Sample Identification					
Samp					
		-			*****
Blank ID					
pui					
Compound					Jt.
					CRQL

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:

Samples with compound concentrations within five times the associated field blank concentration are listed above, these sample results were qualified as not detected, "U".

LDC# 18386 BV SDG #:

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

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

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

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

N N/A

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

N/N/A

Z

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?

F	1		Т	<u> </u>	T			T				T	一	1		Ī	_	Ī	T	Ī				$\neg$
Oualifications	11	M SOL 1 AND ON			ð																			
Accordance Remains	Associated Samples	<b>\</b>				•																		
7000	RPD (CIIIIIS)		( )		( )	( )	( )	( )	( )	( )		( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
USW	%K (LIMITS)	124 (70-120)	(33 (74-124)		( )	( )	( )	( )		( )	( )		( )	( )	)	( )	( )	( )	( )	( )	( )	( )	( )	
MS	%R (Limits)	130 TO-120)	(hel-hL) 1h1	(M1-11-) 521	31-5L) XC1	(	( )	( )		( )				( )	( )	( )	( )	( )	( )		( )	( )	( )	( )
	Compound	u	.5	٧	-9																			
	MS/MSD ID	11 12																						
	Date																							
1	#														<u> </u>			L			L			

LDC# 18380 B2/ SDG# 42 const

# VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

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). Y N N/A N N/A

Qualifications	nos of	-							
Associated Samples	2								
Finding	no conjirmation on	DB-22K							
Annogum on									
ate C									

Comments: See sample calculation verification worksheet for recalculations

### VALIDATION FINDINGS WORKSHEET Field Duplicates

Page:_(	_of
Reviewer:_	BL
2nd reviewer:_	

	Concentrat	on (29/9( )	Diperence
Compound	3	Ly SALU	
F	0.16	THEN THU	10254 5.24 (= 9
G	1.5	0.70	0.8 (=11)
8	0.19	0.26	0.07 (= 5.4)
Q	0.62	0.49	0.13 (=11)
	Concentrat	ion (	
Compound			RPD
Compound			
	Concentra	tion (	
Compound			RPD
Compound			
	Concentra	tion ()	
Compound			RPD
Compound			