



**LABORATORY DATA CONSULTANTS, INC.**

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

March 19, 2008

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:**

<b><u>SDG #</u></b>	<b><u>Fraction</u></b>
F8A250221,	Volatiles, Semivolatiles, Chlorinated Pesticides, Polychlorinated
F8A290158	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



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

Volatiles

*LDC*

## 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.

**Sample Delivery Group (SDG):** F8A250221

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

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 19 soil samples and 3 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8260B for Volatiles.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UU 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 ( $\geq 0.05$ )	All water samples in SDG F8A250221	J (all detects) UJ (all non-detects)	A
1/30/08	Ethanol	0.00855 ( $\geq 0.05$ )	All water samples in SDG F8A250221	J (all detects) UJ (all non-detects)	A

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
2/4/08	Ethanol Acetonitrile	0.00291 ( $\geq 0.05$ ) 0.01869 ( $\geq 0.05$ )	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HJ-04-0' TSB-HR-04-0'*** TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'*** TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'*** TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-0'MS TSB-HR-08-0'MSD 8038049-Blank	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A
2/6/08	Ethanol	0.00366 ( $\geq 0.05$ )	TSB-HJ-07-0'-FD TSB-HR-08-10' TSB-HR-08-10'MS TSB-HR-08-10'MSD 8038277-Blank	J (all detects) UJ (all non-detects)	A

#### IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

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

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

Date	Compound	%D	Associated Samples	Flag	A or P
1/30/08	Bromomethane	48.37592	All water samples in SDG F8A250221	J+ (all detects)	A

Date	Compound	%D	Associated Samples	Flag	A or P
2/5/08 (09:12)	1,1-Dichloroethane Iodomethane Carbon tetrachloride 2-Nitropropane	89.46153 32.31122 28.82024 33.49971	TSB-HJ-05-10' TSB-HJ-05-0' TSB-HR-04-10' TSB-HJ-04-0' TSB-HR-04-0'** TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'*** TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'*** TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-0'MS TSB-HR-08-0'MSD 8038049-Blank	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	A

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

Date	Compound	%D	Associated Samples	Flag	A or P
1/21/08	Iodomethane 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-HJ-04-0' TSB-HR-04-0'** TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'*** TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'*** TSB-HJ-07-10' TSB-HR-08-0' TSB-HR-08-0'MS TSB-HR-08-0'MSD 8038049-Blank	J+ (all detects)	A
2/6/08	Bromomethane	34.53645	TSB-HJ-07-0'-FD TSB-HR-08-10' TSB-HR-08-10'MS TSB-HR-08-10'MSD 8038277-Blank	J+ (all detects)	A
2/6/08	Acetonitrile	27.60270	TSB-HJ-07-0'-FD TSB-HR-08-10' TSB-HR-08-10'MS TSB-HR-08-10'MSD 8038277-Blank	J- (all detects) UJ (all non-detects)	a



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

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

## V. Blanks

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

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

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

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

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

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

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

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

## VI. Surrogate Spikes

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

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

## VII. Matrix Spike/Matrix Spike Duplicates

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

## VIII. Laboratory Control Samples (LCS)

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

## IX. Regional Quality Assurance and Quality Control

Not applicable.

## X. Internal Standards

All internal standard areas and retention times.

## XI. Target Compound Identifications

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

## XII. Compound Quantitation and CRQLs

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

## XIII. Tentatively Identified Compounds (TICs)

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

## XIV. System Performance

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

## XV. Overall Assessment of Data

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

## XVI. Field Duplicates

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

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

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

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

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

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

LDC #: 18386A1  
 SDG #: F8A250221  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 3/10/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

**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
I.	Technical holding times	Δ	Sampling dates: 1/24/08
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	SW	% RSD, r <sup>2</sup>
IV.	Continuing calibration/ICV	SW	ICV ≤ 25
V.	Blanks	SW	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	SW	LCs/D
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	Δ	
XI.	Target compound identification	Δ	Not reviewed for Level III validation.
XII.	Compound quantitation/CRQLs	Δ	Not reviewed for Level III validation.
XIII.	Tentatively identified compounds (TICs)	Δ	Not reviewed for Level III validation.
XIV.	System performance	Δ	Not reviewed for Level III validation.
XV.	Overall assessment of data	A	
XVI.	Field duplicates	SW	D = 11 + 12
XVII.	Field blanks	SW	TB = 16, 17, 18

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

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

soil + water

1 3	TSB-HJ-05-10'	1	† 13	TSB-HJ-07-0**	3	214	TSB-HR-08-10'MS	311	8031135 - Blank	1/30
2 3	TSB-HJ-05-0'	1	124	TSB-HJ-07-0'-FD	3	224	TSB-HR-08-10'MSD	322	8036136 - Blank	2/4
3 3	TSB-HR-04-10'	1	133	TSB-HJ-07-10'	3	23		333	803849 - Blank	2/5/08
4 3	TSB-HJ-04-0'	1	143	TSB-HR-08-0'	3	24		344	8038277 - Blank	2/6/08
5 3	TSB-HR-04-0**	1	154	TSB-HR-08-10'	3	25		355	8031135 - Blank	1/30
6 3	TSB-HJ-04-10'	1	161	TSB-TB-3	w	1	26	36		
7 3	TSB-HR-07-0'	2	171	TSB-TB-2	↓	2	27	37		
8 3	TSB-HR-07-10**	2	181	TSB-TB-1	↓	3	28	38		
9 3	TSB-HR-06-0'	2	193	TSB-HR-08-0'MS			29	39		
10 3	TSB-HR-06-10'	2	203	TSB-HR-08-0'MSD			30	40		

Batch #4 analyzed after ICA

LDC #: 18386A1  
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2  
 Reviewer: FJ  
 2nd Reviewer: W

Method: Volatiles (EPA SW 846 Method 8260B)

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>II. GC/MS instrument performance check</b>				
Were the BFB performance results reviewed and found to be within the specified criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all samples analyzed within the 12 hour clock criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>III. Initial calibration</b>				
Did the laboratory perform a 5 point calibration prior to sample analysis?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a curve fit used for evaluation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did the initial calibration meet the curve fit acceptance criteria of $\geq 0.990$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent relative standard deviations (%RSD) $\leq 30\%$ and relative response factors (RRF) $> 0.05$ ?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Soil instrument calibration</b>				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) $\leq 25\%$ and relative response factors (RRF) $\geq 0.05$ ?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a method blank analyzed at least once every 12 hours for each matrix and concentration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>VI. Surrogate spikes</b>				
Were all surrogate %R within QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
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?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>VII. Matrix spike/Matrix spike duplicates</b>				
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.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a MS/MSD analyzed every 20 samples of each matrix?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>VIII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	



LDC #: 18386A  
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: PT  
 2nd Reviewer: W

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per analytical batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>IX. Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the performance evaluation (PE) samples within the acceptance limits?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>X. Internal Standards</b>				
Were internal standard area counts within -50% or +100% of the associated calibration standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were retention times within + 30 seconds of the associated calibration standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XI. Large Compound Identification</b>				
Were relative retention times (RRT's) within + 0.06 RRT units of the standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were chromatogram peaks verified and accounted for?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XII. Compound Quantitation/CRQLs</b>				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XIII. Reference Identified Compounds (RIS)</b>				
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were relative intensities of the major ions within ± 20% between the sample and the reference spectra?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XIV. System Performance</b>				
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XV. Overall Assessment of Data</b>				
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XVI. Field Duplicates</b>				
Field duplicate pairs were identified in this SDG.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Target compounds were detected in the field duplicates.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XVII. Field Blanks</b>				
Field blanks were identified in this SDG.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Target compounds were detected in the field blanks.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

# 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 chloride**	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	OOO. 1,3,5-Trichlorobenzene	IIII. Isobutyl alcohol
H. 1,1-Dichloroethene**	BB. 1,1,2,2-Tetrachloroethane*	VV. Isopropylbenzene	PPP. trans-1,2-Dichloroethene	JJJJ. Methacrylonitrile
I. 1,1-Dichloroethane*	CC. Toluene**	WW. Bromobenzene	QQQ. cis-1,2-Dichloroethene	KKKK. Propionitrile
J. 1,2-Dichloroethene, total	DD. Chlorobenzene*	XX. 1,2,3-Trichloropropane	RRR. m,p-Xylenes	LLLL. Ethyl ether
K. Chloroform*	EE. Ethylbenzene**	YY. n-Propylbenzene	SSS. o-Xylene	MMMM. Benzyl chloride
L. 1,2-Dichloroethane	FF. Styrene	ZZ. 2-Chlorotoluene	TTT. 1,1,2-Trichloro-1,2,2-trifluoroethane	NNNN.
M. 2-Butanone	GG. Xylenes, total	AAA. 1,3,5-Trimethylbenzene	UUU. 1,2-Dichlorotetrafluoroethane	OOOO.
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	QQQQ.
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	TTTT.
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.

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

VALIDATION FINDINGS WORKSHEET

Initial Calibration

LDC #: 18380A  
SDG #: fire control

Page: 1 of 1  
Reviewer: [Signature]  
2nd Reviewer: [Signature]

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

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

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

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

N N/A Was a curve fit used for evaluation? If yes, what was the acceptance criteria used for evaluation? \_\_\_\_\_

Y N N/A Did the initial calibration meet the acceptance criteria?

Y N N/A Were all %RSDs and RRFs within the validation criteria of  $\leq 30$  %RSD and  $\geq 0.05$  RRF?

#	Date	Standard ID	Compound	Finding %RSD (Limit: $\leq 30.0\%$ )	Finding RRF (Limit: $\geq 0.05$ )	Associated Samples	Qualifications
1	1/21/08	1CAL	PP	0.04510	0.00977	8031135-Blank, All water	J/WJ/A ↓
	1/30/08	1CAL	www	0.00855		↓	↓
	2/4/08	1CAL	www	0.00291	0.01869	803849-Blank, 1-11, 19, 20 13, 14	J/WJ/A
	2/6/08	1CAL	www	0.00366		8038277-Blank, 12, 15, 21, 22	J/WJ/A

VALIDATION FINDINGS WORKSHEET  
Continuing Calibration

LDC #: 18386A  
SDG #: pre cover

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

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".  
 N N/A Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?  
 N N/A Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's?  
 N N/A Were all %D and RRFs within the validation criteria of  $\leq 25\%$  %D and  $\geq 0.05$  RRF?

#	Date	Standard ID	Compound	Finding %D (Limit: $\leq 25.0\%$ )	Finding RRF (Limit: $\geq 0.05$ )	Associated Samples	Qualifications
+	1/21/08	ICV	Iodomethane HH	33.60319 31.0087V		8031135-Blank + all water	J+/A det
+	1/30/08 14:32	CCV	B RR	48.37592	0.04735	↓	J+/A det J/u/A
+	2/4/08	ICV	I	12.08435		803849-Blank, 1-14, 19, 20 13, 14	J+/A det
+	2/6/08	ICV	B EEEE	34.53645 27.60270		8038277-Blank, 12, 15, 21, 2A	J+/A det J- u/A

VALIDATION FINDINGS WORKSHEET  
 Continuing Calibration

LDC #: 18386A1  
 SDG #: fw cover

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

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

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

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

Y/N N/A Were all %D and RRFs within the validation criteria of  $\leq 25\%$  D and  $\geq 0.05$  RRF ?

#	Date	Standard ID	Compound	Finding %D (Limit: $\leq 25.0\%$ )	Finding RRF (Limit: $\geq 0.05$ )	Associated Samples	Qualifications
+	2/5/08	ew	I	89.46153		803849 - blank,	J+ / A det
+	9.12		Iodomethane	32.31122		17, 11, 13, 14,	
+			e	28.82024		19, 20	
+			2-Nitropropane	33.49971			
			EEEE		0.01921		J/uJ/A
	2/5/08	ew	www	0.00259	0.00259		J/uJ/A
	10.14						

LDC #: 18386A  
 SDG #: free cover

**VALIDATION FINDINGS WORKSHEET**  
**Blanks**

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

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

Y/N N/A Was a method blank associated with every sample in this SDG?  
 Y/N N/A Was a method blank analyzed at least once every 12 hours for each matrix and concentration?  
 Y/N N/A Was there contamination in the method blanks? If yes, please see the qualifications below.

Blank analysis date: 1/30/08  
 Conc. units: ng/l

Associated Samples: All water

Compound	Blank ID	Sample Identification			
Vichloromethane Methylene chloride	803135-Blank 0.16	16 0.20/1.00	17 0.14/1.00	18 0.18/1.00	
Acetone					
CRQL					

Blank analysis date: 2/6/08  
 Conc. units: ng/kg

Associated Samples: 12, 15

Compound	Blank ID	Sample Identification			
Vichloromethane Methylene chloride	8038277-Blank 2.8	12 6.1/4	15 4.2/5.50		
Acetone					
CRQL					

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 TlCs 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".

LDC #: 1828677  
 SDG #: per cone  
**VALIDATION FINDINGS WORKSHEET**  
**Field Blanks**

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

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

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

Blank units: ug/l Associated sample units: ug/kg  
 Field blank type: (circle one) Field Blank / Rinsate / Trip Blank / Other: \_\_\_\_\_

Associated Samples: 1-7-6 (ND + 7 SX) *others*

Compound	Blank ID   6	Blank ID	Sample Identification
<del>Dichloromethane</del>	1/24/08	2	6
Methylene-chloride	0.20	-	-
Acetone	4.3	17/2/11	6.8/2/11 14/2/11
Chloroform			
CRQL			

Blank units: ug/l Associated sample units: ug/kg  
 Field blank type: (circle one) Field Blank / Rinsate / Trip Blank / Other: \_\_\_\_\_

Associated Samples: 7-7-10 (ND + 7 SX)

Compound	Blank ID   7	Blank ID	Sample Identification
<del>Dichloromethane</del>	1/24/08		
Methylene-chloride	0.14		
Acetone			
Chloroform			
CRQL			

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

**VALIDATION FINDINGS WORKSHEET**  
**Field Blanks**

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

Blank units: ug/l Associated sample units: ug/l  
 Field blank type: (circle one) Field Blank / Rinsate / Trip Blank / Other: \_\_\_\_\_

Associated Samples: 11 - 15 (ND + 75X)

Compound	Blank ID	Blank ID	Sample Identification
<del>Methylene chloride</del>	<u>1/24/08</u>	<u>13</u>	<u>14</u>
<u>Dichloromethane</u>	<u>0.18</u>	<u>-</u>	<u>-</u>
Acetone	<u>4.9</u>	<u>19/2/10</u>	<u>7.4/2/10</u>
Chloroform			
CRQL			

Blank units: \_\_\_\_\_ Associated sample units: \_\_\_\_\_  
 Field blank type: (circle one) Field Blank / Rinsate / Trip Blank / Other: \_\_\_\_\_ Associated Samples: \_\_\_\_\_

Compound	Blank ID	Blank ID	Sample Identification
<del>Methylene chloride</del>			
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 qualified as not detected, "U". Other contaminants within five times the field blank concentration were also qualified as not detected, "U".



**VALIDATION FINDINGS WORKSHEET**  
**Surrogate Spikes**

Page: 1 of 1  
 Reviewer: D  
 2nd Reviewer:

LDC #: 18386A  
 SDG #: free cover  
**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".  
 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?  
 Y N N/A

#	Date	Sample ID	Surrogate	%Recovery (Limits)	Qualifications
		8036136 - Blank	BFB	125 (63-132) (66-115)	J+/P det
		16	BFB	120 (66-115)	J+/A det email Nonanal only
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

QC Limits (Soil)  
 81-117  
 74-121  
 80-120  
 80-120

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

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





LDC #: 18386 A1  
 SDG #: Sec cover

**VALIDATION FINDINGS WORKSHEET**  
**Field Duplicates**

Page: 1 of 1  
 Reviewer: ST  
 2nd reviewer: W

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

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

Compound	Concentration ( <u>ug/kg</u> )		Difference RPD
	11	12	
dichloromethane	5.8	6.1	0.3 (≤ 5.4)
DDD	0.41	5.34	4.89 (≤ 5.4)

Compound	Concentration ( )		RPD

Compound	Concentration ( )		RPD

Compound	Concentration ( )		RPD

LDC #: 18386A1  
 SDG #: see cover

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

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

**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:

$RRF = (A_x)(C_s)/(A_s)(C_x)$   
 average RRF = sum of the RRFs/number of standards  
 $\%RSD = 100 * (S/X)$   
 $A_x$  = Area of compound,  
 $C_x$  = Concentration of compound,  
 $S$  = Standard deviation of the RRFs  
 $X$  = Mean of the RRFs  
 $A_s$  = Area of associated internal standard  
 $C_s$  = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				RRF (2σ std)	RRF (2σ std)	RRF (2σ std)	Average RRF (initial)	Average RRF (initial)	%RSD	%RSD	
1	KAL	2/4/08	Vinyl chloride (1st internal standard)	0.77522	0.77522	0.73013	0.73013	6.16160	6.16160	6.16160	6.16160
			Ethyl Benzene (2nd internal standard)	1.79313	1.79313	1.80952	1.80952	4.27181	4.27181	4.27181	4.27181
			1,2-DCB (3rd internal standard)	1.53477	1.53477	1.54561	1.54561	3.54081	3.54081	3.54081	3.54081
2			www (1st internal standard)	0.00342	0.00342	0.00291	0.00291	11.51035	11.51035	11.51035	11.51035
			Vinyl chloride (2nd internal standard)	0.50639	0.50639	0.54575	0.54575	13.72254	13.72254	13.72254	13.72254
			1,3,5-Trinitrobenzene (3rd internal standard)	0.88190	0.88190	0.89971	0.89971	9.47511	9.47511	9.47511	9.47511
3			(1st internal standard)								
			(2nd internal standard)								
			(3rd internal standard)								
4			(1st internal standard)								
			(2nd internal standard)								
			(3rd internal standard)								

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

LDC #: 18386A  
 SDG #: per coner

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

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

**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 =  $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$   
 $\text{RRF} = (A_x / C_x) / (A_b / C_b)$

Where: ave. RRF = initial calibration average RRF  
 RRF = continuing calibration RRF  
 $A_x$  = Area of compound,  
 $C_x$  = Concentration of compound,  
 $A_b$  = Area of associated internal standard  
 $C_b$  = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference internal Standard)	Average RRF (initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	ccv	2/5/08 9:12	Vinyl chloride (1st internal standard)	0.73013	0.82924	13.5715	0.82924	13.5715
			Ethyl Benzene (2nd internal standard)	1.80952	1.87688	3.7227	1.87688	3.7227
			1,2 - DCP (3rd internal standard)	1.54561	1.53180	0.89307	1.53180	0.89307
2	ccv	2/5/08 10:14	www (1st internal standard)	0.00291	0.00259	11.3228	0.00259	11.3228
			Dimethyl Disulfide (2nd internal standard)	0.54575	0.58269	6.76765	0.58269	6.76765
			1,2,5- Trichlorobenzene (3rd internal standard)	1.07630	1.09104	1.36954	1.09104	1.36954
3			(1st internal standard)					
			(2nd internal standard)					
			(3rd internal standard)					
4			(1st internal standard)					
			(2nd internal standard)					
			(3rd internal standard)					

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

LDC #: 18386A  
 SDG #: see cover

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

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd reviewer: [Signature]

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found  
 SS = Surrogate Spiked

Sample ID: #5

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8	50	54.6314	109	109	0
Bromofluorobenzene	↓	52.9992	106	106	↓
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 #: 18380A  
 SDG #: per cover

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

Page: 1 of 1  
 Reviewer: f7  
 2nd Reviewer: [Signature]

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

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

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

RPD =  $100 * MSC - MSC / (MSC + MSDC)$       MSC = Matrix spike percent recovery      MSDC = Matrix spike duplicate percent recovery

MS/MSD sample: 19 + 20

Compound	Spike Added (ug/kg)		Sample Concentration (ug/kg)		Spiked Sample Concentration (ug/kg)		Matrix Spike		Matrix Spike Duplicate		MS/MSD	
	MS	MSD	MS	MSD	MS	MSD	Percent Recovery		Percent Recovery		RPD	
	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
1,1-Dichloroethene	52.4	52.9	ND		58.3	59.7	110	110	113	113	2.4	2.4
Trichloroethene					50.4	55.1	96	96	104	104	8.8	8.8
Benzene					56.0	54.0	106	106	102	102	3.6	3.6
Toluene					54.2	55.3	103	103	105	105	1.9	2.0
Chlorobenzene					53.0	54.2	100	100	103	103	2.2	2.2

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 #: 18386A1  
 SDG #: per canon

**VALIDATION FINDINGS WORKSHEET**  
**Laboratory Control Sample Results Verification**

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

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

% Recovery =  $100 \cdot \text{SSC}/\text{SA}$       Where: SSC = Spiked sample concentration  
 SA = Spike added

RPD =  $100 \cdot \frac{\text{LCS} - \text{LCSD}}{\text{LCS} + \text{LCSD}}$

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

LCS ID: 8078049-LCS

Compound	Spike Added (ug/kg)		Spiked Sample Concentration (ug/kg)		LCS		LCSD		Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
1,1-Dichloroethene	50.0	NA	49.5	NA	99	99	99	99						
Trichloroethene			47.1		94	94	94	94						
Benzene			48.2		96	96	96	96						
Toluene			48.4		97	97	97	97						
Chlorobenzene			48.2		96	96	96	96	NA	NA				

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 #: 18380A  
SDG #: mu cover

## VALIDATION FINDINGS WORKSHEET

### Sample Calculation Verification

Page: 1 of 1  
Reviewer: PJ  
2nd reviewer: [Signature]

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

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

Concentration = 
$$\frac{(A_x)(I_s)(DF)}{(A_{is})(RRF)(V_o)(\%S)}$$

A<sub>x</sub> = Area of the characteristic ion (EICP) for the compound to be measured  
A<sub>is</sub> = Area of the characteristic ion (EICP) for the specific internal standard  
I<sub>s</sub> = Amount of internal standard added in nanograms (ng)  
RRF = Relative response factor of the calibration standard.  
V<sub>o</sub> = Volume or weight of sample pruged in milliliters (ml) or grams (g).  
Df = Dilution factor.  
%S = Percent solids, applicable to soils and solid matrices only.

Example:  
Sample I.D. #5 Acetone  
  
Conc. = 
$$\frac{(\quad)(\quad)(\quad)}{(\quad)(\quad)(\quad)(\quad)}$$
  
=

#	Sample ID	Compound	Reported Concentration ( ) ( )	Calculated Concentration ( ) ( )	Qualification
		$\frac{x}{SD} = \left( \frac{10043}{619429} \right) \left( \frac{1}{0.06192} \right)$	- 0.13125		
		$X = 6.529$			
		$final = \frac{6.529}{0.959} = 6.8 \text{ ug/kg}$			

**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.
- UU 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 ( $\geq 0.05$ )	All water samples in SDG F8A290158	J (all detects) UJ (all non-detects)	A
1/30/08	Ethanol	0.00855 ( $\geq 0.05$ )	All water samples in SDG F8A290158	J (all detects) UJ (all non-detects)	A

## IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

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

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

Date	Compound	%D	Associated Samples	Flag	A or P
1/30/08	Bromomethane	48.37592	All water samples in SDG F8A290158	J+ (all detects)	A
2/11/08	Ethanol 2-Methylhexane 3-Ethylpentane n-Heptane	28.63874 27.07574 25.56456 26.94019	All soil samples in SDG F8A290158	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	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	Iodomethane 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 ( $\geq 0.05$ )	All water samples in SDG F8A290158	J (all detects) UJ (all non-detects)	A
2/11/08	Ethanol	0.00649 ( $\geq 0.05$ )	All soil samples in SDG F8A290158	J (all detects) UJ (all non-detects)	A

## V. Blanks

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

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

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

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

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

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

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

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

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

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

## VI. Surrogate Spikes

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

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



## **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:

Compound	Concentration (ug/Kg)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HR-06-0'	TSB-HR-06-0'-FD				
Dichloromethane	7.4	3.0	-	4.4 ( $\leq 5.4$ )	-	-
Toluene	0.54	5.4U	-	4.86 ( $\leq 5.4$ )	-	-

**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)	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-HR-05-0' TSB-HR-05-10'	Ethanol 2-Methylhexane 3-Ethylpentane n-Heptane	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	A	Continuing calibration (%D)
F8A290158	TSB-TB-1-1/28/08 TSB-TB-2-1/28/08 RINSATE-2 TSB-TB-03-1/28/08	Iodomethane Vinyl acetate	J+ (all detects) J+ (all detects)	A	Continuing calibration (ICV %D)
F8A290158	TSB-HJ-10-0' TSB-HJ-10-10' TSB-HR-06-0' TSB-HR-06-0'-FD TSB-HR-06-10' TSB-HJ-08-0' TSB-HJ-08-10' TSB-HR-05-0' TSB-HR-05-10'	Acetonitrile 4-Methyl-2-pentanone 4-Chlorotoluene	J+ (all detects) J+ (all detects) J+ (all detects)	A	Continuing calibration (ICV %D)
F8A290158	TSB-TB-1-1/28/08 TSB-TB-2-1/28/08 RINSATE-2 TSB-TB-03-1/28/08	Dibromomethane	J (all detects) UJ (all non-detects)	A	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-HR-05-0' TSB-HR-05-10'	Ethanol	J (all detects) UJ (all non-detects)	A	Continuing calibration (RRF)

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

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

**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	A
F8A290158	TSB-HJ-10-10'	Dichloromethane	5.2U ug/Kg	A
F8A290158	TSB-HR-06-0'	Dichloromethane	7.4U ug/Kg	A
F8A290158	TSB-HR-06-0'-FD	Dichloromethane	5.4U ug/Kg	A
F8A290158	TSB-HR-06-10'	Dichloromethane	7.3U ug/Kg	A
F8A290158	TSB-HJ-08-0'	Dichloromethane	8.8U ug/Kg	A
F8A290158	TSB-HJ-08-10'	Dichloromethane	5.8U ug/Kg	A
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	A
F8A290158	RINSATE-2	Acetone	8.0U ug/L	A

LDC #: 18386B1  
 SDG #: F8A290158  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III

Date: 3/7/08  
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 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

**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
I.	Technical holding times	A	Sampling dates: 1/28/08
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	SW	% PSD, $r^2 = 20.990$
IV.	Continuing calibration/ICV	SW	ICV = 25
V.	Blanks	SW	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	A	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	SW	R = 12 TB = 10 TB = 11, 13

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:

SOIL + WATER

1	3	TSB-HJ-10-0'	1	11	1	TSB-TB-2-1/28/08	W	2	21	1	8031135	31		1/30	
2	3	TSB-HJ-10-10'	1	12	1	RINSATE-2		3	22	2	8036136	32	nonanal only	2/4	
3	3	TSB-HR-06-0'	D	1	13	1	TSB-TB-03-1/28/08		3	23	3	8043263	33		2/11
4	3	TSB-HR-06-0'-FD	D	1	14		TSB-HR-05-10'MS			24	8'	34			
5	3	TSB-HR-06-10'		1	15		TSB-HR-05-10'MSD			25		35			
6	3	TSB-HJ-08-0'		1	16					26		36			
7	3	TSB-HJ-08-10'		1	17					27		37			
8	3	TSB-HR-05-0'		1	18					28		38			
9	3	TSB-HR-05-10'		1	19					29		39			
10	1	TSB-TB-1-1/28/08	W	1	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 chloride**	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	OOO. 1,3,5-Trichlorobenzene	IIII. Isobutyl alcohol
H. 1,1-Dichloroethene**	BB. 1,1,2,2-Tetrachloroethane*	VV. Isopropylbenzene	PPP. trans-1,2-Dichloroethene	JJJJ. Methacrylonitrile
I. 1,1-Dichloroethane*	CC. Toluene**	WW. Bromobenzene	QQQ. cis-1,2-Dichloroethene	KKKK. Propionitrile
J. 1,2-Dichloroethene, total	DD. Chlorobenzene*	XX. 1,2,3-Trichloropropane	RRR. m,p-Xylenes	LLLL. Ethyl ether
K. Chloroform**	EE. Ethylbenzene**	YY. n-Propylbenzene	SSS. o-Xylene	MMMM. Benzyl chloride
L. 1,2-Dichloroethane	FF. Styrene	ZZ. 2-Chlorotoluene	TTT. 1,1,2-Trichloro-1,2,2-trifluoroethane	NNNN.
M. 2-Butanone	GG. Xylenes, total	AAA. 1,3,5-Trimethylbenzene	UUU. 1,2-Dichlorotetrafluoroethane	OOOO.
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	QQQQ.
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	TTTT.
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.

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

VALIDATION FINDINGS WORKSHEET  
Initial Calibration

LDC #: 1X386B1  
SDG #: see cont

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2nd Reviewer: [Signature]

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".

- X N N/A
- Y N N/A
- X N N/A
- X N N/A
- Y N N/A

Did the laboratory perform a 5 point calibration prior to sample analysis?  
Were percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCC's and SPCC's?  
Was a curve fit used for evaluation? If yes, what was the acceptance criteria used for evaluation? \_\_\_\_\_  
Did the initial calibration meet the acceptance criteria?  
Were all %RSDs and RRFs within the validation criteria of  $\leq 30$  %RSD and  $> 0.05$  RRF ?

#	Date	Standard ID	Compound	Finding %RSD (Limit: $\leq 30.0\%$ )	Finding RRF (Limit: $\geq 0.05$ )	Associated Samples	Qualifications
	<u>1/21/08</u>	<u>ICAL</u>	<u>RR</u>		<u>0.04510</u>	<u>8071135 - Blank,</u>	<u>N/A</u>
			<u>water</u>		<u>0.00977</u>	<u>All water</u>	<u>↓</u>
	<u>1/30/08</u>	<u>ICAL</u>	<u>www</u>		<u>0.00855</u>	<u>↓</u>	<u>↓</u>

# VALIDATION FINDINGS WORKSHEET

## Continuing Calibration

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

LDC #: 183366B  
 SDG #: see cover

**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  $\leq 25\%$  %D and  $\geq 0.05$  RRF?

#	Date	Standard ID	Compound	Finding %D (Limit: $\leq 25.0\%$ )	Finding RRF (Limit: $> 0.05$ )	Associated Samples	Qualifications
+	1/21/08	1CV	1000methane	33.60319		80311 35-Blank	J+/A det
+			HH	31.00872		All water	↓
+	1/30/08	CCV	B	48.37592			J+/A det
	14:32		RR		0.04735		JWS/A
+	2/11/08	CCV	www		0.006491	804 3263-Blank	JWS/A
+	18:16		www	28.63874		All soils	J+/A det
+			2-Methylhexane	27.07574			↓
+			3-Ethylpentane	25.56456			↓
+			n-heptane	26.94019			↓
+	2/11/08	Blank 1CV	EEEE	27.39844			J+/A det
+	16:50		Y	27.93055			↓
+			BBB	25.96800			↓



**VALIDATION FINDINGS WORKSHEET**  
**Blanks**

LDC #: 18386B  
SDG #: pel coned

Page: 1 of 1  
Reviewer: [Signature]  
2nd Reviewer: [Signature]

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

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".  
 N  N/A Was a method blank associated with every sample in this SDG?  
 N  N/A Was a method blank analyzed at least once every 12 hours for each matrix and concentration?  
 N  N/A Was there contamination in the method blanks? If yes, please see the qualifications below.

Blank analysis date: 1/30/08

Conc. units: ug/l Associated Samples: 10 → 13 (all water samples)

Compound	Blank ID	Sample Identification			
<b>1,1,1-trichloroethane</b>	803195-Blank	10	11	12	13
Methylene chloride	0.16	0.21/1.0u	0.19/1.0u	0.12	0.22/1.0u
Acetone					
CRQL					

Blank analysis date: \_\_\_\_\_  
Conc. units: \_\_\_\_\_

Associated Samples: \_\_\_\_\_

Compound	Blank ID	Sample Identification			
<b>1,1,1-trichloroethane</b>					
Methylene chloride					
Acetone					
CRQL					

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

**VALIDATION FINDINGS WORKSHEET**

**Field Blanks**

LDC #: 1X38661  
 SDG #: for cond

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

Blank units: ug/L Associated sample units: ug/kg

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

Associated Samples: All soils

Compound	Blank ID	Sample Identification							
		1	2	3	4	5	6	7	8
Dichloromethane	128108	6.1/U	3.9/5.24	7.4/U	3.0/5.44	7.3/U	8.8/U	5.8/U	7.8/U
Methylene-chloride	8.0	-	-	-	-	-	-	-	15/U
Acetone									
Chloroform									
CRQL									

Blank units: ug/kg Associated sample units: ug/kg

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

Associated Samples: All soils

Compound	Blank ID	Sample Identification							
		1	2	3	4	5	6	7	8
Dichloromethane	128108	9							
Methylene-chloride	12	6.5/U							
Acetone	8.0	<del>6.5/U</del>							
Chloroform									
CRQL									

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

**VALIDATION FINDINGS WORKSHEET**

**Field Blanks**

LDC #: 1828021  
 SDG #: fec coney

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

**Blank units:** ug/L **Associated sample units:** ug/kg  
**Field blank type:** (circle one) Field Blank / Rinsate / Trip Blank / Other: Field Blank

Associated Samples: 1-5 710X + ND

Compound	Blank ID	Blank ID	Sample Identification
<del>Methylene chloride</del>	128108	8	
Dichloromethane	0.21	-	
Acetone	4.0		
Chloroform			
CRQL			

**Blank units:** ug/L **Associated sample units:** ug/kg  
**Field blank type:** (circle one) Field Blank / Rinsate / Trip Blank / Other: Field Blank

Associated Samples: 6-9 (710X + ND)

Compound	Blank ID	Blank ID	Sample Identification
<del>Methylene chloride</del>	128108	8	
Dichloromethane	0.19	-	
Acetone	5.1	15/U	
Chloroform			
CRQL			

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

# VALIDATION FINDINGS WORKSHEET

## Field Blanks

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

Y/N/N/A Were field blanks identified in this SDG?

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

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

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

Associated Samples: 12

Compound	Blank ID	Blank ID	Sample Identification
<b>Dichloro methane</b>	<u>128108</u>	<u>12</u>	
<b>Methylene chloride</b>	<u>0.22</u>	<u>12</u>	
Acetone	<u>4.4</u>	<u>8.0/u</u>	
Chloroform			
CRQL			

Blank units: Associated sample units:

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

Associated Samples:

Compound	Blank ID	Blank ID	Sample Identification
<b>Dichloro methane</b>			
<b>Methylene chloride</b>			
Acetone			
Chloroform			
CRQL			

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

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

**VALIDATION FINDINGS WORKSHEET  
Surrogate Spikes**

Page: 1 of 1  
 Reviewer: PA  
 2nd Reviewer: CA

LDC #: 18386B1  
 SDG #: free lance

**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".

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

#	Date	Sample ID	Surrogate	%Recovery (Limits)	Qualifications
		8036136 - Blank	BFB	124 ( 63-133 )	J+ / P dt
				( 66-115 )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

- SMC1 (TOL) = Toluene-d8
  - SMC2 (BFB) = Bromofluorobenzene
  - SMC3 (DCE) = 1,2-Dichloroethane-d4
  - SMC4 (DFM) = Dibromofluoromethane
- QC Limits (Soil): 81-117, 74-121, 80-120, 80-120  
 QC Limits (Water): 88-110, 86-115, 80-120, 86-118



LDC #: 18386B1  
 SDG #: Sec cover

**VALIDATION FINDINGS WORKSHEET**  
**Field Duplicates**

Page: 1 of 1  
 Reviewer: ST  
 2nd reviewer: [Signature]

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

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

Compound	Concentration ( )		Difference RPD
	3	4	
Dichloromethane	7.4	3.0	4.4 (≤ 5.4)
Toluene	0.54	5.4 u	4.86 (≤ 5.4)

Compound	Concentration ( )		RPD

Compound	Concentration ( )		RPD

Compound	Concentration ( )		RPD

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

Semivolatiles

*LDC*



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

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

**Sample Delivery Group (SDG):** F8A250221

**Sample Identification**

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

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 17 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

## III. Initial Calibration

Initial calibration was performed using required standard concentrations.

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

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

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

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

## IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

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

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

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

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

All of the continuing calibration RRF values were within method and validation criteria.

## V. Blanks

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

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

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

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

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

No field blanks were identified in this SDG.

## VI. Surrogate Spikes

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

## VII. Matrix Spike/Matrix Spike Duplicates

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

## VIII. Laboratory Control Samples (LCS)

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

## IX. Regional Quality Assurance and Quality Control

Not applicable.

## X. Internal Standards

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

## **XI. Target Compound Identifications**

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

## **XII. Compound Quantitation and CRQLs**

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

## **XIII. Tentatively Identified Compounds (TICs)**

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

## **XIV. System Performance**

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

## **XV. Overall Assessment of Data**

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

## **XVI. Field Duplicates**

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

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

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

**BRC Tronox Parcel H  
Semivolatiles - Laboratory Blank Data Qualification Summary - SDG F8A250221**

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



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

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

No Sample Data Qualified in this SDG

LDC #: 18386A2

**VALIDATION COMPLETENESS WORKSHEET**

SDG #: F8A250221

Level III/IV

Laboratory: Test America

Date: 3/11/08

Page: 1 of 1

Reviewer: [Signature]

2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 1/24/08
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	A	%RSD, $r^2$ 20.990
IV.	Continuing calibration/ICV	SW	ICV $\leq 25$
V.	Blanks	SW	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	A	LCS
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	A	Not reviewed for Level III validation.
XII.	Compound quantitation/CRQLs	A	Not reviewed for Level III validation.
XIII.	Tentatively identified compounds (TICs)	A	Not reviewed for Level III validation.
XIV.	System performance	A	Not reviewed for Level III validation.
XV.	Overall assessment of data	A	
XVI.	Field duplicates	ND	D = 11 + 12
XVII.	Field blanks	N	

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

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

D = Duplicate  
TB = Trip blank  
EB = Equipment blank

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

SOIL

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

VALIDATION FINDINGS CHECKLIST

Method: Semivolatiles (EPA SW 846 Method 8270C)

Validation Area	Yes	No	NA	Findings/Comments
<b>Technical Requirements</b>				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>DFTPP Instrument Performance Criteria</b>				
Were the DFTPP performance results reviewed and found to be within the specified criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all samples analyzed within the 12 hour clock criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>Initial Calibration</b>				
Did the laboratory perform a 5 point calibration prior to sample analysis?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a curve fit used for evaluation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did the initial calibration meet the curve fit acceptance criteria of > 0.990?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent relative standard deviations (%RSD) ≤ 30% and relative response factors (RRF) > 0.05?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>Continuing Calibration</b>				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) ≤ 25% and relative response factors (RRF) ≥ 0.05?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>Blanks</b>				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a method blank analyzed for each matrix and concentration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>Surrogate Spikes</b>				
Were all surrogate %R within QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>Matrix Spike/MS/MSD Analyses</b>				
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.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a MS/MSD analyzed every 20 samples of each matrix?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>Actual Analysis Samples</b>				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

LDC #: 18386A2  
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>IX. Regional Quality Assurance (RQA) by SARA</b>				
Were performance evaluation (PE) samples performed?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the performance evaluation (PE) samples within the acceptance limits?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>X. Internal Standards</b>				
Were internal standard area counts within -50% or +100% of the associated calibration standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were retention times within + 30 seconds from the associated calibration standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XI. Pesticide Compound Identification</b>				
Were relative retention times (RRT's) within + 0.06 RRT units of the standard?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were chromatogram peaks verified and accounted for?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XII. Compound Quantification</b>				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XIII. Reference Spectrum Comparison</b>				
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were relative intensities of the major ions within ± 20% between the sample and the reference spectra?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XIV. System Performance</b>				
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XV. Overall Assessment</b>				
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XVI. Field Data</b>				
Field duplicate pairs were identified in this SDG.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Target compounds were detected in the field duplicates.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>XVII. Field Blanks</b>				
Field blanks were identified in this SDG.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Target compounds were detected in the field blanks.	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	

# 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	TTT.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	UUU
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	VVV.
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.

LDC #: 18386A2

SDG #: precamb

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

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

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

Were all %D and RRFs within the validation criteria of  $\leq 25\%$  %D and  $\geq 0.05$  RRF?

# VALIDATION FINDINGS WORKSHEET

## Continuing Calibration

Page: 1 of 1  
Reviewer: AB  
2nd Reviewer: LC

#	Date	Standard ID	Compound	Finding %D (Limit: $\leq 25.0\%$ )	Finding RRF (Limit: $> 0.05$ )	Associated Samples	Qualifications
	2/6/08 20:45	CCV	TT (ccc)	2253152		ALL BIK	None / P

**VALIDATION FINDINGS WORKSHEET**

**Blanks**

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 method blank analyzed for each matrix?
- Y N N/A Was a method blank analyzed for each concentration preparation level?
- Y N N/A Was a method blank associated with every sample?
- Y N N/A Was the blank contaminated? If yes, please see qualification below.

Blank extraction date: 1/29/08 Blank analysis date: 2/6/08

Conc. units: ug/kg Associated Samples: All

Compound	Blank ID	Sample Identification																			
<u>Unknown</u>	<u>8029394 - Blank</u>																				
<u>Aldol concentration</u>	<u>8600 (4.25)</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>8400 (4.2676)</u>	<u>7600 (4.2526)</u>	<u>7800 (4.2534)</u>	<u>9400 (4.2519)</u>	<u>7700 (4.2572)</u>	<u>8300 (4.2632)</u>	<u>8000 (4.2583)</u>	<u>9300 (4.2472)</u>	<u>8200 (4.2524)</u>		

Blank extraction date: 1/29/08 Blank analysis date: 2/6/08

Conc. units: ug/kg Associated Samples: All

Compound	Blank ID	Sample Identification																				
<u>Unknown</u>	<u>8029394 - Blank</u>																					
<u>Aldol concentration</u>	<u>8600 (4.25)</u>	<u>10</u>	<u>11</u>	<u>12</u>	<u>13</u>	<u>14</u>	<u>15</u>	<u>9400 (4.2627)</u>	<u>9600 (4.2678)</u>	<u>8600 (4.269)</u>	<u>9200 (4.2555)</u>	<u>9300 (4.2578)</u>	<u>9100 (4.2595)</u>									

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

# VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

LDC #: 18386A2  
SDG #: for control

Page: 1 of 1  
Reviewer: B  
2nd Reviewer: t

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

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

Y N N/A 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.

Y N N/A Was a MS/MSD analyzed every 20 samples of each matrix?

Y N N/A 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
		16-17	HH	4 (10-9)	0 (10-9)	200 (30)	14	no OUAL WSL in
			PP	4 (16-99)	3 (16-99)	( )	↓	↓
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		

Compound	QC Limits (Soil)	RPD (Soil)	QC Limits (Water)	RPD (Water)	Compound	QC Limits (Soil)	RPD (Soil)	QC Limits (Water)	RPD (Water)
A. Phenol	26-90%	≤ 35%	12-110%	≤ 42%	Acenaphthene	31-137%	≤ 19%	46-118%	≤ 31%
C. 2-Chlorophenol	25-102%	≤ 50%	27-123%	≤ 40%	4-Nitrophenol	11-114%	≤ 50%	10-80%	≤ 50%
E. 1,4-Dichlorobenzene	28-104%	≤ 27%	36-97%	≤ 28%	2,4-Dinitrotoluene	28-89%	≤ 47%	24-96%	≤ 38%
J. N-Nitroso-di-n-propylamine	41-126%	≤ 38%	41-116%	≤ 38%	Pentachlorophenol	17-109%	≤ 47%	9-103%	≤ 50%
R. 1,2,4-Trichlorobenzene	38-107%	≤ 23%	39-98%	≤ 28%	Pyrene	35-142%	≤ 36%	26-127%	≤ 31%
V. 4-Chloro-3-methylphenol	26-103%	≤ 33%	23-97%	≤ 42%					



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

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:

$$RRF = (A_s/C_s)/(A_{is}/C_{is})$$

average RRF = sum of the RRFs/number of standards  
 %RSD = 100 \* (S/X)

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

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				RRF (SD std)	RRF (SD std)	RRF (SD std)	Average RRF (initial)	Average RRF (initial)	%RSD	%RSD	
1	ICAL	1/29/08	Phenol (1st internal standard)	2.66254	2.66254	2.69741	2.69741	6.25349	6.25349	6.25349	6.25349
			Naphthalene (2nd internal standard)	1.10277	1.10277	1.09527	1.09527	10.39092	10.39092	10.39092	10.39092
			Fluorene (3rd internal standard)	1.36978	1.36978	1.34878	1.34878	14.54450	14.54450	14.54450	14.54450
			Pentachlorophenol (4th internal standard)	0.15602	0.15602	0.15050	0.15050	11.84795	11.84795	11.84795	11.84795
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.80769	0.80769	0.81456	0.81456	7.06979	7.06979	7.06979	7.06979
			Benzo(a)pyrene (6th internal standard)	1.21357	1.21357	1.18939	1.18939	3.60258	3.60258	3.60258	3.60258
2			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)								
3			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 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 #: 18380A2  
 SDG #: see cover

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

Page: 1 of 1  
 Reviewer: PF  
 2nd Reviewer: W

**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 =  $100 \cdot (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$       Where:      ave. RRF = initial calibration average RRF  
 RRF =  $(A_x)(C_b) / (A_b)(C_x)$       RRF = continuing calibration RRF  
 $A_x$  = Area of compound,       $A_b$  = Area of associated internal standard  
 $C_x$  = Concentration of compound,       $C_b$  = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	PCAL4350	2/2/08	Phenol (1st internal standard)	2.6974	2.64086	2.09649	2.09649	2.09649
			Naphthalene (2nd internal standard)	1.09527	1.09989	0.42132	0.42132	0.42132
			Fluorene (3rd internal standard)	1.34878	1.34529	0.25866	0.25866	0.25866
			Pentachlorophenol (4th internal standard)	0.15050	0.18441	2.25315	2.25315	2.25315
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.81456	0.79530	2.36440	2.36440	2.36440
			Benzo(a)pyrene (6th internal standard)	1.18939	1.19972	0.86823	0.86823	0.86823
2			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)					
3			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 cover

## VALIDATION FINDINGS WORKSHEET

### Surrogate Results Verification

Page:    of     
 Reviewer:     
 2nd reviewer:   

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found  
 SS = Surrogate Spiked

Sample ID:   #5  

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	50.0	36.4457	73	73	0
2-Fluorobiphenyl		57.8084	76	76	
Terphenyl-d14	↓	46.5125	93	93	↓
Phenol-d5		54.5673	73	73	
2-Fluorophenol		52.0891	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 #: 18386A2  
 SDG #: per count

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

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

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

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

RPD =  $100 * |MS - MSD| / (MS + MSD)$       MS = Matrix spike percent recovery      MSD = Matrix spike duplicate percent recovery

MS/MSD samples: 16 + 17

Compound	Spike Added (ug/kg)		Sample Concentration (ug/kg)	Spiked Sample Concentration (ug/kg)		Matrix Spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		MS/MSD RPD	
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
Phenol	3540	2470	NP	2540	2400	72	72	69	69	5.5	5.5
N-Nitroso-di-n-propylamine				2880	2750	81	81	79	79	4.8	4.8
4-Chloro-3-methylphenol				2710	2550	77	77	73	73	6.0	6.0
Acenaphthene				2570	2370	73	73	68	68	7.9	7.9
Pentachlorophenol				581	661	16	16	19	19	13	13
Pyrene				2830	2650	80	80	76	76	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 #: 18380A2

VALIDATION FINDINGS WORKSHEET

Page: 6 of 7

SDG #: su enet

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

Reviewer: [Signature]

2nd Reviewer: [Signature]

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

LCS/LCSD samples: 8029394 - LCS

Compound	Spike Added (ug/kg)		Spike Concentration (ug/kg)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
Phenol	3330	NA	2300	NA	69	69				
N-Nitroso-di-n-propylamine			2480		74	74				
4-Chloro-3-methylphenol			2520		76	76				
Acenaphthene			2410		72	72				
Pentachlorophenol			2690		81	81				
Pyrene			2620		79	79	NA			

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 #: su cover

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd reviewer: [Signature]

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

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?

$$\text{Concentration} = \frac{(A_x)(I_s)(V_i)(DF)(2.0)}{(A_{is})(RRF)(V_o)(V_1)(\%S)}$$

- $A_x$  = Area of the characteristic ion (EICP) for the compound to be measured
- $A_{is}$  = Area of the characteristic ion (EICP) for the specific internal standard
- $I_s$  = Amount of internal standard added in nanograms (ng)
- $V_o$  = Volume or weight of sample extract in milliliters (ml) or grams (g).
- $V_i$  = Volume of extract injected in microliters (ul)
- $V_1$  = Volume of the concentrated extract in microliters (ul)
- Df = Dilution Factor.
- %S = Percent solids, applicable to soil and solid matrices only.
- 2.0 = Factor of 2 to account for GPC cleanup

Example:

Sample I.D. \_\_\_\_\_

$$\text{Conc.} = \frac{(\quad)(\quad)(\quad)(\quad)(\quad)}{(\quad)(\quad)(\quad)(\quad)(\quad)}$$

= *ND*

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

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



## **I. Technical Holding Times**

All technical holding time requirements were met.

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

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

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

## **III. Initial Calibration**

Initial calibration was performed using required standard concentrations.

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

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

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

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

## **IV. Continuing Calibration**

Continuing calibration was performed at the required frequencies.

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

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

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-HR-05-0' 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 TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
TSB-HJ-10-0'	Unknown aldol condensate (4.2487) Unknown aldol condensate (4.7401)	22000 ug/Kg 350 ug/Kg	22000U ug/Kg 350U ug/Kg
TSB-HJ-10-10'	Unknown aldol condensate (4.2597) Unknown aldol condensate (4.7565)	21000 ug/Kg 350 ug/Kg	21000U ug/Kg 350U ug/Kg
TSB-HR-06-0'	Unknown aldol condensate (4.2595) Unknown aldol condensate (4.751)	23000 ug/Kg 380 ug/Kg	23000U ug/Kg 380U ug/Kg
TSB-HR-06-0'-FD	Unknown aldol condensate (4.2594) Unknown aldol condensate (4.7509)	22000 ug/Kg 360 ug/Kg	22000U ug/Kg 360U ug/Kg
TSB-HR-06-10'	Unknown aldol condensate (4.2583) Unknown aldol condensate (4.7497)	23000 ug/Kg 370 ug/Kg	23000U ug/Kg 370U ug/Kg
TSB-HJ-08-0'	Unknown aldol condensate (4.2624) Unknown aldol condensate (4.7486)	23000 ug/Kg 380 ug/Kg	23000U ug/Kg 380U ug/Kg
TSB-HJ-08-10'	Unknown aldol condensate (4.2588) Unknown aldol condensate (4.7443)	23000 ug/Kg 380 ug/Kg	23000U ug/Kg 380U ug/Kg
TSB-HR-05-0'	Unknown aldol condensate (4.2682) Unknown aldol condensate (4.7597)	22000 ug/Kg 350 ug/Kg	22000U ug/Kg 350U ug/Kg
TSB-HR-05-10'	Unknown aldol condensate (4.247) Unknown aldol condensate (4.7438)	22000 ug/Kg 370 ug/Kg	22000U 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-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-HR-05-0' TSB-HR-05-10' RINSATE-2	N-Hydroxymethylphthalimide	J+ (all detects)	A	Continuing calibration (%D)

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

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

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

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

No Sample Data Qualified in this SDG

LDC #: 18386B2  
 SDG #: F8A290158  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III

Date: 3/6/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	Δ	Sampling dates: 1/28/08
II.	GC/MS Instrument performance check	Δ	
III.	Initial calibration	A	% RSD: r <sup>2</sup> 10.990
IV.	Continuing calibration/ICV	SW	ICV = X
V.	Blanks	SW	
VI.	Surrogate spikes	Δ	
VII.	Matrix spike/Matrix spike duplicates	<del>SW</del> A	
VIII.	Laboratory control samples	SW	
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	Δ	
XI.	Target compound identification	N	
XII.	Compound quantitation/CRQLs	N	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	N	
XV.	Overall assessment of data	Δ	
XVI.	Field duplicates	ND	D = 3+4
XVII.	Field blanks	ND	R = 10

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

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

Soil + water

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. 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	TTT.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	UUU
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	VVV.
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.





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 method blank analyzed for each matrix?
- N N/A Was a method blank analyzed for each concentration preparation level?
- N N/A Was a method blank associated with every sample?
- N N/A Was the blank contaminated? If yes, please see qualification below.

Blank extraction date: 1/31/08 Blank analysis date: 2/08/08

Conc. units: ug/kg Associated Samples: All soils

Compound	Blank ID	Sample Identification								
		1	2	3	4	5	6	7	8	9
Blank	8031299-Blank	-	-	-	-	-	-	-	-	-
unknown	1100 (3.8408)	-	-	-	-	-	-	-	-	-
unknown aldehyde condensate	2000 (4.7522)	21000 (4.2597)	23000 (4.2595)	23000 (4.2594)	23000 (4.2583)	23000 (4.2624)	23000 (4.2588)	23000 (4.2682)	23000 (4.247)	22000 (4.247)
unknown aldehyde condensate	320 (4.749)	350 (4.7401)	380 (4.751)	360 (4.7509)	370 (4.7497)	380 (4.7486)	380 (4.7443)	350 (4.7597)	370 (4.7438)	370 (4.7438)

Blank extraction date: \_\_\_\_\_ Blank analysis date: \_\_\_\_\_ Associated Samples: \_\_\_\_\_

Compound	Blank ID	Sample Identification								
		1	2	3	4	5	6	7	8	9
Blank										

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

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

LDC #: X386B2  
SDG #: see comad

Page: 1 of 1  
Reviewer: P  
2nd Reviewer: [Signature]

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

#	Date	LCS/LCSD ID	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		8029233-105	HH	75 (30-92)	( )	( )	8029233-Blank, # 10	NO QUAL MSD IN
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
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				( )	( )	( )		
				( )	( )	( )		

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

Chlorinated Pesticides

*LDC*

## Laboratory Data Consultants, Inc. Data Validation Report

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

**Sample Delivery Group (SDG):** F8A250221

### Sample Identification

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

\*\*Indicates sample underwent EPA Level IV review

## **Introduction**

This data review covers 17 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8081A for Chlorinated Pesticides.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. GC/ECD Instrument Performance Check

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

## III. Initial Calibration

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

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

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

## IV. Continuing Calibration

Continuing calibration was performed at required frequencies.

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

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



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

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

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

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

## V. Blanks

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

No field blanks were identified in this SDG.

## VI. Surrogate Spikes

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

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

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)	P

## 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:

Compound	Concentration (ug/Kg)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HJ-07-0'**	TSB-HJ-07-0'-FD				
beta-BHC	1.8U	3.4	-	1.6 ( $\leq 1.8$ )	-	-

**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-HJ-04-0' TSB-HR-04-0'** TSB-HJ-04-10' TSB-HR-07-0' TSB-HR-07-10'** TSB-HR-06-0' TSB-HR-06-10' TSB-HJ-07-0'** TSB-HJ-07-10' TSB-HR-08-0'	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)	P	Surrogate spikes (%R)

**BRC Tronox Parcel H  
Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG F8A250221**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel H  
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG F8A250221**

No Sample Data Qualified in this SDG

LDC #: 18386A3a

## VALIDATION COMPLETENESS WORKSHEET

Date: 3/10/08

SDG #: F8A250211

Level III/IV

Page: 1 of 1

Laboratory: Test America

Reviewer: [Signature]

2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	Δ	Sampling dates: 1/24/08
II.	GC/ECD Instrument Performance Check	Δ	
III.	Initial calibration	Δ	
IV.	Continuing calibration/ICV	SW	
V.	Blanks	Δ	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	A	
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	Δ	Not reviewed for Level III validation.
XII.	Compound quantitation and reported CRQLs	Δ	Not reviewed for Level III validation.
XIII.	Overall assessment of data	Δ	
XIV.	Field duplicates	SW	D = 11 & 12
XV.	Field blanks	N	

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

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

D = Duplicate  
TB = Trip blank  
EB = Equipment blank

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

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

LDC #: 18386A39  
 SDG #: su coney

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2  
 Reviewer: FR  
 2nd Reviewer: W

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

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>II. GC/ECD instrument performance check</b>				
Was the instrument performance found to be acceptable?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>III. Initial calibration</b>				
Did the laboratory perform a 5 point calibration prior to sample analysis?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) $\leq$ 20%?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Did the initial calibration meet the curve fit acceptance criteria?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the RT windows properly established?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the required standard concentrations analyzed in the initial calibration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Continuing calibration</b>				
What type of continuing calibration calculation was performed? ___ %D or ___ %R	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were Evaluation mix standards analyzed prior to the initial calibration and sample analysis?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were endrin and 4,4'-DDT breakdowns $\leq$ 15%.0 for individual breakdown in the Evaluation mix standards?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a continuing calibration analyzed daily?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) $\leq$ 15%.0 or percent recoveries 85-115%?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Were all the retention times within the acceptance windows?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a method blank analyzed for each matrix and concentration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were extract cleanup blanks analyzed with every batch requiring clean-up?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Was there contamination in the method blanks or clean-up blanks? If yes, please see the Blanks validation completeness worksheet.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>VI. Surrogate spikes</b>				
Were all surrogate %R within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	

LDC #: 18386A3a  
 SDG #: AC Cones

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

Validation Area	Yes	No	NA	Findings/Comments
<b>VII. Matrix spike/Matrix spike duplicates</b>				
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.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a MS/MSD analyzed every 20 samples of each matrix?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>VIII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IX. Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the performance evaluation (PE) samples within the acceptance limits?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>X. Target compound identification</b>				
Were the retention times of reported detects within the RT windows?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>XI. Compound quantitation/CRQLs</b>				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions, dry weight factors, and clean-up activities applicable to level IV validation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XII. System performance</b>				
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XIII. Overall assessment of data</b>				
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XIV. Field duplicates</b>				
Field duplicate pairs were identified in this SDG.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Target compounds were detected in the field duplicates.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XV. Field blanks</b>				
Field blanks were identified in this SDG.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Target compounds were detected in the field blanks.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:



**VALIDATION FINDINGS WORKSHEET**  
Continuing Calibration

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".  
 What type of continuing calibration calculation was performed?     %D or     RPD  
 Y     N     N/A  
 Were continuing calibration standards analyzed at the required frequencies?  
 Y     N     N/A  
 Did the continuing calibration standards meet the %D / RPD validation criteria of ≤15.0%?  
 Y     N     N/A

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

#	Date	Standard ID	Detector/ Column	Compound	%D / RPD (Limit <u>≤</u> 15.0)	RT (limit)	Associated Samples	Qualifications
+	2/8/08	KCAL587	RTX-CLP1	U	29.5	( )	12	J+/A det
			RTX-CLP2	U	27.4	( )	↓	↓
						( )		
						( )		
+	2/9/08	KCAL661	RTX-CLP1	<del>U</del>	75.4	( )	8029397-Blank, 12, 3, 4, 5 (1-75)	J+/A det
						( )		
						( )		
+	2/9/08	KCAL677	RTX-CLP1	U	31.6	( )	11 6-12, 16, 17 13, 14	J+/A det
						( )		
						( )		
+	2/9/08	KCAL689	RTX-CLP1	U	16.8	( )	15	J+/A det
+				L	16.0	( )		
+				e	15.2	( )		
+				R	15.6	( )		
+				N	17.6	( )		
+				Q	19.4	( )	↓	↓
						( )		
						( )		
						( )		



**VALIDATION FINDINGS WORKSHEET**  
**Surrogate Recovery**

LDC #: 18386A3  
 SDG #: per cover  
 METHOD: GC HPLC

Are surrogates required by the method? Yes     or No    .  
 Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".  
 Y/N N/A Were surrogates spiked into all samples and blanks?  
 Y/N N/A Did all surrogate recoveries (%R) meet the QC limits?

#	Sample ID	Detector/Column	Surrogate Compound	%R (Limits)	Qualifications
	2	not specified	DeB	128 ( 63-117 )	J+ / P det
	15	↓	↓	123 ( ↓ )	J+ / P det

Surrogate Compound	Surrogate Compound	Surrogate Compound	Surrogate Compound	Surrogate Compound
A Chlorobenzene (CBZ)	G Octacosane	M Benzo(e)Pyrene	S 1-Chloro-3-Nitrobenzene	Y Tetrachloro-m-xylene
B 4-Bromofluorobenzene (BFB)	H Ortho-Terphenyl	N Terphenyl-D14	T 3,4-Dinitrofluorene	
C a.a.a-Trifluoroluene	I Fluorobenzene (FBZ)	O Decachlorobiphenyl (DCB)	U Triphenylin	
D Bromochlorobenzene	J n-Triacotane	P 1-methylindanthalene	V Tri-n-octoylin	
E 1,4-Dichlorobutane	K Hexacosane	Q Dichlorophenyl Acetic Acid (DCAA)	W Tributyl Phosphate	
F 1,4-Difluorobenzene (DFB)	L Bromobenzene	R 4-Nitrophenol	X Triphenyl Phosphate	

# VALIDATION FINDINGS WORKSHEET

## Field Duplicates

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd reviewer: [Signature]

LDC #: 18386A36  
 SDG #: per cover

METHOD:  GC  HPLC

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

Compound	Concentration ( <u>ug/kg</u> )		%RPD Limit _____	Qualification Parent only / All Samples
B	1.84	3.4	Difference 1.6	

Compound	Concentration (            )		%RPD Limit _____	Qualification Parent only / All Samples

LDC #: 18 386A32  
 SDG #: file cover

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

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

METHOD: GC / HPLC

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

- CF = A/C
- average CF = sum of the CF/number of standards
- %RSD =  $100 * (S/X)$
- A = Area of compound,
- C = Concentration of compound,
- S = Standard deviation of the CF
- X = Mean of the CFs

#	Standard ID	Calibration Date	Compound	Reported		Recalculated		Reported		Recalculated	
				CF (0.67% std)	CF (0.07% std)	Average CF (Initial)	Average CF (Initial)	%RSD	%RSD		
1	KAL	2/7/08	endosulfan methoxychlor ↓ RTXcup2	568745040	568745040	572386340	572386340	6.294	6.294	6.274	6.274
				240546760	240546760	24502840	24502840	6.787	6.787	6.787	6.787
				264234520	264234520	265059470	265059470	5.585	5.585	5.585	5.585
2				53596400	53596400	55234490	55234490	4.736	4.736	4.736	4.736
3											
4											

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

LDC #: 18306A3a

SDG #: per cover

VALIDATION FINDINGS WORKSHEET  
Continuing Calibration Results Verification

Page: 1 of 1  
Reviewer: [Signature]  
2nd Reviewer: [Signature]

METHOD: GC / HPLC

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

% Difference =  $100 \cdot \frac{\text{ave. CF} - \text{CF}}{\text{ave. CF}}$  Where: ave. CF = initial calibration average CF  
CF = continuing calibration CF  
A = Area of compound  
C = Concentration of compound

#	Standard ID	Calibration Date	Compound	Average CF(ical)/ CCV Conc.	Reported		Recalculated	
					CF/Conc. CCV	%D	CF/Conc. CCV	%D
1	KCAL659	2/9/08	endosulfan ↓ methoxychlor	0.0250	4.9	0.0262	4.9	
				↓	7.4	0.0269	7.4	
	KCAL675	2/9/08	RTX-cur-1 ↓	↓	10.0	0.0275	10.0	
2					9.8	0.0274	9.8	
3								
4								

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

LDC #: 18306A3a  
 SDG #: per cover

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

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

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	<u>BFX-CPI</u>	<u>0.02</u>	<u>0.02117</u>	<u>106</u>	<u>106</u>	<u>106</u>
Decachlorobiphenyl	<u>↓</u>	<u>↓</u>	<u>0.02272</u>	<u>114</u>	<u>114</u>	<u>114</u>
Decachlorobiphenyl						

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

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

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

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

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

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

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

LDC #: 18386 A3a  
 SDG #: per control

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

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

**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 =  $100 * (SSC - SC) / SA$       Where: SSC = Spiked sample concentration      SC = Concentration  
 SA = Spike added

RPD =  $|MS - MSD| * 2 / (MS + MSD)$       MS = Matrix spike percent recovery      MSD = Matrix spike duplicate percent recovery

MS/MSD samples: 16 + 17

Compound	Spike Added (ug/kg)		Sample Concentration (ug/kg)	Spiked Sample Concentration (ug/kg)		Matrix Spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		MS/MSD RPD	
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
gamma-BHC	17.6	17.3	0	18.6	17.1	106	106	98	98	8.9	8.9
4,4'-DDT	↓	17.3	0	21.4	19.1	122	122	110	110	11	11

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 #: 18286A3a  
 SDG #: per control

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

Page: 6 of 7  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

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

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

Where: SSC = Spiked sample concentration  
 SA = Spike added  
 SC = Concentration

RPD =  $|(LCS - LCSD) / ((LCS + LCSD) / 2)| * 100$

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

LCS/LCSD samples: LCS

Compound	Spike Added (ug/L)		Spiked Sample Concentration (ug/L)		LCS		LCSD		LCS		LCSD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
gamma-BHC	16.7	NA	17.2	NA	103	103	NA	NA				
4,4'-DDT	↓	↓	18.7	↓	112	112	NA	NA				

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 #: 18386 A3a  
SDG #: pu cover

### VALIDATION FINDINGS WORKSHEET

#### Sample Calculation Verification

Page: 1 of 1  
Reviewer: [Signature]  
2nd reviewer: [Signature]

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

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?

Example:

Sample I.D. \_\_\_\_\_:

Conc. = ( \_\_\_\_\_ )  
( \_\_\_\_\_ )

= **ND**

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

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. GC/ECD Instrument Performance Check

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

## III. Initial Calibration

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

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

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

## IV. Continuing Calibration

Continuing calibration was performed at required frequencies.

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

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

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

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

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

## V. Blanks

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

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

## VI. Surrogate Spikes

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

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

## **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-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-HR-05-0'	All TCL compounds	J+ (all detects)	P	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  
 SDG #: F8A290158  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**

Level III

Date: 3/6/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

**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	A	Sampling dates: 1/28/08
II.	GC/ECD Instrument Performance Check	A	
III.	Initial calibration	A	
IV.	Continuing calibration/ICV	A SW SW	
V.	Blanks	A	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	A	
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

Validated Samples:

Soil + Water

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' <i>W</i>	13		23		33
4	TSB-HR-06-0'-FD <i>D</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 <i>W</i>	20		30		40

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:



# VALIDATION FINDINGS WORKSHEET

## Surrogate Recovery

Page: 1 of 2

Reviewer: AD

2nd Reviewer: [Signature]

LDC #: 18386 B3a

SDG #: per control

METHOD:  GC  HPLC

Are surrogates required by the method? Yes  or No

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

N  A Were surrogates spiked into all samples and blanks?

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

#	Sample ID	Detector/Column	Surrogate Compound	%R (Limits)	Qualifications
1		not specified	Y	119 ( 55-115 ) 126 ( 63-117 )	J+/Pdut ↓
2		↓	⊖	126 ( 63-117 )	J+/Pdut
3		↓	⊖	121 ( ↓ )	J+/Pdut
4		↓	Y	122 ( 55-115 ) 127 ( 63-117 )	J+/Pdut ↓
5		↓	↓	121 ( ↓ ) 129 ( ↓ )	↓
6		↓	↓	126 ( ↓ ) 132 ( ↓ )	J+/Pdut
7		↓	↓	119 ( ↓ ) 123 ( ↓ )	J+/Pdut
8		↓	↓	124 ( ↓ ) 130 ( ↓ )	J+/Pdut
Surrogate Compound	Surrogate Compound	Surrogate Compound	Surrogate Compound	Surrogate Compound	Surrogate Compound
A Chlorobenzene (CBZ)	G Octacosane	M Benzo(e)Pyrene	S 1-Chloro-3-Nitrobenzene	Y Tetrachloro-m-xylene	
B 4-Bromofluorobenzene (BFB)	H Ortho-Terphenyl	N Terphenyl-D14	T 3,4-Dinitrotoluene		
C a,a-Trifluorotoluene	I Fluorobenzene (FBZ)	O Decachlorobiphenyl (DCB)	U Triphenyltin		
D Bromochlorobenzene	J n-Triacontane	P 1-methylindanthalene	V Tri-n-propyltin		
E 1,4-Dichlorobutane	K Hexacosane	Q Dichlorophenyl Acetic Acid (DCAA)	W Tributyl Phosphate		
F 1,4-Difluorobenzene (DFB)	L Bromobenzene	R 4-Nitrophenol	X Triphenyl Phosphate		



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

Polychlorinated Biphenyls

*LDC*

## 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

\*\*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.
- UU 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

LDC #: 18386A3b  
 SDG #: F8A250221  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 3/7/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 1/24/08
II.	GC/ECD Instrument Performance Check	NA	
III.	Initial calibration	A	
IV.	Continuing calibration/ICV	A	ICV ≤ 15
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	SW	LCS
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	
Xb.	GPC Calibration	N	
XI.	Target compound identification	A	Not reviewed for Level III validation.
XII.	Compound quantitation and reported CRQLs	A	Not reviewed for Level III validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	ND	D = 11 + 12
XV.	Field blanks	N	

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

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

sell

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

LDC #: 18386A3b  
 SDG #: you cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

Method: / GC          HPLC

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>II. Initial calibration</b>				
Did the laboratory perform a 5 point calibration prior to sample analysis?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Did the initial calibration meet the curve fit acceptance criteria?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the RT windows properly established?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Continuing calibration</b>				
What type of continuing calibration calculation was performed? ____%D or ____%R	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a continuing calibration analyzed daily?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all the retention times within the acceptance windows?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a method blank analyzed for each matrix and concentration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>VI. Surrogate spikes</b>				
Were all surrogate %R within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>VII. Matrix spike/Matrix spike duplicates</b>				
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.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a MS/MSD analyzed every 20 samples of each matrix?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>VIII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	

LDC #: 18386A36  
 SDG #: su cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

Validation Area	Yes	No	NA	Findings/Comments
<b>IX. Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?			/	
Were the performance evaluation (PE) samples within the acceptance limits?			/	
<b>X. Target compound identification</b>				
Were the retention times of reported detects within the RT windows?			/	
<b>XI. Compound quantitation/CRQLs</b>				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
<b>XII. System performance</b>				
System performance was found to be acceptable.				
<b>XIII. Overall assessment of data</b>				
Overall assessment of data was found to be acceptable.	/			
<b>XIV. Field duplicates</b>				
Field duplicate pairs were identified in this SDG.	/			
Target compounds were detected in the field duplicates.		/		
<b>XV. Field blanks</b>				
Field blanks were identified in this SDG.		/		
Target compounds were detected in the field blanks.			/	



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

LDC #: 18386A3b  
SDG #: per cover  
METHOD:  GC  HPLC

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

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

#	LCS/LCSD ID	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
	8029396-LCS	✓	118 (80-116)	( )	( )	All + BK	1+ P det no CTL MS IP in
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		

LDC #: 18386A3b  
 SDG #: for cover

**VALIDATION FINDINGS WORKSHEET**  
Initial Calibration Calculation Verification

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

METHOD: GC            HPLC           

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

- CF = A/C
- average CF = sum of the CF/number of standards
- %RSD = 100 \* (S/X)
- A = Area of compound,
- C = Concentration of compound,
- S = Standard deviation of the CF
- X = Mean of the CFs

#	Standard ID	Calibration Date	Compound	Reported		Recalculated		Reported		Recalculated	
				CF (500 std)	CF (50 std)	Average CF (Initial)	Average CF (Initial)	%RSD	%RSD		
1	14A L	9/6/07	Arachol 1260 -   RTX-CLP2	12182	12182	11502	11502	9.589	9.589	9.589	9.589
			Arachol 1260 -   RTX-CLP2	11265	11265	11104	11104	8.799	8.799	8.799	8.799
2											
3											
4											

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

LDC #: 18386A36  
 SDG #: fu coner

**VALIDATION FINDINGS WORKSHEET**  
Continuing Calibration Results Verification

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

METHOD: GC HPLC

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

% Difference =  $100 * (\text{ave. CF} - \text{CF}) / \text{ave. CF}$   
 CF = A/C

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

#	Standard ID	Calibration Date	Compound	Average CF (Ical)/ CCV Conc.	Reported		Recalculated	
					CF/Conc. CCV	%D	CF/Conc. CCV	%D
1	<del>HEA</del> CCV	9/6/07 1/31/08	Araclor 1260-1 R1X cnp 1 Araclor 1260-1 R1X cnp 2	1006.6919 1098.2936	0.7 9.8	1026.69 1098.29	0.7 9.8	
2								
3								
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.

DC #: 18386A3b  
 SDG #: per cover

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

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd reviewer: [Signature]

METHOD: GC HPLC

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

Where: SF = Surrogate Found  
 SS = Surrogate Spiked

% Recovery: SF/SS \* 100

Sample ID: #5

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
DCB	ATX CNPI	20	19.7627	99	99	0

Sample ID:

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

Sample ID:

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

LDC #: 18386 A36  
 SDG #: EV cover

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

Page: 1 of 2  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

METHOD: GC HPLC

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

$\% \text{Recovery} = 100 * ((\text{SSC} - \text{SC}) / \text{SA})$  Where SSC = Spiked sample concentration, SC = Sample concentration, SA = Spike added  
 $\text{RPD} = (((\text{SSCMS} - \text{SSCMSD}) * 2) / (\text{SSCMS} + \text{SSCMSD})) * 100$  MS = Matrix spike, MSD = Matrix spike duplicate  
 MS/MSD samples: 16 + 17

Compound	Spike Added (ug/kg)		Sample Conc. (ug/kg)	Spike Sample Concentration (ug/kg)		Matrix spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		MS/MSD RPD	
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)			---								
Diesel (8015)											
Benzene (8021B)											
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)											
Anthracene (8310)											
HMX (8330)											
2,4,6-Trinitrotoluene (8330)											
Arochlor 1260	176	174	0	212	211	120	121	121	121	0.90	0.5

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.

METHOD: GC  HPLC

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

$$\% \text{Recovery} = 100 * (\text{SSC} - \text{SC}) / \text{SA} \quad \text{Where} \quad \text{SSC} = \text{Spiked concentration} \quad \text{SA} = \text{Spike added} \quad \text{SC} = \text{Sample concentration}$$

$$\text{RPD} = \left( \frac{|\text{SSCLCS} - \text{SSCLCSD}| * 2}{\text{SSCLCS} + \text{SSCLCSD}} \right) * 100$$

LCS = Laboratory Control Sample percent recovery      LCS = Laboratory Control Sample duplicate percent recovery

LCS/LCSD samples: 8 029 3916 - LC >

Compound	Spike Added		Sample Conc.		Spike Sample Concentration		LCS		LCSD		LCS/LCSD	
	LCS	LCSD	LCS	LCSD	LCS	LCSD	Percent Recovery	Recalc.	Percent Recovery	Recalc.	Reported	Recalc.
							Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)												
Diesel (8015)												
Benzene (8021B)												
Methane (RSK-175)												
2,4-D (8151)												
Dinoseb (8151)												
Naphthalene (8310)												
Anthracene (8310)												
HMX (8330)												
2,4,6-Trinitrotoluene (8330)												
Aroclor 1260	167	NA	0	NA	179	NA	107	107	NA	107	NA	

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

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

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

LDC #: 18386A3b  
 SDG #: [Signature]

METHOD: GC HPLC

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 within 10% of the reported results?

Concentration = 
$$\frac{(A)(Fv)(Df)}{(RF)(Vs \text{ or } Ws)(\%S/100)}$$

Example:

Sample ID: \_\_\_\_\_ Compound Name \_\_\_\_\_

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

RF= Average response factor of the compound  
 In the initial calibration

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

Concentration = \_\_\_\_\_

NP

#	Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications

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

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. GC/ECD Instrument Performance Check

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

## III. Initial Calibration

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

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

## IV. Continuing Calibration

Continuing calibration was performed at required frequencies.

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

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

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)	P

## 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  
 SDG #: F8A290158  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III

Date: 3/6/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

**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: 1/28/08
II.	GC/ECD Instrument Performance Check	NA	
III.	Initial calibration	Δ	
IV.	Continuing calibration/ICV	SW	ICV ≤ 15
V.	Blanks	A SW	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	SW	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	Δ	
XIV.	Field duplicates	ND	D = 3 + 4
XV.	Field blanks	ND	R = 10

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

Validated Samples:

Soil + water

1	TSB-HJ-10-0'	11	TSB-HR-05-10'MS	21	8029346-B/K	31	
2	TSB-HJ-10-10'	12	TSB-HR-05-10'MSD	22	8031455-B/K	32	
3	TSB-HR-06-0' D	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. alpha-BHC	I. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	GG.
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH.
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	II.
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	JJ.
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. DB 608	KK.
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. DB 1701	LL.
G. Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	EE.	MM.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	FF.	NN.

Notes:

**VALIDATION FINDINGS WORKSHEET**  
Continuing Calibration

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? 16-X %D or RPD  
 Y  N  N/A Were continuing calibration standards analyzed at the required frequencies?  
 Y  N  N/A Did the continuing calibration standards meet the %D / RPD validation criteria of ≤15.0%?  
Level IV Only  
 Y  N  N/A Were the retention times for all calibrated compounds within their respective acceptance windows?

#	Date	Standard ID	Detector/Column	Compound	%D / RPD (Limit ≤ 15.0)	RT (limit)	Associated Samples	Qualifications
+	2/4/08	PCA151	ATX-CP1	V	16-X	( )	1-4	J <sup>+</sup> /A det QUAL V W X
						( )		
						( )		
						( )		
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**BRC Tronox Parcel H  
Data Validation Reports  
LDC# 18386**

**Metals**

*LDC*

## Laboratory Data Consultants, Inc. Data Validation Report

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

**Sample Delivery Group (SDG):** F8A250221

### Sample Identification

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

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 17 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Methods 6010B, 6020, and 7000 for Metals. The metals analyzed were Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Lithium, Magnesium, Manganese, Molybdenum, Mercury, Nickel, Niobium, Palladium, Phosphorus, Platinum, Potassium, Selenium, Silicon, Silver, Sodium, Strontium, Sulfur, Thallium, Tin, Titanium, Tungsten, Uranium, Vanadium, Zinc, and Zirconium.

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

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

Blanks are summarized in Section III.

Field duplicates are summarized in Section XIII.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UU 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 Lithium	0.099 mg/Kg 14.7 mg/Kg	0.26U mg/Kg 21.0U mg/Kg
TSB-HR-04-10'	Niobium	5.5 mg/Kg	6.6U mg/Kg
TSB-HJ-04-0'	Cadmium Niobium Lithium	0.10 mg/Kg 4.8 mg/Kg 10.7 mg/Kg	0.55U mg/Kg 5.5U mg/Kg 21.8U mg/Kg
TSB-HR-04-0'***	Cadmium Niobium Tungsten Lithium	0.076 mg/Kg 3.3 mg/Kg 0.29 mg/Kg 14.3 mg/Kg	0.13U mg/Kg 5.2U mg/Kg 1.3U mg/Kg 20.9U mg/Kg
TSB-HJ-04-10'	Boron Cadmium Niobium	15.3 mg/Kg 0.064 mg/Kg 5.7 mg/Kg	53.2U mg/Kg 0.27U mg/Kg 6.7U mg/Kg
TSB-HR-07-0'	Cadmium Niobium Lithium	0.069 mg/Kg 4.0 mg/Kg 17.3 mg/Kg	0.27U mg/Kg 5.4U mg/Kg 21.4U mg/Kg
TSB-HR-07-10'***	Niobium	4.4 mg/Kg	6.7U mg/Kg
TSB-HR-06-0'	Cadmium Niobium Lithium	0.11 mg/Kg 3.8 mg/Kg 12.0 mg/Kg	0.28U mg/Kg 5.5U mg/Kg 22.2U mg/Kg
TSB-HR-06-10'	Cadmium Niobium	0.068 mg/Kg 3.4 mg/Kg	0.55U mg/Kg 5.5U mg/Kg
TSB-HJ-07-0'***	Niobium Lithium	3.4 mg/Kg 10.2 mg/Kg	5.4U mg/Kg 21.7U mg/Kg
TSB-HJ-07-0'-FD	Cadmium Niobium Lithium	0.081 mg/Kg 3.5 mg/Kg 12.0 mg/Kg	0.27U mg/Kg 5.3U mg/Kg 21.2U mg/Kg
TSB-HJ-07-10'	Niobium Lithium	3.3 mg/Kg 20.6 mg/Kg	5.4U mg/Kg 21.5U mg/Kg
TSB-HR-08-0'	Cadmium Niobium Lithium	0.099 mg/Kg 3.1 mg/Kg 9.4 mg/Kg	0.27U mg/Kg 5.3U mg/Kg 21.2U mg/Kg
TSB-HR-08-10'	Cadmium Niobium	0.076 mg/Kg 4.9 mg/Kg	0.55U 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	60.6 (75-125)	54.7 (75-125)	-	J- (all detects) UJ (all non-detects)	A
	Phosphorus	31.3 (75-125)	62.5 (75-125)	-	J- (all detects) UJ (all non-detects)	
TSB-HR-08-0'MS/MSD (All samples in SDG F8A250221)	Barium	173.6 (75-125)	150.6 (75-125)	-	J+ (all detects)	A
	Calcium	335.9 (75-125)	411.7 (75-125)	-	J+ (all detects)	
	Chromium	144.1 (75-125)	134.7 (75-125)	-	J+ (all detects)	
	Lead	150.5 (75-125)	-	-	J+ (all detects)	
	Magnesium	160.5 (75-125)	-	-	J+ (all detects)	
	Niobium	190.9 (75-125)	186.9 (75-125)	-	J+ (all detects)	
	Silicon	281.6 (75-125)	225.0 (75-125)	-	J+ (all detects)	
	Strontium	169.6 (75-125)	160.2 (75-125)	-	J+ (all detects)	
	Vanadium	146.9 (75-125)	137.0 (75-125)	-	J+ (all detects)	
	Zinc	131.4 (75-125)	-	-	J+ (all detects)	

#### 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)	P

### 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:

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

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

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

Analyte	Concentration (ug/Kg)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HJ-07-0**	TSB-HJ-07-0'-FD				
Mercury	20.2	7.1U	-	13.1 ( $\leq 36.1$ )	-	-

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

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

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

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

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

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

No Sample Data Qualified in this SDG

LDC #: 18386A4  
 SDG #: F8A250221  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**

Level III/IV

Date: 3/5/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 1/24/08
II.	Calibration	A	
III.	Blanks	SW	
IV.	ICP Interference Check Sample (ICS) Analysis	A	
V.	Matrix Spike Analysis	SW	3MS/MSD
VI.	Duplicate Sample Analysis	N	
VII.	Laboratory Control Samples (LCS)	SW	LCS
VIII.	Internal Standard (ICP-MS)	A	Not reviewed for level 3
IX.	Furnace Atomic Absorption QC	N	N.T Utilized
X.	ICP Serial Dilution	A	
XI.	Sample Result Verification	A	Not reviewed for Level III validation.
XII.	Overall Assessment of Data	A	
XIII.	Field Duplicates	SW	(11, 12)
XIV.	Field Blanks	N	

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

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

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

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



LDC #: 18786A4  
 SDG #: see cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2  
 Reviewer: MH  
 2nd Reviewer: gmg

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

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	✓			
Cooler temperature criteria was met.	✓			
<b>II. Calibration</b>				
Were all instruments calibrated daily, each set-up time?	✓			
Were the proper number of standards used?	✓			
Were all initial and continuing calibration verification %Rs within the 90-110% (80-120% for mercury and 85-115% for cyanide) QC limits?	✓			
Were all initial calibration correlation coefficients > 0.995? (Level IV only)	✓			
<b>III. Blanks</b>				
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.	✓			
<b>IV. ICP Interference Check Sample</b>				
Were ICP interference check samples performed daily?	✓			
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?	✓			
<b>IV. Matrix spike/Matrix spike/duplicates</b>				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	✓			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.		✓		
Were the MS/MSD or duplicate relative percent differences (RPD) < 20% for waters and < 35% for soil samples? A control limit of +/- RL (+/-2X RL for soil) was used for samples that were < 5X the RL, including when only one of the duplicate sample values were < 5X the RL.		✓		
<b>V. Laboratory control samples</b>				
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 80-120% QC limits for water samples and laboratory established QC limits for soils?		✓		
<b>VI. Furnace Atomic Absorption QC</b>				
If MSA was performed, was the correlation coefficients > 0.995?			✓	
Do all applicable analyses 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 #: 18386A4  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: MM  
 2nd Reviewer: ML

Validation Area	Yes	No	NA	Findings/Comments
<b>VII. ICP Serial Dilution</b>				
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the IDL?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<u>&gt; 10<sup>5</sup> x 442 for 200/μg</u>
Were all percent differences (%Ds) < 10%?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>VIII. Internal Standards (EPA SW 845 Method 6020)</b>				
Were all the percent recoveries (%R) within the 30-120% of the intensity of the internal standard in the associated initial calibration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
If the %Rs were outside the criteria, was a reanalysis performed?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>IX. Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the performance evaluation (PE) samples within the acceptance limits?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>X. Sample Result Verification</b>				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XI. Overall assessment of data</b>				
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XII. Field duplicates</b>				
Field duplicate pairs were identified in this SDG.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Target analytes were detected in the field duplicates.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XIII. Field blanks</b>				
Field blanks were identified in this SDG.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Target analytes were detected in the field blanks.	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	

LDC #: 18386A4  
 SDG #: See cover

### VALIDATION FINDINGS WORKSHEET

#### Sample Specific Element Reference

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd reviewer: [Signature]

All circled elements are applicable to each sample.

Sample ID	Matrix	Target Analyte List (TAL)
1-15	Soil	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
2-16,17	Soil	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
1-15	Soil	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
2-16,17	Soil	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		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,
<b>Analysis Method</b>		
ICP		Li, S,
ICP-MS		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si,
ICP-MS		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Zr,
GEAA		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,

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

Analyte	Maximum PB <sup>a</sup> (mg/Kg)	Maximum PB <sup>a</sup> (ug/L)	Maximum ICB/CCB <sup>a</sup> (ug/L)	Blank Action Limit	Sample Identification															
					1	2	3	4	5	6	7	8	9	10						
Al	3.1																			
B	2.4														15.3 / 53.2					
Ca	14.0																			
Cd	0.33		0.036		0.063 / 0.54	0.099 / 0.26			0.10 / 0.55	0.076 / 0.13	0.064 / 0.27	0.069 / 0.27							0.11 / 0.28	0.068 / 0.55
Cr			0.5																	
Co			0.5																	
Fe	5.6																			
Ni			0.5																	
Nb	1.4		6.8				5.5 / 6.6	4.8 / 5.5	3.3 / 5.2	5.7 / 6.7	4.0 / 5.4	4.4 / 6.7							3.8 / 5.5	3.4 / 5.5
P	3.0																			
K	3.9																			
Na	8.2																			
Tl			0.4																	
Ti	0.067		0.6																	
W			0.9							0.29 / 1.3										
Li			7.6			14.7 / 21.0		10.7 / 21.8	14.3 / 20.9		17.3 / 21.4								12.0 / 22.2	

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.

Analyte	Maximum PB* (mg/Kg)	Maximum PB* (ug/L)	Maximum ICB/CCB* (ug/L)	Blank Action Limit	Sample Identification															
					11	12	13	14	15											
Al	3.1																			
B	2.4																			
Ca	14.0																			
Cd			0.036			0.081 / 0.27					0.099 / 0.27									
Cr	0.33		0.5																	
Co			0.5																	
Fe	5.6																			
Ni			0.5																	
Nb	1.4		6.8			3.4 / 5.4	3.5 / 5.3	3.3 / 5.4	3.1 / 5.3	4.9 / 5.5										
P	3.0																			
K	3.9																			
Na	8.2																			
Tl			0.4																	
Ti	0.067		0.6																	
W			0.9																	
Li			7.6			10.2 / 21.7	12.0 / 21.2	20.6 / 21.5	9.4 / 21.2											

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

LDC #: 18386A4  
 SDG #: See lower

Page: 1 of 1  
 Reviewer: mm  
 2nd Reviewer: SM

**VALIDATION FINDINGS WORKSHEET**  
**Matrix Spike/Matrix Spike Duplicates**

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  N/A Were matrix spike percent recoveries (%R) within the control limits of 75-125? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.
- Y  N  N/A Were all duplicate sample relative percent differences (RPD)  $\leq$  20% for water samples and  $\leq$  35% for soil samples?
- Y  N  N/A Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations.

#	MS/MSD ID	Matrix	Analyte	MS %Recovery	MSD %Recovery	RPD (Limits)	Associated Samples	Qualifications
1	16/17	601	Sb	60.6	54.7		A1	J-1/J/A
			Be	173.6	150.6			J-1/J/A
			Ca	335.9	411.7			
			Cd	144.1	134.7			
			Pb	150.5				
			Mg	160.5				
			Mn	190.9	186.9			
			P	31.3	62.5			J-1/J/A
			Si	281.6	225.0			J-1/J/A
			Sr	169.6	160.2			
			V	146.9	137.0			
			Zn	131.4				
			Mn			12.9 (±20)		Ns found (Location)

Comments: See lower

LDC #: 18386A4

SDG #: See cover

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

Page: 1 of 1

Reviewer: MH

2nd Reviewer: MNA

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

#	LCS ID	Matrix	Analyte	%R (limits)	Associated Samples	Qualifications
1	LCS	Soil	Pd Pb	120.5 (80-120) 121.9 ✓	A11 ✓	FF Jt/p

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

LDC#: 18386A4  
 SDG#: See Cover

**VALIDATION FINDINGS WORKSHEET**  
**Field Duplicates**

Page: 1 of 2  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

Y N NA  
Y N NA

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

Compound	Concentration (mg/kg)		(<50) RPD	(mg/Kg) Difference	(mg/Kg) Limits	Qualifications (Parent Only)
	11	12				
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**  
**Field Duplicates**

Page: 2 of 2  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

Y N NA  
Y N NA

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

Compound	Concentration (mg/kg)		(<=50) RPD	(mg/Kg) Difference	(mg/Kg) Limits	Qualifications (Parent Only)
	11	12				
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)	

LDC #: 18386A4  
 SDG #: See case

**VALIDATION FINDINGS WORKSHEET**  
**Initial and Continuing Calibration Calculation Verification**

Page: 1 of 1  
 Reviewer: M4  
 2nd Reviewer: M4

**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:

$\%R = \frac{\text{Found} \times 100}{\text{True}}$  Where, Found = concentration (in ug/L) of each analyte measured in the analysis of the ICV or CCV solution  
 True = concentration (in ug/L) of each analyte in the ICV or CCV source

Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	Recalculated		Reported		Acceptable (Y/N)
					%R	%R	%R	%R	
ICV	ICP (Initial calibration)	S	3960	4000	99.1	99.1	99.1	99.1	Y
	GFAA (Initial calibration)								
ICV	CVAA (Initial calibration)	Hg	2.51	2.5	100.4	100.4	100.4	100.4	Y
CCV	ICP (Continuing calibration)	Li	3995	4000	99.9	99.9	99.9	99.9	Y
	GFAA (Continuing calibration)								
CCV	CVAA (Continuing calibration)	Hg	5.06	5.0	101.2	101.2	101.2	101.2	Y
ICV	ICP/MS (Initial calibration)	Sn	201.35	200	100.7	100.7	100.7	100.7	Y
CCV	ICP/MS (Continuing calibration)	Cu	216.73	200	108.4	108.4	108.4	108.4	Y

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 #: 18386A4  
 SDG #: see com

Page: 1 of 1  
 Reviewer: AW  
 2nd Reviewer: SMZ

**VALIDATION FINDINGS WORKSHEET**  
Level IV Recalculation Worksheet

**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:

$\%R = \frac{\text{Found} \times 100}{\text{True}}$  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:

$RPD = \frac{|S-D|}{(S+D)/2} \times 100$  Where, S = Original sample concentration  
 D = Duplicate sample concentration

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

$\%D = \frac{|I-SDR|}{I} \times 100$  Where, I = Initial Sample Result (mg/L)  
 SDR = Serial Dilution Result (mg/L) (Instrument Reading x 5)

Sample ID	Type of Analysis	Element	Found / S / I (units)	True / D / SDR (units)	Recalculated		Reported		Acceptable (Y/N)
					%R / RPD / %D	%R / RPD / %D			
IC6A5	ICP interference check	Sb	97.93	100	97.9	97.9	97.9	97.9	Y
LC9	Laboratory control sample	Li	215.4	200	107.7	107.7	107.7	107.7	Y
16	Matrix spike	Mo	58.586 (SSR-SR)	53.079	110.4	110.3	110.3	110.3	Y
16/17	Duplicate	Hg	182	186	2.2	2.2	1.9	1.9	Y
14	ICP serial dilution	As	5777.43	555.05	4.0	4.0	4.0	4.0	Y

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

LDC #: 1838644  
 SDG #: sel cov

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

Page: 1 of 2  
 Reviewer: my  
 2nd reviewer: gmk

**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?
- N N/A 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?

Detected analyte results for 5 were recalculated and verified using the following equation:

Concentration =  $\frac{(RD)(FV)(Dil)}{(In. Vol.)(\%S)}$

Recalculation:

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

$$Mn = \frac{861.2 \mu g/L \times 0.12 \times 2}{0.58 \times 0.959} = 359.2 \text{ mg/kg}$$

Sample ID	Analyte	Reported Concentration (mg/kg)	Calculated Concentration (mg/kg)	Acceptable (Y/N)
5	Li	14.3	14.3	Y
	Al	7860	7860	
	As	1.3	1.3	
	Ba	145	145	
	Be	0.55	0.55	
	Cd	0.076	0.076	
	Ca	12600	12600	
	Cr	10.4	10.4	
	Co	6.5	6.5	
	Cu	16.4	16.4	
	Fe	12700	12700	
	Pb	7.0	7.0	
	Mg	7460	7460	
	Mn	359	359	
	Mo	0.41	0.40	
	Ni	17.3	17.3	
	Nb	3.3	3.3	
	Pd	0.25	0.25	
	P	1010	1000	
	K	1840	1840	
	Si	112	112	
	Na	203	203	Y

LDC #: 18386AL  
 SDG #: 6el cover

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

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

**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 Have results been reported and calculated correctly?  
 Y  N  N/A Are results within the calibrated range of the instruments and within the linear range of the ICP?  
 Y  N  N/A Are all detection limits below the CRDL?

Detected analyte results for 5 were recalculated and verified using the following equation:

Concentration =  $\frac{(RD)(FV)(Dil)}{(In. Vol.)(\%S)}$

Recalculation:

$$V = \frac{84.7394 \times 0.12 \times 2}{0.58 \times 0.959} = 35.34 \text{ mg/kg}$$

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

Sample ID	Analyte	Reported Concentration (mg/kg)	Calculated Concentration (mg/kg)	Acceptable (Y/N)
5	Sr	122	122	Y
	Sn	0.084	0.084	Y
	Ti	526	526	Y
	W	0.29	0.29	Y
	U	0.90	0.90	Y
	V	35.3	35.3	Y
	Zn	31.7	31.7	Y
	Hg (mg/kg)	13.6	13.6	Y

**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.
- UU 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)	P
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)	P
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)	P
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-HR-05-0' PBS	J+ (all detects)	P

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

Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-HJ-10-0'	Boron Cadmium Silver Tungsten	10.4 mg/Kg 0.094 mg/Kg 0.072 mg/Kg 0.74 mg/Kg	26.5U mg/Kg 0.13U mg/Kg 0.53U mg/Kg 1.3U mg/Kg
TSB-HJ-10-10'	Boron Cadmium Niobium Silver Tin Tungsten	6.7 mg/Kg 0.090 mg/Kg 4.7 mg/Kg 0.092 mg/Kg 0.50 mg/Kg 0.46 mg/Kg	26.2U mg/Kg 0.13U mg/Kg 6.6U mg/Kg 0.52U mg/Kg 0.52U mg/Kg 1.3U mg/Kg
TSB-HR-06-0'	Boron Niobium Silver Tungsten	4.6 mg/Kg 2.1 mg/Kg 0.090 mg/Kg 0.36 mg/Kg	26.1U mg/Kg 6.5U mg/Kg 0.52U mg/Kg 1.3U mg/Kg
TSB-HR-06-0'-FD	Boron Cadmium Silver Tin	3.9 mg/Kg 0.094 mg/Kg 0.094 mg/Kg 0.46 mg/Kg	27.0U mg/Kg 0.14U mg/Kg 0.54U mg/Kg 0.54U mg/Kg
TSB-HR-06-10'	Boron Cadmium Silver Tin Tungsten	5.2 mg/Kg 0.077 mg/Kg 0.10 mg/Kg 0.47 mg/Kg 0.35 mg/Kg	26.6U mg/Kg 0.13U mg/Kg 0.53U mg/Kg 0.53U mg/Kg 1.3U mg/Kg
TSB-HJ-08-0'	Boron Cadmium Silver Tin Tungsten	4.6 mg/Kg 0.10 mg/Kg 0.11 mg/Kg 0.52 mg/Kg 0.28 mg/Kg	27.0U mg/Kg 0.14U mg/Kg 0.54U mg/Kg 0.54U mg/Kg 1.4U mg/Kg
TSB-HJ-08-10'	Boron Cadmium Silver Tin Tungsten	5.3 mg/Kg 0.10 mg/Kg 0.11 mg/Kg 0.50 mg/Kg 0.33 mg/Kg	27.0U mg/Kg 0.14U mg/Kg 0.54U mg/Kg 0.54U mg/Kg 1.4U mg/Kg
TSB-HR-05-0'	Cadmium Sodium	0.14 mg/Kg 138 mg/Kg	0.54U mg/Kg 218U mg/Kg
TSB-HR-05-10'	Boron Cadmium Silver Tin Tungsten	4.9 mg/Kg 0.071 mg/Kg 0.11 mg/Kg 0.50 mg/Kg 0.27 mg/Kg	26.8U mg/Kg 0.13U mg/Kg 0.54U mg/Kg 0.54U 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 0.51 ug/L 1.0 ug/L 0.67 ug/L	All soil samples in SDG F8A290158

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

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

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

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

The frequency of analysis was met.

The criteria for analysis were met.

#### V. Matrix Spike Analysis

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

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
TSB-HR-05-10'MS/MSD (All soil samples in SDG F8A290158)	Antimony	54.5 (75-125)	57.9 (75-125)	-	J- (all detects) UJ (all non-detects)	A
TSB-HR-05-10'MS/MSD (All soil samples in SDG F8A290158)	Barium	41.2 (75-125)	4.8 (75-125)	-	J- (all detects) R (all non-detects)	A
TSB-HR-05-10'MS/MSD (All soil samples in SDG F8A290158)	Niobium Palladium Magnesium	169.4 (75-125) 127.7 (75-125) -	210.0 (75-125) 128.3 (75-125) 131 (75-125)	- - -	J+ (all detects) J+ (all detects) J+ (all detects)	A

#### VI. Duplicate Sample Analysis

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

#### VII. Laboratory Control Samples (LCS)

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

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

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

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

Raw data were not reviewed for this SDG.

### IX. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

### X. ICP Serial Dilution

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

Diluted Sample	Analyte	%D (Limits)	Associated Samples	Flag	A or P
TSB-HR-05-10'L	Manganese Strontium	10.1 ( $\leq 10$ ) 10.6 ( $\leq 10$ )	All soil samples in SDG F8A290158	J (all detects) J (all detects)	A

### XI. Sample Result Verification

Raw data were not reviewed for this SDG.

### XII. Overall Assessment of Data

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

### XIII. Field Duplicates

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

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

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

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

Analyte	Concentration (ug/Kg)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HR-06-0'	TSB-HR-06-0'-FD				
Mercury	7.0U	9.5	-	2.5 ( $\leq 36.0$ )	-	-



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

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

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

### BRC Tronox Parcel H

### Metals - Laboratory Blank Data Qualification Summary - SDG F8A290158

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

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

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

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

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

LDC #: 18386B4  
 SDG #: F8A290158  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III

Date: 3/5/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 1/28/08
II.	Calibration	SW	
III.	Blanks	SW	
IV.	ICP Interference Check Sample (ICS) Analysis	A	
V.	Matrix Spike Analysis	SW	3 MS/MSD
VI.	Duplicate Sample Analysis	N	
VII.	Laboratory Control Samples (LCS)	SW	LCS
VIII.	Internal Standard (ICP-MS)	N	not reviewed
IX.	Furnace Atomic Absorption QC	N	not utilized
X.	ICP Serial Dilution	SW	
XI.	Sample Result Verification	N	
XII.	Overall Assessment of Data	A	
XIII.	Field Duplicates	SW	(3,4)
XIV.	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: All soil except # 10, 13, 14 Az

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

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

**VALIDATION FINDINGS WORKSHEET**  
**Sample Specific Element Reference**

All circled elements are applicable to each sample.

Sample ID	Matrix	Target Analyte List (TAL)
1-10	Soil/A	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si
2-11, 12	Soil	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si
1-13, 14	A2	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si
1-10	Soil/A	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr
2-11, 12	Soil	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr
1-13, 14	A2	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr
Analysis Method		
ICP		U.S.
ICP-MS		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si
ICP-MS		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Zr
GEAA		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN

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

LDC #: 18286B4  
 SDG #: see cover

# VALIDATION FINDINGS WORKSHEET

## Calibration

Page: 1 of 1  
 Reviewer: MM  
 2nd Reviewer: MA

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

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

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

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%)?

**LEVEL IV ONLY:**

Y  N  N/A Was a midrange cyanide standard distilled?

Y  N  N/A Are all correlation coefficients  $\geq 0.995$ ?

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

#	Date	Calibration ID	Analyte	%R	Associated Samples	Qualification of Data
1	2/5/08	ccv (1028)	Ag	111.4	PBW	J+ J+/p
2	2/5/08	ccv (2153)	B	112.3	AN MA	J
			Nb	111.8	↓	↓
			Ag	112.6		
3	2/6/08	ccv (1477)	Ag	112.7	1-7, PBS	J+ J+/p
4	2/6/08	ccv (357)	Ag	112.4	8,9,11,12	↓
5	2/6/08	ccv (859)	Pd	113.6	1-8, <del>PBS</del> PBS	J+ J+/p

Comments:

Analyte	Maximum PB <sup>a</sup> (mg/Kg)	Maximum PB <sup>a</sup> (ug/L)	Maximum ICB/CCB <sup>a</sup> (ug/L)	Blank Action Limit	Sample Identification														
					1	2	3	4	5	6	7	8	9						
Al	1.9																		
Ba	0.052																		
B			10.6		10.4 / 26.5	6.7 / 26.2	4.6 / 26.1	3.9 / 27.0	5.2 / 26.6	4.6 / 27.0	5.3 / 27.0								4.9 / 26.8
Cd			0.1		0.094 / 0.13	0.090 / 0.13		0.094 / 0.14	0.077 / 0.13	0.10 / 0.14	0.10 / 0.14	0.10 / 0.14	0.14 / 0.54	0.071 / 0.13					
Cr	0.15																		
Nb			6.1			4.7 / 6.6	2.1 / 6.5												
P	1.4																		
K	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	0.11 / 0.54	0.11 / 0.54					0.11 / 0.54
Na	3.4																		138 / 218
Tl	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	0.50 / 0.54	0.50 / 0.54					0.50 / 0.54
Ti	0.077		0.9																
W			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	0.33 / 1.4	0.27 / 1.3						
U																			
Zn	1.3																		

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**

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

**Y**  **N**  **N/A** Were field blanks identified in this SDG?

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

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

**Sampling date:** 1/28/08 **Soil factor applied:** 200X

**Field blank type:** (circle one) Field Blank / Rinsate / Other: R **Associated Samples:** All Soil

Analyte	Blank ID	Sample Identification																		
		1	2	3	4	5	6	7	8	9										
	10	Action Level																		
Cd	0.027	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										
Ca	72.3																			
Fe	32.9																			
Mg	9.2																			
Nb	6.3		4.7 / 6.6	2.1 / 6.5																
Na	21.0								138 / 218											
Sr	0.67																			
Sn	0.51		0.50 / 0.52		0.46 / 0.54	0.47 / 0.53	0.52 / 0.54	0.50 / 0.54		0.50 / 0.54										
Ti	1.0																			
W	0.67	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										

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".

LDC #: 8386A4  
 SDG #: see cover

Page: 1 of 1  
 Reviewer: SM  
 2nd Reviewer: SM

**VALIDATION FINDINGS WORKSHEET**  
Matrix Spike/Matrix Spike Duplicates

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 N/A Were matrix spike percent recoveries (%R) within the control limits of 75-125? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.
- Y N N/A Were all duplicate sample relative percent differences (RPD)  $\leq 20\%$  for water samples and  $\leq 35\%$  for soil samples?
- Y N N/A Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations.

#	MS/MSD ID	Matrix	Analyte	MS %Recovery	MSD %Recovery	RPD (Limits)	Associated Samples	Qualifications
1	11/12	Soil	Sb	54.5	57.9		All soil	J-R/A
			Ba	41.2	4.8			J-R/A
			As	169.4	20.0			J-R/A
			Pd	127.7	128.3			J-R/A
			Mg		131			↓
			Mn			44.2 (±20)		No quat (LCC m)
			Nb			21.4		↓
			Sr			25.5		↓

Comments: Hg = MS/MSD for As, a non-client sample.





LDC#: 18386B4  
 SDG#: See Cover

**VALIDATION FINDINGS WORKSHEET**  
**Field Duplicates**

Page: 1 of 2  
 Reviewer: [Signature]  
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**METHOD:** Metals (EPA Method 6010B/6020/7000)

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

Compound	Concentration (mg/kg)		(<50) RPD	(mg/Kg) Difference	(mg/Kg) Limits	Qualifications (Parent Only)
	3	4				
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#: 18386B4  
 SDG#: See Cover

**VALIDATION FINDINGS WORKSHEET**  
**Field Duplicates**

Page: 2 of 2  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

Y N NA  
Y N NA

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

Compound	Concentration (mg/kg)		(<=50) RPD	(mg/Kg) Difference	(mg/Kg) Limits	Qualifications (Parent Only)
	3	4				
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)	

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

Wet Chemistry

*LDC*



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

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

**Sample Delivery Group (SDG):** F8A250221

**Sample Identification**

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

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 20 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 300.0 for Bromide, Bromine, Chlorate, Chloride, Chlorine, 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.
- UU 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)	A

## V. Duplicates

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

## VI. Laboratory Control Samples

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

## VII. Sample Result Verification

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:

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

Analyte	Concentration (ug/Kg)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HJ-07-0'***	TSB-HJ-07-0'-FD				
Perchlorate	8.6	12.8	-	4.2 ( $\leq 10.8$ )	-	-

**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-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'	Oil & grease	J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8A250221	TSB-HJ-07-0'** TSB-HJ-07-0'-FD	Chloride Chlorine	J (all detects) J (all detects)	A	Field duplicates (Difference)

**BRC Tronox Parcel H  
Wet Chemistry - Laboratory Blank Data Qualification Summary - SDG F8A250221**

No Sample Data Qualified in this SDG

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

No Sample Data Qualified in this SDG

LDC #: 18386A6  
 SDG #: F8A250221  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**

Level III/IV

Date: 3/5/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 1/24/08
IIa.	Initial calibration	A	
IIb.	Calibration verification	A	
III.	Blanks	A	
IV	Matrix Spike/Matrix Spike Duplicates	SW	} MS/MSD/DUP
V	Duplicates	A	
VI.	Laboratory control samples	A	LS/LSD
VII.	Sample result verification	A	Not reviewed for Level III validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	SW	(11, 12)
X	Field blanks	N	

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

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

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

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

LDC #: 18386A6  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2  
 Reviewer: mm  
 2nd Reviewer: MA

Method: Inorganics (EPA Method See cover)

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	✓			
Cooler temperature criteria was met.	✓			
<b>II. Calibration</b>				
Were all instruments calibrated daily, each set-up time?	✓			
Were the proper number of standards used?	✓			
Were all initial calibration correlation coefficients > 0.995?	✓			
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?	✓			
Were titrant checks performed as required? (Level IV only)			✓	
Were balance checks performed as required? (Level IV only)	✓			
<b>III. Blanks</b>				
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.		✓		
<b>IV. Matrix spike/Matrix spike duplicates and Duplicates</b>				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	✓			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.	✓	✓		
Were the MS/MSD or duplicate relative percent differences (RPD) ≤ 20% for waters and ≤ 35% for soil samples? A control limit of ≤ CRDL (≤ 2X CRDL for soil) was used for samples that were < 5X the CRDL, including when only one of the duplicate sample values were < 5X the CRDL.	✓			
<b>V. Laboratory control samples</b>				
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 80-120% (85-115% for Method 300.0) QC limits?	✓			
<b>VI. Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?			✓	
Were the performance evaluation (PE) samples within the acceptance limits?			✓	



LDC #: 1838646  
 SDG #: Sed cover

VALIDATION FINDINGS CHECKLIST

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 Reviewer: MM  
 2nd Reviewer: MM

Validation Area	Yes	No	NA	Findings/Comments
<b>VII. Sample Result Verification</b>				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	✓			
Were detection limits < RL?	✓			
<b>VIII. Parallel Sampling Data</b>				
Overall assessment of data was found to be acceptable.	✓			
Field duplicate pairs were identified in this SDG.	✓			
Target analytes were detected in the field duplicates.	✓			
Field blanks were identified in this SDG.		✓		
Target analytes were detected in the field blanks.			✓	





LDC#: 18386A6  
SDG#: See Cover

**VALIDATION FINDINGS WORKSHEET**  
**Field Duplicates**

Page: ( of )  
Reviewer: [Signature]  
2nd Reviewer: [Signature]

Inorganics, Method: See Cover

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

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

V:\FIELD DUPLICATES\FD\_inorganic\18386A6.wpd

LDC #: 18386M6  
 SDG #: See cover

**Validatin Findings Worksheet**  
**Initial and Continuing Calibration Calculation Verification**

Page: 1 of 1  
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 2nd Reviewer: [Signature]

**Method:** Inorganics, Method See cover  
 The correlation coefficient (r) for the calibration of ClO4 was recalculated. Calibration date: 1/20/08

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

%R =  $\frac{\text{Found} \times 100}{\text{True}}$   
 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

Type of analysis	Analyte	Standard	Conc. (ug/L)	Area	Recalculated		Reported		Acceptable (Y/N)
					r	r <sup>2</sup>	r	r <sup>2</sup>	
Initial calibration	ClO4	s1	1	0.00316	0.9999372	0.999867			Y
		s2	2.5	0.00918					
		s3	5	0.01935					
		s4	10	0.04027					
		s5	20	0.07784					
		s6	40	0.15704					
Calibration verification	ClO4	30	28.0		93.3	NR		NR	Y
Calibration verification	Chlorate	4000	3990.6		99.8	NR		NR	Y
Calibration verification	Br	2000	2029.0		101.45	101.45		101.45	Y

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 #: 18386A6  
 SDG #: See cover

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

Page: 1 of 1  
 Reviewer: my  
 2nd Reviewer: AKA

METHOD: Inorganics, Method See cover

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

$\%R = \frac{\text{Found}}{\text{True}} \times 100$  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:

$RPD = \frac{|S-D|}{(S+D)/2} \times 100$  Where, S = Original sample concentration  
 D = Duplicate sample concentration

Sample ID	Type of Analysis	Element	Found / S (units)	True / D (units)	Recalculated		Reported		Acceptable (Y/N)
					%R / RPD	%R / RPD			
19	Laboratory control sample	NO <sub>3</sub> -N	4.056	4.00	101	101	71	101	Y
20	Matrix spike sample	OTG	1.008 (SSR-SR)	1.420	71	71	1.3	<del>1.3</del>	Y
	Duplicate sample	ce	4.02	4.08	1.5				Y

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



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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. Calibration

### a. Initial Calibration

All criteria for the initial calibration of each method were met.

### b. Calibration Verification

Calibration verification frequency and analysis criteria were met for each method when applicable.

## III. Blanks

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

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

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

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

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

## IV. Matrix Spike/Matrix Spike Duplicates

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

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

## V. Duplicates

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

## VI. Laboratory Control Samples

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

## VII. Sample Result Verification

Raw data were not reviewed for this SDG.

## VIII. Overall Assessment of Data

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

## IX. Field Duplicates

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

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

Analyte	Concentration (ug/Kg)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HR-06-0'	TSB-HR-06-0'-FD				
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	A

LDC #: 18386B6  
 SDG #: F8A290158  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III

Date: 3/5/08  
 Page: 1 of 1  
 Reviewer: WJ  
 2nd Reviewer: MMA

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

907/B / ZPA 1664A

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 1/28/08
IIa.	Initial calibration	A	
IIb.	Calibration verification	A	
III.	Blanks	A	
IV	Matrix Spike/Matrix Spike Duplicates	SW	3 MS/MSD/DUP
V	Duplicates	A	
VI.	Laboratory control samples	A	LCS/LCSD
VII.	Sample result verification	N	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	SW	(3,4)
X	Field blanks	SW	R=10

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

Validated Samples: All (5.1) extent # 10, 18-20 A2

1	TSB-HJ-10-0'	11	TSB-HJ-10-0'MS	21	MS	31	
2	TSB-HJ-10-10'	12	TSB-HJ-10-0'MSD	22		32	
3	TSB-HR-06-0'	13	TSB-HJ-10-0'DUP	23		33	
4	TSB-HR-06-0'-FD	14	TSB-HJ-08-10'MS	24		34	
5	TSB-HR-06-10'	15	TSB-HJ-08-10'DUP	25		35	
6	TSB-HJ-08-0'	16	TSB-HR-05-10'MS	26		36	
7	TSB-HJ-08-10'	17	TSB-HR-05-10'DUP	27		37	
8	TSB-HR-05-0'	18	RINSATE-2MS A2	28		38	
9	TSB-HR-05-10'	19	RINSATE-2MSD	29		39	
10	RINSATE-2 A2	20	RINSATE-2DUP	30		40	

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





LDC #: 188866  
 SDG #: See cover

# VALIDATION FINDINGS WORKSHEET

## Field Blanks

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

METHOD: Inorganics, EPA Method See cover  
 Were field blanks identified in this SDG?  
 Were target analytes detected in the field blanks?  
 Blank units: mg/L Associated sample units: mg/kg  
 Sampling date: 11/28/08 Soil factor applied  
 Field blank type: (circle one) Field Blank / Rinsate / Other: R Associated Samples: All SDG

Analyte	Blank ID	Blank Action Limit	Sample Identification																			
SO4	10																					
	0.10																					

Blank units: \_\_\_\_\_ Associated sample units: \_\_\_\_\_  
 Sampling date: \_\_\_\_\_ Soil factor applied \_\_\_\_\_  
 Field blank type: (circle one) Field Blank / Rinsate / Other: \_\_\_\_\_ Associated Samples: \_\_\_\_\_

Analyte	Blank ID	Blank Action Limit	Sample Identification																			

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".



LDC#: 18386B6  
SDG#: See Cover

**VALIDATION FINDINGS WORKSHEET**  
**Field Duplicates**

Page: 1 of 1  
Reviewer: [Signature]  
2nd Reviewer: [Signature]

Inorganics, Method: See Cover

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

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

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

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

Gasoline Range Organics

*LDC*

## Laboratory Data Consultants, Inc. Data Validation Report

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

**Sample Delivery Group (SDG):** F8A250221

### Sample Identification

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

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 19 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8015 for Gasoline Range Organics.

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

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. Calibration

### a. Initial Calibration

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

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

### b. Calibration Verification

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

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

## III. Blanks

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

No field blanks were identified in this SDG.

## IV. Accuracy and Precision Data

### a. Surrogate Recovery

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

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



## **b. Matrix Spike/Matrix Spike Duplicates**

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

## **c. Laboratory Control Samples**

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

## **V. Target Compound Identification**

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

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

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

## **VII. System Performance**

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

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

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

## **IX. Field Duplicates**

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

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

<b>SDG</b>	<b>Sample</b>	<b>Compound</b>	<b>Flag</b>	<b>A or P</b>	<b>Reason</b>
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

LDC #: 18386A7  
 SDG #: F8A250221  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**

Level III/IV

Date: 3/10/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 1/24/08
IIa.	Initial calibration	A	
IIb.	Calibration verification/ICV	A	ICV = 15
III.	Blanks	ND	
IVa.	Surrogate recovery	SW	
IVb.	Matrix spike/Matrix spike duplicates	SW	
IVc.	Laboratory control samples	A	LCS
V.	Target compound identification	A	Not reviewed for Level III validation.
VI.	Compound Quantitation and CRQLs	A	Not reviewed for Level III validation.
VII.	System Performance	A	Not reviewed for Level III validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	ND	0 = 11 + 12
X.	Field blanks	N	

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

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

901

1 3	TSB-HJ-05-10'	11 1	TSB-HJ-07-0**	21 1	8031129-BLK	31	1/31
2 3	TSB-HJ-05-0'	12 2	TSB-HJ-07-0'-FD	22 2	8030151-BLK	32	1/30
3 3	TSB-HR-04-10'	13 2	TSB-HJ-07-10'	23 3	8030149-BLK	33	1/29
4 3	TSB-HJ-04-0'	14 2	TSB-HR-08-0'	24	-	34	
5 2	TSB-HR-04-0**	15 2	TSB-HR-08-10'	25		35	
6 2	TSB-HJ-04-10'	16 3	TSB-HJ-05-10'MS	26		36	
7 2	TSB-HR-07-0'	17 3	TSB-HJ-05-10'MSD	27		37	
8 2	TSB-HR-07-10**	18 2	TSB-HR-08-0'MS	28		38	
9 2	TSB-HR-06-0'	19 2	TSB-HR-08-0'MSD	29		39	
10 2	TSB-HR-06-10'	20		30		40	

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

LDC #: 18386A7  
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

Method:  GC  HPLC

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>II. Initial calibration</b>				
Did the laboratory perform a 5 point calibration prior to sample analysis?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Did the initial calibration meet the curve fit acceptance criteria?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the RT windows properly established?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Continuing calibration</b>				
What type of continuing calibration calculation was performed? ___%D or %R	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a continuing calibration analyzed daily?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all the retention times within the acceptance windows?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a method blank analyzed for each matrix and concentration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>VI. Surrogate spikes</b>				
Were all surrogate %R within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>VII. Matrix spike/Matrix spike duplicates</b>				
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.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a MS/MSD analyzed every 20 samples of each matrix?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>VIII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

LDC #: 18386A7  
 SDG #: see cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

Validation Area	Yes	No	NA	Findings/Comments
<b>IX. Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?			/	
Were the performance evaluation (PE) samples within the acceptance limits?			/	
<b>X. Target compound identification</b>				
Were the retention times of reported detects within the RT windows?			/	
<b>XI. Compound quantitation/CRQLs</b>				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
<b>XII. System performance</b>				
System performance was found to be acceptable.	/			
<b>XIII. Overall assessment of data</b>				
Overall assessment of data was found to be acceptable.	/			
<b>XIV. Field duplicates</b>				
Field duplicate pairs were identified in this SDG.	/			
Target compounds were detected in the field duplicates.		/		
<b>XV. Field blanks</b>				
Field blanks were identified in this SDG.		/		
Target compounds were detected in the field blanks.			/	





LDC #: 18386A7  
 SDG #: for control

**VALIDATION FINDINGS WORKSHEET**  
Initial Calibration Calculation Verification

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

METHOD: GC        HPLC       

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

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

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

#	Standard ID	Calibration Date	Compound	Reported		Recalculated		Reported		Recalculated	
				CF (1.0 std)	CF (1.0 std)	Average CF (Initial)	Average CF (Initial)	%RSD	%RSD		
1	1CAL	1/9/08	gasoline Range Organics	15189023	15189023	15699364	15699364	8.667	8.667	8.667	8.667
2											
3											
4											

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



LDC #: 18 386A7  
SDG #: fu cover

**VALIDATION FINDINGS WORKSHEET**  
Continuing Calibration Results Verification

Page: 1 of 1  
Reviewer: [Signature]  
2nd Reviewer: [Signature]

METHOD: GC HPLC

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

% Difference =  $100 \cdot (\text{ave. CF} - \text{CF}) / \text{ave. CF}$     Where: ave. CF = initial calibration average CF  
CF = A/C    CF = continuing calibration CF  
A = Area of compound  
C = Concentration of compound

#	Standard ID	Calibration Date	Compound	Average CF(ave)/ CCV Conc.	Reported		Recalculated	
					CF/Conc. CCV	%D	CF/Conc. CCV	%D
1	CV	1/30/8	GRU	1.0	0.9486	5.1	0.9486	5.1
	10930							
2	CV	1/30/8	↓	1.0	0.929138	8.6	0.9138	8.6
	14:54							
3								
4								

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

LDC #: 18386A7  
 SDG #: per cover

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

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd reviewer: [Signature]

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: B-5

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery		Percent Difference
				Reported	Recalculated	
TFI	not spiked	0.04	0.0228	56	57	1

Sample ID:

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

Sample ID:

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

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

METHOD: GC HPLC

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

$$\% \text{Recovery} = 100 * (\text{SSC} - \text{SC}) / \text{SA}$$

Where

SSC = Spiked sample concentration

SA = Spike added

MS = Matrix spike

SC = Sample concentration

MSD = Matrix spike duplicate

$$\text{RPD} = \frac{((\text{SSC} - \text{SSC}(\text{MSD}) * 2) / (\text{SSC}(\text{MS} + \text{SSC}(\text{MSD}))) * 100$$

MS/MSD samples: 183109

Compound	Spike Added (mg)		Sample Conc. (mg)	Spike Sample Concentration (mg)		Matrix spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		MS/MSD RPD	
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)	1.06	1.07	0	0.175	0.994	17	17	94	94	140	140
Diesel (8015)			0.175	0.175							
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 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 #: 18386 A7  
SDG #: fu cover

VALIDATION FINDINGS WORKSHEET

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Reviewer: [Signature]  
2nd Reviewer: [Signature]

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

METHOD: GC HPLC

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

%Recovery =  $100 \cdot ((SSC - SC)/SA)$  Where SSC = Spiked concentration SA = Spike added SC = Sample concentration

RPD =  $(|(SSCLCS - SSCLCSD) \cdot 2| / ((SSCLCS + SSCLCSD))) \cdot 100$

LCS = Laboratory Control Sample percent recovery LCS/D = Laboratory Control Sample duplicate percent recovery

LCS/LCSD samples: 8030151 - LCS

Compound	Spike Added (mg/kg)		Sample Conc. (mg/kg)	Spike Sample Concentration (mg/kg)		LCS		LCS/D	
	LCS	LCS/D		LCS	LCS/D	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)	1.0	NA	0	0.92	NA	92	92	NA	NA
Diesel (8015)									
Benzene (8021B)									
Methane (RSK-175)									
2,4-D (8151)									
Dinoseb (8151)									
Naphthalene (8310)									
Anthracene (8310)									
HMX (8330)									
2,4,6-Trinitrotoluene (8330)									

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

LDC #: 18386A7  
SDG #: flu cover

# VALIDATION FINDINGS WORKSHEET

## Sample Calculation Verification

Page: 1 of 1  
Reviewer: [Signature]  
2nd Reviewer: [Signature]

METHOD: GC HPLC

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 within 10% of the reported results?

Concentration =  $\frac{A(Fv)(Df)}{(RF)(Vs \text{ or } Ws)(\%S/100)}$

Example:

Sample ID: \_\_\_\_\_ Compound Name \_\_\_\_\_

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

RF= Average response factor of the compound  
in the initial calibration

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

Concentration = ND

#	Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications

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

## 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.
- UU 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)	P



### **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**

<b>SDG</b>	<b>Sample</b>	<b>Compound</b>	<b>Flag</b>	<b>A or P</b>	<b>Reason</b>
F8A290158	RINSATE-2	Gasoline range organics	J+ (all detects)	P	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

LDC #: 18386B7  
 SDG #: F8A290158  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III

Date: 3/6/08  
 Page: 1 of 1  
 Reviewer: P  
 2nd Reviewer: [Signature]

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

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

Validation Area			Comments
I.	Technical holding times	A	Sampling dates: <u>1/28/08</u>
IIa.	Initial calibration	A	
IIb.	Calibration verification/ICV	A	<u>ICV ≤ 15</u>
III.	Blanks	A <del>SW</del>	
IVa.	Surrogate recovery	SW	
IVb.	Matrix spike/Matrix spike duplicates	A	
IVc.	Laboratory control samples	A	<u>LCS</u>
V.	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	N	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	ND	<u>D = 3 + 4</u>
X.	Field blanks	ND	<u>R = 10</u>

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:

soil + water

1	TSB-HJ-10-0'	11	TSB-HR-05-10'MS	21	<u>8037078</u>	31	
2	TSB-HJ-10-10'	12	TSB-HR-05-10'MSD	22	<u>8037174</u>	32	
3	TSB-HR-06-0'	13	RINSATE-2MS <u>W</u>	23	<u>8039035</u>	33	
4	TSB-HR-06-0'-FD	14	RINSATE-2MSD <u>W</u>	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 <u>W</u>	20		30		40	

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



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

Diesel Range Organics

*LDC*

## 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

\*\*Indicates sample underwent EPA Level IV review

## Introduction

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

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

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. Calibration

### a. Initial Calibration

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

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

### b. Calibration Verification

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

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

## III. Blanks

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

No field blanks were identified in this SDG.

## IV. Accuracy and Precision Data

### a. Surrogate Recovery

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

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



## **b. Matrix Spike/Matrix Spike Duplicates**

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

## **c. Laboratory Control Samples**

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

## **V. Target Compound Identification**

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

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

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

## **VII. System Performance**

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

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

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

## **IX. Field Duplicates**

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

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

<b>SDG</b>	<b>Sample</b>	<b>Compound</b>	<b>Flag</b>	<b>A or P</b>	<b>Reason</b>
F8A250221	TSB-HR-06-10' TSB-HR-08-10'	Diesel range organics	J- (all detects) UJ (all non-detects)	A	Surrogate spikes (%R)

**BRC Tronox Parcel H  
Diesel Range Organics - Laboratory Blank Data Qualification Summary - SDG  
F8A250221**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel H  
Diesel Range Organics - Field Blank Data Qualification Summary - SDG  
F8A250221**

No Sample Data Qualified in this SDG

LDC #: 18386A8  
 SDG #: F8A250221  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 3/7/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: <u>1/24/08</u>
IIa.	Initial calibration	Δ	
IIb.	Calibration verification/ICV	Δ	<u>ICV ≤ 15</u>
III.	Blanks	Δ	<u>8</u>
IVa.	Surrogate recovery	SW	
IVb.	Matrix spike/Matrix spike duplicates	Δ	
IVc.	Laboratory control samples	A	<u>LCS</u>
V.	Target compound identification	Δ	Not reviewed for Level III validation.
VI.	Compound Quantitation and CRQLs	Δ	Not reviewed for Level III validation.
VII.	System Performance	Δ	Not reviewed for Level III validation.
VIII.	Overall assessment of data	Δ	
IX.	Field duplicates	NP	<u>P = 11 + 12</u>
X.	Field blanks	N	

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

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

SOIL

1	TSB-HJ-05-10'	11	TSB-HJ-07-0***	D	21	<u>8029395</u>	31	
2	TSB-HJ-05-0'	12	TSB-HJ-07-0'-FD	D	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	

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

LDC #: 18386 AS  
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

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Method: GC HPLC

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>II. Initial calibration</b>				
Did the laboratory perform a 5 point calibration prior to sample analysis?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Did the initial calibration meet the curve fit acceptance criteria?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the RT windows properly established?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Continuing calibration</b>				
What type of continuing calibration calculation was performed? ___%D or %R	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a continuing calibration analyzed daily?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all the retention times within the acceptance windows?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a method blank analyzed for each matrix and concentration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>VI. Surrogate spikes</b>				
Were all surrogate %R within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>VII. Matrix spike/Matrix spike duplicates</b>				
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.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a MS/MSD analyzed every 20 samples of each matrix?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>VIII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

LDC #: 18 386A8  
 SDG #: su cover

VALIDATION FINDINGS CHECKLIST

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 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

Validation Area	Yes	No	NA	Findings/Comments
<b>IX. Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?			<input checked="" type="checkbox"/>	
Were the performance evaluation (PE) samples within the acceptance limits?			<input checked="" type="checkbox"/>	
<b>X. Target compound identification</b>				
Were the retention times of reported detects within the RT windows?			<input checked="" type="checkbox"/>	
<b>XI. Compound quantitation/CRQLs</b>				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>			
<b>XII. System performance</b>				
System performance was found to be acceptable.	<input checked="" type="checkbox"/>			
<b>XIII. Overall assessment of data</b>				
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>			
<b>XIV. Field duplicates</b>				
Field duplicate pairs were identified in this SDG.	<input checked="" type="checkbox"/>			
Target compounds were detected in the field duplicates.		<input checked="" type="checkbox"/>		
<b>XV. Field blanks</b>				
Field blanks were identified in this SDG.		<input checked="" type="checkbox"/>		
Target compounds were detected in the field blanks.			<input checked="" type="checkbox"/>	



LDC #: 18386A8  
 SDG #: see cover

# VALIDATION FINDINGS WORKSHEET

## Initial Calibration Calculation Verification

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

METHOD: GC / HPLC

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

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

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

#	Standard ID	Calibration Date	Compound	Reported		Recalculated		Reported		Recalculated	
				CF (SD std)	CF (SD std)	Average CF (Initial)	Average CF (Initial)	%RSD	%RSD		
1	1CAL	1/31/08	Diene	16699	16699	17265	17265	10.435	10.435	10.435	10.435
2											
3											
4											

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

# VALIDATION FINDINGS WORKSHEET

## Continuing Calibration Results Verification

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

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

% Difference =  $100 * (\text{ave. CF} - \text{CF}) / \text{ave. CF}$  Where: ave. CF = initial calibration average CF  
 CF = A/C  
 CF = continuing calibration CF  
 A = Area of compound  
 C = Concentration of compound

#	Standard ID	Calibration Date	Compound	Average CF (cal)/ CCV Conc.	Reported		Recalculated	
					CF/Conc. CCV	%D	CF/Conc. CCV	%D
1	FCAL868	1/31/08	Ding	1000.00	871.965	12.8	871.965	12.8
	879	2/01/08	↓		980.1386	2.0	980.1386	2.0
2	887	2/01/08	↓	1000	1006.5768	0.7	1006.5768	0.7
3								
4								

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



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

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd reviewer: [Signature]

LDC #: 18386A8  
 SDG #: per conrol  
 METHOD: GC HPLC

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

Where: SF = Surrogate Found  
 SS = Surrogate Spiked

% Recovery: SF/SS \* 100

Sample ID: 5

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
o-terphenyl	not spiked	25	19.3267	77	77	0

Sample ID:

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

Sample ID:

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

LDC #: 18386 AG  
SDG #: fu cover

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

Page: 1 of 1  
Reviewer: [Signature]  
2nd Reviewer: [Signature]

**METHOD:** GC HPLC

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

%Recovery =  $100 * (SSC - SC) / SA$       Where      SSC = Spiked sample concentration      SC = Sample concentration  
 RPD =  $\frac{((SSCMS - SSCMSD) * 2) / (SSCMS + SSCMSD)}{SA} * 100$       MS = Matrix spike      MSD = Matrix spike duplicate

MS/MSD samples: 16 + 17

Compound	Spike Added (mg/kg)		Sample Conc. (mg/kg)	Spike Sample Concentration (mg/kg)		Matrix spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		MS/MSD RPD	
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)											
Diesel (8015)	86.7	88.2	0	77.8	82.4	90	90	94	94	50	5.0
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 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 #: 18386A8  
SDG #: fu cover

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

Page: 1 of 1  
Reviewer: [Signature]  
2nd Reviewer: [Signature]

METHOD: GC HPLC

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

$\% \text{Recovery} = 100 * (\text{SSC} - \text{SC}) / \text{SA}$       Where       $\text{SSC} = \text{Spiked concentration}$        $\text{SC} = \text{Sample concentration}$   
 $\text{SA} = \text{Spike added}$

$\text{RPD} = \frac{((\text{SSCLCS} - \text{SSCLCSD}) * 2) / ((\text{SSCLCS} + \text{SSCLCSD})) * 100}$

LCS = Laboratory Control Sample percent recovery      LCSLCS = Laboratory Control Sample duplicate percent recovery

LCS/LCSD samples: 8029395-LCS

Compound	Spike Added (mg/kg)		Sample Conc. (mg/kg)	Spike Sample Concentration (mg/kg)		LCS		LCSLCS	
	LCS	LCSLCS		LCS	LCSLCS	Percent Recovery Reported	Recalc.	Percent Recovery Reported	Recalc.
Gasoline (8015)									
Diesel (8015)	83.3	NA	0	76.5	NA	92	92	NA	
Benzene (8021B)									
Methane (RSK-175)									
2,4-D (8151)									
Dinoseb (8151)									
Naphthalene (8310)									
Anthracene (8310)									
HMX (8330)									
2,4,6-Trinitrotoluene (8330)									

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

LDC #: 18386A8  
SDG #: *for cover*

# VALIDATION FINDINGS WORKSHEET

## Sample Calculation Verification

Page: 1 of 1  
Reviewer: *[Signature]*  
2nd Reviewer: *[Signature]*

METHOD:  GC  HPLC

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 within 10% of the reported results?

Concentration =  $\frac{(A)(FV)(Df)}{(RF)(Vs \text{ or } Ws)(\%S/100)}$

A = Area or height of the compound to be measured  
FV = Final Volume of extract  
Df = Dilution Factor  
RF = Average response factor of the compound  
    In the initial calibration  
Vs = Initial volume of the sample  
Ws = Initial weight of the sample  
%S = Percent Solid

Example:  
Sample ID: \_\_\_\_\_ Compound Name: \_\_\_\_\_

Concentration = \_\_\_\_\_  
*ND*

#	Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications

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

**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.
- UU 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)	P

### **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**

<b>SDG</b>	<b>Sample</b>	<b>Compound</b>	<b>Flag</b>	<b>A or P</b>	<b>Reason</b>
F8A290158	TSB-HR-05-10'	Diesel range organics	J- (all detects) UJ (all non-detects)	A	Surrogate spikes (%R)

**BRC Tronox Parcel H  
 Diesel Range Organics - Laboratory Blank Data Qualification Summary - SDG  
 F8A290158**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel H  
 Diesel Range Organics - Field Blank Data Qualification Summary - SDG  
 F8A290158**

No Sample Data Qualified in this SDG

LDC #: 18386B8  
 SDG #: F8A290158  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III

Date: 3/6/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

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

Validation Area			Comments
I.	Technical holding times	A	Sampling dates: <u>1/28/08</u>
IIa.	Initial calibration	A	
IIb.	Calibration verification/ICV	A	<u>ICV = 15</u>
III.	Blanks	A	
IVa.	Surrogate recovery	SW	
IVb.	Matrix spike/Matrix spike duplicates	A	
IVc.	Laboratory control samples	A	<u>LCSP</u>
V.	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	N	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	ND	<u>D = 3, 4</u>
X.	Field blanks	ND	<u>R = 11</u>

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:

soil + water

1	TSB-HJ-10-0'	11	RINSATE-2	W	21	8031303	31
2	TSB-HJ-10-10'	12	TSB-HJ-10-0'MS		22	8035123	32
3	TSB-HR-06-0' <u>D</u>	13	TSB-HJ-10-0'MSD		23	80329 8039219	33
4	TSB-HR-06-0'-FD <u>D</u>	14	TSB-HR-05-10'MS		24		34
5	TSB-HR-06-10'	15	TSB-HR-05-10'MSD		25		35
6	TSB-HJ-08-0'	16			26		36
7	TSB-HJ-08-10'	17			27		37
8	TSB-HR-05-0'	18			28		38
9	TSB-HR-05-10'	19			29		39
10	TSB-HR-05-10'RE <u>W</u>	20			30		40

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

**VALIDATION FINDINGS WORKSHEET**  
**Surrogate Recovery**

LDC #: 18386B8  
SDG #: pel cover

METHOD: GC HPLC

Are surrogates required by the method? Yes      or No       
Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".  
 N N/A Were surrogates spiked into all samples and blanks?  
 N N/A Did all surrogate recoveries (%R) meet the QC limits?

#	Sample ID	Detector/Column	Surrogate Compound	%R (Limits)	Qualifications
	9	not specified	H	( 73 - 150 )	5/u/A
	8039219-blank	↓	↓	(      )	5/u/P
				(      )	
				(      )	
				(      )	
				(      )	
				(      )	
				(      )	
				(      )	
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				(      )	
				(      )	
				(      )	
				(      )	

Surrogate Compound	Surrogate Compound	Surrogate Compound	Surrogate Compound	Surrogate Compound
A Chlorobenzene (CBZ)	G Octacosane	M Benzo(e)Pyrene	S 1-Chloro-3-Nitrobenzene	Y Tetrachloro-m-xylene
B 4-Bromofluorobenzene (BFB)	H Ortho-Terphenyl	N Terphenyl-D14	T 3,4-Dinitrotoluene	
C a,a-Trifluorotoluene	I Fluorobenzene (FBZ)	O Decachlorobiphenyl (DCB)	U Triphenyltin	
D Bromochlorobenzene	J n-Triacontane	P 1-methylnaphthalene	V Tri-n-propyltin	
E 1,4-Dichlorobutane	K Hexacosane	Q Dichlorophenyl Acetic Acid (DCAA)	W Tributyl Phosphate	
F 1,4-Difluorobenzene (DFB)	L Bromobenzene	R 4-Nitrophenol	X Triphenyl Phosphate	

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

**Dioxins/Dibenzofurans**

*LDC*

## Laboratory Data Consultants, Inc. Data Validation Report

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

**Sample Delivery Group (SDG):** F8A250221

### Sample Identification

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

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 17 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

## **I. Technical Holding Times**

All technical holding time requirements were met.

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

## **II. HRGC/HRMS Instrument Performance Check**

Instrument performance was checked at the required daily frequency.

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

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

## **III. Initial Calibration**

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

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

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

The minimum S/N ratio for each target compound was greater than or equal to 2.5 and 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	P

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:

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HJ-07-0'**	TSB-HJ-07-0'-FD				
OCDD	0.80	0.30	-	0.50 ( $\leq 11$ )	-	-
1,2,3,7,8,9-HxCDF	0.070	0.14	-	0.07 ( $\leq 5.4$ )	-	-
1,2,3,4,6,7,8-HpCDF	0.064	5.3U	-	5.24 ( $\leq 5.4$ )	-	-
OCDF	0.21	11U	-	10.79 ( $\leq 11$ )	-	-

**BRC Tronox Parcel H  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG F8A250221**

SDG	Sample	Compound	Flag	A or P	Reason
F8A250221	TSB-HR-06-0'	2,3,7,8-TCDF (from DB-225)	None	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	A
F8A250221	TSB-HR-04-10'	OCDD OCDF	11U pg/g 11U pg/g	A
F8A250221	TSB-HJ-04-0'	OCDD	11U pg/g	A
F8A250221	TSB-HR-04-0'***	OCDD	10U pg/g	A
F8A250221	TSB-HJ-04-10'	OCDD	11U pg/g	A
F8A250221	TSB-HR-07-0'	OCDD	11U pg/g	A
F8A250221	TSB-HR-07-10'***	OCDD	11U pg/g	A
F8A250221	TSB-HR-06-10'	OCDD	11U pg/g	A
F8A250221	TSB-HJ-07-0'***	OCDD OCDF	11U pg/g 11U pg/g	A
F8A250221	TSB-HJ-07-0'-FD	OCDD	11U pg/g	A
F8A250221	TSB-HJ-07-10'	1,2,3,7,8-PeCDD OCDD	5.4U pg/g 11U pg/g	A
F8A250221	TSB-HR-08-0'	1,2,3,7,8-PeCDD OCDD	5.3U pg/g 11U pg/g	A
F8A250221	TSB-HR-08-10'	OCDD	11U pg/g	A

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

No Sample Data Qualified in this SDG

LDC #: 18386A21

**VALIDATION COMPLETENESS WORKSHEET**

SDG #: F8A250221

Level III/IV

Laboratory: Test America

Date: 3/11/08

Page: 1 of 1

Reviewer: RB2nd Reviewer: RB**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
I.	Technical holding times	A	Sampling dates: 1/24/08
II.	GC/MS Instrument performance check	Δ	
III.	Initial calibration	Δ	
IV.	Routine calibration	Δ	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	A	
VII.	Laboratory control samples	A	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
X.	Target compound identifications	Δ	Not reviewed for Level III validation.
XI.	Compound quantitation and CRQLs	SW	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	N	

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

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

D = Duplicate  
TB = Trip blank  
EB = Equipment blank

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

1	TSB-HJ-05-10**	11	TSB-HJ-07-0**	21	8043106-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-06-0'MS	26		36
7	TSB-HR-07-0'	17	TSB-HR-06-0'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

Notes: \_\_\_\_\_  
 \_\_\_\_\_  
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LDC #: 18386A21  
 SDG #: fire cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 3  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>II. GC/MS Instrument performance check</b>				
Was PFK exact mass 380.9760 verified?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the retention time windows established for all homologues?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers $\leq 25\%$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Is the static resolving power at least 10,000 (10% valley definition)?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the mass resolution adequately check with PFK?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>III. Initial calibration</b>				
Was the initial calibration performed at 5 concentration levels?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent relative standard deviations (%RSD) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did all calibration standards meet the Ion Abundance Ratio criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the signal to noise ratio for each target compound $\geq 2.5$ and for each recovery and internal standard $\geq 10$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Continuing calibration</b>				
Was a routine calibration performed at the beginning and end of each 12 hour period?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a method blank performed for each matrix and concentration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>VI. Matrix spike/Matrix spike duplicates</b>				
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.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>VII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

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VALIDATION FINDINGS CHECKLIST

Page: 2 of 3  
 Reviewer: [Signature]  
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Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>VIII. Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the performance evaluation (PE) samples within the acceptance limits?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>IX. Internal standards</b>				
Were internal standard recoveries within the 40-135% criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the minimum S/N ratio of all internal standard peaks $\geq 10$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>X. Target compound identification</b>				
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?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
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?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
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?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did compound spectra contain all characteristic ions listed in the table attached?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the signal to noise ratio for each target compound and labeled standard $\geq 2.5$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Does the maximum intensity of each specified characteristic ion coincide within $\pm 2$ seconds (includes labeled standards)?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
For PCDF identification, was any signal ( $S/N \geq 2.5$ , at $\pm$ seconds RT) detected in the corresponding PCDPE channel?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was an acceptable lock mass recorded and monitored?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XI. Compound quantitation/CRQLs</b>				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XII. System performance</b>				
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XIII. Overall assessment of data</b>				
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XIV. Field duplicates</b>				
Field duplicate pairs were identified in this SDG.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

LDC #: 18386A21  
SDG #: see cover

VALIDATION FINDINGS CHECKLIST

Page: 3 of 3  
Reviewer: [Signature]  
2nd Reviewer: [Signature]

Validation Area	Yes	No	NA	Findings/Comments
Target compounds were detected in the field duplicates.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
XV. Field blanks				
Field blanks were identified in this SDG.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Target compounds were detected in the field blanks.	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	



# 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:

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LDC #: 18386A21  
 SDG #: see lower

**VALIDATION FINDINGS WORKSHEET**  
**Field Duplicates**

Page: 1 of 1  
 Reviewer: B  
 2nd reviewer: V

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

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

Compound	Concentration (pg/g)		Difference RPD
	11	12	
G	0.80	0.30	0.50 ( $\leq 5.4$ )
N	0.070	0.14	0.07 ( <del>4.11</del> ) ( $\leq 5.4$ )
<del>Q</del>	0.064	5.34	5.24 ( $\leq 5.4$ )
Q	0.21	11.0	10.79 ( $\leq 11$ )

Compound	Concentration ( )		RPD

Compound	Concentration ( )		RPD

Compound	Concentration ( )		RPD

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

**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:

$RRF = (A_x/C_x)/(A_s/C_s)$   
 average RRF = sum of the RRFs/number of standards  
 $\%RSD = 100 * (S/X)$   
 $A_x$  = Area of compound,  $A_s$  = Area of associated internal standard  
 $C_x$  = Concentration of compound,  $C_s$  = Concentration of internal standard  
 $S$  = Standard deviation of the RRFs,  $X$  = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated		
				Average RRF (Initial)	RRF (CS3)	Average RRF (Initial)	RRF (CS3 std)	RRF (CS3 std)	RRF (CS3 std)	%RSD	%RSD	
1	1CA1	2/15/08	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.045	1.040	1.045	1.040	1.040	9.9	9.9	9.9	
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.07584	1.075	1.081	1.075	1.075	11.0	11.0	11.0	
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.053	1.070	1.053	1.070	1.070	7.0	7.0	7.0	
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.976	0.977	0.976	0.977	0.977	7.0	7.0	7.0	
			OCDF ( <sup>13</sup> C-OCDF)	1.102	1.118	1.102	1.118	1.118	8.0	8.0	8.0	
2	1CAL DB-225	3/15/08	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.119	1.205	1.179	1.205	1.205	4.0	4.0	4.0	
			<del>2,3,7,8-TCDD (<sup>13</sup>C-2,3,7,8-TCDD)</del>	1.081	1.125	1.081	1.125	1.125	6.7	6.7	6.7	
			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)									
			<del>OCDF (<sup>13</sup>C-OCDF)</del>									
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)									
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)									
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)									
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)									

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.

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

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: \_\_\_\_\_

LDC #: \_\_\_\_\_  
 SDG #: \_\_\_\_\_

**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 =  $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$       Where:      ave. RRF = initial calibration average RRF  
 RRF =  $(A_x)(C_{is}) / (A_{is})(C_x)$       RRF = continuing calibration RRF  
 $A_x$  = Area of compound,       $A_{is}$  = Area of associated internal standard  
 $C_x$  = Concentration of compound,       $C_{is}$  = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	COB 02255	2/25/08	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.125	1.044	7.2	1.044	7.2
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDF)					
2			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDF)					
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDF)					

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.

**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 =  $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$       Where:    ave. RRF = initial calibration average RRF  
 $\text{RRF} = (A_x)(C_{is}) / (A_{is})(C_x)$       RRF = continuing calibration RRF  
 $A_x$  = Area of compound,       $A_{is}$  = Area of associated internal standard  
 $C_x$  = Concentration of compound,       $C_{is}$  = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	ceV	2/20/08	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.045	0.984	5.7	0.984	5.9
	A08022052	12:34	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.084	1.021	5.9	1.021	5.9
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.053	1.001	4.9	1.001	4.9
2	ceV	2/20/08	1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	0.916	1.030	5.5	1.030	5.8
	A08022152	23:53	OCDF ( <sup>13</sup> C-OCDD)	1.102	1.276	15.8	1.276	15.8
			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)		0.996	4.7	0.996	4.7
3	ceV	2/22/08	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)		1.035	4.5	1.035	4.5
	A08022251	09:19	1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)		1.083	2.8	1.023	2.8
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)		1.021	4.5	1.021	4.5
			OCDF ( <sup>13</sup> C-OCDD)		1.171	6.3	1.171	6.3
			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)		0.989	5.4	0.989	5.4
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)		1.055	2.7	1.055	2.7
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)		1.019	3.2	1.019	3.2
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)		1.032	5.7	1.032	5.7
			OCDF ( <sup>13</sup> C-OCDD)		1.170	6.1	1.170	6.1

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

LDC #: 18386A2 /  
 SDG #: fee covered

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

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer:

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

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

% Recovery =  $100 * (SSR - SR) / SA$       Where: SSR = Spiked sample result, SR = Sample result  
 SA = Spike added

RPD =  $100 * |MSR - MSDR| * 2 / (MSR + MSDR)$       MSR = Matrix spike percent recovery      MSDR = Matrix spike duplicate percent recovery

MS/MSD samples: 16 + 17

Compound	Spike Added (pg/g)		Sample Concentration (pg/g)	Spiked Sample Concentration (pg/g)		Matrix Spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		Reported	Recalculated
	MS	MSD		MS	MSD	Reported	Recalc	Reported	Recalc	RPD	RPD
2,3,7,8-TCDD	22.2	22.2	ND	22.7	27.4	102	102	101	101	1.5	1.5
1,2,3,7,8-PeCDD	111	111	ND	115	116	103	103	105	105	1.3	1.3
1,2,3,4,7,8-HxCDD	111	111	ND	118	110	106	106	99	99	6.8	6.8
1,2,3,4,7,8,9-HpCDF	111	111	0.45	117	111	105	105	100	100	4.9	4.9
OCDF	222	222	2.8	250	250	115	115	112	112	3.1	3.1

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 #: 18386A21  
 SDG #: pu cover

**VALIDATION FINDINGS WORKSHEET**  
Sample Calculation Verification

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd reviewer: [Signature]

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

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

$$\text{Concentration} = \frac{(A_x)(I_s)(DF)}{(A_{is})(RRF)(V_o)(\%S)}$$

- A<sub>x</sub> = Area of the characteristic ion (EICP) for the compound to be measured
- A<sub>is</sub> = Area of the characteristic ion (EICP) for the specific internal standard
- I<sub>s</sub> = Amount of internal standard added in nanograms (ng)
- V<sub>o</sub> = Volume or weight of sample extract in milliliters (ml) or grams (g).
- RRF = Relative Response Factor (average) from the initial calibration
- Df = Dilution Factor.
- %S = Percent solids, applicable to soil and solid matrices only.

Example:

Sample I.D. # 2 , G :

$$\text{Conc.} = \frac{(52145)(2000)}{1739867(0.976)(10)(0.953)}$$

= 6.44 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		# 2 # confirmation			
		= $\frac{8462(1000)}{388684(1.081)(10)(0.953)}$			
		= 2.1 pg/g			

LDC #: \_\_\_\_\_  
 SDG #: \_\_\_\_\_

**VALIDATION FINDINGS WORKSHEET**  
Sample Calculation Verification (additional page)

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

Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate mass <sup>(b)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(b)</sup>	Ion ID	Elemental Composition	Analyte		
1	303.9016	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDF		
	305.8987	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF		409.7788	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF		
	315.9419	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> O	TCDF (S)		417.8250	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> O	HpCDF (S)		
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF (S)		419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDF		
	319.8965	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD		423.7767	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	321.8936	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD		425.7737	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	331.9368	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD (S)		435.8169	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	333.9338	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD (S)		437.8140	M+4	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	375.8364	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDFE		479.7165	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDFE		
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK		[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK		
	2	339.8597	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	OCDF
		341.8567	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		443.7399	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
		351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF (S)		457.7377	M+2	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
353.8970		M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDF (S)	459.7348	M+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDD		
355.8546		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	469.7780	M+2		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
357.8516		M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD	471.7750	M+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD (S)		
367.8949		M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	513.6775	M+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDFE		
369.8919		M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD (S)	[422.9278]	LOCK		C <sub>10</sub> F <sub>17</sub>	PFK		
409.7974		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO	HxCDFE							
[354.9792]		LOCK	C <sub>9</sub> F <sub>13</sub>	PFK							
3		373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDF						
		375.8178	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF						
		383.8639	M	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDF (S)						
	385.8610	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	HxCDF (S)							
	389.8156	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD							
	391.8127	M+4	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD							
	401.8559	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD (S)							
	403.8529	M+4	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD (S)							
	445.7555	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO	OCDFE							
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK							

(e) The following nucleic masses were used:

H = 1.007825  
 C = 12.000000  
<sup>13</sup>C = 13.003355  
 F = 18.9984  
 O = 15.994915  
<sup>35</sup>Cl = 34.968853  
<sup>37</sup>Cl = 36.965903

S = internal/recovery standard

**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.
- UU 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 1,2,3,4,7,8,9-HpCDF OCDF	1.1 pg/L 0.84 pg/L 1.2 pg/L 1.7 pg/L 6.6 pg/L 1.1 pg/L 1.1 pg/L 0.72 pg/L 1.2 pg/L 1.1 pg/L 1.6 pg/L 3.8 pg/L	All water samples in SDG F8A290158

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

Sample	Compound	Reported Concentration	Modified Final Concentration
TSB-HJ-10-10'	OCDD	0.22 pg/g	10U pg/g
TSB-HR-06-0'-FD	OCDD	0.70 pg/g	11U pg/g
TSB-HR-06-10'	OCDD	0.24 pg/g	11U pg/g
TSB-HJ-08-10'	OCDD	0.26 pg/g	11U pg/g
TSB-HR-05-10'	OCDD	0.40 pg/g	11U pg/g
RINSATE-2	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF OCDF	0.84 pg/L 3.9 pg/L 0.35 pg/L 0.56 pg/L 1.7 pg/L	50U pg/L 100U pg/L 50U pg/L 50U pg/L 100U pg/L

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

Rinsate ID	Sampling Date	Compound	Concentration	Associated Samples
RINSATE-2	1/28/08	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF OCDF	0.84 pg/L 3.9 pg/L 0.35 pg/L 0.56 pg/L 1.7 pg/L	All soil samples in SDG F8A290158

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

Sample	Compound	Reported Concentration	Modified Final Concentration
TSB-HJ-10-0'	1,2,3,7,8,9-HxCDF	0.51 pg/g	5.3U pg/g
TSB-HJ-10-10'	OCDD	0.22 pg/g	10U pg/g
TSB-HR-06-0'	1,2,3,4,6,7,8-HpCDD OCDD OCDF	0.16 pg/g 1.5 pg/g 0.62 pg/g	5.2U pg/g 10U pg/g 10U pg/g
TSB-HR-06-0'-FD	OCDD OCDF	0.70 pg/g 0.49 pg/g	11U pg/g 11U pg/g
TSB-HR-06-10'	OCDD	0.24 pg/g	11U pg/g
TSB-HJ-08-0'	OCDD 1,2,3,6,7,8-HxCDF OCDF	1.2 pg/g 0.26 pg/g 1.3 pg/g	11U pg/g 5.4U pg/g 11U pg/g
TSB-HJ-08-10'	OCDD	0.26 pg/g	11U pg/g
TSB-HR-05-0'	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,6,7,8-HxCDF OCDF	1.6 pg/g 9.2 pg/g 0.16 pg/g 1.7 pg/g	5.4U pg/g 11U pg/g 5.4U pg/g 11U pg/g
TSB-HR-05-10'	OCDD	0.40 pg/g	11U pg/g

## VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Although the MS/MSD percent recoveries (%R) were not within QC limits for some compounds, the LCS/LCSD percent recoveries (%R) were within QC limits and no data were qualified.



## VII. Laboratory Control Samples (LCS)

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

## VIII. Regional Quality Assurance and Quality Control

Not applicable.

## IX. Internal Standards

All internal standard recoveries were within QC limits.

## X. Target Compound Identifications

Raw data were not reviewed for this SDG.

## XI. Compound Quantitation and CRQLs

All compound quantitation and CRQLs were within validation criteria with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P
TSB-HJ-10-10'	2,3,7,8-TCDF (from DB-225)	Confirmation was not performed for this compound.	All compounds must be confirmed on the 2nd column per the QAPP.	None	P

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:

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HR-06-0'	TSB-HR-06-0'-FD				
1,2,3,4,6,7,8-HpCDD	0.16	5.4U	-	5.24 ( $\leq 5.4$ )	-	-
OCDD	1.5	0.70	-	0.8 ( $\leq 11$ )	-	-
1,2,3,4,6,7,8-HpCDF	0.19	0.26	-	0.07 ( $\leq 5.4$ )	-	-
OCDF	0.62	0.46	-	0.13 ( $\leq 11$ )	-	-

**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	P	Compound quantitation and CRQLs

**BRC Tronox Parcel H  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG F8A290158**

SDG	Sample	Compound	Modified Final Concentration	A or P
F8A290158	TSB-HJ-10-10'	OCDD	10U pg/g	A
F8A290158	TSB-HR-06-0'-FD	OCDD	11U pg/g	A
F8A290158	TSB-HR-06-10'	OCDD	11U pg/g	A
F8A290158	TSB-HJ-08-10'	OCDD	11U pg/g	A
F8A290158	TSB-HR-05-10'	OCDD	11U pg/g	A
F8A290158	RINSATE-2	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF OCDF	50U pg/L 100U pg/L 50U pg/L 50U pg/L 100U pg/L	A

**BRC Tronox Parcel H  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG F8A290158**

SDG	Sample	Compound	Modified Final Concentration	A or P
F8A290158	TSB-HJ-10-0'	1,2,3,7,8,9-HxCDF	5.3U pg/g	A
F8A290158	TSB-HJ-10-10'	OCDD	10U pg/g	A
F8A290158	TSB-HR-06-0'	1,2,3,4,6,7,8-HpCDD OCDD OCDF	5.2U pg/g 10U pg/g 10U pg/g	A

SDG	Sample	Compound	Modified Final Concentration	A or P
F8A290158	TSB-HR-06-0'-FD	OCDD OCDF	11U pg/g 11U pg/g	A
F8A290158	TSB-HR-06-10'	OCDD	11U pg/g	A
F8A290158	TSB-HJ-08-0'	OCDD 1,2,3,6,7,8-HxCDF OCDF	11U pg/g 5.4U pg/g 11U pg/g	A
F8A290158	TSB-HJ-08-10'	OCDD	11U pg/g	A
F8A290158	TSB-HR-05-0'	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,6,7,8-HxCDF OCDF	5.4U pg/g 11U pg/g 5.4U pg/g 11U pg/g	A
F8A290158	TSB-HR-05-10'	OCDD	11U pg/g	A

LDC #: 18386B21

## VALIDATION COMPLETENESS WORKSHEET

SDG #: F8A290158

Level III

Laboratory: Test America

Date: 3/7/08

Page: 1 of 1

Reviewer: [Signature]

2nd Reviewer: [Signature]

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
I.	Technical holding times	A	Sampling dates: 1/28/08
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	LCS 10
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	SW	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	D = 3 + 4
XV.	Field blanks	SW	R = 10

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

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

D = Duplicate  
TB = Trip blank  
EB = Equipment blank

Validated Samples:

soil + water

1	✓	TSB-HJ-10-0'	11	TSB-HR-06-0'MS	21	8042278-BLK	31
2	✓	TSB-HJ-10-10'	12	TSB-HR-06-0'MSD	22	8045178-BLK	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

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

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:







LDC #: 18386B2/  
 SDG #: per company

**VALIDATION FINDINGS WORKSHEET**  
**Field Blanks**

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

(Y) (N) (A) Were field blanks identified in this SDG?

Blank units: pg/L Associated sample units: pg/g

Sampling date: 1/28/08

Field blank type: (circle one) Field Blank / Rinsate / Other: R Associated Samples: All soils

Compound	Blank ID	Sample Identification											
		1	2	3	4	5	6	7	8	9			
F	0.84	-	-	0.16/5.2u	-	-	-	-	1.6/5.4u	-	-	-	-
G	3.9	-	0.22/10u	1.5/10u	0.70/11u	0.24/11u	1.2/11u	0.26/11u	9.2/11u	0.40/11u	-	-	-
L	0.35	-	-	-	-	-	0.26/5.4u	-	0.16/5.4u	-	-	-	-
N	0.56	0.51/5.3u	-	-	-	-	-	-	-	-	-	-	-
R	1.7	-	-	0.62/10u	0.49/11u	-	1.3/11u	-	1.7/11u	-	-	-	-
CRQL													

Blank units: \_\_\_\_\_ Associated sample units: \_\_\_\_\_

Sampling date: \_\_\_\_\_

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

Compound	Blank ID	Sample Identification											
		1	2	3	4	5	6	7	8	9			
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 #: 182386B 2/  
 SDG #: fee cover

## VALIDATION FINDINGS WORKSHEET

### Field Duplicates

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd reviewer: [Signature]

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

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

Compound	Concentration (pg/g)		Difference RPD
	3	4	
F	0.16	<del>10.84</del> 5.44	<del>10.84</del> 5.24 (≤ 5.4)
G	1.5	0.70	0.8 (≤ 11)
E	0.19	0.26	0.07 (≤ 5.4)
Q	0.62	0.49	0.13 (≤ 11)

Compound	Concentration ( )		RPD

Compound	Concentration ( )		RPD

Compound	Concentration ( )		RPD