



**LABORATORY DATA CONSULTANTS, INC.**

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

March 17, 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 February 29, 2008. Attachment 1 is a summary of the samples that were reviewed for each analysis.

**LDC Project # 18356:**

<b><u>SDG #</u></b>	<b><u>Fraction</u></b>
F8A260143	Volatiles, Semivolatiles, Chlorinated Pesticides, Polychlorinated Biphenyls, Metals, Gasoline Range Organics, Diesel Range Organics, Dioxins/Dibenzofurans, Wet Chemistry

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

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

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

Sincerely,

Erlinda T. Rauto  
Operations Manager/Senior Chemist



**BRC Tronox Parcel H  
Data Validation Reports  
LDC# 18356**

Volatiles

*LDC*

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** BRC Tronox Parcel H  
**Collection Date:** January 25, 2008  
**LDC Report Date:** March 10, 2008  
**Matrix:** Soil/Water  
**Parameters:** Volatiles  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8A260143

TSB-HJ-01-10'	TSB-TB-04-1/25/08
TSB-HJ-09-0'	RINSATE-1
TSB-HJ-09-10'	RINSATE-1RE
TSB-HJ-03-0'	TRIP BLANK-TB-05
TSB-HJ-03-0'-FD	TSB-HJ-02-10'MS
TSB-HJ-03-10'	TSB-HJ-02-10'MSD
TSB-HR-03-0'	TSB-HJ-01-0'MS
TSB-HR-03-10'**	TSB-HJ-01-0'MSD
TSB-HJ-02-0'**	RINSATE-1MS
TSB-HJ-02-10'**	RINSATE-1MSD
TSB-HR-02-0'**	RINSATE-1REMS
TSB-HR-02-10'**	RINSATE-1REMSD
TSB-HJ-11-0'**	
TSB-HJ-11-10'	
TSB-HJ-11-10'-FD	
TSB-HR-01-0'	
TSB-HR-01-10'	
TSB-HJ-01-0'	
TSB-TB-2-1/25/08	
TSB-TB-03-1/25/08	

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 22 soil samples and 10 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 F8A260143	J (all detects) UJ (all non-detects)	A
1/30/08	Ethanol	0.00855 ( $\geq 0.05$ )	TSB-TB-2-1/25/08 TSB-TB-03-1/25/08 TSB-TB-04-1/25/08 RINSATE-1 TRIP BLANK-TB-05 8031135-Blank	J (all detects) UJ (all non-detects)	A
2/6/08	Ethanol	0.00366 ( $\geq 0.05$ )	All soil samples in SDG F8A260143	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	TSB-TB-2-1/25/08 TSB-TB-03-1/25/08 TSB-TB-04-1/25/08 RINSATE-1 TRIP BLANK-TB-05 8031135-Blank	J+ (all detects)	A
2/8/08	Ethanol 2,2-Dimethylpentane 2,4-Dimethylpentane	60.01099 33.49841 32.08794	TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0' TSB-HJ-01-0'MS TSB-HJ-01-0'MSD 8043261-Blank	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	TSB-TB-2-1/25/08 TSB-TB-03-1/25/08 TSB-TB-04-1/25/08 RINSATE-1 TRIP BLANK-TB-05 8031135-Blank	J+ (all detects) J+ (all detects)	A
1/21/08	Vinyl acetate	31.00872	RINSATE-1RE 8036136-Blank	J+ (all detects)	A
2/6/08	Bromomethane	34.53645	All soil samples in SDG F8A260143	J+ (all detects)	A
2/6/08	Acetonitrile	27.60270	All soil samples in SDG F8A260143	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
2/4/08	Dibromomethane	0.04878 ( $\geq 0.05$ )	RINSATE-1RE 8036136-Blank	J (all detects) UJ (all non-detects)	A
1/30/08	Dibromomethane	0.04735 ( $\geq 0.05$ )	TSB-TB-2-1/25/08 TSB-TB-03-1/25/08 TSB-TB-04-1/25/08 RINSATE-1 TRIP BLANK-TB-05 8031135-Blank	J (all detects) UJ (all non-detects)	A
2/7/08	Ethanol	0.00337 ( $\geq 0.05$ )	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'*** TSB-HJ-02-0'*** TSB-HJ-02-10'*** TSB-HR-02-0'*** TSB-HR-02-10'*** TSB-HJ-11-0'*** TSB-HJ-11-10' TSB-HJ-02-10'MS TSB-HJ-02-10'MSD 8039077-Blank	J (all detects) UJ (all non-detects)	A
2/8/08	Ethanol	0.00586 ( $\geq 0.05$ )	TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0' TSB-HJ-01-0'MS TSB-HJ-01-0'MSD 8043261-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
8039077-Blank	2/7/08	Dichloromethane	13 ug/Kg	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'*** TSB-HJ-02-0'*** TSB-HJ-02-10'*** TSB-HR-02-0'*** TSB-HR-02-10'*** TSB-HJ-11-0'*** TSB-HJ-11-10'
8031135-Blank	1/30/08	Dichloromethane	0.16 ug/L	TSB-TB-2-1/25/08 TSB-TB-03-1/25/08 TSB-TB-04-1/25/08 RINSATE-1 TRIP BLANK-TB-05

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-01-10'	Dichloromethane	21 ug/Kg	21U ug/Kg
TSB-HJ-09-0'	Dichloromethane	15 ug/Kg	15U ug/Kg
TSB-HJ-09-10'	Dichloromethane	14 ug/Kg	14U ug/Kg
TSB-HJ-03-0'	Dichloromethane	14 ug/Kg	14U ug/Kg
TSB-HJ-03-0'-FD	Dichloromethane	12 ug/Kg	12U ug/Kg
TSB-HJ-03-10'	Dichloromethane	15 ug/Kg	15U ug/Kg
TSB-HR-03-0'	Dichloromethane	16 ug/Kg	16U ug/Kg
TSB-HR-03-10'***	Dichloromethane	15 ug/Kg	15U ug/Kg
TSB-HJ-02-0'***	Dichloromethane	12 ug/Kg	12U ug/Kg

Sample	Compound TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
TSB-HJ-02-10'***	Dichloromethane	8.8 ug/Kg	8.8U ug/Kg
TSB-HR-02-0'***	Dichloromethane	7.1 ug/Kg	7.1U ug/Kg
TSB-HR-02-10'***	Dichloromethane	8.3 ug/Kg	8.3U ug/Kg
TSB-HJ-11-0'***	Dichloromethane	4.9 ug/Kg	5.2U ug/Kg
TSB-HJ-11-10'	Dichloromethane	6.8 ug/Kg	6.8U ug/Kg
TSB-TB-2-1/25/08	Dichloromethane	0.19 ug/L	1.0U ug/L
TSB-TB-03-1/25/08	Dichloromethane	0.14 ug/L	1.0U ug/L
TSB-TB-04-1/25/08	Dichloromethane	0.19 ug/L	1.0U ug/L
TRIP BLANK-TB-05	Dichloromethane	0.19 ug/L	1.0U ug/L

Samples TSB-TB-2-1/25/08, TSB-TB-03-1/25/08, TSB-TB-04-1/25/08, and TRIP BLANK-TB-05 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
TRIP BLANK-TB-05	1/25/08	Dichloromethane Acetone	0.19 ug/L 4.7 ug/L	RINSATE-1 RINSATE-1RE
TSB-TB-2-1/25/08	1/25/08	Dichloromethane Acetone	0.19 ug/L 4.5 ug/L	TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'***
TSB-TB-03-1/25/08	1/25/08	Dichloromethane Acetone	0.14 ug/L 4.9 ug/L	TSB-HJ-02-0'*** TSB-HJ-02-10'*** TSB-HR-02-0'*** TSB-HR-02-10'***
TSB-TB-04-1/25/08	1/25/08	Dichloromethane Acetone	0.19 ug/L 4.6 ug/L	TSB-HJ-11-0'*** TSB-HJ-11-10' TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0'

Samples "RINSATE-1" and "RINSATE-1RE" were identified as rinsates. No volatile contaminants were found in these blanks with the following exceptions:

Rinsate Blank ID	Sampling Date	Compound	Concentration	Associated Samples
RINSATE-1	1/25/08	Dichloromethane Ethyl ether	12 ug/L 2.2 ug/L	All soil samples in SDG F8A260143
RINSATE-1RE	1/25/08	Dichloromethane Acetone	10 ug/L 1.8 ug/L	All soil samples in SDG F8A260143

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-01-10'	Dichloromethane	21 ug/Kg	21U ug/Kg
TSB-HJ-09-0'	Dichloromethane	15 ug/Kg	15U ug/Kg
TSB-HJ-09-10'	Dichloromethane	14 ug/Kg	14U ug/Kg
TSB-HJ-03-0'	Dichloromethane Acetone	14 ug/Kg 11 ug/Kg	14U ug/Kg 21U ug/Kg
TSB-HJ-03-0'-FD	Dichloromethane	12 ug/Kg	12U ug/Kg
TSB-HJ-03-10'	Dichloromethane Acetone	15 ug/Kg 5.8 ug/Kg	15U ug/Kg 21U ug/Kg
TSB-HR-03-0'	Dichloromethane	16 ug/Kg	16U ug/Kg
TSB-HR-03-10'***	Dichloromethane	15 ug/Kg	15U ug/Kg
TSB-HJ-02-0'***	Dichloromethane	12 ug/Kg	12U ug/Kg
TSB-HJ-02-10'***	Dichloromethane Acetone	8.8 ug/Kg 9.7 ug/Kg	8.8U ug/Kg 21U ug/Kg
TSB-HR-02-0'***	Dichloromethane Acetone	7.1 ug/Kg 9.3 ug/Kg	7.1U ug/Kg 21U ug/Kg

Sample	Compound	Reported Concentration	Modified Final Concentration
TSB-HR-02-10 <sup>**</sup>	Dichloromethane Acetone	8.3 ug/Kg 9.5 ug/Kg	8.3U ug/Kg 21U ug/Kg
TSB-HJ-11-0 <sup>**</sup>	Dichloromethane	4.9 ug/Kg	5.2U ug/Kg
TSB-HJ-11-10 <sup>'</sup>	Dichloromethane	6.8 ug/Kg	6.8U 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

## 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 many compounds, the MS, MSD, or LCS percent recovery (%R) was 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 some compounds, the MS or 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.

## 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-03-0' and TSB-HJ-03-0'-FD and samples TSB-HJ-11-10' and TSB-HJ-11-10'-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-03-0'	TSB-HJ-03-0'-FD				
Dichloromethane	14	12	-	2 ug/Kg ( $\leq 5.3$ )	-	-

Compound	Concentration (ug/Kg)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HJ-11-10'	TSB-HJ-11-10'-FD				
Dichloromethane	6.8	5.3U	-	1.5 ug/Kg ( $\leq 5.3$ )	-	-

**BRC Tronox Parcel H**  
**Volatiles - Data Qualification Summary - SDG F8A260143**

SDG	Sample	Compound	Flag	A or P	Reason
F8A260143	TSB-TB-2-1/25/08 TSB-TB-03-1/25/08 TSB-TB-04-1/25/08 RINSATE-1 RINSATE-1RE TRIP BLANK-TB-05	Dibromomethane	J (all detects) UJ (all non-detects)	A	Initial calibration (RRF)
F8A260143	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'*** TSB-HJ-02-0'*** TSB-HJ-02-10'*** TSB-HR-02-0'*** TSB-HR-02-10'*** TSB-HJ-11-0'*** TSB-HJ-11-10' TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0' TSB-TB-2-1/25/08 TSB-TB-03-1/25/08 TSB-TB-04-1/25/08 RINSATE-1 TRIP BLANK-TB-05	Ethanol	J (all detects) UJ (all non-detects)	A	Initial calibration (RRF)
F8A260143	TSB-TB-2-1/25/08 TSB-TB-03-1/25/08 TSB-TB-04-1/25/08 RINSATE-1 TRIP BLANK-TB-05	Bromomethane	J+ (all detects)	A	Continuing calibration (%D)
F8A260143	TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0'	Ethanol 2,2-Dimethylpentane 2,4-Dimethylpentane	J+ (all detects) J+ (all detects) J+ (all detects)	A	Continuing calibration (%D)
F8A260143	TSB-TB-2-1/25/08 TSB-TB-03-1/25/08 TSB-TB-04-1/25/08 RINSATE-1 TRIP BLANK-TB-05	Iodomethane Vinyl acetate	J+ (all detects) J+ (all detects)	A	Continuing calibration (ICV %D)
F8A260143	RINSATE-1RE	Vinyl acetate	J+ (all detects)	A	Continuing calibration (ICV %D)

SDG	Sample	Compound	Flag	A or P	Reason
F8A260143	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'*** TSB-HJ-02-0'*** TSB-HJ-02-10'*** TSB-HR-02-0'*** TSB-HR-02-10'*** TSB-HJ-11-0'*** TSB-HJ-11-10' TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0'	Bromomethane	J+ (all detects)	A	Continuing calibration (ICV %D)
F8A260143	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'*** TSB-HJ-02-0'*** TSB-HJ-02-10'*** TSB-HR-02-0'*** TSB-HR-02-10'*** TSB-HJ-11-0'*** TSB-HJ-11-10' TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0'	Acetonitrile	J- (all detects) UJ (all non-detects)	A	Continuing calibration (ICV %D)
F8A260143	RINSATE-1RE TSB-TB-2-1/25/08 TSB-TB-03-1/25/08 TSB-TB-04-1/25/08 RINSATE-1 TRIP BLANK-TB-05	Dibromomethane	J (all detects) UJ (all non-detects)	A	Continuing calibration (RRF)

SDG	Sample	Compound	Flag	A or P	Reason
F8A260143	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'** TSB-HJ-02-0'** TSB-HJ-02-10'** TSB-HR-02-0'** TSB-HR-02-10'** TSB-HJ-11-0'** TSB-HJ-11-10' TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0'	Ethanol	J (all detects) UJ (all non-detects)	A	Continuing calibration (RRF)

**BRC Tronox Parcel H  
Volatiles - Laboratory Blank Data Qualification Summary - SDG F8A260143**

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P
F8A260143	TSB-HJ-01-10'	Dichloromethane	21U ug/Kg	A
F8A260143	TSB-HJ-09-0'	Dichloromethane	15U ug/Kg	A
F8A260143	TSB-HJ-09-10'	Dichloromethane	14U ug/Kg	A
F8A260143	TSB-HJ-03-0'	Dichloromethane	14U ug/Kg	A
F8A260143	TSB-HJ-03-0'-FD	Dichloromethane	12U ug/Kg	A
F8A260143	TSB-HJ-03-10'	Dichloromethane	15U ug/Kg	A
F8A260143	TSB-HR-03-0'	Dichloromethane	16U ug/Kg	A
F8A260143	TSB-HR-03-10'**	Dichloromethane	15U ug/Kg	A
F8A260143	TSB-HJ-02-0'**	Dichloromethane	12U ug/Kg	A
F8A260143	TSB-HJ-02-10'**	Dichloromethane	8.8U ug/Kg	A
F8A260143	TSB-HR-02-0'**	Dichloromethane	7.1U ug/Kg	A



SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P
F8A260143	TSB-HR-02-10'***	Dichloromethane	8.3U ug/Kg	A
F8A260143	TSB-HJ-11-0'***	Dichloromethane	5.2U ug/Kg	A
F8A260143	TSB-HJ-11-10'	Dichloromethane	6.8U ug/Kg	A
F8A260143	TSB-TB-2-1/25/08	Dichloromethane	1.0U ug/L	A
F8A260143	TSB-TB-03-1/25/08	Dichloromethane	1.0U ug/L	A
F8A260143	TSB-TB-04-1/25/08	Dichloromethane	1.0U ug/L	A
F8A260143	TRIP BLANK-TB-05	Dichloromethane	1.0U ug/L	A

**BRC Tronox Parcel H  
Volatiles - Field Blank Data Qualification Summary - SDG F8A260143**

SDG	Sample	Compound	Modified Final Concentration	A or P
F8A260143	TSB-HJ-01-10'	Dichloromethane	21U ug/Kg	A
F8A260143	TSB-HJ-09-0'	Dichloromethane	15U ug/Kg	A
F8A260143	TSB-HJ-09-10'	Dichloromethane	14U ug/Kg	A
F8A260143	TSB-HJ-03-0'	Dichloromethane Acetone	14U ug/Kg 21U ug/Kg	A
F8A260143	TSB-HJ-03-0'-FD	Dichloromethane	12U ug/Kg	A
F8A260143	TSB-HJ-03-10'	Dichloromethane Acetone	15U ug/Kg 21U ug/Kg	A
F8A260143	TSB-HR-03-0'	Dichloromethane	16U ug/Kg	A
F8A260143	TSB-HR-03-10'***	Dichloromethane	15U ug/Kg	A
F8A260143	TSB-HJ-02-0'***	Dichloromethane	12U ug/Kg	A
F8A260143	TSB-HJ-02-10'***	Dichloromethane Acetone	8.8U ug/Kg 21U ug/Kg	A

SDG	Sample	Compound	Modified Final Concentration	A or P
F8A260143	TSB-HR-02-0'***	Dichloromethane Acetone	7.1U ug/Kg 21U ug/Kg	A
F8A260143	TSB-HR-02-10'***	Dichloromethane Acetone	8.3U ug/Kg 21U ug/Kg	A
F8A260143	TSB-HJ-11-0'***	Dichloromethane	5.2U ug/Kg	A
F8A260143	TSB-HJ-11-10'	Dichloromethane	6.8U ug/Kg	A

LDC #: 18356A1  
 SDG #: F8A260143  
 Laboratory: Test America

### VALIDATION COMPLETENESS WORKSHEET

Level III/IV

Date: 3/5/08  
 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	A	Sampling dates: 1/25/08
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	SW	% PSD, r <sup>2</sup> 30.990
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	SW	LCS
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	SW	
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	SW	D = 4, 5      14, 15
XVII.	Field blanks	SW	R = 22, 23      TB = 24, 19, 20, 21

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-01-10'	11 3	TSB-HR-02-0**	2	21 1	TSB-TB-04-1/25/08 W	3 31 2	RINSATE-1REMS W
2 3	TSB-HJ-09-0'	12 3	TSB-HR-02-10**	2	22 1	RINSATE-1 W	32 2	RINSATE-1REMSD W
3 3	TSB-HJ-09-10'	13 3	TSB-HJ-11-0**	3	23 2	RINSATE-1RE W	33 1	8031135
4 3	TSB-HJ-03-0' 0	14 3	TSB-HJ-11-10' D	3	24 1	TRIP BLANK-TB-05 W	34 3	8039017
5 3	TSB-HJ-03-0'-FD D	15 4	TSB-HJ-11-10'-FD D	3	25 3	TSB-HJ-02-10'MS	35 2	8036136
6 3	TSB-HJ-03-10'	16 4	TSB-HR-01-0'	3	26 3	TSB-HJ-02-10'MSD	36 4	8043261
7 3	TSB-HR-03-0'	17 4	TSB-HR-01-10'	3	27 4	TSB-HJ-01-0'MS	37	
8 3	TSB-HR-03-10**	18 4	TSB-HJ-01-0'	3	28 4	TSB-HJ-01-0'MSD	38	
9 3	TSB-HJ-02-0**	19 1	2-Nonanal TSB-TB-2-1/25/08 W	1	29 1	RINSATE-1MS W	39	
10 3	TSB-HJ-02-10**	20 1	TSB-TB-03-1/25/08 W	2	30 1	RINSATE-1MSD W	40	

TB VOA's for 1-3 Broken

1/30  
2/7  
2/4  
2/8

LDC #: 18356A1  
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2  
 Reviewer: F7  
 2nd Reviewer: [Signature]

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 > 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 type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>IV. 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>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 checked="" type="checkbox"/>	<input 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 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 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 #: 18356A  
 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 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 type="checkbox"/>	<input checked="" 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. Tentatively identified compounds (TICs)</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 $\pm 20\%$ 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**  
**Field Blanks**

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

LDC #: 1820067  
SDG #: pu covered

**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: Field Blank

Associated Samples: All soils

Compound	Blank ID 22		Blank ID 23		Sample Identification							
	12508	12508	10	1.8	1	2	3	4	5	6	7	8
Dichloromethane												
Methylene chloride												
Ethyl Ether												
Acetone												
Chloroform												
CRQL												

Blank units: ug/L Associated sample units: ug/L  
Field blank type: (circle one) Field Blank / Rinsate / Trip Blank / Other: TB Associated Samples: 22, 23, 7, 10, X

Compound	Blank ID 24		Blank ID		Sample Identification							
	12508	0.19	4.7									
Dichloromethane												
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

## Blanks

LDC #: 18356A  
 SDG #: free cover

Page: 1 of 2  
 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 Was a method blank associated with every sample in this SDG? N/A
- Y Was a method blank analyzed at least once every 12 hours for each matrix and concentration? N/A
- Y Was there contamination in the method blanks? if yes, please see the qualifications below. N/A

Blank analysis date: 2-7-08

Conc. units: ug/kg

Associated Samples: 1-14

Compound	Blank ID	Sample Identification								
<u>8034077-Blank</u>	<u>13</u>	1	2	3	4	5	6	7	8	9
<u>1,1,1-trichloroethane</u>		21/u	15/u	14/u	14/u	12/u	15/u	-16/u	15/u	12/u
<u>Methylene chloride</u>										
<u>Acetone</u>										
<u>CRQL</u>										

Blank analysis date: 2-7-08  
 Conc. units: ug/kg

Associated Samples: 1-14

Compound	Blank ID	Sample Identification								
<u>8034077-Blank</u>	<u>13</u>	10	11	12	13	14				
<u>1,1,1-trichloroethane</u>		8.8/u	7.1/u	8.3/u	4.9/u	5.7	6.8/u			
<u>Methylene chloride</u>										
<u>Acetone</u>										
<u>CRQL</u>										

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

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

## VALIDATION FINDINGS WORKSHEET

### Blanks

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: 1/20/08

Conc. units: ug/L

Associated Samples: 19-22-24

Compound	Blank ID	Sample Identification				
Dichloromethane	SO31135- Blank	19	20	21	22	24
Methylenechloride	0.16	0.19/1.04	0.14/1.04	0.19/1.04	(12)	0.19/1.04
Acetone						
CRQL						

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

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

Compound	Blank ID	Sample Identification				
Dichloromethane						
Methylenechloride						
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".

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

Page: 4 of 4  
Reviewer: [Signature]  
2nd Reviewer: [Signature]

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: Field Blank

Associated Samples: 4-7-8

Compound	Blank ID	Blank ID	Sample Identification
Dichloromethane	1/25/08	4	6
Methylene chloride	0.19	(N)	-
Acetone	4.5	11/2/11	5.8/2/11
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: 9-7-12

Compound	Blank ID	Blank ID	Sample Identification
Dichloromethane	1/25/08	10	11
Methylene chloride	0.14	-	-
Acetone	4.9	9.7/2/11	9.3/2/11 9.5/2/11
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**

Page: 5 of 5  
Reviewer: [Signature]  
2nd Reviewer: [Signature]

LDC #: 18706A1  
SDG #: pre cover

**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:** 474 **Associated sample units:** 474  
**Field blank type:** (circle one) Field Blank / Rinsate / Trip Blank / Other: Field Blank

Associated Samples: 13 - 18 (ND + 70X)

Compound	Blank ID	Blank ID	Blank ID	Sample Identification
<del>Sample Blanks</del>	125.108			
Dichloro methane	0.19			
Methylene chloride	4.6			
Acetone				
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	Blank ID	Sample Identification
<del>Sample Blanks</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".

LDC #: 18356A

VALIDATION FINDINGS WORKSHEET

SDG #: full cover

Surrogate Spikes

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 Were all surrogate %R within QC limits?

Y/N 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?

#	Date	Sample ID	Surrogate	%Recovery (Limits)	Qualifications
		8036136 - Blank	BFB	126 (66-115)	5+ / P NP
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

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

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

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 a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.  
 Was a MS/MSD analyzed every 20 samples of each matrix?  
 Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

Y / N / N/A  
Y / N / N/A

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		<u>25 + 26</u>	<u>B</u>	<u>159 (39-150)</u>	( )	( )	<u>10</u>	<u>NO QUAL MS/DIN</u>
			<u>A</u>	( )	( )	<u>34 (30)</u>		<u>MS/DIN</u>
			<u>C</u>	( )	( )	<u>46</u>		
			<u>D</u>	( )	( )	<u>33</u>		<u>↓</u>
			<u>F</u>	( )	<u>196 (30-150)</u>	<u>38</u>		<u>MS/DIN</u>
			<u>H</u>	( )	( )	<u>49</u>		<u>MS/DIN</u>
			<u>G</u>	( )	( )	<u>48</u>		
			<u>J</u>	( )	( )	<u>45</u>		
			<u>N</u>	( )	( )	<u>44</u>		
			<u>B</u>	( )	( )	<u>44</u>		
			<u>S</u>	( )	( )	<u>36</u>		
			<u>CC</u>	( )	( )	<u>33</u>		
			<u>RRR</u>	( )	( )	<u>41</u>		
			<u>SSS</u>	( )	( )	<u>36</u>		
			<u>EE</u>	( )	( )	<u>39</u>		
			<u>FF</u>	( )	( )	<u>34</u>		
			<u>AA</u>	( )	( )	<u>36</u>		
			<u>III</u>	( )	( )	<u>39</u>	<u>↓</u>	<u>↓</u>

Compound	QC Limits (Soil)	RPD (Soil)	QC Limits (Water)	RPD (Water)
H. 1,1-Dichloroethene	59-172%	< 22%	61-145%	< 14%
S. Trichloroethene	62-137%	< 24%	71-120%	< 14%
V. Benzene	66-142%	< 21%	76-127%	< 11%
CC. Toluene	59-139%	< 21%	76-125%	< 13%
DD. Chlorobenzene	60-133%	< 21%	75-130%	< 13%



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

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

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

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

N N/A  
N N/A  
Y N/A

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		<u>25 + 26</u>	<u>EEE</u>	( )	( )	<u>42 ( 30 )</u>	<u>10</u>	<u>no qual</u>
			<u>CCC</u>	( )	( )	<u>39 ( )</u>		
			<u>ZZZ</u>	( )	( )	<u>43 ( )</u>		
			<u>BBB</u>	( )	( )	<u>38 ( )</u>		
			<u>JJJ</u>	( )	( )	<u>55 ( )</u>		
			<u>PPP</u>	( )	<u>53 (54-135)</u>	<u>64 ( )</u>		
			<u>QQQ</u>	( )	<u>49 (51-143)</u>	<u>39 ( )</u>		
			<u>TTT</u>	( )	<u>47 (49-133)</u>	<u>46 ( )</u>	<u>✓</u>	<u>✓</u>
			<u>VVV</u>	( )	( )	<u>53 ( )</u>		
			<u>p-cymene</u>	( )	( )	<u>48 ( )</u>		
			<u>LL</u>	( )	( )	<u>42 ( )</u>		
			<u>YY</u>	( )	( )	<u>31 ( )</u>		
			<u>KK</u>	( )	( )	<u>38 ( )</u>		
			<u>AAA</u>	( )	( )	<u>58 ( )</u>		
			<u>Iodomethane</u>	( )	( )	<u>37 ( ✓ )</u>		
				( )	( )	<u>62 ( 20 )</u>	<u>✓</u>	<u>✓</u>
				( )	( )	( )		
				( )	( )	( )		
<b>Compound</b>								
H.		1,1-Dichloroethene		QC Limits (Soil)		RPD (Soil)	QC Limits (Water)	RPD (Water)
S.		Trichloroethene		59-172%		< 22%	61-145%	< 14%
V.		Benzene		62-137%		< 24%	71-120%	< 14%
CC.		Toluene		66-142%		< 21%	76-127%	< 11%
DD.		Chlorobenzene		59-139%		< 21%	76-125%	< 13%
				60-133%		< 21%	75-130%	< 13%









LDC #: 18356A/  
 SDG #: See cover

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

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

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 (ug/kg)		Difference RPD
	4	5	
Dichloromethane	14	12	2 ( $\leq 5.3$ )

Compound	Concentration (ug/kg)		Difference RPD
	14	15	
↓	6.8	5.34	1.5 ( $\leq 5.3$ )

Compound	Concentration ( )		RPD

Compound	Concentration ( )		RPD

LDC #: 18356A  
 SDG #: per 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:

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

$A_x$  = Area of compound,  
 $C_{x_i}$  = Concentration of compound,  
 $S$  = Standard deviation of the RRFs  
 $X$  = Mean of the RRFs

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				RRF (SD std)	RRF (SD std)	RRF (SD std)	RRF (SD std)	Average RRF (Initial)	Average RRF (Initial)	%RSD	%RSD
1	1CAL	2/6/08	Vinyl chloride (1st internal standard)	0.61961	0.61961	0.61961	0.61961	0.67623	0.67623	6.75051	6.75051
			Ethyl Benzene (2nd internal standard)	2.68251	2.68251	2.68251	2.68251	2.67278	2.67278	5.11635	5.11635
			1,2-DCB (3rd internal standard)	2.13917	2.13917	2.13917	2.13917	2.22191	2.22191	3.97087	3.97087
2			2,2-Dimethyl pentane (1st internal standard) (2500)	0.00386	0.00386	0.00386	0.00386	0.00366	0.00366	14.57825	14.578
			Ethanol (2nd internal standard)	0.80440	0.80440	0.80440	0.80440	0.76134	0.76134	15.15158	15.15
			1,3,5-TCB (3rd internal standard)	1.81058	1.81058	1.81058	1.81058	1.73263	1.73263	8.90652	8.9065
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 #: 18356A1  
 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 =  $100 \times (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$   
 $\text{RRF} = (A_x / C_x) / (A_b / C_b)$

Where: ave. RRF = initial calibration average RRF

RRF = continuing calibration RRF

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

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

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

C<sub>b</sub> = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference internal Standard)	Average RRF (Initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	XCAL0118	2/7/08	Vinyl chloride	0.67623	0.71037	5.04928	5.04928	5.04928
	11:30		Ethyl Benzene	2.67278	2.80238	4.84908	4.84908	4.84908
			1,2-DCP	2.22191	2.21637	0.24947	0.24947	0.24947
2	XCAL0119	2/7/08	Ethanol	0.00366	0.00337	7.89253	7.89253	7.89253
	11:59		Dimethyl Sulfoxide	0.76134	0.74864	1.66819	1.66819	1.66819
			1,3,5-TCB	1.73263	1.73879	0.35591	0.35591	0.35591
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 #: 18356 A1  
 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: 8

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8	50	49.1718	98	98	0
Bromofluorobenzene	↓	47.0564	94	94	↓
1,2-Dichloroethane-d4	↓	51.6740	103	103	↓
Dibromofluoromethane	↓	56.0272	112	112	↓

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 #: 18356 A1  
 SDG #: per cover

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

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

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

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

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

RPD =  $100 * |MSC - MSDC| * 2 / (MSC + MSDC)$       MSC = Matrix spike percent recovery      MSDC = Matrix spike duplicate percent recovery

MS/MSD sample:       X + 26      

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	Percent Recovery		Percent Recovery		Reported	Recalculated
						Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
1,1-Dichloroethene	52.9	53.6	ND	53.5 55.5	32.6 61	101	101	61	61	49	49
Trichloroethene				48.8	33.9	92	92	63	63	36	36
Benzene				43.9	32.5	83	83	61	61	30	30
Toluene				42.6	30.5	81	81	57	57	33	33
Chlorobenzene				43.0	32.0 53.6	81	81	60 57	60	29	29

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

LDC #: 18 356A1  
 SDG #: full control

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

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

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 =  $|(LCS - LCSD) \cdot 2 / (LCS + LCSD)|$

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 8039017

Compound	Spike Added (ug/l)		Spiked Sample Concentration (ug/l)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
1,1-Dichloroethene	50.0	NA	57.1	NA	114	114				
Trichloroethene			49.8		100	100				
Benzene			50.2		100	100				
Toluene			49.6		99	99				
Chlorobenzene			49.0		98	98	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 #: 1835BA1  
 SDG #: pu cover

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

Page: 1 of 1  
 Reviewer: [Signature]  
 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 =  $\frac{(A_x)(I_s)(DF)}{(A_s)(RRF)(V_o)(\%S)}$

$A_x$  = Area of the characteristic ion (EICP) for the compound to be measured  
 $A_s$  = Area of the characteristic ion (EICP) for the specific internal standard  
 $I_s$  = Amount of internal standard added in nanograms (ng)  
 RRF = Relative response factor of the calibration standard.  
 $V_o$  = 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. #1, E:  
 Conc. =  $\frac{(13.88939)(5)}{(4.99)(0.945)}$   
 = 14.7ug/kg

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		$y = \frac{129531}{709774}$			
		$y = 13.88939$			
		$\frac{48}{50} = \frac{(129531)}{(709774)} / (0.40156) = 0.17668$			

**BRC Tronox Parcel H  
Data Validation Reports  
LDC# 18356**

Semivolatiles

*LDC*

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** BRC Tronox Parcel H  
**Collection Date:** January 25, 2008  
**LDC Report Date:** March 6, 2008  
**Matrix:** Soil/Water  
**Parameters:** Semivolatiles  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8A260143

### Sample Identification

TSB-HJ-01-10'	TSB-HJ-02-10'MSD
TSB-HJ-09-0'	TSB-HJ-01-0'MS
TSB-HJ-09-10'	TSB-HJ-01-0'MSD
TSB-HJ-03-0'	RINSATE-1MS
TSB-HJ-03-0'-FD	RINSATE-1MSD
TSB-HJ-03-10'	
TSB-HR-03-0'	
TSB-HR-03-10'**	
TSB-HJ-02-0'**	
TSB-HJ-02-10'**	
TSB-HR-02-0'**	
TSB-HR-02-10'**	
TSB-HJ-11-0'**	
TSB-HJ-11-10'	
TSB-HJ-11-10'-FD	
TSB-HR-01-0'	
TSB-HR-01-10'	
TSB-HJ-01-0'	
RINSATE-1	
TSB-HJ-02-10'MS	

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 22 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 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 water samples in SDG F8A260143	None	P
2/7/08 (KCAL4379)	Pentachlorophenol	21.13833	All soil samples in SDG F8A260143	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
2/7/08 (KCAL4381)	N-Hydroxymethylphthalimide	26.61142	All soil samples in SDG F8A260143	J- (all detects) UJ (all non-detects)	A

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

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

## 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
8031298-Blank	1/31/08	Unknown aldol condensate (4.2679) Unknown aldol condensate (4.7594)	22000 ug/Kg 330 ug/Kg	All soil samples in SDG F8A260143

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-01-10'	Unknown aldol condensate (4.2684) Unknown aldol condensate (4.7652)	23000 ug/Kg 370 ug/Kg	23000U ug/Kg 370U ug/Kg
TSB-HJ-09-0'	Unknown aldol condensate (4.2734) Unknown aldol condensate (4.7649)	23000 ug/Kg 360 ug/Kg	23000U ug/Kg 360U ug/Kg
TSB-HJ-09-10'	Unknown aldol condensate (4.2635) Unknown aldol condensate (4.7604)	23000 ug/Kg 360 ug/Kg	23000U ug/Kg 360U ug/Kg
TSB-HJ-03-0'	Unknown aldol condensate (4.2732) Unknown aldol condensate (4.7701)	20000 ug/Kg 310 ug/Kg	20000U ug/Kg 310U ug/Kg
TSB-HJ-03-0'-FD	Unknown aldol condensate (4.2626) Unknown aldol condensate (4.7594)	20000 ug/Kg 310 ug/Kg	20000U ug/Kg 310U ug/Kg

Sample	Compound TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
TSB-HJ-03-10'	Unknown aldol condensate (4.2643) Unknown aldol condensate (4.7612)	22000 ug/Kg 350 ug/Kg	22000U ug/Kg 350U ug/Kg
TSB-HR-03-0'	Unknown aldol condensate (4.2599) Unknown aldol condensate (4.7567)	23000 ug/Kg 360 ug/Kg	23000U ug/Kg 360U ug/Kg
TSB-HR-03-10'***	Unknown aldol condensate (4.2604) Unknown aldol condensate (4.7572)	23000 ug/Kg 360 ug/Kg	23000U ug/Kg 360U ug/Kg
TSB-HJ-02-0'***	Unknown aldol condensate (4.2789) Unknown aldol condensate (4.7704)	24000 ug/Kg 370 ug/Kg	24000U ug/Kg 370U ug/Kg
TSB-HJ-02-10'***	Unknown aldol condensate (4.2687) Unknown aldol condensate (4.7602)	25000 ug/Kg 400 ug/Kg	25000U ug/Kg 400U ug/Kg
TSB-HR-02-0'***	Unknown aldol condensate (4.2782) Unknown aldol condensate (4.7644)	24000 ug/Kg 390 ug/Kg	2000U ug/Kg 390U ug/Kg
TSB-HR-02-10'***	Unknown aldol condensate (4.2652) Unknown aldol condensate (4.762)	24000 ug/Kg 370 ug/Kg	24000U ug/Kg 370U ug/Kg
TSB-HJ-11-0'***	Unknown aldol condensate (4.259) Unknown aldol condensate (4.7611)	18000 ug/Kg 270 ug/Kg	18000U ug/Kg 270U ug/Kg
TSB-HJ-11-10'	Unknown aldol condensate (4.2631) Unknown aldol condensate (4.76)	23000 ug/Kg 370 ug/Kg	23000U ug/Kg 370U ug/Kg
TSB-HJ-11-10'-FD	Unknown aldol condensate (4.2606) Unknown aldol condensate (4.7574)	21000 ug/Kg 330 ug/Kg	21000U ug/Kg 330U ug/Kg
TSB-HR-01-0'	Unknown aldol condensate (4.2728) Unknown aldol condensate (4.7643)	25000 ug/Kg 380 ug/Kg	25000U ug/Kg 380U ug/Kg
TSB-HR-01-10'	Unknown aldol condensate (4.2701) Unknown aldol condensate (4.7616)	24000 ug/Kg 380 ug/Kg	24000U ug/Kg 380U ug/Kg
TSB-HJ-01-0'	Unknown aldol condensate (4.2714) Unknown aldol condensate (4.7629)	24000 ug/Kg 390 ug/Kg	24000U ug/Kg 390U ug/Kg

Sample "RINSATE-1" 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. Although the MS percent recoveries (%R) and MS/MSD relative percent differences (RPD) were not within QC limits for some compounds, the MSD percent recoveries (%R) were within QC limits and no data were qualified.

## **VIII. Laboratory Control Samples (LCS)**

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

## **IX. Regional Quality Assurance and Quality Control**

Not applicable.

## **X. Internal Standards**

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

## **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-03-0' and TSB-HJ-03-0'-FD and samples TSB-HJ-11-10' and TSB-HJ-11-10'-FD were identified as field duplicates. No semivolatiles were detected in any of the samples.

**BRC Tronox Parcel H  
Semivolatiles - Data Qualification Summary - SDG F8A260143**

SDG	Sample	Compound	Flag	A or P	Reason
F8A260143	RINSATE-1 TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'*** TSB-HJ-02-0'*** TSB-HJ-02-10'*** TSB-HR-02-0'*** TSB-HR-02-10'*** TSB-HJ-11-0'*** TSB-HJ-11-10' TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0'	Pentachlorophenol	None	P	Continuing calibration (CCC %D)
F8A260143	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'*** TSB-HJ-02-0'*** TSB-HJ-02-10'*** TSB-HR-02-0'*** TSB-HR-02-10'*** TSB-HJ-11-0'*** TSB-HJ-11-10' TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0'	N-Hydroxymethylphthalimide	J- (all detects) UJ (all non-detects)	A	Continuing calibration (%D)

**BRC Tronox Parcel H  
Semivolatiles - Laboratory Blank Data Qualification Summary - SDG F8A260143**

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P
F8A260143	TSB-HJ-01-10'	Unknown aldol condensate (4.2684) Unknown aldol condensate (4.7652)	23000U ug/Kg 370U ug/Kg	A
F8A260143	TSB-HJ-09-0'	Unknown aldol condensate (4.2734) Unknown aldol condensate (4.7649)	23000U ug/Kg 360U ug/Kg	A

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P
F8A260143	TSB-HJ-09-10'	Unknown aldol condensate (4.2635) Unknown aldol condensate (4.7604)	23000U ug/Kg 360U ug/Kg	A
F8A260143	TSB-HJ-03-0'	Unknown aldol condensate (4.2732) Unknown aldol condensate (4.7701)	20000U ug/Kg 310U ug/Kg	A
F8A260143	TSB-HJ-03-0'-FD	Unknown aldol condensate (4.2626) Unknown aldol condensate (4.7594)	20000U ug/Kg 310U ug/Kg	A
F8A260143	TSB-HJ-03-10'	Unknown aldol condensate (4.2643) Unknown aldol condensate (4.7612)	22000U ug/Kg 350U ug/Kg	A
F8A260143	TSB-HR-03-0'	Unknown aldol condensate (4.2599) Unknown aldol condensate (4.7567)	23000U ug/Kg 360U ug/Kg	A
F8A260143	TSB-HR-03-10'***	Unknown aldol condensate (4.2604) Unknown aldol condensate (4.7572)	23000U ug/Kg 360U ug/Kg	A
F8A260143	TSB-HJ-02-0'***	Unknown aldol condensate (4.2789) Unknown aldol condensate (4.7704)	24000U ug/Kg 370U ug/Kg	A
F8A260143	TSB-HJ-02-10'***	Unknown aldol condensate (4.2687) Unknown aldol condensate (4.7602)	25000U ug/Kg 400U ug/Kg	A
F8A260143	TSB-HR-02-0'***	Unknown aldol condensate (4.2782) Unknown aldol condensate (4.7644)	2000U ug/Kg 390U ug/Kg	A
F8A260143	TSB-HR-02-10'***	Unknown aldol condensate (4.2652) Unknown aldol condensate (4.762)	24000U ug/Kg 370U ug/Kg	A
F8A260143	TSB-HJ-11-0'***	Unknown aldol condensate (4.259) Unknown aldol condensate (4.7611)	18000U ug/Kg 270U ug/Kg	A
F8A260143	TSB-HJ-11-10'	Unknown aldol condensate (4.2631) Unknown aldol condensate (4.76)	23000U ug/Kg 370U ug/Kg	A
F8A260143	TSB-HJ-11-10'-FD	Unknown aldol condensate (4.2606) Unknown aldol condensate (4.7574)	21000U ug/Kg 330U ug/Kg	A
F8A260143	TSB-HR-01-0'	Unknown aldol condensate (4.2728) Unknown aldol condensate (4.7643)	25000U ug/Kg 380U ug/Kg	A
F8A260143	TSB-HR-01-10'	Unknown aldol condensate (4.2701) Unknown aldol condensate (4.7616)	24000U ug/Kg 380U ug/Kg	A

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P
F8A260143	TSB-HJ-01-0'	Unknown aldol condensate (4.2714) Unknown aldol condensate (4.7629)	24000U ug/Kg 390U ug/Kg	A

**BRC Tronox Parcel H  
Semivolatiles - Field Blank Data Qualification Summary - SDG F8A260143**

No Sample Data Qualified in this SDG



LDC #: 18356A2

## VALIDATION COMPLETENESS WORKSHEET

SDG #: F8A260143

Level III/IV

Laboratory: Test America

Date: 3/5/08

Page: 1 of 1

Reviewer: F7

2nd Reviewer: 

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

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

	Validation Area		Comments
I.	Technical holding times	Δ	Sampling dates: 1/25/08
II.	GC/MS Instrument performance check	Δ	
III.	Initial calibration	Δ	%RSD, 1 <sup>2</sup> 20.990
IV.	Continuing calibration/ICV	SW	
V.	Blanks	ASW	
VI.	Surrogate spikes	Δ	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	SW	LCS
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	Δ	
XVI.	Field duplicates	ND	D = 4 + 5    14 + 15
XVII.	Field blanks	NP	R = 19

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 + water

1	TSB-HJ-01-10'	11	TSB-HR-02-0**	21	TSB-HJ-02-10'MSD	31	8029233
2	TSB-HJ-09-0'	12	TSB-HR-02-10**	22	TSB-HJ-01-0'MS	32	8031298
3	TSB-HJ-09-10'	13	TSB-HJ-11-0**	23	TSB-HJ-01-0'MSD	33	
4	TSB-HJ-03-0'	14	TSB-HJ-11-10'	24	RINSATE-1MS	34	W
5	TSB-HJ-03-0'-FD	15	TSB-HJ-11-10'-FD	25	RINSATE-1MSD	35	W
6	TSB-HJ-03-10'	16	TSB-HR-01-0'	26		36	
7	TSB-HR-03-0'	17	TSB-HR-01-10'	27		37	
8	TSB-HR-03-10**	18	TSB-HJ-01-0'	28		38	
9	TSB-HJ-02-0**	19	RINSATE-1	29		39	W
10	TSB-HJ-02-10**	20	TSB-HJ-02-10'MS	30		40	

LDC #: 18356A2  
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

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

Method: Semivolatiles (EPA SW 846 Method 8270C)

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

LDC #: 18356A2  
 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"/>			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		<input checked="" type="checkbox"/>		
<b>IX. Regional Stability 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. Internal Standards</b>				
Were internal standard area counts within -50% or +100% of the associated calibration standard?	<input checked="" type="checkbox"/>			
Were retention times within + 30 seconds from the associated calibration standard?	<input checked="" type="checkbox"/>			
<b>XI. Target Compound Identification</b>				
Were relative retention times (RRT's) within + 0.06 RRT units of the standard?	<input checked="" type="checkbox"/>		<input checked="" type="checkbox"/>	
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	<input checked="" type="checkbox"/>		<input checked="" type="checkbox"/>	
Were chromatogram peaks verified and accounted for?	<input checked="" type="checkbox"/>	<input checked="" 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"/>	
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>XIII. Reference Identified Compounds (RICs)</b>				
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?	<input checked="" type="checkbox"/>			
Were relative intensities of the major ions within ± 20% between the sample and the reference spectra?	<input checked="" 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"/>			
<b>XIV. System Performance</b>				
System performance was found to be acceptable.	<input checked="" type="checkbox"/>			
<b>XV. Overall Assessment/Quality</b>				
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>			
<b>XVI. Field Data</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>XVII. 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"/>		

# 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. <del>N-(2-Hydroxy methyl)</del> Phthalimide
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.



**VALIDATION FINDINGS WORKSHEET**

**Blanks**

Page: 1 of 1  
 Reviewer: FB  
 2nd Reviewer: LB

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

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

- Y  N  N/A Was a 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: 1/31/08 Blank analysis date: 2/7/08

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

Compound	Blank ID	Sample Identification											
Unknown aldo condensate	22000 (4.2684)	23000 (4.2635)	20000 (4.2732)	20000 (4.2626)	22000 (4.2643)	23000 (4.2599)	23000 (4.2604)	360 (4.7594)	310 (4.7594)	310 (4.7701)	350 (4.7612)	360 (4.7567)	360 (4.7512)
Unknown aldo condensate	370 (4.7652)	360 (4.7649)	370 (4.7701)	360 (4.7604)	370 (4.7612)	360 (4.7567)	360 (4.7512)	370 (4.7611)	370 (4.7611)	370 (4.7611)	370 (4.7611)	370 (4.7611)	370 (4.7611)
	24000 (4.2789)	24000 (4.2782)	24000 (4.2652)	24000 (4.2659)	24000 (4.2631)	24000 (4.2604)	24000 (4.2628)	370 (4.7604)	370 (4.7611)	370 (4.7611)	370 (4.7611)	370 (4.7611)	370 (4.7611)
	370 (4.7704)	370 (4.7602)	370 (4.762)	370 (4.7611)	370 (4.7611)	370 (4.7574)	370 (4.7643)	370 (4.7611)	370 (4.7611)	370 (4.7611)	370 (4.7611)	370 (4.7611)	370 (4.7611)

Blank extraction date: 1/31/08 Blank analysis date: 2/7/08

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

Compound	Blank ID	Sample Identification											
Unknown aldo condensate	24000 (4.2701)	24000 (4.2714)	24000 (4.2714)	24000 (4.2714)	24000 (4.2714)	24000 (4.2714)	24000 (4.2714)	24000 (4.2714)	24000 (4.2714)	24000 (4.2714)	24000 (4.2714)	24000 (4.2714)	24000 (4.2714)
	380 (4.7616)	370 (4.7629)	370 (4.7629)	370 (4.7629)	370 (4.7629)	370 (4.7629)	370 (4.7629)	370 (4.7629)	370 (4.7629)	370 (4.7629)	370 (4.7629)	370 (4.7629)	370 (4.7629)

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**

Page: 101  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

LDC #: 18356A2  
 SDG #: pu canal

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
		22+23	HH	8 (10-91)	( )	32 (30)	18	no Qual
			BB	( )	( )	32 (30)	↓	↓
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
		24+25	HH	6 (14-94)	( )	77 (20)	19	no Qual
			PP	16 (28-96)	( )	74 (20)	↓	↓
			TT	( )	( )	46 (20)		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		

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%					





LDC #: 18356A2  
 SDG #: Au epny

VALIDATION FINDINGS WORKSHEET  
 Initial Calibration Calculation Verification

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

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

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

$RRF = (A_x/C_x)/(A_b/C_b)$   
 average RRF = sum of the RRFs/number of standards  
 $\%RSD = 100 * (S/X)$   
 $A_x$  = Area of compound,  
 $C_x$  = Concentration of compound,  
 $A_b$  = Area of associated internal standard,  
 $C_b$  = 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	
				RRF (5D std)	RRF (5D std)	RRF (5D std)	RRF (5D std)	Average RRF (initial)	%RSD	Average RRF (initial)	%RSD
1	1CAL	1/29/07	Phenol (1st internal standard)	2.66254	2.66254	2.69741	2.69741	6.25319	6.25319	2.69741	6.25319
			Naphthalene (2nd internal standard)	1.10277	1.10277	1.09527	1.09527	19.39092	10.39092	1.09527	10.39092
			Fluorene (3rd internal standard)	1.36978	1.36978	1.34878	1.34878	14.54450	14.54450	1.34878	14.54450
			Pentachlorophenol (4th internal standard)	0.15602	0.15602	0.15050	0.15050	11.84195	11.84195	0.15050	11.84195
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.8769	0.8769	0.81456	0.81456	7.06979	7.06979	0.81456	7.06979
			Benzo(a)pyrene (6th internal standard)	1.21257	1.21257	1.18939	1.18939	3.60350	3.60350	1.18939	3.60350
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 #: 15336A2  
 SDG #: see cover

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

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

**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 = continuing calibration RRF  
 $\text{RRF} = (A_s)(C_s) / (A_s)(C_s)$  A<sub>s</sub> = Area of associated internal standard  
 C<sub>s</sub> = Concentration of internal standard  
 A<sub>x</sub> = Area of compound, C<sub>x</sub> = Concentration of compound,

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	KCAL4379	2/7/08	Phenol (1st internal standard)	2.69741	2.62250	2.77722	2.62250	2.77722
			Naphthalene (2nd internal standard)	1.09527	1.10100	0.52331	1.10100	0.52331
			Fluorene (3rd internal standard)	1.34818	1.34358	0.38531	1.34358	0.38531
			Pentachlorophenol (4th internal standard)	0.18050	0.18231	2.13833	0.18231	2.13833
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.81456	0.79502	2.398	0.79502	2.398
			Benzo(a)pyrene (6th internal standard)	1.14939	1.21353	2.02920	1.21353	2.02920
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 #: 18356A2  
 SDG #: per cover

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

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

**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: 8

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	50	34.0522	68	68	0
2-Fluorobiphenyl	↓	35.5669	71	71	↓
Terphenyl-d14	↓	44.6857	89	89	
Phenol-d5	75	50.2596	67	67	
2-Fluorophenol	↓	46.9488	63	63	
2,4,6-Tribromophenol	↓	54.1342	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 #: 18 350 A2  
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  
SA = Spike added

SC = Sample concentration

RPD =  $100 * (MS - MSD) / (MS + MSD)$

MS = Matrix spike percent recovery

MSD = Matrix spike duplicate percent recovery

MS/MSD samples: 21 + 22

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	MS	MSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenol	3560	3530	(ug/kg)	(ug/kg)	2390	2480	67	67	70	70	3.5	3.5
N-Nitroso-di-n-propylamine					2580	2720	72	72	77	77	5.4	5.4
4-Chloro-3-methylphenol					2450	2740	74	74	78	78	3.6	3.6
Acenaphthene					2460	2510	69	69	71	71	1.8	1.8
Pentachlorophenol					2640	2460	74	74	70	70	7.0	7.0
Pyrene					2610	2700	74	74	77	77	3.4	3.4

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

LDC #: 18 33042

SDG #: see cover

### VALIDATION FINDINGS WORKSHEET

### Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

Page: 6 of 7

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 =  $100 * (LCS - LCSD) / (LCS + LCSD)$

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

LCS/LCSD samples: 8031298

Compound	Spike Added (ug/kg)		Spike Concentration		LCS		LCSD		Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenol	3330	NA	2180	NA	65	65								
N-Nitroso-di-n-propylamine			2330		70	70								
4-Chloro-3-methylphenol			2420		73	73								
Acenaphthene			2320		69	69								
Pentachlorophenol			2650		80	80								
Pyrene			2400		72	72			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 #: 18350A2

SDG #: no count

# 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?

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

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).

V<sub>i</sub> = Volume of extract injected in microliters (ul)

V<sub>c</sub> = 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. \_\_\_\_\_

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

=  
ND

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

**BRC Tronox Parcel H  
Data Validation Reports  
LDC# 18356**

**Chlorinated Pesticides**

*LDC*

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** BRC Tronox Parcel H  
**Collection Date:** January 25, 2008  
**LDC Report Date:** March 6, 2008  
**Matrix:** Soil/Water  
**Parameters:** Chlorinated Pesticides  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8A260143

### Sample Identification

TSB-HJ-01-10'	TSB-HR-01-10'
TSB-HJ-09-0'	TSB-HJ-01-0'
TSB-HJ-09-0'DL	RINSATE-1
TSB-HJ-09-0'RE	TSB-HJ-01-10'MS
TSB-HJ-09-0'REDL	TSB-HJ-01-10'MSD
TSB-HJ-09-10'	TSB-HJ-09-0'MS
TSB-HJ-03-0'	TSB-HJ-09-0'MSD
TSB-HJ-03-0'-FD	RINSATE-1MS
TSB-HJ-03-10'	RINSATE-1MSD
TSB-HR-03-0'	
TSB-HR-03-0'RE	
TSB-HR-03-10'**	
TSB-HJ-02-0'**	
TSB-HJ-02-10'**	
TSB-HR-02-0'**	
TSB-HR-02-10'**	
TSB-HJ-11-0'**	
TSB-HJ-11-10'	
TSB-HJ-11-10'-FD	
TSB-HR-01-0'	

\*\*Indicates sample underwent EPA Level IV review



## Introduction

This data review covers 26 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 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 with the following exceptions:

Sample	Compound	Total Days From Sample Collection Until Extraction	Required Holding Time (in Days) From Sample Collection Until Extraction	Flag	A or P
TSB-HJ-09-0'RE TSB-HJ-09-0'REDL TSB-HR-03-0'RE	All TCL compounds	19	14	J- (all detects) UJ (all non-detects)	A

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
2/11/08	KCAL735	RTX-CLP1	Toxaphene	26.5	TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0'	J+ (all detects)	A

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
2/11/08	KCAL735	RTX-CLP2	Toxaphene	23.6	TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0'	J+ (all detects)	A
2/14/08	KCAL955	RTX-CLP2	Heptachlor	15.1	TSB-HJ-09-0'RE TSB-HR-03-0'RE TSB-HJ-09-0'MS TSB-HJ-09-0'MSD 8044048-BLK	J+ (all detects)	A
2/14/08	KCAL955	RTX-CLP2	4,4'-DDT	20.4	TSB-HJ-09-0'RE TSB-HJ-09-0'REDL TSB-HR-03-0'RE TSB-HJ-09-0'MS TSB-HJ-09-0'MSD 8044048-BLK	J+ (all detects)	A

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

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
2/12/08	ICV	RTX-CLP1	Toxaphene	20.7	TSB-HJ-09-0'RE TSB-HR-03-0'RE TSB-HJ-09-0'MS TSB-HJ-09-0'MSD 8044048-BLK	J+ (all detects)	A
2/12/08	ICV	RTX-CLP2	Toxaphene	17.8	TSB-HJ-09-0'RE TSB-HR-03-0'RE TSB-HJ-09-0'MS TSB-HJ-09-0'MSD 8044048-BLK	J+ (all detects)	A

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

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

## V. Blanks

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

Sample "RINSATE-1" 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-01-10'	Not specified	Decachlorobiphenyl	118 (63-117)	All TCL compounds	J+ (all detects)	P
TSB-HJ-09-0'	Not specified	Decachlorobiphenyl	127 (63-117)	All TCL compounds	J+ (all detects)	A
TSB-HJ-03-0'	Not specified	Decachlorobiphenyl	121 (63-117)	All TCL compounds	J+ (all detects)	P
TSB-HJ-03-0'-FD	Not specified	Decachlorobiphenyl	118 (63-117)	All TCL compounds	J+ (all detects)	P
TSB-HJ-03-10'	Not specified	Decachlorobiphenyl	131 (63-117)	All TCL compounds	J+ (all detects)	P
TSB-HR-03-0'	Not specified	Decachlorobiphenyl	120 (63-117)	All TCL compounds	J+ (all detects)	A
TSB-HR-03-10'***	RTX-CLP1	Decachlorobiphenyl	121 (63-117)	All TCL compounds	J+ (all detects)	P
TSB-HR-02-0'***	RTX-CLP1	Decachlorobiphenyl	121 (63-117)	All TCL compounds	J+ (all detects)	P
TSB-HR-02-10'***	RTX-CLP1	Decachlorobiphenyl	123 (63-117)	All TCL compounds	J+ (all detects)	P
TSB-HJ-11-0'***	RTX-CLP1	Decachlorobiphenyl	128 (63-117)	All TCL compounds	J+ (all detects)	P
TSB-HJ-11-10'	Not specified	Decachlorobiphenyl Tetrachloro-m-xylene	126 (63-117) 120 (55-115)	All TCL compounds	J+ (all detects)	P
TSB-HJ-11-10'-FD	Not specified	Decachlorobiphenyl Tetrachloro-m-xylene	123 (63-117) 118 (55-115)	All TCL compounds	J+ (all detects)	P
TSB-HR-01-0'	Not specified	Decachlorobiphenyl Tetrachloro-m-xylene	134 (63-117) 120 (55-115)	All TCL compounds	J+ (all detects)	P
TSB-HR-01-10'	Not specified	Decachlorobiphenyl	122 (63-117)	All TCL compounds	J+ (all detects)	P
TSB-HJ-01-0'	Not specified	Decachlorobiphenyl	122 (63-117)	All TCL compounds	J+ (all detects)	P
8034041-BLK	Not specified	Decachlorobiphenyl	130 (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. Although the MS and MSD percent recoveries (%R) were not within QC limits for some compounds, LCS percent recovery (%R) was 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 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 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 with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P
TSB-HJ-09-0' TSB-HJ-09-0'RE	4,4'-DDE 4,4'-DDT	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	A

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-03-0' and TSB-HJ-03-0'-FD and samples TSB-HJ-11-10' and TSB-HJ-11-10'-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 F8A260143**

SDG	Sample	Compound	Flag	A or P	Reason
F8A260143	TSB-HJ-09-0'RE TSB-HJ-09-0'REDL TSB-HR-03-0'RE	All TCL compounds	J- (all detects) UJ (all non-detects)	A	Technical holding times
F8A260143	TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0'	Toxaphene	J+ (all detects)	A	Continuing calibration (%D)
F8A260143	TSB-HJ-09-0'RE TSB-HR-03-0'RE	Heptachlor	J+ (all detects)	A	Continuing calibration (%D)
F8A260143	TSB-HJ-09-0'RE TSB-HJ-09-0'REDL TSB-HR-03-0'RE	4,4'-DDT	J+ (all detects)	A	Continuing calibration (%D)
F8A260143	TSB-HJ-09-0'RE TSB-HR-03-0'RE	Toxaphene	J+ (all detects)	A	Continuing calibration (ICV %D)
F8A260143	TSB-HJ-09-0' TSB-HR-03-0'	All TCL compounds	J+ (all detects)	A	Surrogate spikes (%R)
F8A260143	TSB-HJ-01-10' TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-10'** TSB-HR-02-0'** TSB-HR-02-10'** TSB-HJ-11-0'** TSB-HJ-11-10' TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0'	All TCL compounds	J+ (all detects)	P	Surrogate spikes (%R)
F8A260143	TSB-HJ-09-0' TSB-HJ-09-0'RE	4,4'-DDE 4,4'-DDT	J (all detects) J (all detects)	A	Compound quantitation and CRQLs

**BRC Tronox Parcel H  
Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG  
F8A260143**

No Sample Data Qualified in this SDG



**BRC Tronox Parcel H  
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG F8A260143**

No Sample Data Qualified in this SDG

LDC #: 18356A3a  
 SDG #: F8A260143  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 3/4/08  
 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	SW	Sampling dates: 1/25/08
II.	GC/ECD Instrument Performance Check	A	
III.	Initial calibration	A	
IV.	Continuing calibration/ICV	SW	ICV = 15
V.	Blanks	A	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	SW	
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	SW	Not reviewed for Level III validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	ND	D = 7, 8 18, 19
XV.	Field blanks	ND	R = 23

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-01-10'	11	TSB-HR-03-0'RE	21	TSB-HR-01-10'	31	8034041-BLK
2	TSB-HJ-09-0'	12	TSB-HR-03-10**	22	TSB-HJ-01-0'	32	8044048-BLK
3	TSB-HJ-09-0'DL	13	TSB-HJ-02-0**	23	RINSATE-1 W	33	8029304-BLK
4	TSB-HJ-09-0'RE	14	TSB-HJ-02-10**	24	TSB-HJ-01-10'MS	34	
5	TSB-HJ-09-0'REDL	15	TSB-HR-02-0**	25	TSB-HJ-01-10'MSD	35	
6	TSB-HJ-09-10'	16	TSB-HR-02-10**	26	TSB-HJ-09-0'MS	36	
7	TSB-HJ-03-0' D	17	TSB-HJ-11-0**	27	TSB-HJ-09-0'MSD	37	
8	TSB-HJ-03-0'-FD D	18	TSB-HJ-11-10' D	28	RINSATE-1MS W	38	
9	TSB-HJ-03-10'	19	TSB-HJ-11-10'-FD D	29	RINSATE-1MSD W	39	
10	TSB-HR-03-0'	20	TSB-HR-01-0'	30		40	

LDC #: 18356A3a  
 SDG #: pu cones

VALIDATION FINDINGS CHECKLIST

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

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"/>		
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>			
<b>II. GC/ECD Instrument performance check</b>				
Was the instrument performance found to be acceptable?	<input checked="" type="checkbox"/>			
<b>III. Initial calibration</b>				
Did the laboratory perform a 5 point calibration prior to sample analysis?	<input checked="" type="checkbox"/>			
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) $\leq$ 20%?	<input checked="" type="checkbox"/>			
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?		<input checked="" type="checkbox"/>		
Did the initial calibration meet the curve fit acceptance criteria?			<input checked="" type="checkbox"/>	
Were the RT windows properly established?	<input checked="" type="checkbox"/>			
Were the required standard concentrations analyzed in the initial calibration?	<input checked="" type="checkbox"/>			
<b>IV. Continuing calibration</b>				
What type of continuing calibration calculation was performed? ___%D or ___%R	<input checked="" type="checkbox"/>			
Were Evaluation mix standards analyzed prior to the initial calibration and sample analysis?	<input checked="" type="checkbox"/>			
Were endrin and 4,4'-DDT breakdowns $\leq$ 15%.0 for individual breakdown in the Evaluation mix standards?	<input checked="" type="checkbox"/>			
Was a continuing calibration analyzed daily?	<input checked="" type="checkbox"/>			
Were all percent differences (%D) $\leq$ 15%.0 or percent recoveries 85-115%?		<input checked="" type="checkbox"/>		
Were all the retention times within the acceptance windows?	<input checked="" type="checkbox"/>			
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>			
Was a method blank analyzed for each matrix and concentration?	<input checked="" type="checkbox"/>			
Were extract cleanup blanks analyzed with every batch requiring clean-up?			<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 checked="" type="checkbox"/>		
<b>VI. Surrogate spikes</b>				
Were all surrogate %R within the QC limits?		<input checked="" 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"/>			
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?			<input checked="" type="checkbox"/>	

LDC #: 18356A3a  
 SDG #: per 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 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 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. 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 type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>XV. 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 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**  
**Surrogate Recovery**

LDC #: 18356A3  
 SDG #: per covered

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

METHOD: GC HPLC

Are surrogates required by the method? Yes or No

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

Were surrogates spiked into all samples and blanks?

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

#	Sample ID	Detector/Column	Surrogate Compound	%R (Limits)	Qualifications
1		not specified	θ	118 (63-117)	J <sup>+</sup> /Pdct NP
2		↓	↓	127 ( )	N/A det ND+Det
3, 5		↓	θ	DD (63-117)	no qual 10x PL
6		↓	γ	DD (55-115)	↓
7		↓	θ	121 (63-117)	J <sup>+</sup> /Pdct ND
8		↓	↓	118 ( )	↓ ND
9		↓	↓	131 ( )	↓ ND
10		↓	↓	120 ( )	J <sup>+</sup> /A det ND + Det
12		RX-CVP	↓	121 ( )	J <sup>+</sup> /Pdct ND
15		↓	↓	121 ( )	↓ ND
16		↓	↓	123 ( )	↓ ND

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 Tripentyltin	
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-Nitrobenzol	X Tributienyl Phosphate	



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

LDC #: 18356A2a  
 SDG #: per control

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

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
17		Rtx-cvp1	0	( 63-117 )	J <sup>+</sup> /P det ND
18		not specified	↓	( )	J <sup>+</sup> /P det ND
19		↓	↓	( 55-115 )	↓
20		↓	↓	( )	J <sup>+</sup> /P det ND
21		↓	0	( 63-117 )	J <sup>+</sup> /P det ND
22		↓	0	( )	J <sup>+</sup> /P det ND
8034041-B/R		↓	0	( )	J <sup>+</sup> /P det ND

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-Triacotane	P 1-methylnaphthalene	V Trif-n-propyllin	
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 Trichlorophosphate	

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

METHOD: GC HPLC

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".  
 N  A Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG?  
 N  A Was an MS/MSD analyzed every 20 samples for each matrix or whenever a sample extraction was performed?  
 N  A Were the MS/MSD percent recoveries (%R) and relative percent differences (RPD) within QC limits?

#	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
	26427	Ø	228 (52-140)	( )	( )	2 → 5	no qual
		J	143 (57-125)	47 (57-125)	( )	↓	↓
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
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			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		

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

LDC #: 18356 A34  
SDG #: per cover

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

METHOD:  GC  HPLC

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

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

#	LCS/LCSD ID	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
	8034041-LCS	P	133 (59-123)	( )	( )	8034041 - B/F, 1-13, 6-10, 12-122	NO OUA L
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )		
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			( )	( )	( )		

**VALIDATION FINDINGS WORKSHEET**  
Compound Quantitation and Reported CRQLs

METHOD:  GC  HPLC

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".  
 Level IV/D Only  
 Y N N/A Were CRQLs adjusted for sample dilutions, dry weight factors, etc.?  
 Y N N/A Did the reported results for detected target compounds agree within 10.0% of the recalculated results?

#	Compound Name	Finding	Associated Samples	Qualifications
	<u>1, 8</u>	<u>exceeded cal Range</u>	<u>2, 4</u>	<u>N/A det</u>

Comments: See sample calculation verification worksheet for recalculations

LDC #: 16356 A3a  
 SDG #: per count

**VALIDATION FINDINGS WORKSHEET**  
Initial Calibration Calculation Verification

Page: 1 of 1  
 Reviewer: JPE  
 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:

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

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

#	Standard ID	Calibration Date	Compound	Reported		Recalculated		Reported		Recalculated	
				CF (0.02 std)	CF (0.02 std)	Average CF (Initial)	Average CF (Initial)	%RSD	%RSD		
1	ICAL	2/7/08	endosulfan I methoxychlor ↓ ↓	264234520	264234520	265059470	265059470	5.585	5.585	4.736	4.736
				53596400	53596400	55334490	55334490				
2				568745040	568745040	572386340	572386340	6.294	6.294		
				240546960	240546960	24501240	24501240	6.787	6.787		
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 =  $100 \cdot (\text{ave. CF} - \text{CF}) / \text{ave. CF}$  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(ical)/ CCV Conc.	Reported		Recalculated	
					CF/Conc. CCV	%D	CF/Conc. CCV	%D
1	KCAL717	2/10/08	endosulfan I R1X-C1P1	0.0250	0.0245	2.2	2.2	
			methoxychlor ↓	↓	0.0757	2.6	2.6	
			↓ R1X-C1P2	↓	0.0266	6.4	6.4	
2					0.0259	3.7	3.7	
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.

LDC #: 18356A3  
 SDG #: du 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: 12

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene						
Tetrachloro-m-xylene	<u>ATXCP1</u>	<u>0.020</u>	<u>0.024</u>	<u>120</u>	<u>120</u>	<u>0</u>
Decachlorobiphenyl	<u>↓</u>	<u>↓</u>	<u>0.024</u>	<u>121</u>	<u>121</u>	<u>0</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 #: 18350A3a  
 SDG #: per event

**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  
 SA = Spike added

RPD =  $100 * |MS - MSD| / (MS + MSD)$

MS = Matrix spike percent recovery  
 MSD = Matrix spike duplicate percent recovery

MS/MSD samples: 24 + 25

Compound	Spike Added (ng/kg)		Sample Concentration (ng/kg)	Spiked Sample Concentration (ng/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	17.3	17.6	ND	16.9	15.9	97	97	90	90	5.9	5.9
4,4'-DDT	↓	17.6	↓	18.6	17.0	107	107	96	96	9.1	9.1

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 #: 183 SBABA

SDG #: per copy

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| \* 2 / (LCS + LCSD)

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

LCS/LCSD samples: 8034041 - LCS

Compound	Spike Added (ug/L)		Spiked Sample Concentration (ug/L)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
gamma-BHC	16.7	NA	15.5	NA	93	93				
4,4'-DDT	16.7	↓	17.3	↓	104	104	N/A			

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 #: 18356A3a  
 SDG #: pu cover

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

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

**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: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

**BRC Tronox Parcel H  
Data Validation Reports  
LDC# 18356**

Polychlorinated Biphenyls

*LDC*

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** BRC Tronox Parcel H  
**Collection Date:** January 25, 2008  
**LDC Report Date:** March 6, 2008  
**Matrix:** Soil/Water  
**Parameters:** Polychlorinated Biphenyls  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8A260143

### Sample Identification

TSB-HJ-01-10'	TSB-HJ-02-10'MSD
TSB-HJ-09-0'	TSB-HJ-01-0'MS
TSB-HJ-09-10'	TSB-HJ-01-0'MSD
TSB-HJ-03-0'	RINSATE-1MS
TSB-HJ-03-0'-FD	RINSATE-1MSD
TSB-HJ-03-10'	
TSB-HR-03-0'	
TSB-HR-03-10'**	
TSB-HJ-02-0'**	
TSB-HJ-02-10'**	
TSB-HR-02-0'**	
TSB-HR-02-10'**	
TSB-HJ-11-0'**	
TSB-HJ-11-10'	
TSB-HJ-11-10'-FD	
TSB-HR-01-0'	
TSB-HR-01-10'	
TSB-HJ-01-0'	
RINSATE-1	
TSB-HJ-02-10'MS	

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 22 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 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 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-HR-01-10' TSB-HJ-01-0' TSB-HJ-01-0'MS TSB-HJ-01-0'MSD	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.

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

## **V. Blanks**

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

Sample RINSATE-1 was identified as a rinsate. No polychlorinated biphenyl contaminants were found in this blank.

## **VI. Surrogate Spikes**

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

## **VII. Matrix Spike/Matrix Spike Duplicates**

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

## **VIII. Laboratory Control Samples (LCS)**

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

## **IX. Regional Quality Assurance and Quality Control**

Not applicable.

## **X. Pesticide Cleanup Checks**

### **a. Florisil Cartridge Check**

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

### **b. GPC Calibration**

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

## **XI. Target Compound Identification**

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



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

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

## **XIII. Overall Assessment of Data**

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

## **XIV. Field Duplicates**

Samples TSB-HJ-03-0' and TSB-HJ-03-0'-FD and samples TSB-HJ-11-10' and TSB-HJ-11-10'-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 F8A260143**

SDG	Sample	Compound	Flag	A or P	Reason
F8A260143	TSB-HR-01-10' TSB-HJ-01-0'	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 F8A260143**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel H  
Polychlorinated Biphenyls - Field Blank Data Qualification Summary - SDG F8A260143**

No Sample Data Qualified in this SDG

LDC #: 18356A3b  
 SDG #: F8A260143  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**

Level III/IV

Date: 3/3/08  
 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: 1/25/08
II.	GC/ECD Instrument Performance Check	NA	
III.	Initial calibration	A	
IV.	Continuing calibration/ICV	SW	ICV ≤ 15
V.	Blanks	A	
VI.	Surrogate spikes	A	
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	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 = 4, 5      14 + 15
XV.	Field blanks	ND	R = 19

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-01-10'	11	TSB-HR-02-0**	21	TSB-HJ-02-10'MSD	31	8031454	2/4
2	TSB-HJ-09-0'	12	TSB-HR-02-10**	22	TSB-HJ-01-0'MS	32	8029346	1/30
3	TSB-HJ-09-10'	13	TSB-HJ-11-0**	23	TSB-HJ-01-0'MSD	33		
4	TSB-HJ-03-0' D	14	TSB-HJ-11-10' D	24	RINSATE-1MS W	34		
5	TSB-HJ-03-0'-FD D	15	TSB-HJ-11-10'-FD D	25	RINSATE-1MSD W	35		
6	TSB-HJ-03-10'	16	TSB-HR-01-0'	26		36		
7	TSB-HR-03-0'	17	TSB-HR-01-10'	27		37		
8	TSB-HR-03-10**	18	TSB-HJ-01-0'	28		38		
9	TSB-HJ-02-0**	19	RINSATE-1 W	29		39		
10	TSB-HJ-02-10**	20	TSB-HJ-02-10'MS	30		40		

LDC #: 18356A3b  
 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% 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 checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

LDC #: 18356A-3b  
 SDG #: pu 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

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:



LDC #: 18356A3b  
 SDG #: for con

**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 (12182)	Average CF (Initial)	Average CF (Initial)	%RSD	%RSD		
1	1CAL	9/6/07	1260-1 ↓ RTX-emp1	12182	12182	11502	11502	9.589	9.589	9.589	9.589
2	1CAL	9/6/07	↓ RTX-emp2	11265	11265	11104	11104	8.799	8.799	8.799	8.799
3	1CAL	1/02/08	↓ RTX-emp1	22828	22828	21742	21742	7.597	7.597	7.597	7.597
4	1CAL	1/2/08	↓ RTX-emp2	28897	28897	28014	28014	5.089	5.089	5.089	5.089

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 #: 18256A3b  
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 =  $100 \times (\text{ave. CF} - \text{CF}) / \text{ave. CF}$   
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 (cal)/CCV Conc.	Reported		Recalculated	
					CF/Conc. CCV	%D	CF/Conc. CCV	%D
1	PCAL519	2/4/08	Amoclor 1260 RT(xe?)	1000	918.0254	718.0254	8.2	
2	PCAL530	2/4/08	Amoclor 1260 RT(xe?)	1000	958.0012	958.0012	4.2	
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.

LDC #: 18356A3b  
 SDG #: per canon  
 METHOD: GC HPLC

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

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

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

Where: SF = Surrogate Found  
 SS = Surrogate Spiked

Sample ID: 8

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
DCE	RX OUP	20	19.95082	100	100	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 #: 18356A3b  
 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:

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

$\text{RPD} = \frac{((\text{SSCMS} - \text{SSCMSD}) * 2) / (\text{SSCMS} + \text{SSCMSD}) * 100}{\text{MS}}$  Where MS = Matrix spike

MS/MSD samples: 20 + 21

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)											
Aroclor 1240	177	174		193	168	109	109	97	97	14	14

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 #: 18356A2b

SDG #: fu cover

VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample 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 laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:

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

RPD =  $((SS - SC) / SC) * 2 / ((SS - SC) / SC + SSC)$

LCS = Laboratory Control Sample percent recovery, LCS/Duplicate = Laboratory Control Sample duplicate percent recovery

LCS/LCSD samples: 8031452 - LCS

Compound	Spike Added (ug/kg)		Sample Conc. (ug/kg)		Spike Sample Concentration (ug/kg)		LCS		LCS/Duplicate	
	LCS	LCSD	LCS	LCSD	LCS	LCSD	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)										
Arched 1260	167	NA	0		167	NA	100	100	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

LDC #: 18356AB  
 SDG #: See cover

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: \_\_\_\_\_

**BRC Tronox Parcel H  
Data Validation Reports  
LDC# 18356**

**Metals**

*LDC*

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** BRC Tronox Parcel H  
**Collection Date:** January 25, 2008  
**LDC Report Date:** March 6, 2008  
**Matrix:** Soil/Water  
**Parameters:** Metals  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8A260143

### Sample Identification

TSB-HJ-01-10'	TSB-HJ-02-10'MSD
TSB-HJ-09-0'	TSB-HJ-01-0'MS
TSB-HJ-09-10'	TSB-HJ-01-0'MSD
TSB-HJ-03-0'	RINSATE-1MS
TSB-HJ-03-0'-FD	RINSATE-1MSD
TSB-HJ-03-10'	
TSB-HR-03-0'	
TSB-HR-03-10'**	
TSB-HJ-02-0'**	
TSB-HJ-02-10'**	
TSB-HR-02-0'**	
TSB-HR-02-10'**	
TSB-HJ-11-0'**	
TSB-HJ-11-10'	
TSB-HJ-11-10'-FD	
TSB-HR-01-0'	
TSB-HR-01-10'	
TSB-HJ-01-0'	
RINSATE-1	
TSB-HJ-02-10'MS	

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 22 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.

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 with the following exceptions:

Date	Lab. Reference/ID	Analyte	%R (Limits)	Associated Samples	Flag	A or P
2/5/08	CCV (20:28)	Silver	111.4 (90-110)	All water samples in SDG F8A260143	J+ (all detects)	P
2/5/08	CCV (21:53)	Silver Boron Niobium	112.6 (90-110) 112.3 (90-110) 111.8 (90-110)	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' RINSATE-1 RINSATE-1MS RINSATE-1MSD PBS PBW	J+ (all detects) J+ (all detects) J+ (all detects)	P
2/6/08	CCV (00:29)	Silver	111.6 (90-110)	TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'*** TSB-HJ-02-0'*** TSB-HJ-02-10'*** TSB-HR-02-0'*** TSB-HR-02-10'*** TSB-HJ-11-0'*** TSB-HJ-11-10' TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0' TSB-HJ-02-10'MS TSB-HJ-02-10'MSD TSB-HJ-01-0'MS TSB-HJ-01-0'MSD	J+ (all detects)	P

Date	Lab. Reference/ID	Analyte	%R (Limits)	Associated Samples	Flag	A or P
2/6/08	CCV (1:47)	Silver	112.7 (90-110)	TSB-HR-02-0'*** TSB-HR-02-10'*** TSB-HJ-11-0'*** TSB-HJ-11-10' TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0' TSB-HJ-01-0'MS TSB-HJ-01-0'MSD	J+ (all detects)	P
2/6/08	CCV (15:27)	Palladium	111.8 (90-110)	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' PBS	J+ (all detects)	P
2/6/08	CCV (16:22)	Palladium	112.3 (90-110)	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'*** TSB-HJ-02-0'*** TSB-HJ-02-10'*** TSB-HR-02-0'*** TSB-HJ-02-10'MS TSB-HJ-02-10'MSD 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)	Antimony Cadmium Chromium Iron Sodium Tin Titanium Tungsten	0.27 ug/L 0.065 ug/L 2.3 ug/L 12.6 ug/L 6.6 ug/L 0.48 ug/L 1.3 ug/L 0.27 ug/L	All water samples in SDG F8A260143

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
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 F8A260143
PB (prep blank)	Barium Boron Chromium Molybdenum Niobium Phosphorus Potassium Sodium Thallium Tin Titanium Tungsten Uranium Zinc	0.059 mg/Kg 4.2 mg/Kg 0.20 mg/Kg 0.066 mg/Kg 4.0 mg/Kg 2.1 mg/Kg 2.1 mg/Kg 2.7 mg/Kg 0.35 mg/Kg 0.12 mg/Kg 0.15 mg/Kg 0.39 mg/Kg 0.025 mg/Kg 0.69 mg/Kg	All soil samples in SDG F8A260143
ICB/CCB	Boron Cadmium Niobium Potassium Thallium Tin Titanium Tungsten Lithium	10.6 ug/L 0.1 ug/L 6.1 ug/L 7.3 ug/L 0.5 ug/L 0.2 ug/L 0.5 ug/L 0.7 ug/L 7.6 ug/L	All soil samples in SDG F8A260143

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-1	Cadmium Iron Molybdenum Niobium Sodium Tin Titanium Tungsten	0.075 ug/L 46.0 ug/L 0.60 ug/L 18.0 ug/L 42.8 ug/L 0.70 ug/L 1.5 ug/L 1.7 ug/L	0.50U ug/L 50.0U ug/L 5.0U ug/L 25.0U ug/L 50.0U ug/L 2.0U ug/L 2.0U ug/L 5.0U ug/L
TSB-HJ-01-10'	Boron Cadmium Molybdenum Niobium Thallium Tungsten	13.4 mg/Kg 0.12 mg/Kg 0.51 mg/Kg 9.4 mg/Kg 0.32 mg/Kg 0.85 mg/Kg	26.7U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 9.4J+ mg/Kg 0.53U mg/Kg 1.3U mg/Kg

Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-HJ-09-0'	Boron Cadmium Molybdenum Niobium Thallium Tungsten Lithium	7.0 mg/Kg 0.086 mg/Kg 0.46 mg/Kg 4.6 mg/Kg 0.27 mg/Kg 0.52 mg/Kg 8.4 mg/Kg	27.8U mg/Kg 0.14U mg/Kg 1.4U mg/Kg 6.9U mg/Kg 0.56U mg/Kg 1.4U mg/Kg 11.1U mg/Kg
TSB-HJ-09-10'	Boron Cadmium Molybdenum Niobium Thallium Tungsten	8.0 mg/Kg 0.096 mg/Kg 0.74 mg/Kg 3.4 mg/Kg 0.20 mg/Kg 0.48 mg/Kg	26.6U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 6.6U mg/Kg 0.53U mg/Kg 1.3U mg/Kg
TSB-HJ-03-0'	Boron Cadmium Molybdenum Niobium Thallium Tungsten Lithium	4.3 mg/Kg 0.097 mg/Kg 0.56 mg/Kg 2.2 mg/Kg 0.19 mg/Kg 0.34 mg/Kg 3.5 mg/Kg	26.6U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 6.6U mg/Kg 0.53U mg/Kg 1.3U mg/Kg 10.6U mg/Kg
TSB-HJ-03-0'-FD	Boron Cadmium Molybdenum Thallium Tin Tungsten	3.7 mg/Kg 0.092 mg/Kg 0.46 mg/Kg 0.19 mg/Kg 0.49 mg/Kg 0.32 mg/Kg	26.0U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.52U mg/Kg 0.52U mg/Kg 1.3U mg/Kg
TSB-HJ-03-10'	Boron Cadmium Molybdenum Tin Tungsten Lithium	5.5 mg/Kg 0.077 mg/Kg 0.40 mg/Kg 0.52 mg/Kg 0.33 mg/Kg 9.2 mg/Kg	26.5U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg 10.6U mg/Kg
TSB-HR-03-0'	Cadmium Molybdenum Thallium Tungsten Lithium	0.095 mg/Kg 0.40 mg/Kg 0.19 mg/Kg 0.27 mg/Kg 4.9 mg/Kg	0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg 10.6U mg/Kg
TSB-HR-03-10'***	Boron Cadmium Molybdenum Tungsten	6.3 mg/Kg 0.066 mg/Kg 0.39 mg/Kg 0.29 mg/Kg	26.5U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 1.3U mg/Kg
TSB-HJ-02-0'***	Boron Molybdenum Tungsten Lithium	4.2 mg/Kg 0.91 mg/Kg 0.29 mg/Kg 3.8 mg/Kg	26.2U mg/Kg 1.3U mg/Kg 1.3U mg/Kg 10.5U mg/Kg

Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-HJ-02-10 <sup>**</sup>	Boron Cadmium Molybdenum Tin Tungsten Lithium	7.1 mg/Kg 0.076 mg/Kg 0.85 mg/Kg 0.50 mg/Kg 0.30 mg/Kg 8.9 mg/Kg	26.7U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg 10.7U mg/Kg
TSB-HR-02-0 <sup>**</sup>	Cadmium Molybdenum Tin Tungsten Lithium	0.12 mg/Kg 0.48 mg/Kg 0.48 mg/Kg 0.36 mg/Kg 4.7 mg/Kg	0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg 10.6U mg/Kg
TSB-HR-02-10 <sup>**</sup>	Boron Cadmium Molybdenum Tin Tungsten	6.1 mg/Kg 0.058 mg/Kg 0.47 mg/Kg 0.51 mg/Kg 0.37 mg/Kg	26.5U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg
TSB-HJ-11-0 <sup>**</sup>	Cadmium Molybdenum Tungsten Lithium	0.11 mg/Kg 0.52 mg/Kg 0.30 mg/Kg 6.0 mg/Kg	0.13U mg/Kg 1.3U mg/Kg 1.3U mg/Kg 10.5U mg/Kg
TSB-HJ-11-10'	Boron Cadmium Molybdenum Tin Tungsten Lithium	4.7 mg/Kg 0.093 mg/Kg 0.70 mg/Kg 0.52 mg/Kg 0.37 mg/Kg 10.0 mg/Kg	26.7U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg 10.7U mg/Kg
TSB-HJ-11-10'-FD	Boron Cadmium Molybdenum Thallium Tin	5.2 mg/Kg 0.071 mg/Kg 0.41 mg/Kg 0.19 mg/Kg 0.46 mg/Kg	26.5U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 0.53U mg/Kg
TSB-HR-01-0'	Boron Cadmium Molybdenum Tungsten Lithium	4.1 mg/Kg 0.090 mg/Kg 0.46 mg/Kg 0.31 mg/Kg 5.8 mg/Kg	27.4U mg/Kg 0.14U mg/Kg 1.4U mg/Kg 1.4U mg/Kg 10.9U mg/Kg
TSB-HR-01-10'	Boron Cadmium Molybdenum Tin Tungsten	6.3 mg/Kg 0.068 mg/Kg 0.46 mg/Kg 0.46 mg/Kg 0.31 mg/Kg	26.4U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg
TSB-HJ-01-0'	Molybdenum Thallium Tungsten Lithium	0.76 mg/Kg 0.19 mg/Kg 0.28 mg/Kg 6.8 mg/Kg	1.3U mg/Kg 0.52U mg/Kg 1.3U mg/Kg 10.5U mg/Kg

Sample RINSATE-1 was identified as a rinsate. No metal contaminants were found in this blank with the following exceptions:

Rinsate ID	Sampling Date	Analyte	Concentration	Associated Samples
RINSATE-1	1/25/08	Aluminum Boron Cadmium Calcium Copper Iron Magnesium Manganese Molybdenum Niobium Phosphorus Potassium Silicon Sodium Strontium Thallium Tin Titanium Tungsten Zinc	10.5 ug/L 17.8 ug/L 0.075 ug/L 95.0 ug/L 0.26 ug/L 46.0 ug/L 12.5 ug/L 0.67 ug/L 0.60 ug/L 18.0 ug/L 19.0 ug/L 13.5 ug/L 43.6 ug/L 42.8 ug/L 0.86 ug/L 0.73 ug/L 0.70 ug/L 1.5 ug/L 1.7 ug/L 3.2 ug/L	All soil samples in SDG F8A260143

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-HJ-01-10'	Boron Cadmium Molybdenum Thallium Tungsten	13.4 mg/Kg 0.12 mg/Kg 0.51 mg/Kg 0.32 mg/Kg 0.85 mg/Kg	26.7U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg
TSB-HJ-09-0'	Boron Cadmium Molybdenum Niobium Thallium Tungsten	7.0 mg/Kg 0.086 mg/Kg 0.46 mg/Kg 4.6 mg/Kg 0.27 mg/Kg 0.52 mg/Kg	27.8U mg/Kg 0.14U mg/Kg 1.4U mg/Kg 6.9U mg/Kg 0.56U mg/Kg 1.4U mg/Kg
TSB-HJ-09-10'	Boron Cadmium Molybdenum Niobium Thallium Tungsten	8.0 mg/Kg 0.096 mg/Kg 0.74 mg/Kg 3.4 mg/Kg 0.20 mg/Kg 0.48 mg/Kg	26.6U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 6.6U mg/Kg 0.53U mg/Kg 1.3U mg/Kg

Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-HJ-03-0'	Boron Cadmium Molybdenum Niobium Thallium Tungsten	4.3 mg/Kg 0.097 mg/Kg 0.56 mg/Kg 2.2 mg/Kg 0.19 mg/Kg 0.34 mg/Kg	26.6U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 6.6U mg/Kg 0.53U mg/Kg 1.3U mg/Kg
TSB-HJ-03-0'-FD	Boron Cadmium Molybdenum Thallium Tin Tungsten	3.7 mg/Kg 0.092 mg/Kg 0.46 mg/Kg 0.19 mg/Kg 0.49 mg/Kg 0.32 mg/Kg	26.0U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.52U mg/Kg 0.52U mg/Kg 1.3U mg/Kg
TSB-HJ-03-10'	Boron Cadmium Molybdenum Tin Tungsten	5.5 mg/Kg 0.077 mg/Kg 0.40 mg/Kg 0.52 mg/Kg 0.33 mg/Kg	26.5U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg
TSB-HR-03-0'	Cadmium Molybdenum Thallium Tungsten	0.095 mg/Kg 0.40 mg/Kg 0.19 mg/Kg 0.27 mg/Kg	0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg
TSB-HR-03-10'***	Boron Cadmium Molybdenum Tungsten	6.3 mg/Kg 0.066 mg/Kg 0.39 mg/Kg 0.29 mg/Kg	26.5U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 1.3U mg/Kg
TSB-HJ-02-0'***	Boron Molybdenum Tungsten	4.2 mg/Kg 0.91 mg/Kg 0.29 mg/Kg	26.2U mg/Kg 1.3U mg/Kg 1.3U mg/Kg
TSB-HJ-02-10'***	Boron Cadmium Molybdenum Tin Tungsten	7.1 mg/Kg 0.076 mg/Kg 0.85 mg/Kg 0.50 mg/Kg 0.30 mg/Kg	26.7U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg
TSB-HR-02-0'***	Cadmium Molybdenum Tin Tungsten	0.12 mg/Kg 0.48 mg/Kg 0.48 mg/Kg 0.36 mg/Kg	0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg
TSB-HR-02-10'***	Boron Cadmium Molybdenum Tin Tungsten	6.1 mg/Kg 0.058 mg/Kg 0.47 mg/Kg 0.51 mg/Kg 0.37 mg/Kg	26.5U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg



Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-HJ-11-0**	Cadmium Molybdenum Tungsten	0.11 mg/Kg 0.52 mg/Kg 0.30 mg/Kg	0.13U mg/Kg 1.3U mg/Kg 1.3U mg/Kg
TSB-HJ-11-10'	Boron Cadmium Molybdenum Tin Tungsten	4.7 mg/Kg 0.093 mg/Kg 0.70 mg/Kg 0.52 mg/Kg 0.37 mg/Kg	26.7U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg
TSB-HJ-11-10'-FD	Boron Cadmium Molybdenum Thallium Tin	5.2 mg/Kg 0.071 mg/Kg 0.41 mg/Kg 0.19 mg/Kg 0.46 mg/Kg	26.5U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 0.53U mg/Kg
TSB-HR-01-0'	Boron Cadmium Molybdenum Tungsten	4.1 mg/Kg 0.090 mg/Kg 0.46 mg/Kg 0.31 mg/Kg	27.4U mg/Kg 0.14U mg/Kg 1.4U mg/Kg 1.4U mg/Kg
TSB-HR-01-10'	Boron Cadmium Molybdenum Tin Tungsten	6.3 mg/Kg 0.068 mg/Kg 0.46 mg/Kg 0.46 mg/Kg 0.31 mg/Kg	26.4U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg
TSB-HJ-01-0'	Molybdenum Thallium Tungsten	0.76 mg/Kg 0.19 mg/Kg 0.28 mg/Kg	1.3U mg/Kg 0.52U 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-HJ-02-10'MS/MSD (All soil samples in SDG F8A260143)	Antimony	55.6 (75-125)	59.1 (75-125)	-	J- (all detects) UJ (all non-detects)	A
TSB-HJ-02-10'MS/MSD (All soil samples in SDG F8A260143)	Silicon	25.3 (75-125)	-	-	J- (all detects) R (all non-detects)	A
TSB-HJ-02-10'MS/MSD (All soil samples in SDG F8A260143)	Barium Niobium	208.4 (75-125) 159.2 (75-125)	128.1 (75-125) 187.4 (75-125)	- -	J+ (all detects) J+ (all detects)	A
TSB-HJ-01-0'MS/MSD (All soil samples in SDG F8A260143)	Antimony	60.8 (75-125)	57.1 (75-125)	-	J- (all detects) UJ (all non-detects)	A
TSB-HJ-01-0'MS/MSD (All soil samples in SDG F8A260143)	Barium Niobium Palladium Strontium Chromium	142.2 (70-125) 159.6 (70-125) 132.1 (70-125) 194.3 (70-125) -	139.2 (70-125) 159.8 (70-125) - 170.6 (70-125) 126.3 (70-125)	- - - - -	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	A
TSB-HJ-01-0'MS/MSD (All soil samples in SDG F8A260143)	Phosphorus	61.9 (70-125)	29.6 (70-125)	-	J- (all detects) R (all non-detects)	A

## VI. Duplicate Sample Analysis

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

## VII. Laboratory Control Samples (LCS)

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

LCS ID	Analyte	%R (Limits)	Associated Samples	Flag	A or P
LCS	Niobium Palladium Platinum Tungsten	116.3 (85-115) 121.1 (85-115) 115.1 (85-115) 117.9 (85-115)	All water samples in SDG F8A260143	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	P
LCS	Palladium	131.1 (80-120)	All soil samples in SDG F8A260143	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-03-0' and TSB-HJ-03-0'-FD and samples TSB-HJ-11-10' and TSB-HJ-11-10'-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-03-0'	TSB-HJ-03-0'-FD				
Aluminum	6450	5370	18 ( $\leq 50$ )	-	-	-
Antimony	0.24	0.16	-	0.08 ( $\leq 1.3$ )	-	-
Arsenic	1.9	3.0	-	1.1 ( $\leq 2.7$ )	-	-
Barium	158	133	17 ( $\leq 50$ )	-	-	-
Beryllium	0.53	0.43	-	0.1 ( $\leq 0.27$ )	-	-
Boron	4.3	3.7	-	0.6 ( $\leq 26.6$ )	-	-

Analyte	Concentration (mg/Kg)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HJ-03-0'	TSB-HJ-03-0'-FD				
Cadmium	0.097	0.092	-	0.005 ( $\leq 0.13$ )	-	-
Calcium	12600	35100	94 ( $\leq 50$ )	-	-	-
Chromium	10.5	7.9	-	2.6 ( $\leq 2.7$ )	-	-
Cobalt	7.2	5.3	30 ( $\leq 50$ )	-	-	-
Copper	15.7	12.1	26 ( $\leq 50$ )	-	-	-
Iron	14000	9360	40 ( $\leq 50$ )	-	-	-
Lead	7.8	7.2	8 ( $\leq 50$ )	-	-	-
Magnesium	6300	5680	10 ( $\leq 50$ )	-	-	-
Manganese	362	296	20 ( $\leq 50$ )	-	-	-
Molybdenum	0.56	0.46	-	0.1 ( $\leq 1.3$ )	-	-
Nickel	15.4	10.2	41 ( $\leq 50$ )	-	-	-
Niobium	2.2	2.0U	-	0.2 ( $\leq 6.6$ )	-	-
Palladium	0.26	0.22	-	0.04 ( $\leq 1.0$ )	-	-
Phosphorus	1560	1350	-	210 ( $\leq 530$ )	-	-
Potassium	1870	1570	17 ( $\leq 50$ )	-	-	-
Silicon	431	373	14 ( $\leq 50$ )	-	-	-
Silver	0.093	0.078	-	0.015 ( $\leq 0.53$ )	-	-
Sodium	514	295	54 ( $\leq 50$ )	-	J (all detects)	A
Strontium	125	132	5 ( $\leq 50$ )	-	-	-
Thallium	0.19	0.19	-	0 ( $\leq 0.53$ )	-	-

Analyte	Concentration (mg/Kg)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HJ-03-0'	TSB-HJ-03-0'-FD				
Tin	0.56	0.49	-	0.07 ( $\leq 0.53$ )	-	-
Titanium	667	538	21 ( $\leq 50$ )	-	-	-
Tungsten	0.34	0.32	-	0.02 ( $\leq 1.3$ )	-	-
Uranium	0.71	0.95	-	0.24 ( $\leq 0.27$ )	-	-
Vanadium	40.7	29.2	33 ( $\leq 50$ )	-	-	-
Zinc	31.5	29.0	8 ( $\leq 50$ )	-	-	-
Zirconium	21.1	15.9	-	5.2 ( $\leq 26.6$ )	-	-
Lithium	3.5	1.5U	-	2 ( $\leq 10.6$ )	-	-

Analyte	Concentration (ug/Kg)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HJ-03-0'	TSB-HJ-03-0'-FD				
Mercury	20.4	21.7	-	1.3 ( $\leq 35.4$ )	-	-

Analyte	Concentration (mg/Kg)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HJ-11-10'	TSB-HJ-11-10'-FD				
Aluminum	7930	7390	7 ( $\leq 50$ )	-	-	-
Antimony	0.15	0.17	-	0.02 ( $\leq 1.3$ )	-	-
Arsenic	3.3	3.6	-	0.3 ( $\leq 2.7$ )	-	-
Barium	198	179	10 ( $\leq 50$ )	-	-	-
Beryllium	0.53	0.53	-	0 ( $\leq 0.27$ )	-	-
Boron	4.7	5.2	-	0.5 ( $\leq 26.7$ )	-	-
Cadmium	0.093	0.071	-	0.022 ( $\leq 0.13$ )	-	-

Analyte	Concentration (mg/Kg)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HJ-11-10'	TSB-HJ-11-10'-FD				
Calcium	57400	20900	93 ( $\leq 50$ )	-	J (all detects)	A
Chromium	13.4	11.2	-	2.2 ( $\leq 2.7$ )	-	-
Cobalt	7.7	6.8	12 ( $\leq 50$ )	-	-	-
Copper	16.7	16.8	1 ( $\leq 50$ )	-	-	-
Iron	12500	12900	3 ( $\leq 50$ )	-	-	-
Lead	7.2	7.0	3 ( $\leq 50$ )	-	-	-
Magnesium	10800	10500	3 ( $\leq 50$ )	-	-	-
Manganese	369	330	11 ( $\leq 50$ )	-	-	-
Molybdenum	0.70	0.41	-	0.29 ( $\leq 1.3$ )	-	-
Nickel	15.0	16.3	8 ( $\leq 50$ )	-	-	-
Palladium	0.58	0.55	-	0.03 ( $\leq 1.3$ )	-	-
Phosphorus	1210	1240	-	30 ( $\leq 667$ )	-	-
Potassium	1510	1230	20 ( $\leq 50$ )	-	-	-
Silicon	155	578	-	423 ( $\leq 66.7$ )	J (all detects)	A
Silver	0.11	0.12	-	0.01 ( $\leq 0.53$ )	-	-
Sodium	490	469	4 ( $\leq 50$ )	-	-	-
Strontium	299	230	26 ( $\leq 50$ )	-	-	-
Thallium	0.19U	0.19	-	0 ( $\leq 0.53$ )	-	-
Tin	0.52	0.46	-	0.06 ( $\leq 0.53$ )	-	-
Titanium	675	719	6 ( $\leq 50$ )	-	-	-

Analyte	Concentration (mg/Kg)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HJ-11-10'	TSB-HJ-11-10'-FD				
Tungsten	0.37	0.27U	-	0.1 ( $\leq 1.3$ )	-	-
Uranium	1.2	1.2	-	0 ( $\leq 0.27$ )	-	-
Vanadium	38.6	43.1	11 ( $\leq 50$ )	-	-	-
Zinc	29.7	33.2	11 ( $\leq 50$ )	-	-	-
Zirconium	24.7	24.6	-	0.1 ( $\leq 26.7$ )	-	-
Lithium	10.0	17.1	-	7.1 ( $\leq 10.7$ )	-	-

**BRC Tronox Parcel H  
Metals - Data Qualification Summary - SDG F8A260143**

SDG	Sample	Analyte	Flag	A or P	Reason
F8A260143	TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'*** TSB-HJ-02-0'*** TSB-HJ-02-10'*** TSB-HR-02-0'*** TSB-HR-02-10'*** TSB-HJ-11-0'*** TSB-HJ-11-10' TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0'	Silver	J+ (all detects)	P	Calibration (%R)
F8A260143	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' RINSATE-1	Silver Boron Niobium	J+ (all detects) J+ (all detects) J+ (all detects)	P	Calibration (%R)
F8A260143	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'*** TSB-HJ-02-0'*** TSB-HJ-02-10'*** TSB-HR-02-0'***	Palladium	J+ (all detects)	P	Calibration (%R)
F8A260143	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'*** TSB-HJ-02-0'*** TSB-HJ-02-10'*** TSB-HR-02-0'*** TSB-HR-02-10'*** TSB-HJ-11-0'*** TSB-HJ-11-10' TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0'	Antimony	J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)



SDG	Sample	Analyte	Flag	A or P	Reason
F8A260143	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'** TSB-HJ-02-0'** TSB-HJ-02-10'** TSB-HR-02-0'** TSB-HR-02-10'** TSB-HJ-11-0'** TSB-HJ-11-10' TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0'	Barium Niobium Palladium Strontium Chromium	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8A260143	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'** TSB-HJ-02-0'** TSB-HJ-02-10'** TSB-HR-02-0'** TSB-HR-02-10'** TSB-HJ-11-0'** TSB-HJ-11-10' TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0'	Silicon  Phosphorus	J- (all detects) R (all non-detects) J- (all detects) R (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8A260143	RINSATE-1	Niobium Palladium Platinum Tungsten	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	P	Laboratory control samples (%R)

SDG	Sample	Analyte	Flag	A or P	Reason
F8A260143	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'*** TSB-HJ-02-0'*** TSB-HJ-02-10'*** TSB-HR-02-0'*** TSB-HR-02-10'*** TSB-HJ-11-0'*** TSB-HJ-11-10' TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0'	Palladium	J+ (all detects)	P	Laboratory control samples (%R)
F8A260143	TSB-HJ-03-0' TSB-HJ-03-0'-FD	Sodium	J (all detects)	A	Field duplicates (RPD)
F8A260143	TSB-HJ-11-10' TSB-HJ-11-10'-FD	Calcium	J (all detects)	A	Field duplicates (RPD)
F8A260143	TSB-HJ-11-10' TSB-HJ-11-10'-FD	Silicon	J (all detects)	A	Field duplicates (Difference)

**BRC Tronox Parcel H  
Metals - Laboratory Blank Data Qualification Summary - SDG F8A260143**

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8A260143	RINSATE-1	Cadmium Iron Molybdenum Niobium Sodium Tin Titanium Tungsten	0.50U ug/L 50.0U ug/L 5.0U ug/L 25.0U ug/L 50.0U ug/L 2.0U ug/L 2.0U ug/L 5.0U ug/L	A
F8A260143	TSB-HJ-01-10'	Boron Cadmium Molybdenum Niobium Thallium Tungsten	26.7U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 9.4J+ mg/Kg 0.53U mg/Kg 1.3U mg/Kg	A

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8A260143	TSB-HJ-09-0'	Boron Cadmium Molybdenum Niobium Thallium Tungsten Lithium	27.8U mg/Kg 0.14U mg/Kg 1.4U mg/Kg 6.9U mg/Kg 0.56U mg/Kg 1.4U mg/Kg 11.1U mg/Kg	A
F8A260143	TSB-HJ-09-10'	Boron Cadmium Molybdenum Niobium Thallium Tungsten	26.6U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 6.6U mg/Kg 0.53U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HJ-03-0'	Boron Cadmium Molybdenum Niobium Thallium Tungsten Lithium	26.6U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 6.6U mg/Kg 0.53U mg/Kg 1.3U mg/Kg 10.6U mg/Kg	A
F8A260143	TSB-HJ-03-0'-FD	Boron Cadmium Molybdenum Thallium Tin Tungsten	26.0U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.52U mg/Kg 0.52U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HJ-03-10'	Boron Cadmium Molybdenum Tin Tungsten Lithium	26.5U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg 10.6U mg/Kg	A
F8A260143	TSB-HR-03-0'	Cadmium Molybdenum Thallium Tungsten Lithium	0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg 10.6U mg/Kg	A
F8A260143	TSB-HR-03-10'***	Boron Cadmium Molybdenum Tungsten	26.5U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HJ-02-0'***	Boron Molybdenum Tungsten Lithium	26.2U mg/Kg 1.3U mg/Kg 1.3U mg/Kg 10.5U mg/Kg	A

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8A260143	TSB-HJ-02-10'***	Boron Cadmium Molybdenum Tin Tungsten Lithium	26.7U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg 10.7U mg/Kg	A
F8A260143	TSB-HR-02-0'***	Cadmium Molybdenum Tin Tungsten Lithium	0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg 10.6U mg/Kg	A
F8A260143	TSB-HR-02-10'***	Boron Cadmium Molybdenum Tin Tungsten	26.5U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HJ-11-0'***	Cadmium Molybdenum Tungsten Lithium	0.13U mg/Kg 1.3U mg/Kg 1.3U mg/Kg 10.5U mg/Kg	A
F8A260143	TSB-HJ-11-10'	Boron Cadmium Molybdenum Tin Tungsten Lithium	26.7U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg 10.7U mg/Kg	A
F8A260143	TSB-HJ-11-10'-FD	Boron Cadmium Molybdenum Thallium Tin	26.5U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 0.53U mg/Kg	A
F8A260143	TSB-HR-01-0'	Boron Cadmium Molybdenum Tungsten Lithium	27.4U mg/Kg 0.14U mg/Kg 1.4U mg/Kg 1.4U mg/Kg 10.9U mg/Kg	A
F8A260143	TSB-HR-01-10'	Boron Cadmium Molybdenum Tin Tungsten	26.4U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HJ-01-0'	Molybdenum Thallium Tungsten Lithium	1.3U mg/Kg 0.52U mg/Kg 1.3U mg/Kg 10.5U mg/Kg	A

**BRC Tronox Parcel H  
Metals - Field Blank Data Qualification Summary - SDG F8A260143**

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8A260143	TSB-HJ-01-10'	Boron Cadmium Molybdenum Thallium Tungsten	26.7U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HJ-09-0'	Boron Cadmium Molybdenum Niobium Thallium Tungsten	27.8U mg/Kg 0.14U mg/Kg 1.4U mg/Kg 6.9U mg/Kg 0.56U mg/Kg 1.4U mg/Kg	A
F8A260143	TSB-HJ-09-10'	Boron Cadmium Molybdenum Niobium Thallium Tungsten	26.6U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 6.6U mg/Kg 0.53U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HJ-03-0'	Boron Cadmium Molybdenum Niobium Thallium Tungsten	26.6U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 6.6U mg/Kg 0.53U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HJ-03-0'-FD	Boron Cadmium Molybdenum Thallium Tin Tungsten	26.0U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.52U mg/Kg 0.52U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HJ-03-10'	Boron Cadmium Molybdenum Tin Tungsten	26.5U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HR-03-0'	Cadmium Molybdenum Thallium Tungsten	0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HR-03-10'***	Boron Cadmium Molybdenum Tungsten	26.5U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 1.3U mg/Kg	A

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8A260143	TSB-HJ-02-0'***	Boron Molybdenum Tungsten	26.2U mg/Kg 1.3U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HJ-02-10'***	Boron Cadmium Molybdenum Tin Tungsten	26.7U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HR-02-0'***	Cadmium Molybdenum Tin Tungsten	0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HR-02-10'***	Boron Cadmium Molybdenum Tin Tungsten	26.5U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HJ-11-0'***	Cadmium Molybdenum Tungsten	0.13U mg/Kg 1.3U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HJ-11-10'	Boron Cadmium Molybdenum Tin Tungsten	26.7U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HJ-11-10'-FD	Boron Cadmium Molybdenum Thallium Tin	26.5U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 0.53U mg/Kg	A
F8A260143	TSB-HR-01-0'	Boron Cadmium Molybdenum Tungsten	27.4U mg/Kg 0.14U mg/Kg 1.4U mg/Kg 1.4U mg/Kg	A
F8A260143	TSB-HR-01-10'	Boron Cadmium Molybdenum Tin Tungsten	26.4U mg/Kg 0.13U mg/Kg 1.3U mg/Kg 0.53U mg/Kg 1.3U mg/Kg	A
F8A260143	TSB-HJ-01-0'	Molybdenum Thallium Tungsten	1.3U mg/Kg 0.52U mg/Kg 1.3U mg/Kg	A

LDC #: 18356A4

# VALIDATION COMPLETENESS WORKSHEET

Date: 2/29/08

SDG #: F8A260143

Level III/IV

Page: 1 of 1

Laboratory: Test America

Reviewer: *WJ*

2nd Reviewer: *gnA*

**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: 1/25/08
II.	Calibration	SW	
III.	Blanks	SW	
IV.	ICP Interference Check Sample (ICS) Analysis	A	
V.	Matrix Spike Analysis	SW	3ms/msb
VI.	Duplicate Sample Analysis	N	
VII.	Laboratory Control Samples (LCS)	SW	LCS
VIII.	Internal Standard (ICP-MS)	A	not reviewed for line 3
IX.	Furnace Atomic Absorption QC	N	not 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	(4.5) (14.15)
XIV.	Field Blanks	SW	R=19

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

*All soil event # 19, 24, 25 A2*

1	TSB-HJ-01-10'	11	TSB-HR-02-0***	21	TSB-HJ-02-10'MSD	31	
2	TSB-HJ-09-0'	12	TSB-HR-02-10***	22	TSB-HJ-01-0'MS	32	
3	TSB-HJ-09-10'	13	TSB-HJ-11-0***	23	TSB-HJ-01-0'MSD	33	
4	TSB-HJ-03-0'	14	TSB-HJ-11-10'	24	RINSATE-1MS <i>A2</i>	34	
5	TSB-HJ-03-0'-FD	15	TSB-HJ-11-10'-FD	25	RINSATE-1MSD <i>↓</i>	35	
6	TSB-HJ-03-10'	16	TSB-HR-01-0'	26	PB	36	
7	TSB-HR-03-0'	17	TSB-HR-01-10'	27		37	
8	TSB-HR-03-10***	18	TSB-HJ-01-0'	28		38	
9	TSB-HJ-02-0**	19	RINSATE-1 <i>A2</i>	29		39	
10	TSB-HJ-02-10***	20	TSB-HJ-02-10'MS	30		40	

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

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LDC #: 18356A4  
 SDG #: see cover

VALIDATION FINDINGS CHECKLIST

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

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 #: 18356 A4  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: MM  
 2nd Reviewer: AME

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"/>	7100 X <sub>10</sub> 2 to 100/100
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 846 Method 8020)</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 checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Target analytes were detected in the field blanks.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

LDC #: 18356A4  
 SDG #: See com

**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)
1-19	Soil/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
20, 21	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
22, 23	↓	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
24, 25	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
1-19	Soil/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
20, 21	Soil	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr
22, 23	↓	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr
24, 25	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

**Analysis Method**

ICP	(Li, S)
ICP-MS	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si
ICP-MS	(Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Zr)
GFAA	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 #: 18356A4  
 SDG #: See wmm

VALIDATION FINDINGS WORKSHEET  
 Calibration

Page: 1 of 1  
 Reviewer: JMM  
 2nd Reviewer: MRF

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	2/15/08	ccv (2028)	Ag	111.4	Am Ac	JT LT/P
2	2/15/08	ccv (2153)	Ag	112.6	+13, PBS	↓
			Pd	112.3	↓	↓
			Nb	111.8	↓	↓
2	2/16/08	ccv (2029)	Ag	111.6	4-18, 20-23	JT LT/P
3	2/16/08	ccv (147)	Ag	112.7	11-18, 22, 23	JT LT/P
4	2/16/08	ccv (1527)	Pd	111.8	1-4, PBS	↓
2	2/16/08	ccv (1622)	Pd	112.3	<del>1-11, 20-21</del> , PBS	↓

Comments:

Sample Identification

Analyte	Maximum PB <sup>a</sup> (mg/kg)	Maximum PB <sup>a</sup> ( $\mu\text{g}/\text{L}$ )	Maximum ICB/CCB <sup>a</sup> ( $\mu\text{g}/\text{L}$ )	Blank Action Limit	19																	
Sb		0.27	0.2																			
Cd		0.065	0.1		0.075 / 0.50																	
Cr		2.3																				
Fe		12.6			46.0 / 50.0																	
Mo			0.2		0.60 / 5.0																	
Nb			6.1		18.0 / 25.0																	
Na		6.6			42.8 / 50.0																	
Sn		0.48			0.70 / 2.0																	
Ti		1.3	1.2		1.5 / 2.0																	
W		0.27	0.6		1.7 / 5.0																	

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 <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						
Ba	0.059																			
B	4.2		10.6		13.4 / 26.7	7.0 / 27.8	8.0 / 26.6	4.3 / 26.6	3.7 / 26.0	5.5 / 26.5	6.3 / 26.5	4.2 / 26.2	7.1 / 26.7							
Cd			0.1		0.12 / 0.13	0.086 / 0.14	0.096 / 0.13	0.097 / 0.13	0.092 / 0.13	0.077 / 0.13	0.095 / 0.13	0.066 / 0.13	0.076 / 0.13							
Cr	0.20																			
Mo	0.066				0.51 / 1.3	0.46 / 1.4	0.74 / 1.3	0.56 / 1.3	0.46 / 1.3	0.40 / 1.3	0.40 / 1.3	0.91 / 1.3	0.85 / 1.3							
Nb	4.0		6.1	40	9.4 J+	4.6 / 6.9	3.4 / 6.6	2.2 / 6.6												
P	2.1																			
K	2.1		7.3																	
Na	2.7																			
Tl	0.35		0.5	3.5	0.32 / 0.53	0.27 / 0.56	0.20 / 0.53	0.19 / 0.53	0.19 / 0.52	0.19 / 0.52	0.19 / 0.53	0.19 / 0.53	0.50 / 0.53							
Sn	0.12		0.2																	
Ti	0.15		0.5																	
W	0.39		0.7		0.85 / 1.3	0.52 / 1.4	0.48 / 1.3	0.34 / 1.3	0.32 / 1.3	0.33 / 1.3	0.27 / 1.3	0.29 / 1.3	0.30 / 1.3							
U	0.025																			
Zn	0.69																			
Li			7.6			8.4 / 11.1		3.5 / 10.6		9.2 / 10.6	4.9 / 10.6	3.8 / 10.5	8.9 / 10.7							

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	16	17	18					
Ba	0.059																
B	4.2		10.6		6.1 / 26.5	4.7 / 26.7	5.2 / 26.5	4.1 / 27.4	6.3 / 26.4								
Cd			0.1		0.12 / 0.13	0.058 / 0.13	0.11 / 0.13	0.093 / 0.13	0.071 / 0.13	0.090 / 0.14	0.068 / 0.13						
Cr	0.20																
Mo	0.066				0.48 / 1.3	0.47 / 1.3	0.52 / 1.3	0.70 / 1.3	0.41 / 1.3	0.46 / 1.4	0.46 / 1.3	0.76 / 1.3					
Nb	4.0		6.1	40													
P	2.1																
K	2.1		7.3														
Na	2.7																
Tl	0.35		0.5	3.5					0.19 / 0.53								
Sn	0.12		0.2		0.48 / 0.53	0.51 / 0.53		0.52 / 0.53	0.46 / 0.53		0.46 / 0.53						
Ti	0.15		0.5														
W	0.39		0.7		0.36 / 1.3	0.37 / 1.3	0.30 / 1.3	0.37 / 1.3		0.31 / 1.4	0.31 / 1.3	0.28 / 1.3					
U	0.025																
Zn	0.69																
Li			7.6		4.7 / 10.6		6.0 / 10.5	10.0 / 10.7	5.8 / 10.9	6.8 / 10.5							

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)  
 N/A Were field blanks identified in this SDG?  
 N/A Were target analytes detected in the field blanks?  
**Blank units:** ug/L **Associated sample units:** mg/Kg  
**Sampling date:** 1/25/08 Soil factor applied 200X  
**Field blank type:** (circle one) Field Blank / Rinsate / Other: R Associated Samples: All Soil

Analyte	Blank ID	Action Level	Sample Identification																	
			1	2	3	4	5	6	7	8	9	10								
Al	10.5																			
B	17.8		13.4 / 26.7	7.0 / 27.8	8.0 / 26.6	4.3 / 26.6	3.7 / 26.0	5.5 / 26.5	6.3 / 26.5	4.2 / 26.2	7.1 / 26.7									
Cd	0.075		0.12 / 0.13	0.086 / 0.14	0.096 / 0.13	0.097 / 0.13	0.092 / 0.13	0.077 / 0.13	0.066 / 0.13	0.095 / 0.13	0.076 / 0.13									
Ca	95.0																			
Cu	0.26																			
Fe	46.0																			
Mg	12.5																			
Mn	0.67																			
Mo	0.60		0.51 / 1.3	0.46 / 1.4	0.74 / 1.3	0.56 / 1.3	0.46 / 1.3	0.40 / 1.3	0.39 / 1.3	0.40 / 1.3	0.85 / 1.3									
Nb	18.0			4.6 / 6.9	3.4 / 6.6	2.2 / 6.6														
P	19.0																			
K	13.5																			
Si	43.6																			
Na	42.8																			
Sr	0.86																			
Tl	0.73		0.32 / 0.53	0.27 / 0.56	0.20 / 0.53	0.19 / 0.53	0.19 / 0.52	0.19 / 0.53	0.19 / 0.53	0.19 / 0.53										
Sn	0.70							0.49 / 0.52	0.52 / 0.53		0.50 / 0.53									
Ti	1.5																			
W	1.7		0.85 / 1.3	0.52 / 1.4	0.48 / 1.3	0.34 / 1.3	0.32 / 1.3	0.33 / 1.3	0.29 / 1.3	0.27 / 1.3	0.30 / 1.3									
Zn	3.2																			

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".

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

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

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

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

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

**Sampling date:** 1/25/08 Soil factor applied 200X

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

Analyte	Blank ID	Action Level	Sample Identification																	
			11	12	13	14	15	16	17	18	19	20	21	22	23	24				
Al	10.5																			
B	17.8		6.1 / 26.5	4.7 / 26.7	5.2 / 26.5	4.1 / 27.4	6.3 / 26.4													
Cd	0.075		0.12 / 0.13	0.058 / 0.13	0.11 / 0.13	0.093 / 0.13	0.071 / 0.13	0.090 / 0.14	0.068 / 0.13											
Ca	95.0																			
Cu	0.26																			
Fe	46.0																			
Mg	12.5																			
Mn	0.67																			
Mo	0.60		0.48 / 1.3	0.47 / 1.3	0.52 / 1.3	0.70 / 1.3	0.41 / 1.3	0.46 / 1.4	0.46 / 1.3	0.76 / 1.3										
Nb	18.0																			
P	19.0																			
K	13.5																			
Si	43.6																			
Na	42.8																			
Sr	0.86																			
Tl	0.73							0.19 / 0.53												
Sn	0.70		0.48 / 0.53	0.51 / 0.53	0.52 / 0.53	0.46 / 0.53	0.46 / 0.53													
Ti	1.5																			
W	1.7		0.36 / 1.3	0.37 / 1.3	0.30 / 1.3	0.37 / 1.3	0.31 / 1.4	0.31 / 1.3	0.28 / 1.3											
Zn	3.2																			

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".



**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?
- N 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	20121	Soil	Sb	55.6	59.1		All Soil	J-1/J/A
			Ba	208.4	128.1		↓	J-1/J/A
			Nb	159.2	187.4		↓	J-1/J/A
			Si	25.3				J-1/J/A
2	22123	Soil	Sb	60.8	57.1		All Soil	J-1/J/A
			Ba	142.2	139.2		↓	J-1/J/A
			Nb	159.6	159.8		↓	J-1/J/A
			Pd	132.1				J-1/J/A
			P	61.9	29.6		↓	J-1/J/A
			Si	194.3	170.6		↓	J-1/J/A
			Cu		126.3			↓
			<del>P</del>		29.6			

Comments:

LPC #: 18356A4  
 SDG #: 11 822 CSM

VALIDATION FINDINGS WORKSHEET  
 Matrix Spike/Matrix Spike Duplicates

Page: 2 of 2  
 Reviewer: LMV  
 2nd Reviewer: amg

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

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

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

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.

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

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	20/21	Soil	Pb			22.0	01) Soil	No qual (CEST)
			Ba			28.7		
			Ca			21.9		
			Si			21.4		
			Sr			50.7		
2	22/23	Soil	Ca			25.4		
			Hg			26.2		

Comments:

LDC #: 18356A4  
 SDG #: See cover

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

Page: 1 of 1  
 Reviewer: MH  
 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".  
 (Y) N N/A Was a laboratory control sample (LCS) analyzed for each matrix in this SDG?  
 (Y) (N) N/A Were all aqueous LCS percent recoveries (%R) within the control limits of 80-120% and all soil LCS %R within laboratory established control limits.  
**LEVEL IV ONLY:** See Lab files, TS  
 (Y) 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	AL	Nb Pd	116.3 (85-115)	All AL	J+ Jt/p
			Pt	121.1	↓	↓
			W	115.7	↓	↓
				119.9		
2	LCS	Soil	Pd	131.7 (80-120)	All Soil	J+ Jt/p

Comments:

LDC#: 18356A4  
 SDG#: See Cover

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

Page: 1 of 4  
 Reviewer: [Signature]  
 2nd Reviewer: OM

**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)	(mg/Kg)	(mg/Kg)	Qualifications (Parent Only)
	4	5	RPD	Difference	Limits	
Aluminum	6450	5370	18			
Antimony	0.24	0.16		0.08	( ≤1.3)	
Arsenic	1.9	3.0		1.1	( ≤2.7)	
Barium	158	133	17			
Beryllium	0.53	0.43		0.1	( ≤0.27)	
Boron	4.3	3.7		0.6	( ≤26.6)	
Cadmium	0.097	0.092		0.005	( ≤0.13)	
Calcium	12600	35100	94			
Chromium	10.5	7.9		2.6	( ≤2.7)	
Cobalt	7.2	5.3	30			
Copper	15.7	12.1	26			
Iron	14000	9360	40			
Lead	7.8	7.2	8			
Magnesium	6300	5680	10			
Manganese	362	296	20			
Molybdenum	0.56	0.46		0.1	( ≤1.3)	
Nickel	15.4	10.2	41			
Niobium	2.2	2.0U		0.2	( ≤6.6)	
Palladium	0.26	0.22		0.04	( ≤1.0)	

LDC#: 18356A4  
 SDG#: See Cover

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

Page: 2 of 4  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

**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)
	4	5				
Phosphorus	1560	1350		210	( ≤530)	
Potassium	1870	1570	17			
Silicon	431	373	14			
Silver	0.093	0.078		0.015	( ≤0.53)	
Sodium	514	295	54			J det / A
Strontium	125	132	5			
Thallium	0.19	0.19		0	( ≤0.53)	
Tin	0.56	0.49		0.07	( ≤0.53)	
Titanium	667	538	21			
Tungsten	0.34	0.32		0.02	( ≤1.3)	
Uranium	0.71	0.95		0.24	( ≤0.27)	
Vanadium	40.7	29.2	33			
Zinc	31.5	29.0	8			
Zirconium	21.1	15.9		5.2	( ≤26.6)	
Lithium	3.5	1.5U		2	( ≤10.6)	
Mercury (ug/Kg)	20.4	21.7		1.3	( ≤35.4)	

Compound	Concentration (mg/kg)		(<50) RPD	(mg/Kg) Difference	(mg/Kg) Limits	Qualifications (Parent Only)
	14	15				
Aluminum	7930	7390	7			

LDC#: 18356A4  
 SDG#: See Cover

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

Page: 3 of 4  
 Reviewer: [Signature]  
 2nd Reviewer: gnh

**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)	(mg/Kg)	(mg/Kg)	Qualifications (Parent Only)
	14	15	RPD	Difference	Limits	
Antimony	0.15	0.17		0.02	( ≤1.3)	
Arsenic	3.3	3.6		0.3	( ≤2.7)	
Barium	198	179	10			
Beryllium	0.53	0.53		0	( ≤0.27)	
Boron	4.7	5.2		0.5	( ≤26.7)	
Cadmium	0.093	0.071		0.022	( ≤0.13)	
Calcium	57400	20900	93			J det / A
Chromium	13.4	11.2		2.2	( ≤2.7)	
Cobalt	7.7	6.8	12			
Copper	16.7	16.8	1			
Iron	12500	12900	3			
Lead	7.2	7.0	3			
Magnesium	10800	10500	3			
Manganese	369	330	11			
Molybdenum	0.70	0.41		0.29	( ≤1.3)	
Nickel	15.0	16.3	8			
Palladium	0.58	0.55		0.03	( ≤1.3)	
Phosphorus	1210	1240		30	( ≤667)	
Potassium	1510	1230	20			

LDC#: 18356A4  
 SDG#: See Cover

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

Page: 4 of 4  
 Reviewer: [Signature]  
 2nd Reviewer: gnh

**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)
	14	15				
Silicon	155	578		423	( ≤66.7)	J det / A
Silver	0.11	0.12		0.01	( ≤0.53)	
Sodium	490	469	4			
Strontium	299	230	26			
Thallium	0.19U	0.19		0	( ≤0.53)	
Tin	0.52	0.46		0.06	( ≤0.53)	
Titanium	675	719	6			
Tungsten	0.37	0.27U		0.1	( ≤1.3)	
Uranium	1.2	1.2		0	( ≤0.27)	
Vanadium	38.6	43.1	11			
Zinc	29.7	33.2	11			
Zirconium	24.7	24.6		0.1	( ≤26.7)	
Lithium	10.0	17.1		7.1	( ≤10.7)	

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

**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:

$\%R = \frac{\text{Found}}{\text{True}} \times 100$  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	
TW	ICP (Initial calibration)	Li	4012	4000	100.3	100.3	100.3	100.3	Y
	GFAA (Initial calibration)								
TW	CVAA (Initial calibration)	Hg	2.51	2.50	100.4	100.4	100.4	100.4	Y
CCV	ICP (Continuing calibration)	S	39780	40000	99.5	99.5	99.4	99.4	N
	GFAA (Continuing calibration)								
CCV	CVAA (Continuing calibration)	Hg	5.09	5.0	101.8	101.8	101.8	101.8	Y
TW	ICPMS (Initial calibration)	<del>S</del> Cu	208.6	200	104.3	104.3	104.3	104.3	N
CCV	ICPMS (Continuing calibration)	V	209.73	200	104.9	104.9	104.9	104.9	N

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 #: 18356A4  
 SDG #: Cell cover

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

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

**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:

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

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:

$$RPD = \frac{|S-DL|}{(S+D)/2} \times 100$$

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		Acceptable (Y/N)
					%R / RPD / %D	%R / RPD / %D	
7584	ICP interference check	cd	108.15	100	108.2	108.2	Y
761	Laboratory control sample	Si	73.88	100	73.9	73.9	
70	Matrix spike	B (SSR-SR)	96.29	106.68	90.3	90.3	
20/21	Duplicate	Na	3334	3759	12.0	12.0	
10	ICP serial dilution	SY	483.3	456.23	5.9	5.9	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 #: 18356A4  
 SDG #: cel cov

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

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

**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 10 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

$$Be = \frac{0.9158 \mu g/L \times 0.1 \times 2.5}{0.158 \times 0.9374} = 0.4884 \mu g/L$$

Sample ID	Analyte	Reported Concentration (mg/kg)	Calculated Concentration (mg/kg)	Acceptable (Y/N)
10	Li	8.9	8.9	Y
	Al	6860	6860	Y
	Sb	0.19	0.19	
	As	3.7	3.7	
	Ba	126	126	
	Be	0.49	0.49	
	B	7.1	7.1	
	Cd	0.076	0.076	
	Ca	39100	39100	
	Cr	10.7	10.7	
	Co	6.3	6.3	
	Cu	14.8	14.8	
	Fe	12000	12000	
	Pb	6.3	6.3	
	Mg	9000	9000	
	Mn	254	254	
	Mo	0.85	0.85	
	Ni	135	13.5	
	Pd	0.51	0.51	
	P	1070	1070	
	K	1230	1230	
	Si	265	265	Y

LDC #: 18356A4  
 SDG #: See cover

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

Page: 2 of 2  
 Reviewer: MH  
 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".

- 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 10 were recalculated and verified using the following equation:

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

Recalculation:

$$Zn = \frac{54.29 \mu\text{g} / 0.1 \times 25}{0.58 \times 0.9374} = 28.96 \mu\text{g} / \mu\text{g}$$

- 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 (µg/µg)	Calculated Concentration (µg/µg)	Acceptable (Y/N)
10	Ag	0.13	0.13	Y
	Na	736	736	
	Sr	243	243	
	Sn	0.50	0.50	
	Ti	6.04	6.04	
	W	0.30	0.29	
	U	1.7	1.7	
	V	36.6	36.6	
	Zn	29.0	29.0	
	Zr	23.2	23.2	Y

**BRC Tronox Parcel H  
Data Validation Reports  
LDC# 18356**

Wet Chemistry

*LDC*

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

**Project/Site Name:** BRC Tronox Parcel H  
**Collection Date:** January 25, 2008  
**LDC Report Date:** March 6, 2008  
**Matrix:** Soil/Water  
**Parameters:** Wet Chemistry  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8A260143

**Sample Identification**

TSB-HJ-01-10'	TSB-HJ-09-0'MSD
TSB-HJ-09-0'	TSB-HJ-02-10'MS
TSB-HJ-09-10'	TSB-HJ-02-10'DUP
TSB-HJ-03-0'	TSB-HJ-01-0'MS
TSB-HJ-03-0'-FD	TSB-HJ-01-0'DUP
TSB-HJ-03-10'	RINSATE-1MS
TSB-HR-03-0'	RINSATE-1DUP
TSB-HR-03-10'**	
TSB-HJ-02-0'**	
TSB-HJ-02-10'**	
TSB-HR-02-0'**	
TSB-HR-02-10'**	
TSB-HJ-11-0'**	
TSB-HJ-11-10'	
TSB-HJ-11-10'-FD	
TSB-HR-01-0'	
TSB-HR-01-10'	
TSB-HJ-01-0'	
RINSATE-1	
TSB-HJ-09-0'MS	

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 24 soil samples and 3 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.

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.

Sample "RINSATE-1" was identified as a rinsate. No contaminant concentrations were found in this blank with the following exceptions:

Rinsate ID	Sampling Date	Analyte	Concentration	Associated Samples
RINSATE-1	1/25/08	Sulfate	0.067 mg/L	All soil samples in SDG F8A260143

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-03-0'	Sulfate	5.1 mg/Kg	5.3U mg/Kg
TSB-HR-01-0'	Sulfate	2.2 mg/Kg	5.5U 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)	Flag	A or P
TSB-HJ-02-10'MS (All soil samples in SDG F8A260143)	Oil & grease	62 (75-125)	-	-	J- (all detects) UJ (all non-detects)	A
TSB-HJ-02-10'MS (TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' TSB-HJ-02-10'**)*)	Sulfate	44 (75-125)	-	-	J- (all detects) UJ (all non-detects)	A
TSB-HJ-01-0'MS (All soil samples in SDG F8A260143)	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-03-0' and TSB-HJ-03-0'-FD and samples TSB-HJ-11-10' and TSB-HJ-11-10'-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-03-0'	TSB-HJ-03-0'-FD				
Chloride	4.4	0.85	-	3.55 ( $\leq 2.1$ )	J (all detects)	A

Analyte	Concentration (mg/Kg)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HJ-03-0'	TSB-HJ-03-0'-FD				
Chlorine	8.7	1.7	-	7 ( $\leq 4.3$ )	J (all detects)	A
Fluoride	0.60	0.70	-	0.1 ( $\leq 1.1$ )	-	-
Nitrate as N	10.3	1.9	138 ( $\leq 50$ )	-	J (all detects)	A
Sulfate	92.3	87.4	5 ( $\leq 50$ )	-	-	-

Analyte	Concentration (ug/Kg)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HJ-03-0'	TSB-HJ-03-0'-FD				
Perchlorate	19.9	284	-	264.1 ( $\leq 42.5$ )	J (all detects)	A

Analyte	Concentration (mg/Kg)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HJ-11-10'	TSB-HJ-11-10'-FD				
Chloride	10.8	8.9	-	1.9 ( $\leq 2.1$ )	-	-
Chlorine	21.5	17.7	-	3.8 ( $\leq 4.3$ )	-	-
Fluoride	1.3	1.7	-	0.4 ( $\leq 1.1$ )	-	-
Nitrate as N	1.2	0.97	21 ( $\leq 50$ )	-	-	-
Sulfate	24.4	29.1	-	4.7 ( $\leq 5.3$ )	-	-

Analyte	Concentration (ug/Kg)		RPD (Limits)	Difference (Limits)	Flag	A or P
	TSB-HJ-11-10'	TSB-HJ-11-10'-FD				
Perchlorate	3.6U	170	-	166.4 ( $\leq 42.7$ )	J (all detects) UJ (all non-detects)	A

**BRC Tronox Parcel H  
Wet Chemistry - Data Qualification Summary - SDG F8A260143**

SDG	Sample	Analyte	Flag	A or P	Reason
F8A260143	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' TSB-HJ-03-0'-FD TSB-HJ-03-10' TSB-HR-03-0' TSB-HR-03-10'*** TSB-HJ-02-0'*** TSB-HJ-02-10'*** TSB-HR-02-0'*** TSB-HR-02-10'*** TSB-HJ-11-0'*** TSB-HJ-11-10' TSB-HJ-11-10'-FD TSB-HR-01-0' TSB-HR-01-10' TSB-HJ-01-0'	Oil & grease	J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8A260143	TSB-HJ-01-10' TSB-HJ-09-0' TSB-HJ-09-10' TSB-HJ-03-0' TSB-HJ-02-10'***	Sulfate	J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8A260143	TSB-HJ-03-0' TSB-HJ-03-0'-FD	Chloride Chlorine Perchlorate	J (all detects) J (all detects) J (all detects)	A	Field duplicates (Difference)
F8A260143	TSB-HJ-03-0' TSB-HJ-03-0'-FD	Nitrate as N	J (all detects)	A	Field duplicates (RPD)
F8A260143	TSB-HJ-11-10' TSB-HJ-11-10'-FD	Perchlorate	J (all detects) UJ (all non-detects)	A	Field duplicates (Difference)

**BRC Tronox Parcel H  
Wet Chemistry - Laboratory Blank Data Qualification Summary - SDG F8A260143**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel H  
Wet Chemistry - Field Blank Data Qualification Summary - SDG F8A260143**

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8A260143	TSB-HR-03-0'	Sulfate	5.3U mg/Kg	A

<b>SDG</b>	<b>Sample</b>	<b>Analyte</b>	<b>Modified Final Concentration</b>	<b>A or P</b>
F8A260143	TSB-HR-01-0'	Sulfate	5.5U mg/Kg	A

LDC #: 18356A6  
 SDG #: F8A260143  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**

Level III/IV

Date: 3/3/08  
 Page: 1 of 1  
 Reviewer: W  
 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) / EPA 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: 1/25/08
IIa.	Initial calibration	A	
IIb.	Calibration verification	A	
III.	Blanks	A	
IV.	Matrix Spike/Matrix Spike Duplicates	SW A <del>SW</del>	2 MS / MSO / MSO
V.	Duplicates	A	
VI.	Laboratory control samples	A	LCs / LCSD
VII.	Sample result verification	A	Not reviewed for Level III validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	SW	(4.5) (14.15)
X.	Field blanks	SW	R=19

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  
*any soil event #19, 26, 27 A*

1	TSB-HJ-01-10'	11 ✓	TSB-HR-02-0***	21	TSB-HJ-09-0'MSD	31	
2	TSB-HJ-09-0'	12 ✓	TSB-HR-02-10***	22	TSB-HJ-02-10'MS	32	
3	TSB-HJ-09-10'	13 ✓	TSB-HJ-11-0***	23	TSB-HJ-02-10'DUP	33	
4	TSB-HJ-03-0'	14 ✓	TSB-HJ-11-10'	24	TSB-HJ-01-0'MS	34	
5 ✓	TSB-HJ-03-0'-FD	15 ✓	TSB-HJ-11-10'-FD	25	TSB-HJ-01-0'DUP	35	
6 ✓	TSB-HJ-03-10'	16 ✓	TSB-HR-01-0'	26	RINSATE-1MS	36	
7 ✓	TSB-HR-03-0'	17 ✓	TSB-HR-01-10'	27	RINSATE-1DUP	37	
8 ✓	TSB-HR-03-10***	18 ✓	TSB-HJ-01-0'	28	MS	38	
9 ✓	TSB-HJ-02-0***	19	RINSATE-1	29		39	
10	TSB-HJ-02-10***	20	TSB-HJ-09-0'MS	30		40	

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

LDC #: 18356 A6  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

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

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 #: 18356 Ab  
 SDG #: Lee Creek

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 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. Overall Assessment of Data</b>				
Overall assessment of data was found to be acceptable.	✓			
<b>IX. Field Duplicates</b>				
Field duplicate pairs were identified in this SDG.	✓			
Target analytes were detected in the field duplicates.	✓			
<b>X. Field Blanks</b>				
Field blanks were identified in this SDG.	✓			
Target analytes were detected in the field blanks.	✓			

LDC #: 18356A6  
 SDG #: See Cover

## VALIDATION FINDINGS WORKSHEET Sample Specific Analysis Reference

Page: 1 of 1  
 Reviewer: MN  
 2nd reviewer: MK

All circled methods are applicable to each sample.

Sample ID	Matrix	Parameter
1-19	Soil/A2	pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate) TOC (C)O CR <sup>6+</sup> TKN (O+G)/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
26.27	A2	pH Br Bromine (C) Chlorine (F) NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate) TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
20.21	Soil	pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC (C)O CR <sup>6+</sup> TKN O+G/TPH
22.23	↓	pH Br Bromine (C) Chlorine (F) NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN (O+G)/TPH
24.25	↓	pH Br Bromine (C) Chlorine (F) NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN (O+G)/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH
		pH Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate TOC ClO <sub>4</sub> CR <sup>6+</sup> TKN O+G/TPH

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



LDC #: 18356A6  
 SDG #: See cover

VALIDATION FINDINGS WORKSHEET  
 Field Blanks

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

METHOD: Inorganics, EPA Method See cover  
 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: mg/L Associated sample units: mg/kg  
 Sampling date: 1/25/08 Soil factor applied: 100  
 Field blank type: (circle one) Field Blank / Rinsate / Other: R Associated Samples: Am Soil

Analyte	Blank ID	Blank Action Limit	Sample Identification																	
SO4	19		7	16																
	0.067		5.1/5.3	2.2/5.5																

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#: 18356A6  
 SDG#: See Cover

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

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

Inorganics, Method: See Cover

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

Analyte	Concentration (mg/Kg)		RPD ( $\leq 50$ )	Difference	Limits	Qualification (Parent only)
	4	5				
Chloride	4.4	0.85		3.55	( $\leq 2.1$ )	J det / A
Chlorine	8.7	1.7		7	( $\leq 4.3$ )	J det / A
Fluoride	0.60	0.70		0.1	( $\leq 1.1$ )	
Nitrate as N	10.3	1.9	138			J det / A
Perchlorate (ug/Kg)	19.9	284		264.1	( $\leq 42.5$ )	J det / A
Sulfate	92.3	87.4	5			

Analyte	Concentration (mg/Kg)		RPD ( $\leq 50$ )	Difference	Limits	Qualification (Parent only)
	14	15				
Chloride	10.8	8.9		1.9	( $\leq 2.1$ )	
Chlorine	21.5	17.7		3.8	( $\leq 4.3$ )	
Fluoride	1.3	1.7		0.4	( $\leq 1.1$ )	
Nitrate as N	1.2	0.97	21			
Perchlorate (ug/Kg)	<del>42.7</del> 37.64	170		<del>127.3</del> 166.4	( $\leq 42.7$ )	J / UJ / A
Sulfate	24.4	29.1		4.7	( $\leq 5.3$ )	

LDC #: 18356A6  
 SDG #: See cover

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

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

Method: Inorganics, Method See cover

The correlation coefficient (r) for the calibration of Cl was recalculated. Calibration date: 2/4/08

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

%R =  $\frac{\text{Found} \times 100}{\text{True}}$  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. (mg/L)	Area	Recalculated		Reported		Acceptable (Y/N)
					r or r <sup>2</sup>	r or r <sup>2</sup>			
Initial calibration	Cl	s1	0.2	0.033	0.9998819	0.999880			Y
		s2	0.5	0.075					
		s3	1	0.154					
		s4	2.5	0.386					
		s5	5	0.794					
<u>2/4/08 1519</u> <u>204 [Signature]</u> Calibration verification	<u>CO3</u>	<u>4000</u>	<u>3927.6</u>		<u>98</u>	<u>98</u>		<u>Y</u>	
<u>2/4/08 1519</u> <u>CCV</u> Calibration verification	<u>F</u>	<u>1000</u>	<u>1009.7</u>		<u>101</u>	<u>100.98</u>		<u>Y</u>	
<u>CCV</u> Calibration verification	<u>CO4</u>	<u>100</u>	<u>101.6</u>		<u>102</u>	<u>102</u>		<u>Y</u>	

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

LDC #: 18356A6  
 SDG #: See cover

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

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

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			
107	Laboratory control sample	074	1173	1330	88	88	88	88	Y
20	Matrix spike sample	004	2832 (SSR-SR)	2770	102	102	102	102	Y
23	Duplicate sample	505	127	120	57	57	55	55	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.



**BRC Tronox Parcel H  
Data Validation Reports  
LDC# 18356**

Gasoline Range Organics

*LDC*

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** BRC Tronox Parcel H  
**Collection Date:** January 25, 2008  
**LDC Report Date:** March 6, 2008  
**Matrix:** Soil/Water  
**Parameters:** Gasoline Range Organics  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8A260143

### Sample Identification

TSB-HJ-01-10'	TSB-HJ-01-10'MSD
TSB-HJ-09-0'	TSB-HJ-02-10'MS
TSB-HJ-09-10'	TSB-HJ-02-10'MSD
TSB-HJ-03-0'	TSB-HJ-01-0'MS
TSB-HJ-03-0'-FD	TSB-HJ-01-0'MSD
TSB-HJ-03-10'	RINSATE-1MS
TSB-HR-03-0'	RINSATE-1MSD
TSB-HR-03-10'**	
TSB-HJ-02-0'**	
TSB-HJ-02-10'**	
TSB-HR-02-0'**	
TSB-HR-02-10'**	
TSB-HJ-11-0'**	
TSB-HJ-11-10'	
TSB-HJ-11-10'-FD	
TSB-HR-01-0'	
TSB-HR-01-10'	
TSB-HJ-01-0'	
RINSATE-1	
TSB-HJ-01-10'MS	

\*\*Indicates sample underwent EPA Level IV review



## Introduction

This data review covers 24 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.

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**

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-1" was identified as a rinsate. No gasoline range organic contaminants were found in this blank.

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

### **a. Surrogate Recovery**

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

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

Matrix spike (MS) 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-03-0' and TSB-HJ-03-0'-FD and samples TSB-HJ-11-10' and TSB-HJ-11-10'-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 F8A260143**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel H  
Gasoline Range Organics - Laboratory Blank Data Qualification Summary - SDG  
F8A260143**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel H  
Gasoline Range Organics - Field Blank Data Qualification Summary - SDG  
F8A260143**

No Sample Data Qualified in this SDG

LDC #: 18356A7  
 SDG #: F8A260143  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**

Level III/IV

Date: 3/4/08  
 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	Δ	Sampling dates: <u>1/25/08</u>
IIa.	Initial calibration	Δ	
IIb.	Calibration verification/ICV	Δ	<u>ICV ≤ 15</u>
III.	Blanks	Δ	
IVa.	Surrogate recovery	Δ	
IVb.	Matrix spike/Matrix spike duplicates	Δ	
IVc.	Laboratory control samples	Δ	<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	ND	<u>D = 4 + 5      * 14 + 15</u>
X.	Field blanks	N	<u>R = 19</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: \*\* Indicates sample underwent Level IV validation

soil + water

1	1	TSB-HJ-01-10'	11	2	TSB-HR-02-0**	21	1	TSB-HJ-01-10'MSD	31	1	8031129	1/31
2	1	TSB-HJ-09-0'	12	3	TSB-HR-02-10***	22	2	TSB-HJ-02-10'MS	32	2	8035215	2/4
3	2	TSB-HJ-09-10'	13	4	TSB-HJ-11-0**	23	1	TSB-HJ-02-10'MSD	33	3	8037078	2/5
4	1	TSB-HJ-03-0'	14	3	TSB-HJ-11-10'	24	3	TSB-HJ-01-0'MS	34	4	8037174	2/6
5	2	TSB-HJ-03-0'-FD	15	3	TSB-HJ-11-10'-FD	25	3	TSB-HJ-01-0'MSD	35	5	8039150	2/8
6	2	TSB-HJ-03-10'	16	3	TSB-HR-01-0'	26	3	RINSATE-1MS	36			
7	2	TSB-HR-03-0'	17	3	TSB-HR-01-10'	27	5	RINSATE-1MSD	37			
8	2	TSB-HR-03-10***	18	3	TSB-HJ-01-0'	28			38			
9	2	TSB-HJ-02-0**	19	5	RINSATE-1	29			39			
10	2	TSB-HJ-02-10**	20	1	TSB-HJ-01-10'MS	30			40			

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

LDC #: 18356A7  
 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.	/			
Cooler temperature criteria was met.	/			
<b>II. Initial calibration</b>				
Did the laboratory perform a 5 point calibration prior to sample analysis?	/			
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?	/			
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?			/	
Did the initial calibration meet the curve fit acceptance criteria?			/	
Were the RT windows properly established?	/			
<b>IV. Continuing calibration</b>				
What type of continuing calibration calculation was performed? ___%D or %R	/			
Was a continuing calibration analyzed daily?	/			
Were all percent differences (%D) < 15% or percent recoveries 85-115%?	/			
Were all the retention times within the acceptance windows?	/			
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	/			
Was a method blank analyzed for each matrix and concentration?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.			/	
<b>VI. Surrogate spikes</b>				
Were all surrogate %R within the QC limits?	/			
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?			/	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?			/	
<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.	/			
Was a MS/MSD analyzed every 20 samples of each matrix?	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	/			
<b>VIII. 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 QC limits?	/			

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 SDG #: su cover

VALIDATION FINDINGS CHECKLIST

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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 #: 18356A7  
 SDG #: see comment

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

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 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 (std)	CF (std)	Average CF (Initial)	Average CF (Initial)	%RSD	%RSD		
1	1CAL	1/9/08	GRU	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 #: 18356A7  
 SDG #: fu cover

### VALIDATION FINDINGS WORKSHEET Continuing Calibration Results Verification

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

METHOD: GC [Signature] 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 =  $100 \times \frac{(\text{ave. CF} - \text{CF})}{\text{ave. CF}}$   
 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	Reported		Recalculated		
				CF/Conc. CCV	Average CF (Ical)/CCV Conc.	CF/Conc. CCV	%D	%D
1	2/4/08 LEAL911B	2/4/08 0848 0917	GRD	0.9692	1.0	0.9692	3.1	3.1
2	10A1937B	2/5/08 0430	GRD	0.9769	1.0	0.9769	2.3	2.3
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**

LDC #: 18356A7  
 SDG #: per canal  
 METHOD: GC HPLC

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

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: 8

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
TFT	not spiked	0.04	0.04246 0.04246	106	106	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

**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:

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

SSC = Spiked sample concentration  
 SA = Spike added

SC = Sample concentration

RPD =  $\frac{((SSCMS - SSCMSD) \cdot 2) / ((SSCMS + SSCMSD)) \cdot 100}{}$

MS = Matrix spike

MSD = Matrix spike duplicate

MS/MSD samples: 22 + 23

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)	1.06	1.06	ND	1.04	1.04	98	98	99	99	0.58	0
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 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:

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

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

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

LCS/LCSD samples: 8035215 - 105

Compound	Spike Added ( <u>1.0</u> )		Spike Sample Concentration ( <u>1.0</u> )		Sample Conc. ( <u>0</u> )		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)	1.0	NA	1.0	NA	0		100	100	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 #: 18 356A7

SDG #: Lee Coney

**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: \_\_\_\_\_

**BRC Tronox Parcel H  
Data Validation Reports  
LDC# 18356**

Diesel Range Organics

*LDC*

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** BRC Tronox Parcel H  
**Collection Date:** January 25, 2008  
**LDC Report Date:** March 6, 2008  
**Matrix:** Soil/Water  
**Parameters:** Diesel Range Organics  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8A260143

### Sample Identification

TSB-HJ-01-10'	TSB-HJ-01-10'MS
TSB-HJ-01-10'RE	TSB-HJ-01-10'MSD
TSB-HJ-09-0'	TSB-HJ-02-10'MS
TSB-HJ-09-10'	TSB-HJ-02-10'MSD
TSB-HJ-03-0'	TSB-HJ-01-0'MS
TSB-HJ-03-0'-FD	TSB-HJ-01-0'MSD
TSB-HJ-03-10'	RINSATE-1MS
TSB-HR-03-0'	RINSATE-1MSD
TSB-HR-03-10'**	
TSB-HJ-02-0'**	
TSB-HJ-02-10'**	
TSB-HR-02-0'**	
TSB-HR-02-10'**	
TSB-HJ-11-0'**	
TSB-HJ-11-10'	
TSB-HJ-11-10'-FD	
TSB-HR-01-0'	
TSB-HR-01-10'	
TSB-HJ-01-0'	
RINSATE-1	

\*\*Indicates sample underwent EPA Level IV review



## Introduction

This data review covers 25 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 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.
- 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-1 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
RINSATE-1	ortho-Terphenyl	46 (52-150)	Diesel range organics	J- (all detects) UJ (all non-detects)	P
TSB-HJ-01-10'	ortho-Terphenyl	71 (73-150)	Diesel range organics	J- (all detects) UJ (all non-detects)	A

Sample	Surrogate	%R (Limits)	Compound	Flag	A or P
TSB-HJ-01-10'RE	ortho-Terphenyl	66 (73-150)	Diesel range organics	J- (all detects) UJ (all non-detects)	A
TSB-HJ-09-0'	ortho-Terphenyl	59 (73-150)	Diesel range organics	J- (all detects) UJ (all non-detects)	P
8030074-BLK	ortho-Terphenyl	51 (52-150)	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) 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-03-0' and TSB-HJ-03-0'-FD and samples TSB-HJ-11-10' and TSB-HJ-11-10'-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 F8A260143**

SDG	Sample	Compound	Flag	A or P	Reason
F8A260143	RINSATE-1 TSB-HJ-09-0'	Diesel range organics	J- (all detects) UJ (all non-detects)	P	Surrogate recovery (%R)
F8A260143	TSB-HJ-01-10' TSB-HJ-01-10'RE	Diesel range organics	J- (all detects) UJ (all non-detects)	A	Surrogate recovery (%R)

**BRC Tronox Parcel H  
 Diesel Range Organics - Laboratory Blank Data Qualification Summary - SDG  
 F8A260143**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel H  
 Diesel Range Organics - Field Blank Data Qualification Summary - SDG  
 F8A260143**

No Sample Data Qualified in this SDG

LDC #: 18356A8  
 SDG #: F8A260143  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**

Level III/IV

Date: 3/3/08  
 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	Δ	Sampling dates: 1/25/08
IIa.	Initial calibration	Δ	
IIb.	Calibration verification/ICV	A	ICV ≤ 15
III.	Blanks	Δ	
IVa.	Surrogate recovery	SW	
IVb.	Matrix spike/Matrix spike duplicates	Δ	
IVc.	Laboratory control samples	A	LC3
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	ND	D = 5, 6      15, 16
X.	Field blanks	ND	R = 20

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-01-10'	11	TSB-HJ-02-10'***	21	TSB-HJ-01-10'MS	31	8031301	2/5
2	TSB-HJ-01-10'RE	12	TSB-HR-02-0'***	22	TSB-HJ-01-10'MSD	32	8037073	2/6
3	TSB-HJ-09-0'	13	TSB-HR-02-10'***	23	TSB-HJ-02-10'MS	33	8030074	2/1
4	TSB-HJ-09-10'	14	TSB-HJ-11-0'***	24	TSB-HJ-02-10'MSD	34		
5	TSB-HJ-03-0'	15	TSB-HJ-11-10'	25	TSB-HJ-01-0'MS	35		
6	TSB-HJ-03-0'-FD	16	TSB-HJ-11-10'-FD	26	TSB-HJ-01-0'MSD	36		
7	TSB-HJ-03-10'	17	TSB-HR-01-0'	27	RINSATE-1MS	37		
8	TSB-HR-03-0'	18	TSB-HR-01-10'	28	RINSATE-1MSD	38		
9	TSB-HR-03-10'***	19	TSB-HJ-01-0'	29		39		
10	TSB-HJ-02-0'***	20	RINSATE-1	30		40		

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

LDC #: 18356A8  
 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 checked="" type="checkbox"/>	<input type="checkbox"/>	
Were the RT windows properly established?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>III. 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% 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>IV. 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>V. 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 checked="" type="checkbox"/>	<input type="checkbox"/>	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?	<input type="checkbox"/>	<input checked="" 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"/>	
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>VII. 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 #: 18356A8  
 SDG #: pu 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

## Surrogate Recovery

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

LDC #: 18350A8  
 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".

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

#	Sample ID	Detector/Column	Surrogate Compound	%R (Limits)	Qualifications
	20	not specified	H	46 ( 52-150 )	J- / UJ / P ND
	1	↓	↓	71 ( 73-150 )	J- / UJ / A ND
	2	↓	↓	66 ( )	J- / UJ / A ND
	3	↓	↓	59 ( )	J- / UJ / <del>A</del> P ND
	8030074-BIK	↓	↓	51 ( 52-150 )	J- / UJ / P ND

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 Tripentyltin	
D Bromochlorobenzene	J n-Triacontane	P 1-methylbiphenylene	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	

LDC #: 18356A8  
 SDG #: per could

**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 =  $100 \cdot (S/X)$   
 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 (100-std)	CF (100-std)	Average CF (Initial)	Average CF (Initial)	%RSD	%RSD		
1	KAL	1/31/08	DRD	15615	15615	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.

LDC #: 18352A8  
 SDG #: for 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(cal)/CCV Conc.	Reported		Recalculated	
					CF/Conc. CCV	%D	CF/Conc. CCV	%D
1	ECAL905	2/4/08	DRO	1000	913.23	8.7	913.23	8.7
2	ECAL917	2/5/08	↓	↓	1047.8227	4.8	1047.8227	4.8
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.

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

LDC #: 183306A8  
 SDG #: per coner  
 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  
 #8 9

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

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
<u>o-terphenyl</u>	<u>not specified</u>	<u>25.0</u>	<u>22.0434</u>	<u>88</u>	<u>88</u>	<u>0</u>

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 #: 12356 AX  
 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, SA = Spike added  
 $RPD = ((SSCMS - SSCMSD) * 2) / (SSCMS + SSCMSD) * 100$  MS = Matrix spike, MSD = Matrix spike duplicate

MS/MSD samples: 2 | 1 | 2

Compound	Spike Added		Sample Conc.	Spike Sample Concentration		Matrix spike		Matrix Spike Duplicate		MS/MSD												
	MS	MSD		MS	MSD	Percent Recovery		Percent Recovery		RPD												
	Reported	Recalc.		Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.											
Gasoline (8015)																						
Diesel (8015)	87.4	88.9	ND	63.7	61.7	73	73	69	69	3.3	3.3											
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 #: 18350 AX  
 SDG #: fu cover

VALIDATION FINDINGS WORKSHEET  
 Laboratory Control Sample/Laboratory Control Sample 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 laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:

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

RPD =  $((SSCLCS - SSCLCSD) * 2) / (SSCLCS + SSCLCSD) * 100$  LCS = Laboratory Control Sample percent recovery, LCSD = Laboratory Control Sample duplicate percent recovery

LCS/LCSD samples: 8031301-105

Compound	Spike Added (mg/kg)		Spike Sample Concentration (mg/kg)		Sample Conc. (mg/kg)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	---	---	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)												
Diesel (8015)	83.3	NA	61.3	NA	0		74	74	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 #: 18356 AY  
SDG #: Lee Casey

# 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:

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

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

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

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

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

ND

#	Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications

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

**BRC Tronox Parcel H  
Data Validation Reports  
LDC# 18356**

**Dioxins/Dibenzofurans**

*LDC*



## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** BRC Tronox Parcel H  
**Collection Date:** January 25, 2008  
**LDC Report Date:** March 7, 2008  
**Matrix:** Soil/Water  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** EPA Level III & IV  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** F8A260143

### Sample Identification

TSB-HJ-01-10'  
TSB-HJ-09-0'  
TSB-HJ-09-10'  
TSB-HJ-03-0'  
TSB-HJ-03-0'-FD  
TSB-HJ-03-10'  
TSB-HR-03-0'  
TSB-HR-03-10'\*\*  
TSB-HJ-02-0'\*\*  
TSB-HJ-02-10'\*\*  
TSB-HR-02-0'\*\*  
TSB-HR-02-10'  
TSB-HJ-11-0'  
TSB-HJ-11-10'  
TSB-HJ-11-10'-FD  
TSB-HR-01-0'  
TSB-HR-01-10'  
TSB-HJ-01-0'  
RINSATE-1

\*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 18 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.

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.
- 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) for samples on which EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by EPA Level III criteria.

## **III. Initial Calibration**

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

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

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

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

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

Routine calibration was performed at the required frequencies.

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

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

## **V. Blanks**

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

Sample RINSATE-1 was identified as a rinsate. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank.

## VI. Matrix Spike/Matrix Spike Duplicates

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

## VII. Laboratory Control Samples (LCS)

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

## VIII. Regional Quality Assurance and Quality Control

Not applicable.

## IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
TSB-HJ-03-10'	<sup>13</sup> C-OCDD	38 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

## X. Target Compound Identifications

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

## XI. Compound Quantitation and CRQLs

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

## XII. System Performance

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

### **XIII. Overall Assessment of Data**

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

### **XIV. Field Duplicates**

Samples TSB-HJ-03-0' and TSB-HJ-03-0'-FD and samples TSB-HJ-11-10' and TSB-HJ-11-10'-FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples.

**BRC Tronox Parcel H  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG F8A260143**

SDG	Sample	Compound	Flag	A or P	Reason
F8A260143	TSB-HJ-03-10'	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R)

**BRC Tronox Parcel H  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG  
F8A260143**

No Sample Data Qualified in this SDG

**BRC Tronox Parcel H  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG F8A260143**

No Sample Data Qualified in this SDG

LDC #: 18356A21  
 SDG #: F8A260143  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 9/6/08  
 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	Δ	Sampling dates: 1/25/08
II.	GC/MS Instrument performance check	Δ	
III.	Initial calibration	Δ	
IV.	Routine calibration	Δ	
V.	Blanks	Δ	
VI.	Matrix spike/Matrix spike duplicates	N	client specified
VII.	Laboratory control samples	Δ	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	Δ	Not reviewed for Level III validation.
XI.	Compound quantitation and CRQLs	Δ	Not reviewed for Level III validation.
XII.	System performance	Δ	Not reviewed for Level III validation.
XIII.	Overall assessment of data	Δ	
XIV.	Field duplicates	ND	D = 4, 5      14, 15
XV.	Field blanks	ND	R = 19

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-01-10'	11	TSB-HR-02-0**	21	8036238	31
2	TSB-HJ-09-0'	12	TSB-HR-02-10'	22	8037221	32
3	TSB-HJ-09-10'	13	TSB-HJ-11-0'	23		33
4	TSB-HJ-03-0' D	14	TSB-HJ-11-10' D	24		34
5	TSB-HJ-03-0'-FD D	15	TSB-HJ-11-10'-FD D	25		35
6	TSB-HJ-03-10'	16	TSB-HR-01-0'	26		36
7	TSB-HR-03-0'	17	TSB-HR-01-10'	27		37
8	TSB-HR-03-10**	18	TSB-HJ-01-0'	28		38
9	TSB-HJ-02-0**	19	RINSATE-1	29		39
10	TSB-HJ-02-10**	20		30		40

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

LDC #: 18356A2  
 SDG #: see cond

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2  
 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.	/			
Cooler temperature criteria was met.	/			
<b>II. GC/MS Instrument performance check</b>				
Was PFK exact mass 380.9760 verified?	/			
Were the retention time windows established for all homologues?	/			
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers $\leq 25\%$ ?	/			
Is the static resolving power at least 10,000 (10% valley definition)?	/			
Was the mass resolution adequately check with PFK?	/			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	/			
<b>III. Initial calibration</b>				
Was the initial calibration performed at 5 concentration levels?	/			
Were all percent relative standard deviations (%RSD) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	/			
Did all calibration standards meet the Ion Abundance Ratio criteria?	/			
Was the signal to noise ratio for each target compound $\geq 2.5$ and for each recovery and internal standard $\geq 10$ ?	/			
<b>IV. Continuing calibration</b>				
Was a routine calibration performed at the beginning and end of each 12 hour period?	/			
Were all percent differences (%D) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	/			
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	/			
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	/			
Was a method blank performed for each matrix and concentration?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?		/		
<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.			/	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?			/	
<b>VII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	/			



LDC #: 18356A21  
 SDG #: see 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 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 #: 18350A21  
SDG #: 2nd cover

### VALIDATION FINDINGS CHECKLIST

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

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

VALIDATION FINDINGS WORKSHEET

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

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

Notes:

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

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

$RRF = (A_x)(C_{is}) / (A_{is})(C_x)$   
 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,  
 $A_{is}$  = Area of associated internal standard  
 $C_{is}$  = Concentration of internal standard  
 $X$  = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (Initial)	%RSD	Average RRF (Initial)	%RSD	RRF (CS3 std)	%RSD	RRF (CS3 std)	%RSD
1	ICAL	2/7/08	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.114	1.23	1.116	1.23	1.13	1.23	1.1344	1.23
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.244	7.84	1.244	7.84	1.21	7.84	1.2084	7.84
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	0.974	3.17	0.974	3.17	0.95	3.17	0.9514	3.17
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.074	5.61	1.074	5.61	1.14	5.61	1.135	5.61
			OCDF ( <sup>13</sup> C-OCDD)	3.024	6.72	3.024	6.72	2.93	6.72	2.9348	6.72
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-OCDD)								
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-OCDD)								

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

LDC #: 8356 A21  
 SDG #: per manual

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

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

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 =  $(A_x)(C_s) / (A_s)(C_x)$       RRF = continuing calibration RRF  
 $A_x$  = Area of compound,       $A_s$  = Area of associated internal standard  
 $C_x$  = Concentration of compound,       $C_s$  = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	07FED8A105	2/7/08	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.114	1.10	1.09806	1.6	1.6
	-51		2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.244	1.15	1.15344	7.3	7.3
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	0.974	0.98	0.9751	0.1	0.1
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.074	1.07	1.0168	5.3	5.3
			OCDF ( <sup>13</sup> C-OCDD)	3.024	3.02	3.024	0	0
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-OCDD)					
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-OCDD)					

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



Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte		
1	303.9016	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>9</sub> <sup>37</sup> ClO	HpCDF		
	305.8987	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> C <sub>10</sub>	TCDF		409.7788	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> C <sub>10</sub> O	HpCDF		
	315.9419	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF (S)		417.8250	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</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> C <sub>10</sub>	TCDF (S)		419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> C <sub>10</sub> O	HpCDF (S)		
	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>6</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	321.8936	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> C <sub>10</sub> <sub>2</sub>	TCDD		425.7737	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <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> Cl <sub>2</sub> O <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>4</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD (S)		
	375.8364	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDFE		479.7165	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>7</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDFE		
	[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>35</sub> Cl <sub>9</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO	OCDF
		341.8567	M+4	C <sub>12</sub> H <sub>35</sub> 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>35</sub> Cl <sub>4</sub> <sup>37</sup> C <sub>10</sub>		PeCDF (S)		457.7377	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
		353.8970	M+4	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF (S)		459.7348	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD
355.8546		M+2	C <sub>12</sub> H <sub>35</sub> 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	C <sub>12</sub> H <sub>35</sub> 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>35</sub> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	513.6775	M+4		C <sub>12</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDFE		
369.8919		M+4	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> 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>35</sub> 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> Cl <sub>2</sub> O	HxCDF (S)						
	389.8156	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD							
	391.8127	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							
	401.8559	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)							
	403.8529	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</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> Cl <sub>2</sub> O	OCDFE							
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK							

(a) 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



