

### Laboratory Data Consultants, Inc.

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Northgate Environmental Management, Inc.

December 14, 2010

1100 Quail Street Ste. 102 Newport Beach, CA 92660 ATTN: Ms. Cindy Arnold

SUBJECT: Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada,

Data Validation

Dear Ms. Arnold,

Enclosed are the final validation reports for the fractions listed below. These SDGs were received on November 23, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

### **LDC Project # 24449:**

SDG_#	<u>Fraction</u>
280-6290-1, 280-6385-1 280-6415-1, 280-7549-1	Volatiles, Semivolatiles, Chlorinated Pesticides, Metals, Wet Chemistry

The data validation was performed under Stage 2B/4 guidelines. The analyses were validated using the following documents, as applicable to each method:

- Standard Operating Procedures (SOP) 40, Data Review/Validation, BRC 2009
- Quality Assurance Project Plan Tronox LLC Facility, Henderson Nevada, June 2009
- NDEP Guidance, May 2006
- USEPA, Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, June 2008
- USEPA, Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, October 2004

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

Sincerely,

Erlinda T. Rauto

Operations Manager/Senior Chemist

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LDC #: 24449 SDG #: 280-6290-1, 280-6385-1, 280-6415-1, 280-7549-1 Page: 1 of 1 Reviewer: JE 2nd Reviewer: BC

### Tronox Northgate Henderson Worksheet

EDD Area	Yes	No	NA	Findings/Comments
I. Completeness				
Is there an EDD for the associated Tronox validation report?	Χ			
II. EDD Qualifier Population				
Were all qualifiers from the validation report populated into the EDD?	X	10,000		
III. EDD Lab Anomalies				
Were EDD anomalies identified?		Х		
If yes, were they corrected or documented for the client?				See EDD_discrepancy_ form_LDC24449_111310.doc
IV. EDD Delivery				
Was the final EDD sent to the client?	Х			

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

Project/Site Name: Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

Collection Date: September 17, 2010

LDC Report Date: December 8, 2010

Matrix: Soil/Water

Parameters: Volatiles

Validation Level: Stage 2B & 4

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 280-7549-1

### Sample Identification

TB-09172010 1

SSA07-05-9.5BPC

SSAO8-05-9.5BPC\*\*

SSAO8-05-9.5BPC FD

SSAO8-08-9.5BPC

SSA07-08-0.5BPC

SSA07-08-0BPC

SSA07-07-0BPC

SSA07-07-0.5BPC

SSAO8-06-0BPC

SSAO8-06-0.5BPC

SSA08-09-0BPC

SSAO8-09-0.5BPC

SSAO8-12-0BPC\*\*

SSAO8-12-0.5BPC

SSAO8-12-0BPC\_FD

SSA07-05-9.5BPCMS

SSAO7-05-9.5BPCMSD

SSAO7-07-0BPCMS

SSAO7-07-0BPCMSD

<sup>\*\*</sup>Indicates sample underwent Stage 4 review

### Introduction

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

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008).

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.

Samples indicated by a double asterisk on the front cover underwent a Stage 4 review. A Stage 2B review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Stage 2B criteria since this review is based on QC data.

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- 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 with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P
All samples in SDG 280-7549-1	All TCL compounds	Freezer storing samples went out of temperature control limits for 11 hours.	Cooler temperature must be 4±2°C.	J- (all detects) UJ (all non-detects)	А

### 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 30.0% for all compounds.

Average relative response factors (RRF) for all compounds were within method and validation criteria with the following exceptions:

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
9/27/10	tert-Butyl alcohol	0.0042 (≥0.05)	TB-09172010_1 MB280-33890/6	J (all detects) UJ (all non-detects)	Α

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
8/31/10	tert-Butyl alcohol	0.0243 (≥0.05)	SSAO7-05-9.5BPC SSAO8-05-9.5BPC** SSAO8-05-9.5BPC_FD SSAO8-08-9.5BPC SSAO7-08-0.5BPC SSAO7-08-0.5BPC SSAO7-07-0.5BPC SSAO8-06-0.5BPC SSAO8-06-0.5BPC SSAO8-09-0.5BPC SSAO8-09-0.5BPC SSAO8-09-0.5BPC SSAO8-12-0.5BPC SSAO8-12-0.5BPC SSAO8-12-0.5BPC SSAO8-12-0.5BPC SSAO8-12-0.5BPC SSAO7-05-9.5BPCMS SSAO7-05-9.5BPCMS SSAO7-07-0BPCMS SSAO7-07-0BPCMS SSAO7-07-0BPCMSD MB280-32921/3-A 9/24 MB280-32921/3-A 9/25 MB280-33216/1-A	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) and 25.0% for all other compounds.

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

All of the continuing calibration relative response factors (RRF) were within method and validation criteria with the following exceptions:

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
9/24/10	tert-Butyl alcohol	0.0192 (≥0.05)	SSAO7-05-9.5BPC SSAO8-05-9.5BPC** SSAO8-05-9.5BPC_FD SSAO8-08-9.5BPC SSAO7-08-0.5BPC SSAO7-08-0BPC SSAO7-07-0BPC SSAO7-07-0-5BPC SSAO7-05-9.5BPCMS SSAO7-07-05-9.5BPCMS SSAO7-07-0BPCMS SSAO7-07-0BPCMS SSAO7-07-0BPCMSD MB280-32921/3-A 9/24	J (all detects) UJ (all non-detects)	Α .

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
9/25/10	tert-Butyl alcohol	0.0253 (≥0.05)	SSAO8-06-0BPC SSAO8-06-0.5BPC SSAO8-12-0.5BPC MB280-32921/3-A 9/25	J (all detects) UJ (all non-detects)	Α
9/27/10	tert-Butyl alcohol	0.0226 (≥0.05)	SSAO8-09-0BPC SSAO8-09-0.5BPC SSAO8-12-0BPC** SSAO8-12-0BPC_FD MB280-33216/1-A	J (all detects) UJ (all non-detects)	А
9/30/10	tert-Butyl alcohol	0.0034 (≥0.05)	TB-09172010_1 MB280-33890/6	J (all detects) UJ (all non-detects)	А

### V. Blanks

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

Method Blank ID	Analysis Date	Compound TIC (RT in minutes)	Concentration	Associated Samples
MB280-32921/3-A 9/24	9/24/10	Methylene chloride	1.98 ug/Kg	SSAO7-05-9.5BPC SSAO8-05-9.5BPC** SSAO8-05-9.5BPC_FD SSAO8-08-9.5BPC SSAO7-08-0.5BPC SSAO7-08-0BPC SSAO7-07-0BPC SSAO7-07-0.5BPC
MB280-32921/3-A 9/25	9/25/10	Methylene chloride	1.53 ug/Kg	SSAO8-06-0BPC SSAO8-06-0.5BPC SSAO8-12-0.5BPC
MB280-33216/1-A 9/27/		Hexachlorobutadiene Methylene chloride Naphthalene	0.883 ug/Kg 1.47 ug/Kg 0.696 ug/Kg	SSAO8-09-0BPC SSAO8-09-0.5BPC SSAO8-12-0BPC** SSAO8-12-0BPC_FD
MB280-33890/6	9/30/10	Methylene chloride	0.399 ug/L	TB-09172010_1

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	Compound TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
SSAO7-05-9.5BPC	Methylene chloride	2.0 ug/Kg	2.0U ug/Kg
SSAO8-05-9.5BPC**	Methylene chloride	0.99 ug/Kg	0.99U ug/Kg

Sample	Compound TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
SSAO8-05-9.5BPC_FD	Methylene chloride	1.0 ug/Kg	1.0U ug/Kg
SSAO8-08-9.5BPC	Methylene chloride	1.1 ug/Kg	1.1U ug/Kg
SSA07-08-0.5BPC	Methylene chloride	0.69 ug/Kg	0.69U ug/Kg
SSA07-08-0BPC	Methylene chloride	1.1 ug/Kg	1.1U ug/Kg
SSAO7-07-0BPC	Methylene chloride	1.3 ug/Kg	1.3U ug/Kg
SSA07-07-0.5BPC	Methylene chloride	1.0 ug/Kg	1.0U ug/Kg
SSAO8-06-0BPC	Methylene chloride	1.9 ug/Kg	1.9U ug/Kg
SSAO8-06-0.5BPC	Methylene chloride	1.1 ug/Kg	1.1U ug/Kg
SSAO8-12-0.5BPC	Methylene chloride	1.3 ug/Kg	1.3U ug/Kg
SSAO8-09-0BPC	Methylene chloride Naphthalene	1.8 ug/Kg 1.2 ug/Kg	1.8U ug/Kg 1.2U ug/Kg
SSAO8-09-0.5BPC	Methylene chloride Naphthalene	1.2 ug/Kg 0.60 ug/Kg	1.2U ug/Kg 0.60U ug/Kg
SSAO8-12-0BPC**	Methylene chloride	1.2 ug/Kg	1.2U ug/Kg
SSAO8-12-0BPC_FD	Methylene chloride	0.78 ug/Kg	0.78U ug/Kg
TB-09172010_1	Methylene chloride	0.79 ug/L	0.79U ug/L

Sample TB-09172010\_1 was identified as a trip blank. No volatile contaminants were found in this blank with the following exceptions:

Trìp Blank ID	Sampling Date	Compound	Concentration	Associated Samples
TB-09172010_1	9/17/10	Acetone Methylene chloride	2.5 ug/L 0.79 ug/L	All soil samples in SDG 280-7549-1

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	Compound	Reported Concentration	Modified Final Concentration
SSAO8-05-9.5BPC**	Methylene chloride	0.99 ug/Kg	0.99U ug/Kg
SSAO8-05-9.5BPC_FD	Methylene chloride	1.0 ug/Kg	1.0U ug/Kg
SSAO8-08-9.5BPC	Methylene chloride	1.1 ug/Kg	1.1U ug/Kg
SSAO7-08-0.5BPC	Acetone Methylene chloride	4.7 ug/Kg 0.69 ug/Kg	4.7U ug/Kg 0.69U ug/Kg
SSA07-08-0BPC	Methylene chloride	1.1 ug/Kg	1.1U ug/Kg
SSAO7-07-0BPC	Methylene chloride	1.3 ug/Kg	1.3U ug/Kg
SSAO7-07-0.5BPC	Methylene chloride	1.0 ug/Kg	1.0U ug/Kg
SSAO8-06-0.5BPC	Methylene chloride	1.1 ug/Kg	1.1U ug/Kg
SSAO8-09-0.5BPC	Methylene chloride	1.2 ug/Kg	1.2U ug/Kg
SSAO8-12-0BPC**	Acetone Methylene chloride	5.0 ug/Kg 1.2 ug/Kg	5.0U ug/Kg 1.2U ug/Kg
SSAO8-12-0.5BPC	Methylene chloride	1.3 ug/Kg	1.3U ug/Kg
SSAO8-12-0BPC_FD	Methylene chloride	0.78 ug/Kg	0.78U 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.

### 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 relative percent differences (RPD) were not within QC limits for several compounds, the MS/MSD percent recoveries (%R) were within QC limits and no data were qualified.

### VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Although the LCS percent recovery (%R) was not within QC limits for one compound, the LCSD 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. 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 Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

### XII. Project Quantitation Limit

All project quantitation limits were within validation criteria for samples on which a Stage 4 review was performed.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-7549-1	All compounds reported below the PQL.	J (all detects)	А

Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

### XIII. Tentatively Identified Compounds (TICs)

Tentatively identified compounds were not reported by the laboratory.

### XIV. System Performance

The system performance was acceptable for samples on which a Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

### XV. Overall Assessment of Data

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

### XVI. Field Duplicates

Samples SSAO8-05-9.5BPC\*\* and SSAO8-05-9.5BPC\_FD and samples SSAO8-12-0BPC\*\* and SSAO8-12-0BPC\_FD were identified as field duplicates. No volatiles were detected in any of the samples with the following exceptions:

:	Concentra	ation (ug/Kg)			181	
Compound	SSAO8-05-9.5BPC**	SSAO8-05-9.5BPC_FD	RPD (Limits)	Difference (Limits)	Flags	AorP
Chloroform	0.44	0.34	-	0.10 (≤6.4)	-	-
Methylene chloride	le 0.99 1.0		-	0.01 (≤3.2)	<u>-</u>	_

	Concentra	ation (ug/Kg)				
Compound	SSAO8-12-0BPC**	SSA08-12-0BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
1,1-Dichloroethene	0.55	2.8U	-	2.25 (≤2.8)	-	-
2-Butanone	13U	3.2	-	9.80 (≤13)		-
Acetone	5.0	16	-	11.00 (≤13)	_	-
Methylene chloride	1.2	0.78	-	0.42 (≤3.1)	-	-
Naphthalene	7.5	3.9	-	3.60 (≤3.1)	J (all detects)	Α

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Volatiles - Data Qualification Summary - SDG 280-7549-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-7549-1	TB-09172010_1 SSAO7-05-9.5BPC SSAO8-05-9.5BPC** SSAO8-05-9.5BPC_FD SSAO8-08-9.5BPC SSAO7-08-0.5BPC SSAO7-07-0BPC SSAO7-07-0.5BPC SSAO8-06-0BPC SSAO8-06-0.5BPC SSAO8-09-0BPC SSAO8-09-0BPC SSAO8-09-0.5BPC SSAO8-12-0BPC** SSAO8-12-0BPC_FD	07-05-9.5BPC 08-05-9.5BPC** 08-05-9.5BPC_FD 08-08-9.5BPC 07-08-0.5BPC 07-08-0BPC 07-07-08-0BPC 07-07-0.5BPC 08-06-0BPC 08-06-0.5BPC 08-06-0.5BPC 08-09-0.5BPC 08-09-0BPC 08-12-0BPC** 08-12-0BPC**		А	Cooler temperature (o)
280-7549-1	TB-09172010_1 SSAO7-05-9.5BPC SSAO8-05-9.5BPC** SSAO8-05-9.5BPC_FD SSAO8-08-9.5BPC SSAO7-08-0.5BPC SSAO7-07-08-0.5BPC SSAO7-07-0.5BPC SSAO8-06-0.5BPC SSAO8-06-0.5BPC SSAO8-09-0BPC SSAO8-09-05BPC SSAO8-12-0BPC** SSAO8-12-0BPC_FD	SAO7-05-9.5BPC SAO8-05-9.5BPC_** SAO8-05-9.5BPC_FD SAO8-08-9.5BPC SAO7-08-05BPC SAO7-07-08-05BPC SAO7-07-07-08-05BPC SAO8-06-08-06 SAO8-06-05BPC SAO8-06-05BPC SAO8-09-05BPC SAO8-09-05BPC SAO8-09-05BPC SAO8-12-05BPC		Α	Initial calibration (RRF) (c)
280-7549-1	SSAO7-05-9.5BPC SSAO8-05-9.5BPC** SSAO8-05-9.5BPC_FD SSAO8-08-9.5BPC SSAO7-08-0.5BPC SSAO7-07-0BPC SSAO7-07-0.5BPC SSAO8-06-0BPC SSAO8-06-0.5BPC SSAO8-12-0.5BPC SSAO8-09-0.5BPC SSAO8-09-0.5BPC SSAO8-12-0.5BPC SSAO8-12-0.5BPC	tert-Butyl alcohol	J (all detects) UJ (all non-detects)	A	Continuing calibration (RRF) (c)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-7549-1	TB-09172010_1 SSAO7-05-9.5BPC SSAO8-05-9.5BPC** SSAO8-05-9.5BPC_FD SSAO8-08-9.5BPC SSAO7-08-0.5BPC SSAO7-07-08-0.5BPC SSAO7-07-0.5BPC SSAO8-06-0BPC SSAO8-06-0.5BPC SSAO8-09-0BPC SSAO8-09-0.5BPC SSAO8-12-0BPC** SSAO8-12-0BPC_FD	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (PQL) (sp)
280-7549-1	SSAO8-12-0BPC** SSAO8-12-0BPC_FD	Naphthalene	J (all detects)	А	Field duplicates (Differences) (fd)

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Volatiles - Laboratory Blank Data Qualification Summary - SDG 280-7549-1

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
280-7549-1	SSAO7-05-9.5BPC	Methylene chloride	2.0U ug/Kg	А	bl
280-7549-1	SSAO8-05-9.5BPC**	Methylene chloride	0.99U ug/Kg	Α	bl
280-7549-1	SSAO8-05-9.5BPC_FD	Methylene chloride	1.0U ug/Kg	А	bl
280-7549-1	SSAO8-08-9.5BPC	Methylene chloride	1.1U ug/Kg	А	bl
280-7549-1	SSAO7-08-0.5BPC	Methylene chloride	0.69U ug/Kg	А	bl
280-7549-1	SSAO7-08-0BPC	Methylene chloride	1.1U ug/Kg	Α	bl
280-7549-1	SSAO7-07-0BPC	Methylene chloride	1.3U ug/Kg	Α	bl
280-7549-1	SSAO7-07-0.5BPC	Methylene chloride	1.0U ug/Kg	Ą	ы
280-7549-1	SSAO8-06-0BPC	Methylene chloride	1.9U ug/Kg	А	bl
280-7549-1	SSAO8-06-0.5BPC	Methylene chloride	1.1U ug/Kg	Α	bl
280-7549-1	SSAO8-12-0.5BPC	Methylene chloride	1.3U ug/Kg	А	ы

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
280-7549-1	SSAO8-09-0BPC	Methylene chloride Naphthalene	1.8U ug/Kg 1.2U ug/Kg	А	bl
280-7549-1	SSAO8-09-0.5BPC	Methylene chloride Naphthalene	1.2U ug/Kg 0.60U ug/Kg	Ā	bi
280-7549-1	SSAO8-12-0BPC**	Methylene chloride	1.2U ug/Kg	Α	bl
280-7549-1	SSAO8-12-0BPC_FD	Methylene chloride	0.78U ug/Kg	А	bl
280-7549-1	TB-09172010_1	Methylene chloride	0.79U ug/L	А	bl

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Volatiles - Field Blank Data Qualification Summary - SDG 280-7549-1

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
280-7549-1	SSAO8-05-9.5BPC**	Methylene chloride	0.99U ug/Kg	А	bt
280-7549-1	SSAO8-05-9.5BPC_FD	Methylene chloride	1.0U ug/Kg	А	bt
280-7549-1	SSAO8-08-9.5BPC	Methylene chłoride	1.1U ug/Kg	А	bt
280-7549-1	SSAO7-08-0.5BPC	Acetone Methylene chloride	4.7U ug/Kg 0.69U ug/Kg	А	bt
280-7549-1	SSAO7-08-0BPC	Methylene chloride	1.1U ug/Kg	Α	bt
280-7549-1	SSAO7-07-0BPC	Methylene chloride	1.3U ug/Kg	Α .	bt
280-7549-1	SSAO7-07-0.5BPC	Methylene chloride	1.0U ug/Kg	А	bt
280-7549-1	SSAO8-06-0.5BPC	Methylene chloride	1.1U ug/Kg	А	bt
280-7549-1	SSAO8-09-0.5BPC	Methylene chloride	1.2U ug/Kg	Α	bt
280-7549-1	SSAO8-12-0BPC**	Acetone Methylene chloride	5.0U ug/Kg 1.2U ug/Kg	А	bt
280-7549-1	SSAO8-12-0.5BPC	Methylene chloride	1.3U ug/Kg	А	bt
280-7549-1	SSAO8-12-0BPC_FD	Methylene chloride	0.78U ug/Kg	А	bt

### **Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET**

LDC #: SDG #: 280-7549-1 Stage 2B/4 Laboratory: Test America

Reviewer: 2nd Reviewer:

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

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

	Validațion Area		Comments
1.	Technical holding times	S¥	Sampling dates: 9/17 /10
11.	GC/MS Instrument performance check	À	
III.	Initial calibration	SW	7 RSD
IV.	Continuing calibration/ICV	SN)	CW/IW & 25 Z
V.	Blanks	ZM)	
VI:	Surrogate spikes	<u> </u>	
VII.	Matrix spike/Matrix spike duplicates	SW)	
VIII.	Laboratory control samples	SW	LCS /D
IX.	Regional Quality Assurance and Quality Control	N	· ·
<u>X</u> .	Internal standards	A	
XI.	Target compound identification	4	Not reviewed for Stage 2B validation.
XII.	Compound quantitation/CRQLs	A	Not reviewed for Stage 2B validation.
XIII.	Tentatively identified compounds (TICs)	N	Not reviewed for Stage 2B validation.
XIV.	System performance	A	Not reviewed for Stage 2B validation.
XV.	Overall assessment of data	A	
XVI.	Field duplicates	SW	$D_1 = 3.4$ $D_2 = 14.16$
XVII.	Field blanks	SM	TB =/

Note:

A = Acceptable

N = Not provided/applicable

ND = No compounds detected

D = Duplicate TB = Trip blank

SW = See worksheet

R = Rinsate FB = Field blank

EB = Equipment blank

Validated Samples:

\*\* Indicates sample underwent Stage 4 validation

	VV AJ	41	7	5011				
ک 1	TB-09172010_1	W		74 SSAO8-06-0.5BPC S	+ 21	MB 260- 32921/3-A	9/24 3/01:	n
2	SSAO7-05-9.5BPC	S	12	SSAO8-09-0BPC	122 >	280-32921/3-4	^/2 -374;	a .
3 1	SSAO8-05-9.5BPC** /	J.	13	SSAO8-09-0.5BPC	+ 3 23	280-33216/1	<i>7</i> 5	
4	SSAO8-05-9.5BPC_FD P		14 14	SSAO8-12-0BPC** ∅γ	24 Y	280- 33371/1-4	<b>\</b> 34	(kkk,mmm)
5	SSAO8-08-9.5BPC		15	∳ SSAO8-12-0.5BPC	+ 25 5	280- 33890/6	35	
6	SSAO7-08-0.5BPC		16 16	SSAO8-12-0BPC_FD 🎷	26	,	36	
7	SSAO7-08-0BPC		17	SSAO7-05-9.5BPCMS	27		37	
8	SSAO7-07-0BPC		18 <sup>/</sup>	SSAO7-05-9.5BPCMSD	28		38	
9 1	SSA07-07-0.5BPC		19	SSAO7-07-0BPCMS	29		39	
10	SSAO8-06-0BPC	J	20	SSAO7-07-0BPCMSD	30		40	

Page: 1 of  $\nearrow$ Reviewer:  $\sqrt{VG}$ 2nd Reviewer:

Method: Volatiles (EPA SW 846 Method 8260B)

Method: Volatiles (EPA SVV 846 Method 82608)	<del></del>	T	Ī	1
Validation Area	Yes	No	NA	Findings/Comments
J. Technical holding times	· -	ı	Γ	
All technical holding times were met.		ļ		
Cooler temperature criteria was met.	1		<u> </u>	
II: GC/MS Instrument performance check	Τ	ı	ı	
Were the BFB performance results reviewed and found to be within the specified criteria?	-			
Were all samples analyzed within the 12 hour clock criteria?		}	************	
III. Initial calibration	· · · · · · · · · · · · · · · · · · ·	ı	· · · · · · · · · · · · · · · · · · ·	
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?				
IV. Continuing calibration				
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?				
V. Blanks				
Was a method blank associated with every sample in this SDG?	_			
Was a method blank analyzed at least once every 12 hours for each matrix and concentration?		,		
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		•		
VI. Surrögate spikes		,		
Were all surrogate %R within QC limits?				
If the percent recovery (%R) for one or more surrogates was out of QC limits, was a reanalysis performed to confirm samples with %R outside of criteria?				
VII: Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.		,		
Was a MS/MSD analyzed every 20 samples of each matrix?				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?		/		
VIII. Laboratory control samples				
Was an LCS analyzed for this SDG?				

LDC #: 2444 B /

### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 2
Reviewer: 776
2nd Reviewer: \_\_\_\_\_

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per analytical batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		_		
IX: Regional Quality Assurance: and: Quality Control:	ı	I		Г
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?			_/	
X. Internal standards	r			
Were internal standard area counts within -50% or +100% of the associated calibration standard?				
Were retention times within + 30 seconds of the associated calibration standard?		*************	********	
XI. Target:compound identification				
Were relative retention times (RRT's) within + 0.06 RRT units of the standard?				
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	/			
Were chromatogram peaks verified and accounted for?			*******	
XII. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?		/		
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?		<u></u>	٠	
XIII Tentatively identified compounds (TICs)	7			
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?				
Were relative intensities of the major ions within ± 20% between the sample and the reference spectra?			/	
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?			/	
XIV. System performance		•		
System performance was found to be acceptable.	$\angle$			
XV. Overall assessment of data				
Overall assessment of data was found to be acceptable.				
XVI. Field duplicates	,			
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.				
XVII - Fjeld blanks	<i>/</i>			
Field blanks were identified in this SDG.	/	,		
Target compounds were detected in the field blanks.				

# TARGET COMPOUND WORKSHEET

METHOD: VOA (EPA SW 846 Method 8260B)

A. Chloromethane*	U. 1,1,2-Trichloroethane	OO. 2,2-Dichloropropane	III. n-Butylbenzene	CCCC.1-Chlorohexane
B. Bromomethane	V. Benzene	PP. Bromochloromethane	JJJ. 1,2-Dichlorobenzene	DDDD. Isopropyl alcohol
C. Vinyl choride**	W. trans-1,3-Dichloropropene	QQ. 1,1-Dichloropropene	KKK. 1,2,4-Trichlorobenzene	EEEE. Acetonitrile
D. Chloroethane	X. Bromoform*	RR. Dibromomethane	LLL. Hexachlorobutadiene	FFFF. Acrolein
E. Methylene chloride	Y. 4-Methyl-2-pentanone	SS. 1,3-Dichloropropane	MMM. Naphthalene	GGGG. Acrylonitrile
F. Acetone	Z. 2-Hexanone	TT. 1,2-Dibromoethane	NNN. 1,2,3-Trichlorobenzene	HHHH. 1,4-Dioxane
. G. Carbon disulfide	AA. Tetrachloroethene	UU. 1,1,1,2-Tetrachloroethane	OOO. 1,3,5-Trichlorobenzene	III. 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, ais-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. Chloroform⁴	EE. Ethylbenzene**	YY. n-Propylbenzene	SSS. o-Xylene	MMMM. Benzyl chloride
L. 1,2-Dichloroethane	FF. Styrene	ZZ. 2-Chlorotoluene	TTT. 1,1,2-Trichloro-1,2,2-trifluoroethane	NNNN.
M. 2-Butanone	GG. Xylenes, total	AAA. 1,3,5-Trimethylbenzene	UUU. 1,2-Dichlorotetrafluoroethane	0000
N. 1.1.1-Trichloroethane	HH. Vinyl acetate	BBB. 4-Chlorotoluene	VVV. 4-Ethyltoluene	
O. Carbon tetrachloride	II. 2-Chloroethylvinyl ether	CCC. tert-Butylbenzene	WWW. Ethanol	<b>ටටටට</b>
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. ais-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-tsopropy/toluene	AAAA. Ethyl tert-butyl ether	ບນບບ.
T. Dibromochloromethane	NN. Methyl ethyl ketone	HHH. 1,4-Dichlorobenzene	BBBB. tert-Amyl methyl ether	vvvv.

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

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### VALIDATION FINDINGS WORKSHEET **Technical Holding Times**

	Page:_	!	_of/	
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All circled dates have exceeded the technical holding times.
Y N N/A Were all cooler temperatures within validation crit

_	1) N/A were all cooler temperatures within validation criteria?	
ſ		
$\ $	METHOD: GC/MS VOA (EPA SW 846 Method 8260B)	
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METHOD : GC/	MS VOA (EPA S'	W 846 Method	1 8260B)		<del></del>	<del></del>	1
Sample ID	Matrix	Preserved	Sampling Date	Extraction date	Analysis date	Total # of Days	Qualifi
AIJ	Freez	ers st	ring sample	went out of mits for 11			J-/WJ
	tem	peratur	e control li	mits for 11	honrs		(
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### **TECHNICAL HOLDING TIME CRITERIA**

Water unpreserved:

Aromatic within 7 days, non-aromatic within 14 days of sample collection.

Water preserved:

Both within 14 days of sample collection. Both within 14 days of sample collection.

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## VALIDATION FINDINGS WORKSHEET Initial Calibration

Page: 1 of 1 Reviewer: 2nd Reviewer:

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

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

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

N N/A

Were percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCC's and SPCC's? Was a curve fit used for evaluation?  $\frac{r^2 - 2}{r^2} \cdot \frac{1}{r} = \frac{1}{r^2} \cdot \frac{1}{r^2}$ 

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

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

	ī		<del></del>	<del>-</del>	Τ.	<del></del>	ī	11	<del>-</del>	<del>-</del>	<del>  -</del>	 T -	<del>-</del>	7	1	<del>-</del>	<u> </u>	1	T	<u> </u>	<del></del>	_	Т	<del></del>
Qualifications	J/11/2	7			721/3-9 9/20	125																		
Associated Samples	411 M+MB 280-3384 //				AII S + MB 260-32921/3-9 %	Mb 280- 32 921 /3-4 9	MB 280-332/16/1-4																	
Finding RRF (Limit: >0.05)	0.0047			1	0,0243																			
Finding %RSD (Limit: <30.0%)					•																			
Сотропи	222				222													-						
Standard ID	1CA1-MS1				1CA1-1NS J																			
Date	9/27/10				8/21/10	,																		
<b>*</b>																					ĺ	ļ		

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## VALIDATION FINDINGS WORKSHEET Continuing Calibration

Page: Reviewer: · 2nd Reviewer:\_

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

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

	Qualifications	164 J/45/A/C								-		
Were all %D and RRFs within the validation criteria of $\leq$ 25 %D and $\geq$ 0.05 RRF?	Associated Samples	80-32921/24	10, 11, 15, MB 286- 32921 /A.A. 11, 01		1x-14, 16, 1115 -30- 32-16/1-4	9/0682E-08CAU+1	,					
of <25 %D and >0.05 RRF ?	Finding RRF (Limit: >0.05)		0,0253		0.0226	6.0034		•				
sponse raciors (rviv riteria of ≤25 %D an	Finding %D (Limit: <25.0%)											
within the validation of	Punodwoo	722	722		46.2	222						
Were all %D and RRFs within the validation criteria	Standard ID	J 1225	J1258		VIOL 6 9	ms4125						
Y N/A We	# Date	9 /24 ho	9/25/10		1/4/160	9/20/E						

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## VALIDATION FINDINGS WORKSHEET Blanks

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Page:	Reviewer:	2nd Reviewer.

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

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

Was a method blank associated with every sample in this SDG? YN <del>Z</del>

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:  $\frac{q}{2} \frac{2d}{l_0} \frac{l_0}{l_0} > 3$ Y N/A

2 0 Ξ Sample Identification 9 1 く Associated Samples: 0,99 2 م, 0 \* 1666 - 080 Blank ID 1.98 ىلا Compound Methylene chloride Conc. units: Acetone

¥

3.96

Blank analysis date: $\frac{7-3}{6}$ 10 11 . Conc. units: $\frac{16}{6}$ 76.	Q+ . # 0		Associated Samples:	SI 11 01	
Compound	Blank ID	-		Sample identification	
giu .	01 A-2 12 pce - 38c 10	5-A 10 1	21		
Methylene chloride E	1. 53	1.9 /4 1.1	14 1.3/4		
Acetone					
CBO					

Blank analysis date: 9/25/10 11:26

3.66

¥

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

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

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2449
LDC#:

# VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Page: Reviewer:

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

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A" Was a method blank associated with every sample in this SDG? 

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

Was there contamination in the method blanks? If yes, please see the qualifications below.

6 R) 12-14 Associated Samples: Blank analysis date: 9

Conc. units:

792. 2.94

シタ Sample Identification  $\leq$ w 6 0.78. Associated Samples: 7 4 <u>ה</u> D. 60 /V ₹ 4 1.2 M N. 8.1 280-33216 969.0 Blank ID 0. 883 47 9/20 AD 0 3 MM 17 2 Compound Blank analysis date:\_\_ Methylene chloride Conc. units: Aeetono 7010

Sample Identification 0.79 116 266 - 37890-Blank 1D 399 M Methylene chloride Acetone

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

ᇛ

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

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## VALIDATION FINDINGS WORKSHEET Field Blanks

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Page:	Reviewer:	2nd Reviewer:

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

Were field blanks identified in this SDG?	Were target compounds detected in the field blank	: "9 /L Associated sample units: 45 / kg
Y N/N/A	YN N/A	Blank units:

9 hz/m Sampling date:\_

ę La

ρί

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

(94)

ó 4 26 (2) Sample Identification Associated Samples: 0.69 7 1.0/11 2 0,99 2.0 Blank ID ų N 0.79 Compound Methylene chloride Chloroform Acetene -

Sampling date: Field Blank / Rinsate / Trip Blank / Other. Associated sample units: Blank units:

as above Same

Associated Samples:

(79)

						•		
Compound	Blank ID				St	Sample Identification	tỉon	
	-	h	71	(3	14	5	91	
Metbylene-chloride +	2.5	(kg)	(210)	<u>(£)</u>	5.8/4	(S)	(33)	
Acetone	0.79	グニー	8,1	h/z1	1,2/4	1.3/4	10.78/4	
Chloroform			)	,		-		
		·						

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

LDC #: 24449 D)

## VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Page: Reviewer: 2nd Reviewer:

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

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?

				MS (m) U (n)	[3	MSD (1 imits)	Į.	<u>a</u>	RPD (Limits)	Associated Samples	ples	Qualifications	suoi
#	211			/ek (Lilli	(CI)		1		( T. " ')	7		Nin OAC P	MS/MSB 9R
¥	17/1	118	RPD	tor 101		compounds.	1 X X	ન	1				
				<b>)</b>		<u> </u>							
				)		)			<u></u>				
				)	( ·		^	_	( )				-+
	19	02/	RPD A	for several		donno annelo	なる	かれずる	Minits )	8		<b>+</b>	<del></del>
						<b>,</b>			7				
					^	)	<u> </u>		( )				
				-		_	(		( )				
					-		^		( )				
				-	_		^		( )				
							^		( )				
					1	_	(		( )				
							(		( )				
					^		^		( )				
						-	^		( )				
					^		^		( )			-	
							^		( )				
									( )				
			Compound	đ		QC Limits (Soil)	(Soil)		RPD (Soil)		QC Limits (Water)		RPD (Water)
	I	1.1-Dichloroethene	Ð			59-172%	%		< 22%		61-145%		< 14%
	, o	Trichloroethene				62-137%	%		< 24%		71-120%		< 14%
	>	Benzene				66-142%	%		< 21%		76-127%		< 11%
	     	Toluene				59-139%	%		< 21%		76-125%		< 13%
		Chlorohenzene				60-133%	%:		< 21%		75-130%		< 13%

7107 2. D but spiked ¥.

LDC#: 29449 D/

## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Page: Reviewer 2nd Reviewer:

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

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

4/5

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

	-2				<del>-</del>			<del></del>			- 1		_											
Qualifications	N.O																							
Associated Samples	1-9 MB 280-32921/5.A																							
		)		7	_	_	^	1	_	_	)	^	^	^	^	^	) [	^	(	^	^	^	(	^
RPD (Limits)	)	)	)	)	)	•	)	)	)	)	)	)	)	)	)	)	)	)	)	)	~	)	)	)
			^	`	)	~	_	7	1	^	`	)	_	(	_	_	(	~	(	(	^	^	^	`
LCSD %R (Limits)	~	)	)	)	)	)	<b>)</b>	)	)	•	)	)	)	) `		)	)	)	)	)	•	)	)	)
LCS %R (Limits)	(2) (71-130)	)	( )	( )	( )	( )	( )		( )		( )	( )		( )	(	( )	( )	( )	( )	( )	( )	( )	( )	( )
Compound	A VV																							
TCS/LCSD ID	LCS 16 280-32921 / 2-1A																						,	
Date																								
*																								

### LDC#:24449D1

### VALIDATION FINDINGS WORKSHEET Field Duplicates

Page:_	<u>lof_/</u>
Reviewer:	
2nd Reviewer:_	
	4

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

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

	Concentrat	ion (ug/Kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Compound	3	4	RPD	Difference	Limits	(Parent Only)
Chloroform	0.44	0.34		0.10	(≤6.4)	
Methylene chloride	0.99	1.0	_	0.01	(≤3.2)	

	Concentrati	ion (ug/Kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Compound	214	416	RPD	Difference	Limits	(Parent Only)
1,1-Dichloroethene	0.55	2.8U		2.25	(<2.8)	
2-Butanone	13 <b>U</b>	3.2		9.80	(<13)	
Acetone	5.0	16		11.00	(s 13)	
Methylene chloride	1.2	0.78		0.42	(~3,1)	
Naphthalene	7.5	3.9	·	3.60	(<3.1)	Jdets/A (fd)

V:\FIELD DUPLICATES\24449D1.wpd

LDC#. 28449 D)

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

₽ Page: Reviewer: 2nd Reviewer:

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

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

 $RRF = (A_x)(C_{is})/(A_{is})(C_x)$ 

average RRF = sum of the RRFs/number of standards

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

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

%RSD = 100 \* (S/X)

S= Standard deviation of the RRFs

Standard ID	Calibration Date	Compound (IS)	RRF (RRF 50 std)	RRF (RRF 50 std)	Average RRF (Initial)	Average RRF (Initial)	%RSD	RSD %RSD
ICAL	8/31/2010 Acetone	Acetone (IS1)	1) 0.0551	0.0551	0.0538	0.0538	6.9	6.84
GC MSV J		Chlorobenzene (IS2)	2) 2.8714	2.8714	2.8329	2.8329	4.0	4.00
		1,1,2,2-TCA (IS3)	3) 1.0214	1.0214	1.0017	1.0017	3.3	3.34
	<u>                                     </u>							
							•	
	ı							

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#

50/200     553331     2512       50/50     1798867     626       50/50     1086352     1063	Conc IS/Cpd	Area cpd	Area IS
1798867 1 1086352 1	50/200	553331	2512
1086352	50/50	1798867	929
	50/50	1086352	1063

331

Conc	Acetone	Chlorobenzene	1,1,2,2-TCA
N		3.0150	1.0270
20	0.0600	2.8949	1.0063
10	0.0539	2.7978	0.9683
20	0.0492	2.8109	0.9462
20	0.0551	2.8714	1.0214
100	0.0533	2.7961	1.0029
200	0.0513	2.6444	1.0401
×	0.0538	2.8329	1.0017
S	0.0037	0.1133	0.0335

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# 74449 D/

# VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: Yof Y Reviewer: JVG

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:

 $RRF = (A_x)(C_{is})/(A_{is})(C_x)$ 

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

					Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
		Calibration			RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
#	Standard ID	Date	Compound (IS)	(1S)	(RRF 10 std)	(RRF 10 std)	(Initial)	(Initial)		
	ICAL	9/29/2010 NR		(181)						
2	GC MSV P		NR (1	(1S2)						
3			Naphthalene (I	(153)	1.2733	1.2733	1.3000	1.3000	7.1	7.10
4		,								
သ		-								
9										

	ľ	_	_	Γ	ı
Area IS			376987		
Area cpd			384026		
Conc IS/Cpd	12.5/10	12.5/10	12.5/10		

Naphthalene	1.3763	1.1461	1.2594	1.2733	1.3633	1.3817	1.3000	0.0922
Chlorobenzene							#DIV/0!	#DIV/0i
Acetone							i0/\\IQ#	#DIV/0!
Conc	_	7	S	0	9	99	×	S

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.

## Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

. Page: 1 of 1

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

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

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

Where:

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF

RRF = (Ax)(Cis)/(Ais)(Cx)

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

Ax = Area of compound

Ais = Area of associated internal standard Cis = Concentration of internal standard Cx = Concentration of compound,

						Reported	Recalculated	Reported	Recalculated
		Calibration			Average RRF	RRF	RRF	۵%	WD
#	Standard ID	Date	Compound	(IS)	(Initial)	(CCV)	(ccv)		
-	J1225	9/24/2010	Acetone	(1S1)	0.054	0.046	0.046	13.9	13.9
	GC MSV J		Chlorobenzene	(IS2)	2.833	2.689	2.689	5.1	5.1
			1,1,2,2-TCA	(183)	1.002	0.928	0.928	7.3	7.3
2	J1258	9/25/2010 Acetone		(1S1)	0.054	0.050	0.050	7.2	7.2
	GC MSV J		ည	(182)	2.833	2.970	2.970	4.9	4.9
			1,1,2,2-TCA	(183)	1.002	1.050	1.050	4.9	4.9
က	P1404	9/29/2010 Naphtha	Naphthalene	(183)	1.3000	1.2727	1.2727	2.1	2.1
	MSP								

		CCV1		CCV2		CCV3	
Compound	Cis/Cx	Ax	Ais	Ax	Ais	Ax	Ais
Acetone	50/200	452929	2445776	467969	2342985	425368	417786
Chlorobenzene	50/50	1674423	622768	1758812	592094		
1,1,2,2-TCA	20/20	953941	1027682	1069360	1018037		

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

Page:_	lof
Reviewer:	JVG
2nd reviewer:	0-
	f

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

Sample ID: + 3

SS = Surrogate Spiked

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8	50	52.2	104	104	a
Bromofluorobenzene		46. 5	93	93	
1,2-Dichloroethane-d4		48.4	97	17	
Dibromofluoromethane	<u> </u>	55.7	l I I	110	1

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		<u></u>		<u> </u>	

LDC#: >4449 D/ SDG#: SQ (?~

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

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

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

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

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

Where:

SC = Sample concentration

SSC = Spiked sample concentration SA = Spike added

MSDC = Matrix spike duplicate concentration

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

MSC = Matrix spike concentration

17 /18 MS/MSD sample:

	Š	ike	Sample	Spiked Sample	ample	Matrix Spike	Spike	Matrix Spike Duplicate	Duplicate	MS/MSD	ASD
Compound	(7. <b>A</b>	Added (UG // )	Concentration (MG / K)	Concentration	ration	Percent Recovery	ecovery	Percent Recovery	scovery	RPD	٠,
	NS W	MSD	Q	MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	* Recalculate
1,1-Dichloroethene	52.7	2.9%	Q.	48.8	31.9	93	63	د>	28	44	9
Trichloroethene		ĺ		42.1	8-62	68	58	<i>۲</i> %	1 × 8	45	8
Benzene				49.4	21.7	16	λb	28	68	XX	8
Toluene				48.7	20.7	٨١	16	43	62	4	6
Chlorobenzene		ۍـــــــــــــــــــــــــــــــــــــ		ج 'اع	33.7	66	6	88	D8	46	q

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#: 2464 1/

# VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Results Verification

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

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

% Recovery = 100 \* SSC/SA W

Where: SSC = Spiked sample concentration SA = Spike added

RPD = ILCSC - LCSDC I \* 2/(LCSC + LCSDC)

LCS ID: US\$ 280- 3294 /1,2-A (9/24)

	Š	oike	Spiked	Sample	SUI	S	I CSD	Q.	l CS/	CS/I CSD
	Ϋ́,	Added	Concentration	itration			ı			
Compound	3	/1 /	( <mark>1</mark> /2)		Percent Recovery	ecovery	Percent Recovery	есоуегу	22	RPD
	SDI	1 GSD	1.05	LGSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
1,1-Dichloroethene	S, S	50.0	1.85	58.1	116	911	116	116	<u>و</u>	a
Trichloroethene			53.5	15.K	16.7	(87	8.4/	X0/		
Benzene			5.5.7	54.1	167	(67	801	108	>	7
Toluene			52.7	53.7	105	105	167	107	>	>
Chlorobenzene		<u>→</u>	53.6	54.4	(87	401	109	109	1	_
				,		//				
						,				
										:
	•									

Comments: Refer to Laboratory Control Sample findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results. LDC#: 24449 D7

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	of ]
Reviewer:	JVG
2nd reviewer:	•/
	Γ

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

Y N N/A Y N N/A Were all reported results recalculated and verified for all level IV samples?

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

Concentration =  $(A_{\bullet})(I_{\bullet})(DF)$ 

(A<sub>b</sub>)(RRF)(V<sub>o</sub>)(%S)

A<sub>x</sub> = Area of the characteristic ion (EICP) for the compound to be measured

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

I<sub>s</sub> = Amount of internal standard added in nanograms (ng)

RRF = Relative response factor of the calibration standard.

V<sub>o</sub> = Volume or weight of sample pruged in milliliters (ml) or grams (g).

Df = Dilution factor.

%S = Percent solids, applicable to soils and solid matrices

Example:

Sample I.D ± 14 F Manage

Conc. = (20560) (50) (5m) (2411490) (0.054)(8.1238) (0.984)

= 4.94

~ 5.0 ns/kg

	only.		Reported Concentration	Calculated Concentration	
#	Sample ID	Compound	()	( )	Qualification
<b> </b>					
		<u> </u>			
ļ					
<u> </u>				<u> </u>	<del> </del>
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					· · · · · · · · · · · · · · · · · · ·
		·			

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 9, 2010

LDC Report Date:

December 8, 2010

Matrix:

Soil/Water

Parameters:

Semivolatiles

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

SSAJ3-05-12BPCMSD

Sample Delivery Group (SDG): 280-6290-1

## Sample Identification

SSAQ3-03-10BPC SB03-24BPC SSAQ3-03-1BPC EB-08092010 SSAQ3-03-5BPC EB-08102010 SSAQ4-08-10BPC\*\* SSAJ3-05-12BPCMS

SSAQ4-08-10BPC\_FD

SSAQ4-08-1BPC SSAQ4-08-5BPC

SSAJ2-06-1BPC

SSAJ2-06-3BPC

SSAJ2-06-5BPC

SSAJ3-05-12BPC

SSAJ3-05-16BPC\*\*

SSAJ3-05-1BPC

SSAJ3-05-5BPC

SSAJ3-05-8BPC

SSAJ3-07-12BPC

SSAJ3-07-17BPC

SSAJ3-07-1BPC

SSAJ3-07-5BPC

SSAJ3-07-8BPC

<sup>\*\*</sup>Indicates sample underwent Stage 4 review

## Introduction

This data review covers 23 soil samples and 2 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 the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008).

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.

Samples indicated by a double asterisk on the front cover underwent a Stage 4 review. A Stage 2B review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Stage 2B criteria since this review is based on QC data.

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- 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 30.0% for all compounds.

In the case where the laboratory used a calibration curve to evaluate the compounds, all coefficients of determination (r<sup>2</sup>) were greater than or equal to 0.990.

Average relative response factors (RRF) for all compounds were within method and validation criteria.

## 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) and 25.0% for all other compounds.

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

All of the continuing calibration relative response factors (RRF) 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
MB 280 -27100/1-A	8/16/10	Dimethylphthalate	44.9 ug/Kg	SSAQ3-03-10BPC SSAQ3-03-1BPC SSAQ3-03-1BPC SSAQ4-08-10BPC** SSAQ4-08-10BPC_FD SSAQ4-08-1BPC SSAJ2-06-1BPC SSAJ2-06-1BPC SSAJ2-06-5BPC SSAJ3-05-12BPC SSAJ3-05-12BPC SSAJ3-05-16BPC** SSAJ3-05-16BPC** SSAJ3-07-12BPC SSAJ3-07-12BPCMS SSAJ3-05-12BPCMSD

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	Compound TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
SSAQ3-03-10BPC	Dimethylphthalate	88 ug/Kg	88U ug/Kg
SSAQ4-08-10BPC**	Dimethylphthalate	56 ug/Kg	56U ug/Kg
SSAQ4-08-10BPC_FD	Dimethylphthalate	36 ug/Kg	36U ug/Kg
SSAQ4-08-1BPC	Dimethylphthalate	50 ug/Kg	50U ug/Kg
SSAQ4-08-5BPC	Dimethylphthalate	47 ug/Kg	47U ug/Kg
SSAJ2-06-1BPC	Dimethylphthalate	30 ug/Kg	30U ug/Kg
SSAJ2-06-3BPC	Dimethylphthalate	33 ug/Kg	33U ug/Kg
SSAJ2-06-5BPC	Dimethylphthalate	45 ug/Kg	45U ug/Kg
SSAJ3-05-12BPC	Dimethylphthalate	43 ug/Kg	43U ug/Kg
SSAJ3-05-16BPC**	Dimethylphthalate	81 ug/Kg	81U ug/Kg
SSAJ3-05-1BPC	Dimethylphthalate	64 ug/Kg	64U ug/Kg

Sample	Compound TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
SSAJ3-05-5BPC	Dimethylphthalate	64 ug/Kg	64U ug/Kg
SSAJ3-07-12BPC	Dimethylphthalate	81 ug/Kg	81U ug/Kg
SSAJ3-07-17BPC	Dimethylphthalate	81 ug/Kg	81U ug/Kg
SSAJ3-07-1BPC	Dimethylphthalate	67 ug/Kg	67U ug/Kg
SSAJ3-07-5BPC	Dimethylphthalate	70 ug/Kg	70U ug/Kg
SSAJ3-07-8BPC	Dimethylphthalate	63 ug/Kg	63U ug/Kg

Samples EB-08092010 and EB-08102010 were identified as equipment blanks. No semivolatile contaminants were found in these blanks.

## VI. Surrogate Spikes

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

## 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) ) and relative percent differences (RPD) were within QC limits.

## IX. Regional Quality Assurance and Quality Control

Not applicable.

## X. Internal Standards

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

## XI. Target Compound Identifications

All target compound identifications were within validation criteria for samples on which a Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

## XII. Project Quantitation Limit

All compound quantitation and CRQLs were within validation criteria for samples on which a Stage 4 review was performed with the following exceptions:

Sample	Compound	Finding	Flag	A or P
SSAQ3-03-5BPC SSAQ4-08-1BPC SSAQ4-08-5BPC	Benzo(b)fluoranthene Benzo(k)fluoranthene	Due to lack of resolution between these compounds in the samples, the laboratory performed the quantitation using the total peak area.	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р

The reported results for the compounds listed above are biased high. The actual values of these compounds are lower than the values reported by the laboratory.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6290-1	All compounds reported below the PQL.	J (all detects)	А

Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

## XIII. Tentatively Identified Compounds (TICs)

Tentatively identified compounds were not reported by the laboratory.

## XIV. System Performance

The system performance was acceptable for samples on which a Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

## XV. Overall Assessment

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

## XVI. Field Duplicates

Samples SSAQ4-08-10BPC\*\* and SSAQ4-08-10BPC\_FD were identified as field duplicates. No semivolatiles were detected in any of the samples with the following exceptions:

	Concentra	tion (ug/Kg)					
Compound	SSAQ4-08-10BPC**	SSAQ4-08-10BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P	
Dimethylphthalate	56	36	-	20 (≤350)	-	-	
Hexachlorobenzene	450	470	-	20 (≤350)	-	-	
Octachlorostyrene	140	160	-	.20 (≤350)	-	-	

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-6290-1

SDG 280-6290-1	Sample SSAQ3-03-5BPC	Compound Benzo(b)fluoranthene	Flag J (all detects)	A or P	Reason (Code) Project Quantitation Limit
	SSAQ4-08-1BPC SSAQ4-08-5BPC	Benzo(k)fluoranthene	UJ (all non-detects) J (all detects) UJ (all non-detects)		(q)
280-6290-1	SSAQ3-03-10BPC SSAQ3-03-1BPC SSAQ3-03-5BPC SSAQ4-08-10BPC** SSAQ4-08-10BPC_FD SSAQ4-08-18PC SSAQ4-08-5BPC SSAJ2-06-18PC SSAJ2-06-5BPC SSAJ3-05-12BPC SSAJ3-05-12BPC SSAJ3-05-16BPC** SSAJ3-05-16BPC** SSAJ3-05-18PC SSAJ3-07-17BPC SSAJ3-07-18PC SSAJ3-07-18PC SSAJ3-07-18PC SSAJ3-07-5BPC SSAJ3-07-5BPC SSAJ3-07-8BPC SSAJ3-07-8BPC SSAJ3-07-8BPC EB-08092010 EB-08102010	All compounds reported below the PQL.	J (all detects)	Α	Project Quantitation Limit (sp)

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-6290-1

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
280-6290-1	SSAQ3-03-10BPC	Dimethylphthalate	88U ug/Kg	А	bl
280-6290-1	SSAQ4-08-10BPC**	Dimethylphthalate	phthalate 56U ug/Kg		bl
280-6290-1	SSAQ4-08-10BPC_FD	Dimethylphthalate	36U ug/Kg	А	bl
280-6290-1	SSAQ4-08-1BPC	Dimethylphthalate	50U ug/Kg	А	ld
280-6290-1	SSAQ4-08-5BPC	Dimethylphthalate	47U ug/Kg	Α	bl
280-6290-1	SSAJ2-06-1BPC	Dimethylphthalate	30U ug/Kg	А	bl
280-6290-1	SSAJ2-06-3BPC	Dimethylphthalate	33U ug/Kg	А	bl
280-6290-1	SSAJ2-06-5BPC	Dimethylphthalate	45U ug/Kg	. A	bl

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
280-6290-1	SSAJ3-05-12BPC	Dimethylphthalate	43U ug/Kg	А	bl
280-6290-1	SSAJ3-05-16BPC**	Dimethylphthalate	81U ug/Kg	Α	bl
280-6290-1	SSAJ3-05-1BPC	Dimethylphthalate	64U ug/Kg	А	bl
280-6290-1	SSAJ3-05-5BPC	Dimethylphthalate	64U ug/Kg	Α _	lď
280-6290-1	SSAJ3-07-12BPC	Dimethylphthalate	81U ug/Kg	А	bl
280-6290-1	SSAJ3-07-17BPC	Dimethylphthalate	81U ug/Kg	А	þí
280-6290-1	SSAJ3-07-1BPC	Dimethylphthalate	67U ug/Kg	Α	þl
280-6290-1	SSAJ3-07-5BPC	Dimethylphthalate	70U ug/Kg	Α	bl
280-6290-1	SSAJ3-07-8BPC	Dimethylphthalate	63U ug/Kg	Α	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Equipment Blank Data Qualification Summary - SDG 280-6290-1

No Sample Data Qualified in this SDG

## **Tronox Northgate Henderson**

## **VALIDATION COMPLETENESS WORKSHEET**

280-6290-1 SDG #: Laboratory: Test America

24449A2a

LDC #:

Stage 2B/4

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

2nd Reviewer:

Page:\_\_

Reviewer:

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

	Validation Area		Comments
l.	Technical holding times	Ă	Sampling dates: 8/09 /p
II.	GC/MS Instrument performance check	A	/
III.	Initial calibration	A	6/6 RSD 12
IV.	Continuing calibration/ICV	A	CW/W E252
V.	Blanks	SW	
VI.	Surrogate spikes	JVCSW A	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	LCS 10
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	A	Not reviewed for Stage 2B validation.
XII.	Compound quantitation/CRQLs	SM)	Not reviewed for Stage 2B validation.
XIII.	Tentatively identified compounds (TICs)	N	Not reviewed for Stage 2B validation.
XIV.	System performance	Ą	Not reviewed for Stage 2B validation.
XV.	Overall assessment of data	4	
XVI.	Field duplicates	MS	D = 4,5
XVII.	Field blanks	NB	S8-21 EB = 22, 23

A = Acceptable Note:

N = Not provided/applicable

SW = See worksheet

R = Rinsate FB = Field blank D = Duplicate

TB = Trip blank

EB = Equipment blank

Validated Samples:

\*\* Indicates sample underwent Stage 4 validation Water

ND = No compounds detected

,	7011	<u>,                                     </u>	rvat et					
+ 1	SSAQ3-03-10BPC S	11	SSAJ3-05-12BPC	21	SB03-24BPC	ک	<b>3</b> 1 ]	MB 280 - 27100/1-A
2	SSAQ3-03-1BPC	12	SSAJ3-05-16BPC**	22 }	EB-08092010	W	32	MB 780 - 27168/1-A
<i>⊁</i> 3	SSAQ3-03-5BPC	13	SSAJ3-05-1BPC	23	EB-08102010		3 33	MB 280- 26860/1-A
<u>+</u> 4	SSAQ4-08-10BPC** /	14	SSAJ3-05-5BPC	24	SSAJ3-05-12BPCMS	5	34	/
5	SSAQ4-08-10BPC_FD D	_ 15	SSAJ3-05-8BPC	25	SSAJ3-05-12BPCMSD	$\bot$	35	
6	SSAQ4-08-1BPC	16	SSAJ3-07-12BPC	26			36	
7	SSAQ4-08-5BPC	17	SSAJ3-07-17BPC	27			37	
8	SSAJ2-06-1BPC	18	SSAJ3-07-1BPC	28			38	
9	SSAJ2-06-3BPC	19	SSAJ3-07-5BPC	29			39	
10	SSAJ2-06-5BPC V	20	SSAJ3-07-8BPC	30			40	

LDC #: 2444 Aza

## VALIDATION FINDINGS CHECKLIST

Page: \ of \ \( \frac{2}{VC} \)
Reviewer: \ \ \( \frac{1}{VC} \)

Method: Semivolatiles (EPA SW 846 Method 8270C)

Validation Area	Yes	No	NA	Findings/Comments
Il Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.				
III. GC/MS Instrument performance check				
Were the DFTPP performance results reviewed and found to be within the specified criteria?	_	_		
Were all samples analyzed within the 12 hour clock criteria?				
ill. Initial calibration				ANTE CALCULATION CONTRACTOR CONTR
Did the laboratory perform a 5 point calibration prior to sample analysis?	_			
Were all percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?	_			
Was a curve fit used for evaluation?	_			
Did the initial calibration meet the curve fit acceptance criteria of ≥ 0.990?				
Were all percent relative standard deviations (%RSD) $\leq$ 30% and relative response factors (RRF) $\geq$ 0.05?				
IV. Continuing calibration.				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?		1		
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?	,			
M. Blanks				
Was a method blank associated with every sample in this SDG?	/			
Was a method blank analyzed for each matrix and concentration?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/.		
VI. Surrogate spikes				
Were all surrogate %R within 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?				
VII. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.				
Was a MS/MSD analyzed every 20 samples of each matrix?				
Were the MS/MSD percent recoveries (%R) and the relative percent differences RPD) within the QC limits?				
/III. Laboratory control samples	40	(4) ji ji	30049 344	Bray Atagan Andrews
Was an LCS analyzed for this SDG?				

## VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
Reviewer: 100
2nd Reviewer: 1

	T		1	
Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?	<del>                                     </del>			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		<u> </u>		
IX. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?	<u> </u>		L	
Were the performance evaluation (PE) samples within the acceptance limits?	<u> </u>			
X: Internal standards				
Were internal standard area counts within -50% or +100% of the associated calibration standard?				
Were retention times within ± 30 seconds from the associated calibration standard?				
XI: Target compound: identification				and the fact of the second
Were relative retention times (RRT's) within ± 0.06 RRT units of the standard?				
Did compound spectra meet specified EPA "Functional Guidelines" criteria?				
Were chromatogram peaks verified and accounted for?				
XII. Compound quantilation/CRQLs				Application of a constraint, security is seen a
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XIII. Tentatively identified compounds (ITICs)	-6.6			
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?				
. Were relative intensities of the major ions within $\pm$ 20% between the sample and the reference spectra?				
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?				
XIV. System performance				
System performance was found to be acceptable.			T	
XV: Overall assessment of data : 10 - 10 - 10 - 10 - 10 - 10 - 10 - 10		ed e		
Overall assessment of data was found to be acceptable.	7			
XVI:(Field, duplicates				
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.				
XVII. Field blanks				
Field blanks were identified in this SDG.	/	X1502 V		. 12 см. на много технопионалисти по из 1996 година предоставления (1994—1994 година 1995—1997—1995 година (19 Статими по пределения по предоставления по предоставления по предоставления по предоставления по предоставления
Target compounds were detected in the field blanks.				

## VALIDATION FINDINGS WORKSHEET

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

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	ODO. 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-Chiorophenyi-phenyi ether	BBB, 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC, Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	TTT.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	UUU
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)/iluoranthene	WW.
0. 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.

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## VALIDATION FINDINGS WORKSHEET

Blanks

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

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

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

Y N/A Was a method blank analyzed for each concentration preparation level? Y N/A Was a method blank associated with every sample?

Was the blank contaminated? If yes, please see qualification below.

All S except 21

(88)

Compound	Blank ID				Š	Sample Identification	tion			
	A-700122-026	K-A 1	4	د	9	7	8	6	Q/	41
)) **	44.9	n/ 88	56 /u	N 28	n/ 05	N 44	My oc	33/4	45 M	43/4
									-	
Blank extraction date:	Blank analysis date:	ysis date:	Sam	Same as above	No me	.1				
Conc. units:			Associa	Associated Samples:				くず)		

Compound	Blank ID				S	Sample Identification	ation			
grip .	46 280- 27100 / -A	-4 12	13	14	31	17	18	- 1	20	
æ	44.9	b/ 18	64 /4	64 /4	n/18	81, 14	107 h	10 /4 63/4	63/u	
	-	`								
								_		

## LDC# 2444 AZA

# VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

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

Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation? Pease see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N/A. Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound? A'N'A

Qualifications	J/45/0 (4)										
Associated Samples	phed	ara,									
Finding	666, ### pe~Ks unres	tab used total peak area	to quantitution								
Sample ID	367								-		
Date											
#											

Comments: See sample calculation verification worksheet for recalculations

LDC#:24449A2a

## VALIDATION FINDINGS WORKSHEET Field Duplicates

Page:	
Reviewer: 2nd Reviewer:	_JVG_
Zilu Keviewei	₩

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

YN NA YN NA

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

	Concentrati	on (ug/Kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Compound	4	5	RPD	Difference	Limits	(Parent Only)
Dimethyl phthalate	56	36		20	(<350)	
Hexachlorobenzene	450	470		20	(350)	
Octachlorostyrene	140	160		20	(<350)	

V:\FIELD DUPLICATES\24449A2a.wpd

LDC# 2444 A A 20

# VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: 1 of 1 Reviewer: JVG 2nd Reviewer: 2

METHOD: GC/MS SVOA (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:

 $RRF = (A_x)(C_{is})/(A_{is})(C_x)$ 

average RRF = sum of the RRFs/number of standards

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

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

%RSD = 100 \* (S/X)

#

		Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
Calibration		RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
Standard ID Date Compound (Internal Standard)	$\overline{}$	 ( 50 std)	( 50 std)	(Initial)	(Initial)		
7/21/2010 1,4-Dioxane (IS1)		0.5607	0.5607	0.5706	0.5707	4.1	4.13
Naphthalene (1S2)		1.0611	1.0611	1.0093	1.0093	5.7	5.70
Fluorene (IS3)		1.3101	1.3101	1.2473	1.2473	5.3	5.25
Hexachlorobenzene (IS4)	ene	0.2418	0.2418	0.2300	0.2300	3.8	3.81
Chrysene (1S5)		1.1089	1.1089	1.0581	1.0581	6.7	6.75
Benzo(a)pyrene (IS6)		1.1425	1.1425	1.0793	1.0794	8.5	8.53

Area IS	160417	616036	358588	534527	565669	542046
Area cpd	112429	817090	587234	161541	784054	774079
nc IS/Cpd	40/50	40/20	40/20	40/20	40/20	40/50

Conc	1,4-Dioxane	Naphthalene	Fluorene	Hexachloro	Chrysene	Benzo(a)py
4.00	0.6209	1.0632	1.2493		1.1443	0.8934
10.00	0.5673	1.0390	1.2573	0.2339	1.1045	0.9948
20.00	0.5842	1.0490	1.3209	0.2330	1.1007	1.0754
50.00	0.5607	1.0611	1.3101	0.2418	1.1089	1.1425
80.00	0.5523	1.0236	1.2953	0.2310	1.0810	1.1683
120.00	0.5455	0.9799	1.2298	0.2266	0.9887	1.1297
160.00	0.5731	0.9540	1.1898	0.2305	0.9795	1.1318
200.00	0.5612	0.9045	1.1261	0.2131	0.9573	1.0989
# ×	0.5707	1.0093	1.2473	0.2300	1.0581	1.0794
S	0.0236	0.0575	0.0655	0.0088	0.0714	0.0920
_						

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 WORSHEET Continuing Calibration Results Verification

Page of Reviewer: JVG 2nd Reviewer:

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

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

Where:

ave. RRF = initial calibration average RRF

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF

RRF = (Ax)(Cis)/(Ais)(Cx)

RRF = continuing calibration RRF

Ax = Area of compound Ai

Ais = Area of associated internal standard

Cx = Concentration of compound Cis = Concentration of internal standard

		Calibration			Average RRF	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Date	Compound (Reference IS)	(S)	(Initial RRF)	(CC RRF)	(CC RRF)	Q%	Q%
-	K5828	08/23/10	1,4-Dioxane	(1S1)	0.5706	0.5422	0.5422	5.0	5.0
			Naphthalene	(182)	1.0093	1.1210	1.1210	11.1	11.1
			Fluorene	(183)	1.2473	1.4290	1.4290	14.6	14.6
			Hexachlorobenzene	(1S4)	0.2300	0.2640	0.2640	14.8	14.8
			Chrysene	(32)	1.0581	1.0684	1.0684	1.0	1.0
			Benzo(a)pyrene	(186)	1.0793	1.2357	1.2357	14.5	14.5
2	K5872	08/24/10	1,4-Dioxane.	(IS1)	0.5706	0.5200	0.5200	8.9	8.9
			Naphthalene	(IS2)	1.0093	1.1283	1.1283	11.8	11.8
	:		Fluorene	(183)	1.2473	1.4081	1.4081	12.9	12.9
			Hexachlorobenzene	(IS4)	0.2300	0.2632	0.2632	14.5	14.5
			Chrysene	(185)	1.0581	1.0891	1.0891	2.9	2.9
			Benzo(a)pyrene	(186)	1.0793	1.2311	1.2311	14.1	14.1

		-	CCV1		CCV2	
Compound (Reference IS)	IS)	Concentration	Area Cpd	Area IS	Area Cpd	Area IS
		(IS/Cpd)				
1,4-Dioxane	(IS1)	40/80	206796	190700	195412	187881
Naphthalene	(IS2)	40/80	1621366	723208	1580575	700454
Fluorene	(1S3)	40/80	1195169	418177	1158634	411418
Hexachlorobenzene	(IS4)	40/80	372526	705597	353417	671295
Chrysene	(IS5)	40/80	1824855	854050	1707482	783883
Benzo(a)pyrene	(156)	40/80	1845845	746891	1671727	678968

LDC#: 24 849 A 26

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

Page:_	<u>lof_1</u>
Reviewer:	JVG
2nd reviewer:	- 0,
_	

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: # 4

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	180	576	58	28	O
2-Fluorobiphenyl		71.4	71	71	
Terphenyl-d14		94.0	94	94	
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			11.7		
Terphenyl-d14					
Phenol-d5					
2-Fluorophenoi					
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#: 214 49 A 22 SDG #: See Cores

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

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

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

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

Where:

SC = Sample concentation

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

SSC = Spiked sample concentration SA = Spike added

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

RPD = I MSC - MSC I \* 2/(MSC + MSDC) MS/MSD samples:

Phenol N-Nitroso-di-n-propylamine Acenaphthene Acenaphthene	·-·	Sample	Spiked (	Sample	Matrix Spike	Spike	Matrix Spike Duplicate	Duplicate	MS/WSD	GS.
so-di-n-propylamine o-3-methylphenol bithene 26.8.0		Concentration (145, /c.)	Concentration ( NS / K-1)	tration	Percent Recovery	есочегу	Percent Recovery	всоуегу	RPD	0
so-di-n-propylamine o-3-methylphenol 26.8.0	MSD	0	MS	0 MSD	Reported	Recalc	Renorted	Recalc	Renorted	Recalculated
2680										
2680										
2680										
Oeniachiorophenol	2690	0	2170	2050	8/	\$)	7.6	76	7	Q
Pyrene   24 60   74	2690		2400	22.72	<b>68</b>	89	<b>48</b>	7∕8	7	23
						. —				

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# 2444 AVA

SDG #: Ste C-1

# VALIDATION FINDINGS WORKSHEET

Page: 1 of 1

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

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

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

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

% Recovery = 100 \* (SC/SA

Where: SSC = Spike concentration SA = Spike added

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

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

27100/2-A 780-Z LCS/LCSD samples:

		Recalculat ed				1						
CS/ICSD	RPD	Reci										
101		Reported										
csD	Зесочегу	Recalc.										
10	Percent	Percent Recovery	Reported							\		
SDI	Percent Recovery	Recalc.				8)		82			:	
	Percent	Percent	Reported				(۶		84			
Spike	Concentration $(u\varsigma, h\varsigma_\zeta)$	LCSD				NA		>				
ď	Concen (45)	SOT				2060		1990				
ike	Added ( 1/2 / / / )	rcsD				A.A.						
<i>\fotation</i>	A A	rcs				2540		7.4.				
	Compound		Phenol	N-Nitroso-di-n-propylamine	4-Chloro-3-methylphenol	Acenaphthene	Pentachlorophenol	Pyrene				

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#: 24449 Am

## VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	<u>l</u> of <u>1</u>
Reviewer:	W
2nd reviewer:	Q

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

$\langle Y \rangle$	N	N/A
$\bigvee$	X	N/A

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

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

Concentration =  $(A_{\bullet})(I_{\bullet})(V_{\bullet})(DF)(2.0)$  $(A_{i\bullet})(RRF)(V_{\circ})(V_{i})(%S)$ 

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, = Volume of the concentrated extract in microliters (ul)

Df = Dilution Factor.

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

Example:

Conc. = (5694cl)(40)(1ml)(0)(0)

\_ 449,7

~ 450 ns/kg

2.0	= Factor of 2 to accoun	nt for GPC cleanup			
#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
⊩—					
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## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 9 through August 10, 2010

LDC Report Date:

December 8, 2010

Matrix:

Soil/Water

Parameters:

Chlorinated Pesticides

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6290-1

## Sample Identification

SSAJ3-05-12BPC

SSAJ3-05-16BPC\*\*

SSAJ3-05-1BPC

SSAJ3-05-5BPC

SSAJ3-05-8BPC

SSAJ3-07-12BPC

SSAJ3-07-17BPC

SSAJ3-07-1BPC

SSAJ3-07-5BPC

SSAJ3-07-8BPC

SB03-24BPC

EB-08102010

SSAJ3-05-12BPCMS

SSAJ3-05-12BPCMSD

<sup>\*\*</sup>Indicates sample underwent Stage 4 review

## Introduction

This data review covers 13 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8081A for Chlorinated Pesticides.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008).

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.

Samples indicated by a double asterisk on the front cover underwent a Stage 4 review. A Stage 2B review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Stage 2B criteria since this review is based on QC data.

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. GC/ECD Instrument Performance Check

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

## III. Initial Calibration

Initial calibration of single compounds were 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 selected compounds.

A curve fit, based on the initial calibration, was established for quantitation for selected compounds. The coefficient of determination (r²) was greater than or equal to 0.990.

Retention time windows were evaluated and considered technically acceptable for samples on which a Stage 4 review was performed. Raw data were not evaluated for the samples on which a Stage 2B 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 20.0% QC limits for all compounds.

The percent difference (%D) of the second source calibration standard were less than or equal to 20.0% for all compounds.

Retention times (RT) of all compounds in the calibration standards were within QC limits for samples on which a Stage 4 review was performed. Raw data were not evaluated for the samples on which a Stage 2B review was performed.

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

## V. Blanks

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

Sample EB-08102010 was identified as an equipment blank. 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:

				T		<del></del> _
Sample	Column	Surrogate	%R (Limits)	Compound	Flag	AorP
SSAJ3-05-1BPC	Not specified	Decachlorobiphenyl	422 (63-124)	All TCL compounds except beta-BHC Hexachlorobenzene	J+ (all detects)	А
SSAJ3-05-8BPC	Not specified	Decachlorobiphenyl	226 (63-124)	All TCL compounds	J+ (all detects)	Р
SSAJ3-07-1BPC	Not specified	Decachlorobiphenyl	1450 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	А
SSAJ3-07-5BPC	Not specified	Decachlorobiphenyl	143 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	А
SSAJ3-07-5BPC (2X)	Not specified	Decachlorobiphenyl	160 (63-124)	Hexachiorobenzene	J+ (all detects)	А
SSAJ3-07-8BPC	Not specified	Decachlorobiphenyl	150 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	А
SSAJ3-07-8BPC (2X)	Not specified	Decachlorobiphenyl	158 (63-124)	Hexachlorobenzene	J+ (all detects)	А
SB03-24BPC	Not specified	Tetrachloro-m-xylene Decachlorobiphenyl	501 (59-115) 4170 (63-124)	All TCL compounds except beta-BHC Hexachlorobenzene 4,4'-DDD 4,4'-DDE 4,4'-DDT	J+ (all detects)	Α

## 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) and relative percent differences (RPD) 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 Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

## XII. Project Quantitation Limit

All compound quantitation and CRQLs were within validation criteria for samples on which an Stage 4 review was performed.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6290-1	All compounds reported below the PQL.	J (all detects)	А

Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

## XIII. Overall Assessment of Data

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

## XIV. Field Duplicates

No field duplicates were identified in this SDG.

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Data Qualification Summary - SDG 280-6290-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6290-1	SSAJ3-05-1BPC	All TCL compounds except beta-BHC Hexachlorobenzene	J+ (all detects)	A	Surrogate recovery (%R) (s)
280-6290-1	SSAJ3-05-8BPC	All TCL compounds	J+ (all detects)	P	Surrogate recovery (%R) (s)
280-6290-1	SSAJ3-07-1BPC SSAJ3-07-5BPC SSAJ3-07-8BPC	All TCL compounds except Hexachlorobenzene	J+ (all detects)	А	Surrogate recovery (%R) (s)
280-6290-1	SSAJ3-07-5BPC (2X) SSAJ3-07-8BPC (2X)	Hexachlorobenzene	J+ (all detects)	Α	Surrogate recovery (%R) (s)
280-6290-1	SB03-24BPC	All TCL compounds except beta-BHC Hexachlorobenzene 4,4'-DDD 4,4'-DDE 4,4'-DDT	J+ (all detects)	А	Surrogate recovery (%R) (s)
280-6290-1	SSAJ3-05-12BPC SSAJ3-05-16BPC** SSAJ3-05-1BPC SSAJ3-05-5BPC SSAJ3-07-12BPC SSAJ3-07-17BPC SSAJ3-07-17BPC SSAJ3-07-18PC SSAJ3-07-5BPC SSAJ3-07-8BPC SSAJ3-07-8BPC SB03-24BPC EB-08102010	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG 280-6290-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG 280-6290-1

No Sample Data Qualified in this SDG

## **Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET**

Stage 2B/4

DG #: <u>280-6290-1</u>	S
aboratory: Test America	

2nd Reviewer:

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

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

	Validation Area		Comments
l.	Technical holding times	4	Sampling dates: 8/9-10/10
II.	GC/ECD Instrument Performance Check	A	
111.	Initial calibration	LA	0/8RSD 52090 1 r=
IV.	Continuing calibration/ICV	A	5/884D = 209+ 1 r=
V.	Blanks	À	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A-	US/D
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 Stage 2B validation.
XII.	Compound quantitation and reported CRQLs	A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	_ A	·
XIV.	Field duplicates	Ν	
XV.	Field blanks	M	EB212

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

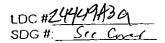
D = Duplicate

TB = Trip blank

EB = Equipment blank

Validated Samples: \*\* Indicates sample underwent Stage 4 validation

1	SSAJ3-05-12BPC -	11	SB03-24BPC	21	31	280-26986-BIKS
2	SSAJ3-05-16BPC**	12	EB-08102010 W	22	32	200-27027-BLA
3	SSAJ3-05-1BPC	13	SSAJ3-05-12BPCMS 5	23	33	280-27469-BLK
4	SSAJ3-05-5BPC	14	SSAJ3-05-12BPCMSD	24	34	
5	SSAJ3-05-8BPC	15		25	35	
6	SSAJ3-07-12BPC	16		26	36	
7	SSAJ3-07-17BPC	17		27	37	
8	SSAJ3-07-1BPC	18		28	38	
9	SSAJ3-07-5BPC	19		29	39	
10	SSAJ3-07-8BPC V	20		30	40	



## VALIDATION FINDINGS CHECKLIST

Page: \_\_| 01 2 Reviewer: \_\_| 2 2nd Reviewer: \_\_|

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

Method: Pesticides/PCBs (EPA SW 846 Method 8081/808	4) T	<del>i</del>	<del></del>	<u> </u>				
Validation Area	Yes	No	NA		Find	ings/Con	iments	
i Technical holding times	njah <u>Diwiz</u>		: : :					<del>.</del>
All technical holding times were met.						<del></del>		···
Cooler temperature criteria was met.								<del></del>
II. GC/ECD Instrument performance check								
Was the instrument performance found to be acceptable?		L				y .	<del> </del>	
III. Inktat calibration	((é.:. T		T	<u></u>	* *	<u> </u>		<u> </u>
Did the laboratory perform a 5 point calibration prior to sample analysis?			<u> </u>		<del></del>			
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations $(\%RSD) \leq 20\%$ ?								
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?								
Did the initial calibration meet the curve fit acceptance criteria?				-		·	·	
Were the RT windows properly established?				: 				
Were the required standard concentrations analyzed in the initial calibration?		e Casa Milia	la čast	2585818161	118900000		5 - 40 Jan	NATE OF
IV. Continuing calibration								
What type of continuing calibration calculation was performed? %D or %R								
Were Evaluation mix standards analyzed prior to the initial calibration and sample analysis?		)			<del></del>	<del></del>		
Were endrin and 4,4'-DDT breakdowns ≤ 15% for individual breakdown in the Evaluation mix standards?		-			<del></del>			·
Was a continuing calibration analyzed daily?				•				
Were all percent differences (%D) < 20% or percent recovieries 80-120%?								_
Were all the retention times within the acceptance windows?								
y. Slanks								
Was a method blank associated with every sample in this SDG?								
Was a method blank analyzed for each matrix and concentration?							· · ·	
Were extract cleanup blanks analyzed with every batch requiring clean-up?								
Was there contamination in the method blanks or clean-up blanks? If yes, please see the Blanks validation completeness worksheet.								
Q1.Surrogade spikes								
Were all surrogate %R within the QC limits?								
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?			ſ	,				
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?				Soosianianna <sup>5</sup>				**********
VII. Matrix spike/Matrix spike triplicates.								

LDC #: 24449A3 a SDG #: See Cores

## VALIDATION FINDINGS CHECKLIST

Page: 2 of 2 Reviewer: 14 2nd Reviewer: 1

Validation Area	Yes	No	NA	Findings/Comments
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water	1		<u> </u>	
Was a MS/MSD analyzed every 20 samples of each malrix?	_			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII. Laboratory control samples		1. 1. 1. 1. 1.		
Was an LCS analyzed for this SDG?				
Was an LCS analyzed per extraction batch?	S			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX: Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
X. Target compound identification	ام ا			
Were the retention times of reported detects within the RT windows?		abu V Tribbu da		
XI. Compound quantitation/CRQLs				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions, dry weight factors, and clean-up activities applicable to level IV validation?				
XII. System performance				
System performance was found to be acceptable.				
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.				
XIV: Field duplicates				
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.				
XV. Field blanks				
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-8HC	I. Dieldrin	Q. Endrin ketone	Y. Aroclar-1242	GG. Chlordane
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	2. Aroclor-1248	HH. Chlordane (Technical)
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	H.
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	13.
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. 2,4'-DDD	KK
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. 2.4. DDE	77
G. Heptachlor epoxide	O. 4.4'-DDT	W. Aroclor-1221	EE. 2.4+DDT	MM.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	FF. Hexachlorobenzene	NN

Notes:

C:\Users\rthompson\AppData\Loca\\Microsoft\\Vindows\Temporary Internet Files\Content Outlook\366E0K9Q\COMPLST-3S wpg

SDG # 2444943

## VALIDATION FINDINDS WORKSHEET Surrogate Recovery

Page: ( of ) Reviewer: Ad 2nd Reviewer:

METHOD: GC Pesticides/PCB's (EPA SW 846 Method 8081/8082)

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

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

Were surrogates spiked into all samples and blanks?

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

#	Date	Sample ID	Column	Surrogate Compound	%R (I	%R (Limits)	Ouslifferstions
		3/4	ZM/	8	7.8%	(63-124 ) Italy	K/A CAU axout BITT
		(clam)				-	
		3(5×) (1)	NS	8	364	21/2-174 ) NO 9 Le	12.55
		1				-	
		Sak	SM	В	226	St. L. + 17 ( 121-62)	(3) 4/3
1					1		Į.
		3/40	WS	0	1450	84-124 1 7+ de 45	15/ 4 ( ALL except FF) 18
						( )	
		8 (SOX)	45	4	0	Sq-115 No 21	1. 01:12 XX
		(du+s)	<b>\</b>	В	00+1	74 )	
		9 (do se)	7/5	8	143	(63-124) (+1, 1/2)	15/4 ( MI DU. OF TI
		۲ ∤					
		9(21) (11)	705	B	09/	13-174 ) Ttole 45	45/4 ( FF 204) (c)
		(100)		\	,	^	
		10/4/67	MS	G	05/	(62-124) T+a	ets 14 (AU +X/ap+ 127)
		`				( / )	\
		· 10/2×)	MC	75	158	Hab I 1 + 06+6	5 14 (FF ONL) (S)
		(ABP)				1	
_		11 (dets)	NS	<i>H</i>	105	(59-115) It do	Jut / A His axount B. FE MI
_		`	7	8	4170	)	
<del> </del>							<u> </u>
+		11 (200x)	MS	\forall \for	232	(59-115) 100 2	J17.5X
		(dets)	- 6	15	065/5	(63-124)	
	Designation	Surrogate Compound	Compound	Recovery QC Limits (Soil)	: Limits (Soil)	Recovery OC Limits (Water)	Comments
	¥	Tetrachloro-m-xylene	vlene			1	

でン

SDG# 244443a\_

VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

EPA 8081 Pesticides METHOD:

0 Parameter:

Linear

Order of regression:

y	4.00	10	25	50	75	100	
a se	33015	78046	193282	386784	5.82E+05	7.56E+05	
Points	Point 1	Point 2	Point 3	Point 4	Point 5	Point 6	
Compound	0						
Channel	Ch. A						
Date	11-Aug-10				<del></del> !		

Regression Output: Regression Output:	Output:	Reported	
Constant	4036.64703	U	
R Squared	0.99968	= Z <sub>v</sub> J	0.99990
	7		
,		p(X)	a(X^2)
X Coefficient(s)	7594.47393		

DEMOHANTS LDC #

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: 2 of
Reviewer: 2nd Reviewer:

see cover

EPA 8081 Pesticides METHOD:

Parameter:

Linear

Order of regression:

			_		_	_	
y conc	4.00	10	25	50	75	100	
x area	47007	113757	:		8.65E+05	1.14E+06	
Points	Point 1	Point 2	Point 3	Point 4	Point 5	Point 6	
Compound	D				,		
Channel	Ch. A						
Date	11-Aug-10				J		

Regression Output: Regression Output:	)utput:	Reported	
Constant	2335.32120	# O	
R Squared	9866.0	r^2 =	0.99900
		p(X)	a(X^2)
X Coefficient(s)	11398.00028		

SDG#: 24449432 SDG#: 548 COVE

## Validatin Findings Worksheet Initial Calibration Calculation Verification

Page: 3 of UReviewer: 2

Method: EPA 8081 Pesticides

Compound:

0

		3	8	(X^2)
Date	Column	Response	Conc	Conc
8/11/2010	В	59795.00	4.000	16
		137046	10	100
		321682	25	625
		607290	50	2500
		883436	75	5625
		1123921	100	10000
			•	

### Regression Output

Constant	J	8705.2718
Std Err of Y Est		
R Squared		0.9999607
Degrees of Freedom		
	co.	q
X Coefficient(s)	1.2932E+04	-1.766E+01
Std Err of Coef.		
Correlation Coefficient		0.999980
Coefficient of Determination (r^2)		0.999961

LDC #: 24449#39 SDG #: 528 CONP

Validatin Findings Worksheet Initial Calibration Calculation

Method: EPA 8081 Pesticides

Compound:

Ω

						,		
(X^2)	Conc	16	100	625	2500	5625	10000	
X	Conc	4.000	10	25	50	75	100	
ω	Response	97207.00	224034	524202	979408	1404491	1780195	
	Column	80						
	Date	8/11/2010					•	

Ä	
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Re	

Constant	S	15757.2149
Std Err of Y Est		
R Squared		0.9999847
Degrees of Freedom		
	, co	q
X Coefficient(s)	2.1051E+04	-3.406E+01
Std Err of Coef.		
Correlation Coefficient		0.999992
Coefficient of Determination (r^2)		0.999985



# Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Reviewer: Page: 2nd Reviewer:\_

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

Percent difference (%D) = 100 \* (N - C)/N

Where: N = Initial Calibration Factor or Nominal Amount (ng)

C = Calibration Factor from Continuing Calibration Standard or \_\_Calculated Amount (ng)

ă			Ī	T	Ī		Ī	T					Ī	T	1
Recalculated	σ%	2.)	1.0	6.4	40										
Reported	0%	2.1	4	5%	2.5										
Recalculated	CF(Cono	49.0	4.05	96.8	46.5										
Reported	CF/Corrections	49.0	p.05	8.9h	S%										
Average OF	လွှေ ဂဝဝ	50.0	50.0	50.0	\$.0										
	Compound	D (DA)	) ()	D (2/2)	٥ آ										
Calibration	Date/Time	_	00/600	1		;			1					<u> </u>	L
	Standard ID	0144149													
-	#	-				2				<u>د</u>		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#: 2444943a SDG#: SEE Carr

### VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page:_	of L
Reviewer:	AY
2nd reviewer:	9/
	/

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:

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	<u> </u>
Telrachloro-m-xylene	I A	20.00	16.8018	84	84	0
Tetrachloro-m-xylene	1/2	20.00	15.5759	73	7-8	6
Decachlorobiphenyl	12	20,00	16-8013 18.38	21 92	92	0
Decachlorobiphenyl	B	20.00	18.1175	91	91	0

Sample ID:

Surrogate	Surrogate Column		Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference	
				Reported	Recalculated		
Tetrachloro-m-xylene							
Tetrachtoro-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:	 

10C# 44447#38 SDG#526 COLAT

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

Page: Reviewer: 2nd Reviewer:\_

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

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

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

Where

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

SC = Sample concentration

MSD = Matrix spike duplicate

RPD =(((SSCMS - SSCMSD) 1/2) / (SSCMS + SSCMSD))\*100 MS/MSD samples:\_

Gamma-BHC Aroclor 1260  Camma-BHC Aroclor 1260  Aroclor 1260  (UT) P. 4   7-2 M)  Aroclor 1260	30 00	18 ( MSD	Reported Reca	Recovery	Percent F	Percent Recovery	ć	
MS MSD MSD MSD 17.2 17.2 17.2 17.2 17.2 17.2 17.2 17.3 17.3 17.3 17.3 17.3 17.3 17.3 17.3		MSD 7:2/	Reported				RPD	٥
14.0 17.2 17.2 17.4 17.2 18.0 18.0 17.2 17.2 17.2 17.2 17.2 17.2 17.2 17.2		5.9/	100	Recalc.	Reported	Recalc.	Reported	Recalc.
60	5/	6.9/	0	38	28	80	6	a
			06	90	20	8	a	2
				:				
			3					

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.

2nd Reviewer: Reviewer:

LDC#: ユギザセのオタへ VALIDATION FINDINGS WORKSHEET SDG#: Specan Laboratory Control Sample/Laboratory Control Sample Duplicate Results Verification

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

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

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

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

SC = Concentration

LCS/LCSD samples:

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

577-98677-087

-	dS.	ike Lea	Spike	d Sample		rcs	-	LCSD	/SOT	TCS/ICSD
Compound	18p)	Jellar	Sono (A)	Concentation (US/)	Percent	Percent Recovery	Percent	Percent Recovery	2	RPD
	rcs C	LCSD	SOT	dson ,	Reported	Recalc.	Reported	Recalc.	Reported	Recolo
gamma-BHC	7.9)	n [g	14.8	nla	16	9				
4,4'-DDT	7.9]	৸৸	15.8	na	90	38				
Aroclor 1260										

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 #: 24449A3 <	•
SDG #: See Cone	

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	)_of
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•	- 1

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

N/N	N/A
MN	N/A

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

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

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

*	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
				<b>j</b>	
L			1		
li					
		•			
			<del> </del>		
				<u> </u>	

Note:	

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

Collection Date:

August 12 through August 13, 2010

LDC Report Date:

**December 8, 2010** 

Matrix:

Soil/Water

Parameters:

Chlorinated Pesticides

Validation Level:

Stage 2B & 4

BDT-4-S-15-6BPC

BDT-4-S-15-8BPC

EB-08122010

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6385-1

### Sample Identification

BDT-4-S-10-10BPC BDT-4-S-10-12BPC BDT-4-S-10-14BPC BDT-4-S-10-16BPC BDT-4-S-10-18BPC\*\* BDT-4-S-10-2BPC BDT-4-S-10-4BPC BDT-4-S-10-6BPC BDT-4-S-10-8BPC SSAL8-02-10BPC\*\* SSAL8-02-1BPC SSAL8-02-5BPC BDT-4-S-15-10BPC BDT-4-S-15-10BPC FD BDT-4-S-15-12BPC BDT-4-S-15-14BPC BDT-4-S-15-16BPC BDT-4-S-15-18BPC\*\*

BDT-4-S-15-2BPC BDT-4-S-15-4BPC

SSAL8-02-1BPCMS SSAL8-02-1BPCMSD

<sup>\*\*</sup>Indicates sample underwent Stage 4 review

### Introduction

This data review covers 24 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8081A for Chlorinated Pesticides.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008).

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.

Samples indicated by a double asterisk on the front cover underwent a Stage 4 review. A Stage 2B review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Stage 2B criteria since this review is based on QC data.

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/ECD Instrument Performance Check

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

### III. Initial Calibration

Initial calibration of single compounds were 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 selected compounds.

A curve fit, based on the initial calibration, was established for quantitation for selected compounds. The coefficient of determination (r²) was greater than or equal to 0.990.

Retention time windows were evaluated and considered technically acceptable for samples on which a Stage 4 review was performed. Raw data were not evaluated for the samples on which a Stage 2B 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 20.0% QC limits with the following exceptions:

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
8/21/10	048F4801.D	A	4,4'-DDD	22.6	BDT-4-S-10-4BPC BDT-4-S-10-6BPC BDT-4-S-10-8BPC SSAL8-02-10BPC** SSAL8-02-1BPC SSAL8-02-5BPC BDT-4-S-15-10BPC BDT-4-S-15-10BPC_FD BDT-4-S-15-12BPC BDT-4-S-15-14BPC BDT-4-S-15-16BPC	J+ (all detects)	А

The percent difference (%D) of the second source calibration standard were less than or equal to 20.0% for all compounds.

Retention times (RT) of all compounds in the calibration standards were within QC limits for samples on which a Stage 4 review was performed. Raw data were not evaluated for the samples on which a Stage 2B review was performed.

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

### V. Blanks

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

Sample EB-08122010 was identified as an equipment blank. 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
BDT-4-S-10-12BPC		Decachlorobiphenyl	131 (63-124)	All TCL compounds except 4,4'-DDE 4,4'-DDT	J+ (all detects)	A
BDT-4-S-10-2BPC	Not specified	Decachlorobiphenyl	136 (63-124)	All TCL compounds except 4,4'-DDE 4,4'-DDT	J+ (all detects)	А
SSAL8-02-10BPC**	Not specified	Decachlorobiphenyl	536 (63-124)	All TCL compounds except 4,4'-DDE 4,4'-DDT Hexachlorobenzene	J+ (all detects)	Α
SSAL8-02-1BPC	Not specified	Tetrachloro-m-xylene Decachlorobiphenyl	132 (59-115) 5260 (63-124)	All TCL compounds except 4,4'-DDE 4,4'-DDT Hexachlorobenzene	J+ (all detects)	Α
SSAL8-02-5BPC	Not specified	Decachlorobiphenyl	283 (63-124)	All TCL compounds except 4,4'-DDE 4,4'-DDT Hexachlorobenzene	J+ (all detects)	Α
BDT-4-S-15-10BPC	Not specified	Decachlorobiphenyl	167 (63-124)	All TCL compounds except 4,4'-DDE	J+ (all detects)	Α
BDT-4-S-15-10BPC_FD	Not specified	Decachlorobiphenyl	196 (63-124)	All TCL compounds except 4,4'-DDE 4,4'-DDT	J+ (all detects)	А

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
BDT-4-S-15-4BPC	Not specified	Decachlorobiphenyl		All TCL compounds except 4,4'-DDE 4,4'-DDT	J+ (all detects)	Α
BDT-4-S-15-8BPC	Not specified	Decachlorobiphenyl	134 (63-124)	All TCL compounds except 4,4'-DDE	J+ (all detects)	А
BDT-4-S-15-8BPC (2X)	Not specified	Decachlorobiphenyl	134 (63-124)	4,4'-DDE	J+ (all detects)	А

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

LCS ID (Associated . Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Flag	A or P
280-27268-LCS/D (EB-08122010)	Toxaphene	152 (63-118)	135 (63-118)	- -	J+ (all detects)	Р

### 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 Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

### XII. Project Quantitation Limit

All compound quantitation and CRQLs were within validation criteria for samples on which an Stage 4 review was performed.

The sample results for detected compounds from the two columns were within 40% relative percent difference (RPD) with the following exceptions:

Sample	Compound	RPD	Flag	A or P
SSAL8-02-10BPC**	4,4'-DDD beta-BHC Endrin ketone Methoxychlor	79.8 66.2 112.3 192.3	J (all detects) J (all detects) J (all detects) J (all detects)	A

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6385-1	All compounds reported below the PQL.	J (all detects)	А

Raw data were not evaluated for the samples reviewed by Stage 2B 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 BDT-4-S-15-10BPC and BDT-4-S-15-10BPC\_FD were identified as field duplicates. No chlorinated pesticides were detected in any of the samples with the following exceptions:

	Concent	ration (ug/Kg)		`		
Compound	BDT-4-S-15-10BPC	BDT-4-S-15-10BPC_FD	RPD (Limits)	Difference (Limits)	Flags	AorP
4,4'-DDE	110	130	17 (≤50)	-	-	-

	Concent					
Compound	BDT-4-S-15-10BPC	BDT-4-S-15-10BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
4,4'-DDT	33	36	9 (≤50)	-	<b>.</b>	
beta-BHC	2.6	3.5	-	0.9 (≤1.8)	-	-
Hexachlorobenzene	12	13	8 (≤50)		-	
Methoxychlor	0.77	1.6	ī	0.8 (≤2.5)	-	-
Endrin ketone	1.8U	0.83	-	0.3 (≤1.8)	-	-

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Data Qualification Summary - SDG 280-6385-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6385-1	BDT-4-S-10-4BPC BDT-4-S-10-6BPC BDT-4-S-10-8BPC SSAL8-02-10BPC** SSAL8-02-1BPC SSAL8-02-5BPC BDT-4-S-15-10BPC BDT-4-S-15-10BPC_FD BDT-4-S-15-12BPC BDT-4-S-15-14BPC BDT-4-S-15-16BPC	4,4'-DDD	J+ (all detects)	A	Continuing calibration (%D) (c)
280-6385-1	BDT-4-S-10-12BPC BDT-4-S-10-2BPC BDT-4-S-15-10BPC_FD BDT-4-S-15-4BPC	All TCL compounds except 4,4'-DDE 4,4'-DDT	J+ (all detects)	А	Surrogate recovery (%R) (s)
280-6385-1	SSAL8-02-10BPC** SSAL8-02-1BPC SSAL8-02-5BPC	All TCL compounds except 4,4'-DDE 4,4'-DDT Hexachlorobenzene	J+ (all detects)	Α	Surrogate recovery (%R) (s)
280-6385-1	BDT-4-S-15-10BPC BDT-4-S-15-8BPC	All TCL compounds except 4,4'-DDE	J+ (all detects)	А	Surrogate recovery (%R) (s)
280-6385-1	BDT-4-S-15-8BPC (2X)	4,4'-DDE	J+ (all detects)	А	Surrogate recovery (%R) (s)
280-6385-1	EB-08122010	Toxaphene	J+ (all detects)	P	Laboratory control samples (%R) (I)
280-6385-1	SSAL8-02-10BPC**	4,4'-DDD beta-BHC Endrin ketone Methoxychlor	J (all detects) J (all detects) J (all detects) J (all detects)	А	Project Quantitation Limit (RPD) (dc)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6385-1	BDT-4-S-10-10BPC BDT-4-S-10-12BPC BDT-4-S-10-14BPC BDT-4-S-10-16BPC BDT-4-S-10-16BPC BDT-4-S-10-18BPC** BDT-4-S-10-2BPC BDT-4-S-10-6BPC BDT-4-S-10-6BPC BDT-4-S-10-6BPC SSAL8-02-10BPC** SSAL8-02-18PC SSAL8-02-5BPC BDT-4-S-15-10BPC BDT-4-S-15-10BPC BDT-4-S-15-12BPC BDT-4-S-15-16BPC BDT-4-S-15-16BPC BDT-4-S-15-16BPC BDT-4-S-15-16BPC BDT-4-S-15-6BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG 280-6385-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG 280-6385-1

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET**

Stage 2B/4

Date: 10-6-10
Page: <u>/</u> of <u>/</u>
Reviewer:
2nd Reviewer: Q

SDG #: 280-6385-1 Laboratory: Test America

LDC #: 24449B3a

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
1.	Technical holding times	A	Sampling dates: 8/12-13/10
11.	GC/ECD Instrument Performance Check	A	, ,
III.	Initial calibration	A	12 % RSD = 20 % ICV/CCV = 20 %
IV.	Continuing calibration/ICV	SW	ICV/CCV = 20 16
V.	Blanks	A	/
VI.	Surrogate spikes	8W	
VII.	Matrix spike/Matrix spike duplicates	50	
VIII.	Laboratory control samples	SW	
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 Stage 2B validation.
XII.	Compound quantitation and reported CRQLs	SW	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	FP= 13+14
XV.	Field blanks	Δiγ	FP= 13+14 EB=223

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 Stage 4 validation

1	BDT-4-S-10-10BPC	11	SSAL8-02-1BPC		21	BDT-4-S-15-6BPC 5	31	280-27469-BU
2	BDT-4-S-10-12BPC 1	12	SSAL8-02-5BPC		22	BDT-4-S-15-8BPC S	32	280-27268-BUK
3	BDT-4-S-10-14BPC	13_	BDT-4-S-15-10BPC		23	لـر/ EB-08122010	33	280-27479-BUK
4	BDT-4-S-10-16BPC	14	BDT-4-S-15-10BPC_FD	];	24	SSAL8-02-1BPCMS 🔑	34	,
5 722	BDT-4-S-10-18BPC**	15	BDT-4-S-15-12BPC		25	SSAL8-02-1BPCMSD_S	35	
6	BDT-4-S-10-2BPC	16	BDT-4-S-15-14BPC		26		36	
7	BDT-4-S-10-4BPC	17	BDT-4-S-15-16BPC		27		37	<u></u>
8	BDT-4-S-10-6BPC	18	BDT-4-S-15-18BPC**		28		38	
9	BDT-4-S-10-8BPC	19	BDT-4-S-15-2BPC		29		39	
10 <sup>12</sup>	SSAL8-02-10BPC**	20	BDT-4-S-15-4BPC	<i>,</i>	30_		40	

LDC #: 24449163a SDG #: Sec Come

### VALIDATION FINDINGS CHECKLIST

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

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

Validation Area	Yes	No	NA	ļ	Findings/C	omments	
l. Technical holding limes							
All technical holding times were met.							
Cooler temperature criteria was met.							
II. GC/EC0 Instrument performance check			(3-30 14-30 14-31				
Was the instrument performance found to be acceptable?		r					
III. Inittal calibration							
Did the laboratory perform a 5 point calibration prior to sample analysis?						<u></u>	
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?						<del></del>	
Did the initial calibration meet the curve fit acceptance criteria?		·					
Were the RT windows properly established?					· 		
Were the required standard concentrations analyzed in the initial calibration?				<del> 20.</del> .		en en en en <del>Elle e</del> n en e	
IV. Continuing calibration							
What type of continuing calibration calculation was performed?%R						-	
Were Evaluation mix standards analyzed prior to the initial calibration and sample analysis?							
Were endrin and 4,4'-DDT breakdowns ≤ 15% for individual breakdown in the Evaluation mix standards?							
Was a continuing calibration analyzed daily?							
Were all percent differences (%D) ≤ 20% or percent recovieries 80-120%?					·		
Were all the retention times within the acceptance windows?							***************************************
V. Blanks							
Was a method blank associated with every sample in this SDG?	h					<u></u> -	
Was a method blank analyzed for each matrix and concentration?				_			
Were extract cleanup blanks analyzed with every batch requiring clean-up?							
Was there contamination in the method blanks or clean-up blanks? If yes, please see the Blanks validation completeness worksheet.	_						
И. Starogale spikes							
Were all surrogate %R within the QC limits?							
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?				·			
ff any %R was less than 10 percent, was a reanalysis performed to confirm %R?							× × × × × × × × × × × × × × × × × × ×
All Natrix spike/Matrix spike duplicates							

LDC #: 2444913 a SDG #: See Googe

### VALIDATION FINDINGS CHECKLIST

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

		T	<del></del>	T
Validation Area	Yes	No	NA	Findings/Comments
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water	7			
Was a MS/MSD analyzed every 20 samples of each matrix?				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII. Laboratory control samples:				
Was an LCS analyzed for this SDG?	/_			<u> </u>
Was an LCS analyzed per extraction batch?	_			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?			<u> </u>	
IX: Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
X. Target compound identification				
Were the retention times of reported detects within the RT windows?			30 am 1	
XI. Compound quantifation/CRQLs				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions, dry weight factors, and clean-up activities applicable to level IV validation?				
XII System performance				
System performance was found to be acceptable.				
XIII: Overall assessment of data				
Overall assessment of data was found to be acceptable.		^		
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.				
CV. Field blanks				
Field blanks were identified in this SDG.	4	`	,	
Farget compounds were detected in the field blanks.				

# VALIDATION FINDINGS WORKSHEET

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

A alpha-BHC	1. Dieldrin	O. Endrin ketone	Y. Aroclor-1242	GG Chlordane	
B beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH Chlordane (Technical)	
C delta-8HC	K. Endrin	S aipha-Chlordane	AA Aroclor-1254	=	
D gamma-BHC	L. Endosulfan II	T gamma-Chlordane	B8 Aroclor-1260	JJ.	
E. Heptachior	M. 4.4'-DDO	U. Toxaphene	CC 2.4:DDD	K.	
F Aldrin	N. Endosulfan sulfate	V Aroclor-1016	DD 2.4-DDE	1	
G. Heptachlor epoxide	O. 4,4'-DDT	VV. Aroclor-1221	EE. 2.4'-DDT	MM	
H Endosulfan I	P Methoxychlor	X Aroclor-1232	FF. Hexachlorobenzene	NN.	

C:\Users\rthompson\AppData\Local\Microsoft\V\rdows\Temporary Internet Files\Content Outlook\366E0K9Q\COMPLST.3S wca

Notes:

SDG # LDC #:

### VALIDATION FINDINGS WORKSHEET Continuing Calibration

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

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

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

Were Evaluation mix standards run before initial calibration and before samples?

Were Endrin & 4,4'-DDT breakdowns acceptable in the Evaluation Mix standard (<15.0% for individual breakdowns)? Was at least one standard run daily to verify the working curve?

Did the continuing calibration standards meet the percent difference (%D) of <20.0%?

pwel IV/D Only Y NA

N N/A N N/A

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

	1	ī	ī	<del>-</del>	ī	<del></del>	1	Т	<del></del>	т	<del>_</del>	T	_	T	_	1	T	T	<del></del>	_	$\overline{}$		<del></del>	<del></del>	7
Qualifications	7+dets/# (C)												THE PROPERTY OF THE PROPERTY O	AND THE RESIDENCE OF THE PROPERTY OF THE PROPE		man managan da ang m								The state of the s	GG. Chlordane HH. Chlordane (Technical) obenzene
Samples		+ + 100																							CC. 24-DDD DD 24-DDE EE 24-DDT FF Hexachlorobenzene
Associated Samples	+(·t	(20,4	_																						Y Aroclor-1242 Z Aroclor-1248 AA Aroclor-1254 BB Aroclor-1260
	^	^	_	^	_	-	_	_	-	_	^	_	~	_	^	^	^	-	-	_	_	_	_	_	
RT (Limits)	)	)	)		_		~		_	)		)	_	`	)	)		_					. )	`	U. Toxaphene V. Aroclor-1016 W. Aroclor-1221 X. Aroclor-1232
	<u></u>					:																			O. Endrin ketone R. Endrin aldehyde S. alpha-Chlordane T. gamma-Chlordane
%D nit ≤ 20.0)	22.6		:																						
(Li	2													3											an sulfate
Compound	M				į																				M 4.4.DDD N Endosulfan sulfate O 4.DDT P Methoxychlor
Column (	-4															ı									1. Dieldna J. 4.4DDE K. Endra L. Endosuifaa II
Č										-															
Standard ID	018 F4801.D																								E Heptachior F Aldrin G. Heptachlor epoxide H. Endosulfan I
Date	01/11/8	,																							A alpha-BHC B beta-BHC C deta-BHC D gamma-BHC
#							•					:													A alph B bets C delt D gam

LDC #: 244491054 SDG # SER COUNT

### VALIDATION FINDINDS WORKSHEET Surrogate Recovery

Page: Reviewer. 2nd Reviewer:

METHOD: GC Pesticides/PCB's (EPA SW 846 Method 8081/8082)

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? N N/A

# Date	Sample ID	Column	Surrogate Compound	%R (I	%R (Limits)	Qualifications
	(54-6)-8	WS	B	131	190+C (83-124)	5/4
	(St. M. S)	4/5	В	136	(63-124) J+24	It obys/A (ALL except JO) (S)
	(D(dA)		В	576	(62-124) J+ J+S	S/A (ALL except 5,0, FF) (S
	10 (10x) (das)		B	0725	(63-124) NO g	quel, dil258
	(1)	57	4 0	132	(59-115) 7+ dets.	5/4 (ALL except 7,0, F7) (8
	(1) (100×)	SSN	A Q	5810	( 63-124 ) 100	2 2 2 11/2 5 X
	12-(MS)	7 %	6	283	( h2129)	45/ ALL except 5,0, Ft) (5
	(XOI) [1	\N.	2	765	(63-126) no a	guel, 01:175x
	13 (AUS)	SW	B	49/	(63-12y) J-7	dets/A (ALL except I) (S
	13 (5x) El	SW .	B	165	63-124 , NO guel	ud 101/25x
	14 (24s)	NS	9	961	5400 ( 421-89)	5/4 (ALL except 5,0) (S
Designation	on Surrogate Compound	punoduo	Recovery Q(	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A B	Tetrachloro-m-xylene	/lene				
_	יייקיבט יטוויסטסס	1811				

### LDC #. 244791839 SDG #: 20 Can

### VALIDATION FINDINDS WORKSHEET Surrogate Recovery

Reviewer. 2 Page:

METHOD: GC Pesticides/PCB's (EPA SW 846 Method 8081/8082)

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

Were surrogates spiked into all samples and blanks?

Y ( ) N/A

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

			,				
*	Date	Sample ID	Column	Surrogate Compound	%R(	%R (Limits)	Qualifications
		(5h4)(xs) h)	1 1	В	861	(63.124)	HOSLE DITSX
						,	
		20(000)	NS	8	290	(23-124)	It along 14. (ALL except J.O) (
						( )	
		20(50x)	W	Q	730	(63-124)	NO 9100 JILYSX
						``	
		-				( )	
		22	NS	Ø	134	(63-124)	Jt dets (A (ALL except 3) (S
		- II				( )	
		22 (24)	MS	P	134	(63-124)	J+ 20+5/4 (ML4 7) /5)
					<b>,</b>	(	
						(	
					,		
						(	
1						( )	
						( )	
1						( )	
						( )	
	Designation	on Surrogate Compound	punodwo	Recovery Q(	Recovery QC Limits (Soil)	Recovery QC	Recovery QC Limits (Water)
ĺ	A	Tetrachloro-m-xylene	lene				
	В	Decachlorobiphenyl	υγί				

LDC #: 24449 B3Q SDG # Sep and

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

Page: Reviewer. 2nd Reviewer:

YN N/A

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?

Was an MS/MSD analyzed every 20 samples for each matrix or whenever a sample extraction was performed? Were the MS/MSD percent recoveries (%R) and relative percent differences (RPD) within QC limits?

Qualifications		1 1 1	See a toches																						
Associated Samples	//																								
RPO (Limits)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(	( )	( )	,	(	,	-	( )	( )	( )	(	( )	( )	{ ,	,
MSD %R (Limits)	(			domat.	( )		( )	( )	( )	( )	( )	(	( )	( )	( )	( )	( )	(	( )	( )	( )	( )	( )	( )	)
MS %R (Limits)	( )	( )	(2	1 A++C			<i>-</i>	1	( )	( )	( )		( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	{	( )	,
Compound					<i>,</i>				\																
# MS/MSD ID	24/25		-																						

### **Quality Control Results**

Client: Northgate Environmental Management Inc.

Job Number: 280-6385-1

Matrix Spike/

Matrix Spike Duplicate Recovery Report - Batch: 280-27469

Method: 8081A Preparation: 3550C

MS Lab Sample ID:

280-6385-11

Analysis Batch: 280-28568

Client Matrix:

Solid

Instrument ID: GCS\_P2 Lab File ID: 065F6501.D

Dilution:

1.0

Initial Weight/Volume:

30.1 g

Date Analyzed:

Final Weight/Volume:

10000 uL

Date Prepared:

08/21/2010 0814 08/18/2010 1850

Injection Volume: Column ID:

**PRIMARY** 

MSD Lab Sample ID:

280-6385-11

Analysis Batch: 280-28568

Instrument ID: GCS\_P2

Client Matrix:

Solid

Prep Batch: 280-27469

Lab File ID: 066F6601.D

30.1 g

Dilution:

1.0

Prep Batch: 280-27469

Initial Weight/Volume: Final Weight/Volume:

10000 uL

Date Analyzed: Date Prepared: 08/21/2010 0830 08/18/2010 1850

Injection Volume: Column ID:

**PRIMARY** 

		<u>%</u>	Rec.						
Analyte		MS	MSD	Limit		RPD	RPD Limit	MS Qual	MSD Qual
4,4'-DDD M		115	122	57 - 1	18	7	20 <del>J + J</del>	H-LA	F
4,4'-DDE "▼"	no gral	-22500	-21200	61 - 1	15	14	15 + 6 + 130	۶ E4 <del>S دم</del>	X+C+301
4,4'-DDT 🕖	, b	-4260	-3470	53 - 1	25	16	29	E 4	E4
Aldrin	UCSOF	82	85	60 - 1	15	3	50		
alpha-BHC 🛧	1	93	154	54 - 1	15	45	17 <del>उ।</del> बर्	3/A	F
کہ alpha-Chlordane	<b>\</b>	0	0	60 - 1	15 ·	NC	18 J-/a	-√. -√.UF	UF
beta-BHC B		269	376	58 - 1	15	25	مماد + <del>1</del> 7	¥£ F	EF
delta-BHC <u></u>		111	129	62 - 1	15	15	19 ]	P141	F
Dieldrin I	` \	0	0	63 - 1	17	NC	25-3-/18	<del>/</del> 4 U F	UF
Endosulfan I 🖁 🖁	1	0	0	55 - 1	15	NC	26	UF	UF
Endosulfan II		0	0	60 - 1	15	NC	20	UF	UF
Endosulfan sulfate		351	0	58 - 1	18	NC	22	EF	UF
Endrin Ҡ	}	458	458	61 - 1	21	0	30	EF	EF
Endrin aldehyde	}	85	68	54 - 1	15	23	29		_,
Endrin ketone		106	70	61 - 1	18	13	20	E	E
gamma-BHC (Lindane	)	90	98	59 - 1	15	8	24		_
gamma-Chlordane	T	347	487	60 - 1	15	33	21	EF	EF
Heptachlor		82	92	61 - 1	15	11	18		
Heptachlor epoxide @	<b>b</b>	0	0	62 - 1	12	NC	18	UF	UF
Hexachlorobenzene	F   ,	-4220	-3220	50 - 1	30	36	25	E 4	E4F
Methoxychlor P		0	0	52 - 1	23	NC	23	UF	UF
Surrogate	<u> </u>	erica de laboración de construcción de constru	MS % Rec		MSD %	Rec	Acce	eptance Limits	<b>:</b>
DCB Decachlorobipher	nyl		5530	ΕX	5220	ΕX	n. St. vermeler er i ser som er en	3 - 124	P. POPPS Photo Prompt West on A. gordina
Tetrachloro-m-xylene			141	Х	168	X	5	9 - 115	

Surrogate	MS % Rec		MSD % Re		Acceptance Limits
DCB Decachlorobiphenyl	10100	EX	9480	EX	63 - 124
Tetrachloro-m-xylene	104		158	X	59 - 115

### **Quality Control Results**

Client: Northgate Environmental Management Inc.

Job Number: 280-6385-1

Matrix Spike/

Matrix Spike Duplicate Recovery Report - Batch: 280-27469

Method: 8081A Preparation: 3550C

MS Lab Sample ID:

280-6385-11

Analysis Batch: 280-28568

Instrument ID: GCS P2

Client Matrix:

Dilution:

Solid 1.0

Prep Batch: 280-27469

Lab File ID:

067F6701.D

Initial Weight/Volume: Final Weight/Volume:

10000 uL

Date Analyzed:

08/21/2010 0847

Injection Volume:

Date Prepared:

08/18/2010 1850

Column ID:

**PRIMARY** 

MSD Lab Sample ID:

280-6385-11

Analysis Batch: 280-28568

Instrument ID: GCS\_P2

Client Matrix:

Solid

Lab File ID:

Dilution:

Prep Batch: 280-27469

068F6801.D

1.0

Initial Weight/Volume: Final Weight/Volume:

30.1 g 10000 uL

Date Analyzed: Date Prepared:

08/21/2010 0903 08/18/2010 1850

Injection Volume: Column ID:

**PRIMARY** 

		<u>%</u>	Rec.						
Analyte		MS	MŞD	Lim	it	RPD	RPD Limit	MS Qual	MSD Qual
, was a sustain a group of the control of the sustain and the	noncel		(300)(1) p.m. 474 (2000)(1000)(1000)(1000)		Mark Million & Advanced Communication	*********	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	Mar appear to some a construction of the section of	MARKET AND THE STATE OF THE STA
Toxaphene $igcup$	20 9 6 - 2	13000	13300	54 -	135	2	23	EF	EF
Surrogate	us		MS % Rec		MSD %	Rec	Acci	eptance Limit	S
DCB Decachlorobiphenyl	ok		4820	ΕX	5270	EX	E	3 - 124	and an analysis of the second
Tetrachloro-m-xylene			108		183	Χ	5	9 - 115	

LDC #: 29% SDG#18

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

2nd Reviewer:\_

METHOD: XGC HPLC

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

N/A N/A

Level IVID Only

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

	Qualifications	T-445/0 (2)																							
	Associated Samples	先し出り	(MN)																						
	RPD (Limits)	( )	( )	( )				)		( )	(				~	)		( )		( )	,	)	(	-	
CSD	r K	(2)-(6)-(6)	`	( )	( )	( )	( )	( )	,	( )	(	( )	(	( )	(	(	, ,	(	~	(		( )	-	( )	
LCS	Oliver, car	1	)	( )	( )	( )	( )	( )		( )	(	( )	( )	( )	`		, ,	( )	( )	( )		( )	( )	( )	
Compound																						İ			
# rcs/rcsp ip	184-17268, (55/1)																								

LDC # 246 SDG#X METHOD: GC\_ HPLC

# Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

Reviewer; 2

Page:

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". Y(R) N/A. Did the percent difference of detected compounds between two columns./detectors < 40%?

	J de	/		7			The state of the s					7		
(%RPD)%D Between Two Columns/Detectors Limit (< 40%)	74.8	66.2	112.3	1933	Turning Addition			1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1		THE THE TAXABLE PARTY OF TAXABLE				
Sample ID	(0													
# Compound Name	X	S	Ø	0										

100 #: 2447 1100 a. SDG #: 54 (OLOR

VALIDATION FINDINGS WORKSHEET

Reviewer: 2

Page;

Field Duplicates

METHOD: CONTROL HPLC

Were field duplicate pairs Identified in this SDG?

Which is a ware target compounds detected in the field duplicate pairs?

Compound	Concentration (	48/129	%RPD	Qualification
	B	<i>b)</i>	Limits	Parent only / All Samples
( <del>-</del>	017	130	1	
0	33	36	8	
0;	2.6	3.5	0.9 (0.01)	(8/5)
7	77	3		
1	5.77	9./	130	(42.5)
Ø	0-7-2-B	0.83	1(2.4)	(4/4)
	81			/9:/-
	Concentration (	(	%RPO	Qualfleation
			Limit	Parent only / All Samples

LDC # 24444634 SDG# see cover

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

EPA 8081 Pesticides METHOD:

0 Parameter:

Order of regression:

Linear

				×	ý
Date	Channel	Compound	Points	агеа	conc
11-Aug-10	Ch. A	0	Point 1	33015	4.00
			Point 2	78046	10
•			Point 3	193282	25
•			Point 4	386784	50
•			Point 5	5.82E+05	75
	•		Point 6	7.56E+05	100

Regression Output: Regression Output:		Reported	
Constant	4036.64703	0	
R Squared	0.99968	r^2 =	06660
		p(X)	a(X^2)
X Coefficient(s) 7594.47393			

Reviewer: M

24449132

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: 2 of Reviewer: 2nd Reviewer:

see cover PDC # EPA 8081 Pesticides METHOD:

Ω Parameter: Order of regression:

Linear

Date	Channel	Compound	Points	x area	y
11-Aug-10	Ch. A	O	Point 1	47007	4.00
_			Point 2	113757	10
			Point 3	286925	25
			Point 4	575154	50
		,	Point 5	8.65E+05	75
			Point 6	1.14E+06	100
į					

Re	Regression Output: Regression Output:	Reported
Constant	2335.32120	11 0
R Squared	98660	r^2 = 0 9990r
		b(X) a(X^2)
X Coefficient(s)	11398.00028	

Validatin Findings Worksheet Initial Calibration Calculation Verification

Page: 3 of U Reviewer: 2nd Reviewer:

Method: EPA 8081 Pesticides

SDG#: 2444 9034 SDG#: 546 COM

Compound:

0

Date         Column         Response         Conc         Conc           8/11/2010         B         59795.00         4.000         16           137046         10         100           607290         50         2500           883436         75         5625           1123921         1000         10000					
Column         Response         Conc           B         59795.00         4.000           137046         10           321682         25           607290         50           883436         75           1123921         100			ε	8	(X^2)
B 59795.00 4.000  137046 10  321682 25  607290 50  1123921 100	Date	Column	Response	Conc	Conc
10 25 50 50 75 100	8/11/2010	В	59795.00	4.000	91
25 50 75 100			137046	10	100
50 75 100			321682	25	625
100			607290	50	2500
100			883436	75	5625
			1123921	100	10000

Regression Output	utput	
Constant	Ü	8705.2718
Std Err of Y Est		
R Squared		0.9999607
Degrees of Freedom		
	æ	q
X Coefficient(s)	1.2932E+04	-1.766E+01
Std Err of Coef.		
Correlation Coefficient		0.999980
Coefficient of Determination (r^2)		0.999961

Validatin Findings Worksheet Initial Calibration Calculation Verification

Page: 4
Reviewer: 2nd Reviewer

Method: EPA 8081 Pesticides

LDC#: 244491829 SDG#: 528 CONPE

Compound:

Ω

(X^2)	Conc	16	. 100	625	2500	5625	10000	
(X)	Conc	4.000	10	25	50	75	100	
(λ)	Response	97207.00	224034	524202	979408	1404491	1780195	
	Column	æ					,	
	Date	8/11/2010						

Indino noissaiday	urpur	
Constant	S	15757.2149
Std Err of Y Est		
R Squared		0.9999847
Degrees of Freedom		
	æ	q
X Coefficient(s)	2.1051E+04	-3.406E+01
Std Err of Coef.		
Correlation Coefficient		0.999992
Coefficient of Determination (r^2)		0.999985

SDG #: See COLL LDC #: 24491

# Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Page: Reviewer. 2nd Reviewer:

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

Percent difference  $(%D) = 100 \cdot (N - C)/N$ 

Where:

N = 1 Initial Calibration Factor or Nominal Amount (ng) G = 1 Calibration Factor from Continuing Calibration Standard or \_ Calculated Amount (ng)

		Calibration		4	Reported	Recalculated	Reported	Recalculated
∓t	Standard ID	Date/Time .	Compound	Acc Wene	CRCCore	CFICON	Φ%	۵%
-	035F3501.D	8/21/10	D (OGA)	50.0	1.05	50.1	1.0	7.0
			ව	20.05	19.4	hbh	7.7	7-1
			P (ChB)	25.0	50,8	7:25	0.3	0.3
			0 7	0.05	50.2	8.7	0.5	20
2	CHBKHBD].	01/12/8	D (GAA)	50.0	5/3	57.3	3.6	2.6
			7 0	20.0	\$.05	\$23.8	1.4	7-7
			D (ChB)	5.00	51.6	5/2	んな	N. 1
-			0 7	90	53.6	53.6	7.2	7.7
m	OC/F6101.	170	D (QQ.4)	0.05	8.65	3.6%	0.5	0,0
		21/17/0	7 0	0.05	9.66	49.6	9.8	8.0
			D (CRB)	50.0	50.7	7.25	9.0	4.0
			0 7	50,0	51.8	57.8	3.6	W. 8
4	BOS FOSOL	D 9/2 /	D (cht)	0.25	48.0	6.3	2.2	22
.		0/22/0	2 0	50.05	495	5.45	0.1.2	0.7
			D (0/16)	50.0	47.4	447	53	5.3
-			7	52.0	470	44.0	5.9	K.3

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

SDG #: 500 5000

## Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET



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

Percent difference (%D) = 100 \* (N - C)/N

Where:  $N = _{\text{initial Calibration Factor or}}$  Nominal Amount (ng)  $C = _{\text{colibration Factor from Continuing Calibration Standard or}$  Calculated Amount (ng)

				,	Reported	Recalculated	Reported	Recalculated
<b>#</b>	Standard ID	Callbration Date/Time	Compound	Average CF/ CCV 860c	CF/GAC CCV	CHICODE	ď%	<b>0%</b>
	018F18017	_	D (PAA)	50.0	49.3	C/b/s	1.4	1.9
		8/22/10	70	0.25	2.7	50.0	0.5	کار معار
<u> </u>			D (CLB)	200	4347	643	# 4.2	4.2
			Jo 0	50.0	44.5	445	(/)	[7]
٥	Clost Pyol	lL	(A)(V) 4	8.0	49.1	1.65	1.8	6./
<u> </u>		01/82/8	71.0	795	5.66	44.3	/.4	6-7
			D (Cha)	505	47.4	かせん	/-5	5-1
			0 4	0,025	2.8.8	18.0	2.4	24
က								,
		_						
4								
<u>L</u>								

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 #: 244491339 SDG #: 500 COLO

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

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it,
'Q'

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

The percent recoveries (%R) of surrogates were recalculated for the compounds feentified below using the following calculation	The percent recoverie	s (%R) of surrogales	were recalculated for the	e compounds ide	enlified below usin	g the followin	g calculation:
--	-----------------------	----------------------	---------------------------	-----------------	---------------------	----------------	----------------

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Sample ID: 5

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene	A	90.00	16.3495	82	82	0
Tetrachloro-m-xylene	B	20.00	16.8845	84	84	0
Decachlorobiphenyl	A	20.00	19-1099	96	94	0
Decachlorobiphenyl	n	70.00	19.4936	97	97	U

Sample ID:

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Telrachloro-m-xylene						
Telrachioro-m-xylene						
Decachlorobiphenyl		<u> </u>				
Decachlorobiphenyl						

Sample ID:

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

Sample ID:

ampic io.	7.T.T.					
Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene						
Tetrachloro-m-xylene						
Decachlorobiphenyl						
Decachlorobiphenyl						

Notes:	 	

LDC#.24449BJA SDG#.446.CO.A.A.

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

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

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

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

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

Where

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

SC = Sample concentration

MSD = Matrix spike duplicate

MS/MSD samples:

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

Recalc.  $e_{j}$ MS/MSD RPD Reported Recalc. Matrix Spike Duplicate Percent Recovery 341 00 Reported 1470 B Recalc. 24-20-0 Percent Recovery 145 Matrix spike Reported 027 90 140 MSD 668 Spike Sample Concentration SΕ 15.6 400 Sample M 840 ИMSD 4 7 Spike Added £ 43 Compound Gamma-BHC Aroclor 1260 4,4'-DDT

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. UDC #: 244963 ק SDG #: ביילאנגל אונויא Control Sample/Laboratory Control Sample Duplicate Results Verification

Reviewer: Page:

2nd Reviewer:

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

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

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

SSC = Spiked sample concentration SA = Spike added Where:

SC = Concentration

RPD = 1 LCS - LCSD 1 \* 2/(LCS + LCSD)

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

LCS/LCSD samples:\_

280-27469 -16

Concentration   Percent Recovery   Percent Recove		dS.	ik•	Spiked	Sample	SOT	Ş	CCSD	ds:	/SOT	LCS/LCSD
LCS	punodi	(V)	<b>K</b>	(Sonce)	ntration (2.	Percent F	lecovery	Percent F	Recovery	R	RPD
16.2 114 12.9 114 . 30 16.2 114 13.8 114 85 160 13.8 114 85		רכצ	O LCSD	CS C	CSD	Reported	Recalc.	Reported	Recalc.	Reported	Receic.
60 13.8 n/k 855	오	7.91	2/4	12.9	nla	08					
Aroclor 1260  Ar	4,4'-DDT	7.9/	44	13.8	nk	38	1				
	560										
								,			
					·				,	_	
					-						

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 #: 24449B39 SDG #: <u>Eal Co</u>ul

## VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	 Lof		
Reviewer:	110	- 7	
2nd reviewer:	0	J	

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

(8)	<u>N</u>	N/A
$   \overline{A} $	Ν	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?

y=nx+b
b=4036.64
M=7594.47

Example:
Sample I.D:
Conc. = (49672-4036.65) (100L) (1x) (7594.47) (30.55) (.924)
(759447) / (30.56) (.924)
= 2.1 vs/kg
0

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

Note:	 	- 	
<del></del>	 		 <del></del>

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

Collection Date: August 13 through August 16, 2010

**LDC Report Date:** December 8, 2010

Matrix: Soil/Water

Parameters: Chlorinated Pesticides

Validation Level: Stage 2B & 4

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6415-1

## Sample Identification

BDT-4-N-10-16BPC EB-08162010 BDT-4-N-10-18BPC\*\* EB-08132010 BDT-4-N-15-2BPC BDT-4-N-10-2BPC BDT-4-N-15-2BPC FD BDT-4-N-10-4BPC BDT-4-N-15-4BPC BDT-4-N-10-6BPC BDT-4-N-15-6BPC BDT-4-N-10-8BPC BDT-4-S-20-2BPC BDT-4-N-15-2BPC FDMS BDT-4-S-20-4BPC BDT-4-N-15-2BPC FDMSD BDT-4-N-10-2BPCMS BDT-4-S-20-6BPC BDT-4-S-20-8BPC BDT-4-N-10-2BPCMSD BDT-4-S-20-10BPC

BDT-4-S-20-12BPC BDT-4-S-20-14BPC BDT-4-S-20-16BPC BDT-4-S-20-18BPC\*\* BDT-4-S-20-8BPC\_FD BDT-4-N-10-10BPC BDT-4-N-10-12BPC BDT-4-N-10-14BPC

BDT-4-N-10-14BPC\_FD

<sup>\*\*</sup>Indicates sample underwent Stage 4 review

## Introduction

This data review covers 28 soil samples and 2 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 the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008).

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.

Samples indicated by a double asterisk on the front cover underwent a Stage 4 review. A Stage 2B review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Stage 2B criteria since this review is based on QC data.

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

## I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/ECD Instrument Performance Check

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

## III. Initial Calibration

Initial calibration of single compounds were 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 selected compounds.

A curve fit, based on the initial calibration, was established for quantitation for selected compounds. The coefficient of determination (r²) was greater than or equal to 0.990.

Retention time windows were evaluated and considered technically acceptable for samples on which a Stage 4 review was performed. Raw data were not evaluated for the samples on which a Stage 2B 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 20.0% QC limits for all compounds.

The percent difference (%D) of the second source calibration standard were less than or equal to 20.0% for all compounds.

Retention times (RT) of all compounds in the calibration standards were within QC limits for samples on which a Stage 4 review was performed. Raw data were not evaluated for the samples on which a Stage 2B review was performed.

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

### V. Blanks

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

Samples EB-08162010 and EB-08132010 was identified as equipment blanks. No chlorinated pesticide contaminants were found in these blanks.

## VI. Surrogate Spikes

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

## 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) and relative percent differences (RPD) 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 Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

## XII. Project Quantitation Limit

All compound quantitation and CRQLs were within validation criteria for samples on which an Stage 4 review was performed.

The sample results for detected compounds from the two columns were within 40% relative percent difference (RPD) with the following exceptions:

Sample	Compound	RPD	Flag	A or P
BDT-4-N-10-18BPC**	beta-BHC	53.2	J (all detects)	А

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6415-1	All compounds reported below the PQL.	J (all detects)	А

Raw data were not evaluated for the samples reviewed by Stage 2B 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 BDT-4-N-15-2BPC and BDT-4-N-15-2BPC\_FD and samples BDT-4-S-20-8BPC and BDT-4-S-20-8BPC\_FD and samples BDT-4-N-10-14BPC and BDT-4-N-10-14BPC\_FD were identified as field duplicates. No chlorinated pesticides were detected in any of the samples with the following exceptions:

	Concent	ration (ug/Kg)				
Compound	BDT-4-N-15-2BPC	BDT-4-N-15-2BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
beta-BHC	0.72	0.66U	-	0.06 (≤1.8)	-	-
Hexachlorobenzene	21	9.2	78 (≤50)	-	J (all detects)	Α

	Concent	Concentration (ug/Kg)				
Compound	BDT-4-S-20-8BPC	BDT-4-S-20-8BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
4,4'-DDE	4.4	1.5	-	2.9 (≤1.8)	J (all detects)	А
4,4'-DDT	1.3	0.62U	-	0.58 (≤1.8)	-	-
Hexachlorobenzene	0.44	0.29U	-	0.15 (≤1.8)	-	•

	Concent	ration (ug/Kg)				
Compound	BDT-4-N-10-14BPC	BDT-4-N-10-14BPC_FD	RPD (Limits)	Difference (Limits)	Flags	AorP
beta-BHC	1.6	1.6	•	0 (≤1.8)	-	-

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Data Qualification Summary - SDG 280-6415-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6415-1	BDT-4-N-10-18BPC**	beta-BHC	J (all detects)	A	Project Quantitation Limit (RPD) (dc)
280-6415-1	EB-08162010 EB-08132010 BDT-4-N-15-2BPC BDT-4-N-15-2BPC_FD BDT-4-N-15-4BPC BDT-4-N-15-6BPC BDT-4-S-20-4BPC BDT-4-S-20-6BPC BDT-4-S-20-6BPC BDT-4-S-20-10BPC BDT-4-S-20-10BPC BDT-4-S-20-14BPC BDT-4-S-20-14BPC BDT-4-S-20-18BPC** BDT-4-S-20-18BPC** BDT-4-N-10-12BPC BDT-4-N-10-14BPC BDT-4-N-10-14BPC BDT-4-N-10-14BPC BDT-4-N-10-16BPC BDT-4-N-10-16BPC BDT-4-N-10-16BPC BDT-4-N-10-16BPC BDT-4-N-10-16BPC BDT-4-N-10-16BPC BDT-4-N-10-6BPC BDT-4-N-10-6BPC BDT-4-N-10-6BPC BDT-4-N-10-6BPC BDT-4-N-10-6BPC BDT-4-N-10-6BPC BDT-4-N-10-6BPC BDT-4-N-10-6BPC BDT-4-N-10-6BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
280-6415-1	BDT-4-N-15-2BPC BDT-4-N-15-2BPC_FD	Hexachlorobenzene	J (all detects)	A	Field differences (RPD) (fd)
280-6415-1	BDT-4-S-20-8BPC BDT-4-S-20-8BPC_FD	4,4'-DDE	J (all detects)	А	Field duplicates (Difference) (fd)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG 280-6415-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG 280-6415-1

No Sample Data Qualified in this SDG

## **Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET**

Stage 2B/4

Date: <u>12-6-10</u>
Page: 1 of /
Reviewer: //
2nd Reviewer:

SDG #: 280-6415-1 Laboratory: Test America

LDC #: 24449C3a

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
],	Technical holding times	A	Sampling dates: 8/13/10, 8/16/10
II.	GC/ECD Instrument Performance Check	A	, , , , ,
JII.	Initial calibration	A	12, 10RSD = 2090
IV.	Continuing calibration/ICV	A	12 /0RSD = 20 90 TEV/CCV = 20 90
V.	Blanks	A	, , , , , , , , , , , , , , , , , , , ,
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	4	
VIII.	Laboratory control samples	A	LESID
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 Stage 2B validation.
XII.	Compound quantitation and reported CRQLs	SW	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	FD=3+4 1/5+16,19+20 FB=1,2
XV.	Field blanks	N	FB= 1,2

Note: A = Acceptable

ND = No compounds detected R = Rinsate

D = Duplicate TB = Trip blank

N = Not provided/applicable SW = See worksheet

FB = Field blank

EB = Equipment blank

Validated Samples: \*\* Indicates sample underwent Stage 4 validation

1	EB-08162010 4)	11	BDT-4-S-20-10BPC 5	21	BDT-4-N-10-16BPC (		31	280-27602-BLK
2	EB-08132010 W	12	BDT-4-S-20-12BPC	22 nA	BDT-4-N-10-18BPC**		32	280-27479-1664
3	BDT-4-N-15-2BPC	13	BDT-4-S-20-14BPC	23	BDT-4-N-10-2BPC		33	280-27799-BLKS
4	BDT-4-N-15-2BPC_FD /	14	BDT-4-S-20-16BPC	24	BDT-4-N-10-4BPC		34	
5	BDT-4-N-15-4BPC	15 <sup>1</sup> V	BDT-4-S-20-18BPC**	25	BDT-4-N-10-6BPC		35	
6	BDT-4-N-15-6BPC	16	BDT-4-S-20-8BPC_FD	26	BDT-4-N-10-8BPC		36	
7	BDT-4-S-20-2BPC	17	BDT-4-N-10-10BPC	27_	BDT-4-N-15-2BPC_FDMS		37	
8	BDT-4-S-20-4BPC	18	BDT-4-N-10-12BPC	28	BDT-4-N-15-2BPC_FDMS	•	38	
9	BDT-4-S-20-6BPC	19	BDT-4-N-10-14BPC	29	BDT-4-N-10-2BPCMS		39	
10	BDT-4-S-20-8BPC	20	BDT-4-N-10-14BPC_FD	30	BDT-4-N-10-2BPCMSD	/	40	

LDC #: 24449039 SDG #: Sec Cores

## VALIDATION FINDINGS CHECKLIST

Page: \_1 of 2 Reviewer: \_ // 2nd Reviewer: \_ g \_\_.

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

Validation Area	Yes	No	NA	Findings/Comments
); Technical holding limes				
	1			
All technical holding times were met.				
Cooler temperature criteria was met.  II: GC/ECD:Instrument performance check				
<u> </u>		<u> </u>	[	
Was the instrument performance found to be acceptable?  III Initial calibration	<b>م</b> الإيلاداء			
Did the laboratory perform a 5 point calibration prior to sample analysis?	1	ļ		
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?	1/			
Were the required standard concentrations analyzed in the initial calibration?	<u> 1</u>	i denia un	21 12-14	
IV. Continuing calibration	ı .			
What type of continuing calibration calculation was performed? %D or%R				
Were Evaluation mix standards analyzed prior to the initial calibration and sample analysis?				
Were endrin and 4,4-DDT breakdowns ≤ 15% for individual breakdown in the Evaluation mix standards?				
Was a continuing calibration analyzed daily?	1	, .		
Were all percent differences (%D) ≤ 20% or percent recovieries 80-120%?				
Were all the retention times within the acceptance windows?			j	
V. Blanks	,			
Was a method blank associated with every sample in this SDG?	1			
Was a method blank analyzed for each matrix and concentration?	1			
Were extract cleanup blanks analyzed with every batch requiring clean-up?				
Was there contamination in the method blanks or clean-up blanks? If yes, please see the Blanks validation completeness worksheet.				
VI Starrogale spikes				
Were all surrogate %R within the QC limits?				
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?				
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?				
All Martiny crystal Martiny solike displicates				

LDC #: 244403.a.

## VALIDATION FINDINGS CHECKLIST

Page 2 of 2
Reviewer WA
2nd Reviewer

					<u></u>
Validation Area	Yes	No	NA	Findings/Comments	
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water	/				
Was a MS/MSD analyzed every 20 samples of each matrix?	//				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				A 3.00 B	
VIII. Laboratory control samples	1				
Was an LCS analyzed for this SDG?					
Was an LCS analyzed per extraction batch?	1				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?					
IX. Regional Quality Assurance and Quality Control					
Were performance evaluation (PE) samples performed?	<u></u>				_
Were the performance evaluation (PE) samples within the acceptance limits?		20.000			
X. Target compound identification	ı				
Were the retention times of reported detects within the RT windows?					
XI. Compound quantitation/CRQLs					
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions, dry weight factors, and clean-up activities applicable to level IV validation?					
XII. System performance					
System performance was found to be acceptable.					
XIII. Overall assessment of data					
Overall assessment of data was found to be acceptable.					**********
XIV: Field duplicates					
Field duplicate pairs were identified in this SDG.					
Target compounds were detected in the field duplicates.					
XV: Field blanks		,			
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)

А аюна-внс	I. Dieldrin	O Endrin kelone	Y. Aroclor-1242	GG Chlordane
B beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH Chlordane (Technical)
C celta-BHC	K. Endrin	S alpha-Chlordane	AA Aroclor-1254	
О уатта-ВНС	L Endosulfan II	T gamma-Chlordane	BB Aroclor-1260	JJ
E Heptachlor	M. 4,4'-DDD	U Toxaphene	CC 2.4:DDD	XX
F Aldrin	N Endosulfan sulfate	V Aroctor-1016	OD 2.4.0DE	r.
G Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	EE. 2.4-DDT	MM
H Endosulfan I	P Methoxychlor	X Aroclor-1232	FF. Hexachlorobenzene	ZZ

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Notes:

LDC # 2444963 SDG #: 561C METHOD: № GC HPLC

## Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

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

Page:

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

YMNNA. Did the percent difference of detected compounds between two columns./detectors <40%? Did the percent difference of detected compounds between two columns./detectors < 40%?

	Compound Name	Sample ID	Limit (< 40%)	Qualifications
	8	77	<i>Ý.ES</i>	T 645/4 (1)
				X / 2/25
-				
	,			

# VALIDATION FINDINGS WORKSHEET

LDC #: 25/14/9/34

SDG #Sae COU

Page: 012

Reviewer: 12

Field Duplicates

METHOD: CC HPLC
N N/A Were field duplicate pairs identified in this SDG?
N N/A Were target compounds detected in the field duplicate pairs?

	Concentration	Concentration (US/Reg.)	%RPD	Qualification
punoduoo	W	h	rlmit SO	Parent ohly / All Samples
B	64.0	0.660	n. (n. (n. 1/2/0)	(0)
FF	2.1	9.3		(A) (A) (A) (A)
			2	A JOHO V COLV

	Concentration ( US/Kg )	(2/kg)	The port	Qualification
	c/	9/	Limit <u>- Lo</u>	Parent only / All Samples
+	6.6	/5/	2.9	Jdrts/A (fd)
0	7.3	0620	990	
tr 17	0,44	0.29 V	0.15	

LDC #: 4447654 SDG #: 500 Check

# VALIDATION FINDINGS WORKSHEET

Page: 2012

Reviewer: 2nd reviewer:

Field Duplicates

Parent only / All Samples Qualification "RPOP. A . LImit 2/8 0 2 Concentration ( VS/kg METHOD: CG HPLC
NNA Were field duplicate pairs identified in this SDG?
N N/A Were target compounds detected in the field duplicate pairs? 61 Compound

Trick and Co	Concentration (	%RPD	Qualification
		LIMIT	Parent only / All Samples

LDC # 24449C3 <... SDG# see cover

VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

EPA 8081 Pesticides METHOD:

۵

Parameter:

Linear

Order of regression:

y conc	4.00	10	25	90	92	100	
x area	47007	113757	286925	575154	8.65E+05	1.14E+06	
Points	Point 1	Point 2	Point 3	Point 4	Point 5	Point 6	
Compound	O				•		
Channel	Ch. A						
Date	11-Aug-10		•				

Regression Output: Regression Output:	itput:	Reported	
Constant	2335.32120	# O	
R Squared	0.99986	r^2 =	0.99900
		(X)q	a(X^2)
X Coefficient(s)	11398.00028		

Page: 1 of 4 Reviewer: 12

24449c3 q see cover

LDC #

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: 2-of
Reviewer: 5
2nd Reviewer: 5

EPA 8081 Pesticides METHOD:

Parameter: D

Order of regression:

Linear

	Channel	Compound	Points	area ×	conc
11-Aug-10	Ch. A	٥	Point 1	47007	4.00
	-		Point 2	113757	10
			Point 3	286925	52
			Point 4	575154	90
		•	Point 5	8.65E+05	15
			Point 6	1.14E+06	100

Regression Output: Regression Output:	Reported
Constant 2335.32120	E 0
R Squared 0.99986	r^2 = 0.99900
	b(X) a(X^2)
X Coefficient(s)	

Page: 3 of 4 Reviewer: U44 2nd Reviewer: 8

Validatin Findings Worksheet Initial Calibration Calculation Verification

Method: EPA 8081 Pesticides

LDC #: 24471/34 SDG #: 54 (Bulp

Compound:

0

		3	8	(X^2)
Date	Column	Response	Conc	Сопс
8/11/2010	В	59795.00	4.000	16
<u>.</u>		137046	10	100
		321682	25	625
		607290	50	2500
		883436	75	5625
		1123921	100	10000

	٠
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-	3
C	7
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- 5	"
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	_

Regression Output	utput	
Constant	U	8705.2718
Std Err of Y Est		
R Squared		0.9999607
Degrees of Freedom		
	co.	q
X Coefficient(s)	1.2932E+04	-1.766E+01
Std Err of Coef.		
		-
Correlation Coefficient		0.999980
Coefficient of Determination (r^2)		0.999961

Validat

LDC #: 24449C

Validatin Findings Worksheet Initial Calibration Calculation Verification

Reviewer: Ad-2nd Reviewer: Q

Page: 4 of 4

Method: EPA 8081 Pesticides

Compound:

0

H			<u> </u>			F		1
(X^2)	Conc	16	100	625	2500	5625	10000	
(X)	Conc	4.000	10	25	20	75	100	•
(λ)	Response	59795.00	137046	321682	607290	883436	1123921	
	Column	В					•	
	Date	8/11/2010						

Regression Output

Constant	ပ	8705.2718
Std Err of Y Est		
R Squared		0.9999607
Degrees of Freedom		
	æ	q
X Coefficient(s)	1.2932E+04	-1.766E+01
Std Err of Coef.		
Correlation Coefficient		0.999980
Coefficient of Determination (r^2)		0.999961

SDG #: 50 Course LDC # 2444PLZ

## Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Page: Reviewer:

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

Percent difference  $(%D) = 100 \cdot (N \cdot C)/N$ 

Where: N = \_\_ Initial Calibration Factor or \_\_ Nominal Amount (ng)
C = \_\_ Calibration Factor from Continuing Calibration Standard or \_\_ Calculated Amount (ng)

:		Callbration		Average CF/	Reported	Recalculated	Reported	Receiculated
**	Standard ID	Date/Time	Compound	ccv con	CF/Cane	CFIEGO	Q%	σ%
-	CAST-450	0/8/2/0	D (UA)	50.0	1.6/-	1.64	20%	8.1
1			O CAM)	500	43.9	23.0	<i>hi/</i>	h'/
			D (Ch B)	59.0	<i>47.4</i>	pth	S)	
			0 (Chro)	30.0	488	8,87	24	24
7								
	020F28d	1) rd 2/	D (Ch.A)	\$ C. C.	49.9	49.4	le C	4,0
	•	0	À	9.0	5/0	21.9	3.0	3.00
			D (OM)	\$0.40	48.6	186	17.6	4-7
ო			7	50.0	5.7	242	25	2.5
		-						
- 1	033F3301.D	01)76/8	48) A	500	\$2.3	50.3	5,0	1,4
			7	50.0	52.)	175	4.2	7.5
4			( ( ( ( ( ( ( ( ( ( ( ( ( ( ( ( ( ( (	0-28	5.85	18.5	1.5	3.1
			9	20.0	2/.0	57.0	2.0	2,0
								•

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 #:_	24449039
	See cover

## VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page:_	1 of [
Reviewer:	M
2nd reviewer:_	0.
	<b>پر</b>

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: 22

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	ļ
Tetrachioro-m-xylene	I A	20.00	15.9915	90	80	0
Tetrachloro-m-xylene	B	20.00	14.8173	7-4	74	0
Decachlorobiphenyl	A	20.00	19.4560	97	97	0
Decachlorobiphenyl	B	20.00	17.4009	87	87	0

Sample ID

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

Notes:			· ·	 
<del></del>	<del> </del>	<del></del>		

LDC # 2444 46-29 SDG # See COLR

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

2nd Reviewer: Page: Reviewer:

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

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

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

Where

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

SC = Sample concentration

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

MS/MSD samples:\_

MSD = Matrix spike duplicate

$\overline{}$		<u> </u>	1 <del></del>	<del></del>	Т	<del>-</del>	<del></del>		т	T	ì	1	<del></del>	T	i	1	_
MS/MSD RPD	Recalc.		_														
MS/I	RF	Reported	_	_	<u> </u>									:			
Duplicate	ecovery	Recalc.	12	2	1												
Matrix Spike Duplicate	Percent Recovery	Reported	18	E													
spike	ecovery	Recalc.	28	90				:									
Matrix spike	Percent Recovery	Reported	13	24													
ample	tration (2)	O MSD	13.4	13.8													
Spike Sample		MS	75.5	13.9													
Sample	(VS/KG.)	0-	Q×	S													
ke	46.7	U MSD	1.6	16.6	_												
Spike	( 00/0	MS	16.6	166													
	Compound		Gamma-BHC	4,4'-DDT	Aroclor 1260												

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#: <u>2444</u>9c3ん SDG#*SacColl*A <u>Laboratory Control Sample/Laboratory Control Sample Duplicate Results Verification</u>

of	Ø	9
Page:	Reviewer	2nd Reviewer:

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

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

% Recovery = 100\* (SSC-SC)/SA Where: SSC = Spiked seri

Where: SSC = Spiked sample concentration SA = Spike added

SC = Concentration

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

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

LCS/LCSD samples: 280-274

		] .	<u></u>	Τ	Ī	T	Τ	<del></del>	T-	Γ	<u> </u>	Τ
rcs/rcsp	RPD	Recalc										
LCS/I		Reported										
rcsp	Percent Recovery	Recalc.										
רכ	Percent	Reported										
S	.ecovary	Recalc.	8	10								
rcs	Percent Recovery	Reported	B	90								
Sample	(US/kd)	C LCSD	1/4	1/4								
Spiked	30) 30)	SOT	13.9	/2:/								
pike	(Vg /kg).	Ccsp	nlg	n G								
S At (UR	37)	CS	16-7	4.4								
	Compound		gamma-BHC	4,4'-DDT	Aroclor 1260							

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 #: 24449C 32 SDG #: Fee Cover

## VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	(_of
Reviewer:	PT .
2nd reviewer:	

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

YN 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?

ycax +6xxx.

a = -1.354 E1

b= 9.5298 E3

c= 90660254

Area = 
$$y = 43360$$
  
 $y = 3.62$ 

Example:

Sample I.D. 22 B:

Conc. = (43362) (om) (w) (3.62) (om) (w) (3.52) (.896)

= 1.3 vz/kg V

,					· <u> </u>
*	Sampio ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
			<del></del>		
			ļ		
			13 ox Keg		

Note:	 		<del> </del>	 <del></del>	
	 ·	·		<del></del>	<del></del>
	 		<del> </del>	 	

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 9 through August 10, 2010

LDC Report Date:

December 2, 2010

Matrix:

Soil/Water

Parameters:

Metals

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6290-1

## Sample Identification

SSAQ3-03-10BPC	SSAQ5-06-3BPC_FD	SSAJ3-05-12BPCMSD
SSAQ3-03-1BPC	SSAJ2-06-1BPC	
SSAQ3-03-5BPC	SSAJ2-06-3BPC	
SSAQ4-08-10BPC**	SSAJ2-06-5BPC	
SSAQ4-08-10BPC_FD	SSAJ3-05-12BPC	
SSAQ4-08-1BPC	SSAJ3-05-16BPC**	
SSAQ4-08-5BPC	SSAJ3-05-1BPC	
SSAQ4-10-10BPC	SSAJ3-05-5BPC	
SSAQ4-10-1BPC	SSAJ3-05-8BPC	
SSAQ4-10-5BPC	SSAJ3-07-12BPC	
SSAQ5-05-1BPC	SSAJ3-07-17BPC	
SSAQ5-05-2BPC	SSAJ3-07-1BPC	
SSAQ5-05-2BPC_FD	SSAJ3-07-5BPC	
SSAQ5-05-3BPC	SSAJ3-07-8BPC	
RSAQ5-1BPC	SB03-24BPC	
RSAQ5-2BPC	EB-08092010	•
RSAQ5-3BPC**	EB-08102010	
SSAQ5-06-1BPC	SSAQ3-03-10BPCMS	
SSAQ5-06-2BPC	SSAQ3-03-10BPCMSD	
SSAQ5-06-3BPC	SSAJ3-05-12BPCMS	

<sup>\*\*</sup>Indicates sample underwent Stage 4 review

### Introduction

This data review covers 39 soil samples and 2 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 Antimony, Arsenic, Barium, Beryllium, Cadmium, Chromium, Cobalt, Copper, Lead, Mercury, Molybdenum, Nickel, Selenium, Silver, Thallium, Vanadium, and Zinc.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004).

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.

Samples indicated by a double asterisk on the front cover underwent a Stage 4 review. A Stage 2B review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Stage 2B criteria since this review is based on QC data.

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a faise positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- 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. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

## III. Calibration

An initial calibration was performed.

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

## IV. Blanks

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

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
PB (prep blank)	Zinc	0.430 mg/Kg	SB03-24BPC
ICB/CCB	Antimony Selenium	3.42 ug/L 5.55 ug/L	SB03-24BPC
PB (prep blank)	Barium	0.710 ug/L	All water samples in SDG 280-6290-1
ICB/CCB	Selenium	8.29 ug/L	All water samples in SDG 280-6290-1

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
EB-08092010	Barium Selenium	0.59 ug/L 9.4 ug/L	10U ug/L 15U ug/L
EB-08102010	Barium	1.1 ug/L	10U ug/L

Samples EB-08092010 and EB-08102010 were identified as equipment blanks. No metal contaminants were found in these blanks with the following exceptions:

Equipment Blank ID	Sampling Date	Analyte	Concentration	Associated Samples
EB-08092010	8/9/10	Barium Selenium	0.59 ug/L 9.4 ug/L	No associated samples in this SDG
EB-08102010	8/10/10	Barium	1.1 ug/L	No associated samples in this SDG

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

The frequency of analysis was met.

The criteria for analysis were met.

## VI. 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.

## VII. Duplicate Sample Analysis

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

## VIII. Laboratory Control Samples (LCS)

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

## IX. Internal Standards

All internal standard percent recoveries (%R) were within QC limits.

## X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

## XI, ICP Serial Dilution

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

## XII. Sample Result Verification and Project Quantitation Limit

All sample result verifications were acceptable for samples on which a Stage 4 review was performed.

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6290-1	All analytes reported below the PQL.	J (all detects)	А

Raw data were not evaluated for the samples reviewed by Stage 2B 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 SSAQ4-08-10BPC\*\* and SSAQ4-08-10BPC\_FD, samples SSAQ5-05-2BPC and SSAQ5-05-2BPC\_FD, and samples SSAQ5-06-3BPC and SSAQ5-06-3BPC\_FD were identified as field duplicates. No metals were detected in any of the samples with the following exceptions:

	Concentrat	ion (mg/Kg)				-
Analyte	SSAQ4-08-10BPC**	SSAQ4-08-10BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	15	14	7 (≤50)	-	-	-

	Concentration (mg/Kg)					
Analyte	SSAQ5-05-2BPC	SSAQ5-05-2BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	3.1	3.1	0 (≤50)	·	<del>-</del>	-

	Concentration (mg/Kg)					<del>_</del> :
Analyte	SSAQ5-06-3BPC	SSAQ5-06-3BPC	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	3.2	3.5	9 (≤50)	-	-	-

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Metals - Data Qualification Summary - SDG 280-6290-1

SDG	Sample	Analyte	Flag	A or P	Reason
280-6290-1	SSAQ3-03-10BPC SSAQ3-03-18PC SSAQ3-03-5BPC SSAQ4-08-10BPC** SSAQ4-08-10BPC_FD SSAQ4-08-1BPC SSAQ4-08-5BPC SSAQ4-10-10BPC SSAQ4-10-10BPC SSAQ4-10-5BPC SSAQ5-05-1BPC SSAQ5-05-2BPC_FD SSAQ5-05-2BPC_FD SSAQ5-05-3BPC RSAQ5-3BPCC RSAQ5-3BPCC RSAQ5-06-3BPC SSAQ5-06-3BPC SSAJ2-06-3BPC SSAJ2-06-3BPC SSAJ2-06-3BPC SSAJ3-05-16BPC** SSAJ3-05-16BPC SSAJ3-05-18PC SSAJ3-05-18PC SSAJ3-05-18PC SSAJ3-05-18PC SSAJ3-05-18PC SSAJ3-07-17BPC SSAJ3-07-18PC SB03-24BPC EB-08092010 EB-08102010	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (PQL) (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Metals - Laboratory Blank Data Qualification Summary - SDG 280-6290-1

SDG	Sample	Analyte	Modified Final Concentration	A or P	Code
280-6290-1	EB-08092010	Barium Selenium	10U ug/L 15U ug/L	Α	bi
280-6290-1	EB-08102010	Barium	10U ug/L	Α	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Metals – Equipment Blank Data Qualification Summary - SDG 280-6290-1

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson** VALIDATION COMPLETENESS WORKSHEET

Stage 2B/4

	Date: 12-1-10	_
2nd	Page: \ of \ Reviewer: \ \ Reviewer: \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	/

SDG #: 280-6290-1 Laboratory: Test America

24449A4

LDC #:\_

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 8/9-10/10
11.	ICP/MS Tune	A	
111.	Calibration	A	
IV.	Blanks	Su	
V.	ICP Interference Check Sample (ICS) Analysis	A	
VI.	Matrix Spike Analysis	A	ms Q
VII.	Duplicate Sample Analysis	$\mathcal{N}_{\perp}$	
VIII.	Laboratory Control Samples (LCS)	A	LCS
IX.	Internal Standard (ICP-MS)	A	
X.	Furnace Atomic Absorption QC	$\mathcal{N}$	Notutined
XI.	ICP Serial Dilution	A	
XII.	Sample Result Verification	7)	Not reviewed for Stage 2B validation.
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	SW	(4,5), (12,13), (20,21)
ΧV	Field Blanks	SW	FB= 36,37

Note:

A = Acceptable

N = Not provided/applicable

ND = No compounds detected R = Rinsate

D = Duplicate

TB = Trip blank

SW = See worksheet

FB = Field blank

EB = Equipment blank

Validated Samples: \*\* Indicates sample underwent Stage 4 validation

	Sollwares		<del> </del>				
1	SSAQ3-03-10BPC 5	11	SSAQ5-05-1BPC 5	21	SSAQ5-06-3BPC_FD 5	31	SSAJ3-07-17BPC 5
2	SSAQ3-03-1BPC	12	SSAQ5-05-2BPC	22	SSAJ2-06-1BPC	32	SSAJ3-07-1BPC
3	SSAQ3-03-5BPC	13	SSAQ5-05-2BPC_FD	23	SSAJ2-06-3BPC	33	SSAJ3-07-5BPC
4	SSAQ4-08-10BPC**	14	SSAQ5-05-3BPC	24	SSAJ2-06-5BPC	342	SSAJ3-07-8BPC
5	SSAQ4-08-10BPC_FD	15	RSAQ5-1BPC	25	SSAJ3-05-12BPC	353	SB03-24BPC
6	SSAQ4-08-1BPC	16	RSAQ5-2BPC	26	SSAJ3-05-16BPC**	364	EB-08092010 W
7	SSAQ4-08-5BPC	17	RSAQ5-3BPC**	27	SSAJ3-05-1BPC	37 4	EB-08102010 W
8	SSAQ4-10-10BPC	182	SSAQ5-06-1BPC	28	SSAJ3-05-5BPC	38	SSAQ3-03-10BPCMS S
9	SSAQ4-10-1BPC	19	SSAQ5-06-2BPC	29	SSAJ3-05-8BPC	39	SSAQ3-03-10BPCMSD
10	SSAQ4-10-5BPC	20	SSAQ5-06-3BPC	30	SSAJ3-07-12BPC	40	SSAJ3-05-12BPCMS
						41	SSAJ3-05-12BPCMSD

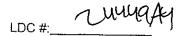
Notes:	& BM
	₽®5
	Gox ?
24449A4W wnd	(DOS)

LDC#: 2449A4

### VALIDATION FINDINGS CHECKLIST

Page: of Reviewer: 2nd Reviewer:

Method:Metals (EPA SW 846 Method 6010B/7000/6020)				
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.		-		
Cooler temperature criteria was met.			Ì	·
II. ICP/MS Tune				
Were all isotopes in the tuning solution mass resolution within 0.1 amu?		,		
Were %RSD of isotopes in the tuning solution ≤5%?				
III. Calibration				
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) QC limits?		^		
Were all initial calibration correlation coefficients > 0.995?				
IV. Blanks				
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.		<u></u>		•
V. ICP Interference Check Sample				•
Were ICP interference check samples performed daily?				
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?				
VI. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.		)		
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.				
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq$ 20% for waters and $\leq$ 35% for soil samples? A control limit of +/- RL(+/-2X RL for soil) was used for samples that were $\leq$ 5X the RL, including when only one of the duplicate sample values were $\leq$ 5X the RL.				
VII. Laboratory control samples				•
Was an LCS anaylzed for this SDG?				
Was an LCS analyzed per extraction batch?	7			
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?				· ·



### **VALIDATION FINDINGS CHECKLIST**

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Validation Area	Yes	No	NA	Findings/Comments
VIII. Furnace Atomic Absorption QC				
If MSA was performed, was the correlation coefficients > 0.995?				
Do all applicable analysies have duplicate injections? (Level IV only)				
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)			/	
Were analytical spike recoveries within the 85-115% QC limits?				
IX. ICP Serial Dilution				
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the MDL (ICP)/>100X the MDL(ICP/MS)?				
Were all percent differences (%Ds) < 10%?				
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.				
X. Internal Standards (EPA SW 846 Method 6020/EPA 200.8)		<del></del>		
Were all the percent recoveries (%R) within the 30-120% (6020)/60-125% (200.8) of the intensity of the internal standard in the associated initial calibration?				
If the %Rs were outside the criteria, was a reanalysis performed?				
XI. Regional Quality Assurance and Quality Control		<del></del>		
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?			/	
XII. Sample Result Verification			,	
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.				
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.	1	Ĺ		
Target analytes were detected in the field duplicates.				
XV. Field blanks				
Field blanks were identified in this SDG.	1			
Target analytes were detected in the field blanks.	1			

LDC #: 24449A4

### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

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All circled elements are applicable to each sample.

Sample ID Ma	
1-34	Al, Sb (As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Sì, CN,
35	Al, Sb)As, Ba Be Ca, Ca Cr)Co, Cu Fe Pb, Mg, Mn (Hg, Ni) K, Se, Ag Na, Ti, V, Zn, Mo, B, Si, CN',
3631	Al, Sb, (As, Ba) Be, Co, Ca, Cr, Co, Cu, Fe, (Pb) Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
Q03641	Al, Sb. (As) Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb. As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN.
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Ai, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN
	Analysis Method
ICP	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe(Pb), Mg, Mn, Hg, (Ni) K, Se, Ag, Na, Tl, V, Zn, Mo) B, Si, CN;
ICP-MS	Al, Sb, (As) Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
GEAA	Al Sh As Ba Be Cd Ca Cr Co Cu Fe Ph Mg Mn Hg Ni K Se Ag Na Tl V Zn Mo B Si CN

Comments: Mercury by CVAA if performed

### LDC #: 24449A4

METHOD: Trace metals (EPA SW 864 Method 6010B/6020/7000)

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

Soil preparation factor applied: 100x x 5x dil

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Associated Samples: No Qualifiers Sample Concentration units, unless otherwise noted: mg/Kg Action Limit Maximum ICB/CCB<sup>a</sup> (ug/L) 3.42 5.55 Maximum PB<sup>a</sup> (ug/L) Maximum PB<sup>a</sup> (mg/Kg) 0.430 Analyte S Š Z

Sample Con	centration ur	nits, unless c	Sample Concentration units, unless otherwise noted: ug/l	ted: ug/L	As	Associated Samples:	s: All Water	
Analyte	Maximum PB <sup>a</sup> (mg/Kg)	Maximum PB <sup>a</sup> (ug/L)	Maximum ICB/CCB <sup>a</sup> (ug/L)	Action	36	37		
Ва		0.710			0.59 / 10	1.1 / 10		
Se			8.29		9.4 / 15			

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 analysis of each element.

LDC #: 24449A4

## VALIDATION FINDINGS WORKSHEET

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2nd Reviewer:

Field Blanks

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

Were field blanks identified in this SDG? YN NA

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

Blank units: ug/L Associated sample units: mg/Kg Sampling date: 8/9/10 Soil factor applied 100x Field blank type: (circle one) Field Blank / Rinsate / Other.

EB

No associated samples Associated Samples: Sample Identification No Qualifiers Action Level Blank ID 0.59 9.4 99 Analyte Se Ва

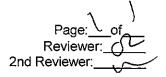
No associated samples Associated Samples: 100x Field blank type: (circle one) Field Blank / Rinsate / Other.\_ Soil factor applied \_ Sampling date: 8/10/10

	tion		
	Sample Identifical		
	Sa		
)			
		No Qualifiers	
		Action Level	
	Blank ID	37	1.1
	Analyte		Ba

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#: 24449A4

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



METHOD: Metals (EPA Method 6020/7000)

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

	Concentration	on (mg/Kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Compound	4	5	RPD	Difference	Limits	(Parent Only)
Arsenic	15	14	7			

V:\FIELD DUPLICATES\FD\_inorganic\24449A4.wpd

	Concentration	on (mg/Kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Compound	12	13	RPD	Difference	Limits	(Parent Only)
Arsenic	3.1	3.1	0			

	Concentration	on (mg/Kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Compound	20	21	RPD	Difference	Limits	(Parent Only)
Arsenic	3.2	3.5	9			

LDC# SYMPHY

# VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

Page: of Beviewer: CS

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 = Found x 100

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

Reported %R 0 Recalculated % (0) True (ug/L) Found (ug/L) <u>5</u> Element ICP/MS (Initial calibration) Type of Analysis ICPI(Initial calibration) Standard ID

Acceptable (Y/N)

 $\overline{S}$ 

5

8

**%** 

AS

ICP/MS (Continuing calibration)

3

ICP (Continuing calibration)

CVAA (Initial calibration)

CVAA (Continuing calibration)

GFAA (Continuing calibation)

GFAA (Initial calibration)

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

LPC#: 244/897

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

2nd Reviewer.\_ Reviewer.

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

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

 $%R = Found \times 100$ True

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

True = Concentration of each analyte in the source.

RPD = <u>[S-D]</u> × 100 (S+D)/2

Where,

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

S = Original sample concentration D = Duplicate sample concentration

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

%D = [I-SDR] x 100

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

					Recalculated	Reported	
Sample ID	Type of Analysis	Element	Found 1 S 11	True / D / SDR (units)	%R / RPD / %D	%R/RPD/%D	Acceptable (Y/N)
7588	ICP interference check	As	phroge	100 mg/L	)-101	الاد(	)-
527	Laboratory control sample		18'0	700	95	95	
38	Matrix spike		(SSR-SR)	9,61	95	95	
BUX	Duplicate		6172	1.12	ک	h	
>	ICP serial dilution	$\rightarrow$	88	3.28	-		

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#: Unughy

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

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2nd reviewer:_	~

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

Y N N/ Y N N/ Y N N/	A Have results b A Are results wit A Are all detection	vior all questions answered "N". Not ap leen reported and calculated correctly? hin the calibrated range of the instrument on limits below the CRDL?	nts and within the line	ear range of the IC	P?
Detected equation	I analyte results for :		were recalcu		
=V = n, Vol. =	(In. Vol.)  Raw data concent Final volume (ml)	Recalculation: tration or weight (G)	(1,09g)(0,0	1000 P)	. = 14,63 m
#	Sample ID	Analyte	Reported Concentration (mg/ks	Calculated Concentration	Acceptable (Y/N)
	니	A5	15	15	<u> </u>
				•	
			1		<u> </u>
Note:					

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

Project/Site Name: Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

Collection Date: August 12 through August 13, 2010

**LDC Report Date:** December 10, 2010

Matrix: Soil/Water

Parameters: Arsenic and Manganese

Validation Level: Stage 2B & 4

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6385-1

### Sample Identification

BDT-4-S-10-10BPC BDT-4-S-15-6BPC BDT-4-S-10-12BPC BDT-4-S-15-8BPC BDT-4-S-10-14BPC EB-08122010 BDT-4-S-10-16BPC SSAL8-02-1BPCMS BDT-4-S-10-18BPC\*\* SSAL8-02-1BPCMSD BDT-4-S-10-2BPC BDT-4-S-15-10BPCMS BDT-4-S-10-4BPC BDT-4-S-15-10BPCMSD BDT-4-S-10-6BPC EB-08122010MS BDT-4-S-10-8BPC EB-08122010MSD SSAL8-02-10BPC\*\* SSAL8-02-1BPC SSAL8-02-5BPC BDT-4-S-15-10BPC BDT-4-S-15-10BPC FD BDT-4-S-15-12BPC BDT-4-S-15-14BPC BDT-4-S-15-16BPC BDT-4-S-15-18BPC\*\* BDT-4-S-15-2BPC BDT-4-S-15-4BPC

<sup>\*\*</sup>Indicates sample underwent Stage 4 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 Methods 6020 for Arsenic and Manganese.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004).

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.

Samples indicated by a double asterisk on the front cover underwent a Stage 4 review. A Stage 2B review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Stage 2B criteria since this review is based on QC data.

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- 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. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

### III. Calibration

An initial calibration was performed.

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

### IV. Blanks

Method blanks were reviewed for each matrix as applicable. No arsenic or manganese contaminants were found in the initial, continuing and preparation blanks with the following exceptions:

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
PB (prep blank)	Manganese	0.151 mg/Kg	BDT-4-S-10-10BPC BDT-4-S-10-12BPC BDT-4-S-10-14BPC BDT-4-S-10-16BPC BDT-4-S-10-18BPC** BDT-4-S-10-2BPC BDT-4-S-10-4BPC BDT-4-S-10-6BPC BDT-4-S-10-8BPC SSAL8-02-10BPC** SSAL8-02-1BPC SSAL8-02-1BPC
PB (prep blank)	Manganese	0.0464 mg/Kg	BDT-4-S-15-10BPC BDT-4-S-15-10BPC_FD BDT-4-S-15-12BPC BDT-4-S-15-14BPC BDT-4-S-15-16BPC BDT-4-S-15-18BPC** BDT-4-S-15-2BPC BDT-4-S-15-6BPC BDT-4-S-15-6BPC BDT-4-S-15-6BPC

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
ICB/CCB	Manganese	1.45 ug/L	BDT-4-S-10-6BPC BDT-4-S-10-8BPC SSAL8-02-10BPC** SSAL8-02-1BPC SSAL8-02-5BPC

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

Samples EB-08122010 and EB-08132010 (from SDG 280-6415-1) were identified as equipment blanks. No arsenic or manganese contaminants were found in these blanks with the following exceptions:

Equipment Blank ID	Sampling Date	Analyte	Concentration	Associated Samples
EB-08132010	8/13/10	Manganese	2.5 ug/L	BDT-4-S-10-10BPC BDT-4-S-10-12BPC BDT-4-S-10-14BPC BDT-4-S-10-16BPC BDT-4-S-10-18BPC** BDT-4-S-10-2BPC BDT-4-S-10-4BPC BDT-4-S-10-6BPC BDT-4-S-10-6BPC BDT-4-S-15-10BPC BDT-4-S-15-10BPC BDT-4-S-15-10BPC BDT-4-S-15-16BPC BDT-4-S-15-15-16BPC BDT-4-S-15-15-15-15-15-15-15-15-15-15-15-15-15-
EB-08122010	8/12/10	Manganese	1.0 ug/L	SSAL8-02-10BPC** SSAL8-02-1BPC SSAL8-02-5BPC

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

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

The frequency of analysis was met.

The criteria for analysis were met.

### VI. 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.

### VII. Duplicate Sample Analysis

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Internal Standards

All internal standard percent recoveries (%R) were within QC limits.

### X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

### XI. ICP Serial Dilution

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

### XII. Sample Result Verification and Project Quantitation Limit

All sample result verifications were acceptable for samples on which a Stage 4 review was performed.

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6385-1	All analytes reported below the PQL.	J (all detects)	Α

Raw data were not evaluated for the samples reviewed by Stage 2B 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 BDT-4-S-15-10BPC and BDT-4-S-15-10BPC\_FD were identified as field duplicates. No arsenic and manganese were detected in any of the samples with the following exceptions:

	Concentra	tion (mg/Kg)			***	
Analyte	BDT-4-S-15-10BPC	BDT-4-S-15-10BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	3.9	3.8	3 (≤50)	-	-	-
Manganese	690	760	10 (≤50)	-	-	-

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic and Manganese - Data Qualification Summary - SDG 280-6385-1

SDG	Sample	Anaiyte	Flag	A or P	Reason
280-6385-1	BDT-4-S-10-10BPC BDT-4-S-10-12BPC BDT-4-S-10-14BPC BDT-4-S-10-16BPC BDT-4-S-10-16BPC BDT-4-S-10-18BPC** BDT-4-S-10-8BPC BDT-4-S-10-8BPC BDT-4-S-10-8BPC BDT-4-S-10-8BPC SSAL8-02-10BPC** SSAL8-02-10BPC** SSAL8-02-15PC BDT-4-S-15-10BPC BDT-4-S-15-10BPC BDT-4-S-15-10BPC BDT-4-S-15-16BPC BDT-4-S-15-16BPC BDT-4-S-15-8BPC BDT-4-S-15-8BPC BDT-4-S-15-8BPC BDT-4-S-15-8BPC BDT-4-S-15-8BPC BDT-4-S-15-8BPC BDT-4-S-15-8BPC BDT-4-S-15-8BPC EB-08122010	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (PQL) (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic and Manganese - Laboratory Blank Data Qualification Summary - SDG 280-6385-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic and Manganese – Equipment Blank Data Qualification Summary - SDG 280-6385-1

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET**

LDC #: 24449B4 280-6385-1 Stage 2B/4 SDG #: Laboratory: Test America

Date:   2-   -	(
Page: <u></u> ∫of ∫	
Reviewer:	
2nd Reviewer:	

METHOD: As & Mn (EPA SW 846 Method 6020)

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

	Validation Area		Comments
l.	Technical holding times	A	Sampling dates: 8/12-13/10
11.	ICP/MS Tune	A	
111.	Calibration	A	
IV.	Blanks	SW	
V.	ICP Interference Check Sample (ICS) Analysis	A	
VI.	Matrix Spike Analysis	A	mSD
VII.	Duplicate Sample Analysis	N	
VIII.	Laboratory Control Samples (LCS)	A	LCS
IX.	Internal Standard (ICP-MS)	À	
Χ	Furnace Atomic Absorption QC	$\mathcal{N}$	Notulitzed
XI.	ICP Serial Dilution	A	'
XII.	Sample Result Verification	A	Not reviewed for Stage 2B validation.
XIII.	Overall Assessment of Data	A	·
XIV.	Field Duplicates	SW	(13,14)
χV	Field Blanks	SV	EB= 23, EB-08132010 (SDGN: 280-6415-1)

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

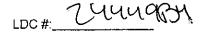
TB = Trip blank

EB = Equipment blank

Validated Samples: \*\* Indicates sample underwent Stage 4 validation

	501/water				
1	BDT-4-S-10-10BPC	11	SSAL8-02-1BPC 5	21	BDT-4-S-15-6BPC 5 31 PBW
2	BDT-4-S-10-12BPC	12	SSAL8-02-5BPC	22	BDT-4-S-15-8BPC J 32 (PBS)
3	BDT-4-S-10-14BPC	13	BDT-4-S-15-10BPC	23	P EB-08122010 W 33 DSSZ
4	BDT-4-S-10-16BPC	14	BDT-4-S-15-10BPC_FD	24	SSAL8-02-1BPCMS 5 34
5	BDT-4-S-10-18BPC**	15	BDT-4-S-15-12BPC	25	SSAL8-02-1BPCMSD 35
6	BDT-4-S-10-2BPC	16	BDT-4-S-15-14BPC	26	BDT-4-S-15-10BPCMS 36
7	BDT-4-S-10-4BPC	17	BDT-4-S-15-16BPC	27	BDT-4-S-15-10BPCMSD 37
8	BDT-4-S-10-6BPC	18	BDT-4-S-15-18BPC**	28	EB-08122010MS W 38
9	BDT-4-S-10-8BPC	19	BDT-4-S-15-2BPC	29	EB-08122010MSD \(\mathcal{D}\) 39
10	SSAL8-02-10BPC**	20	BDT-4-S-15-4BPC \(\begin{array}{c} \psi \\ \psi \\ \end{array}	30	40

Notes:	

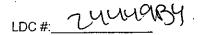


### **VALIDATION FINDINGS CHECKLIST**

Page: of Reviewer: 2nd Reviewer:

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

Method: Metals (EPA SW 846 Method 6010B/7000/6020)			<u> </u>	
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times	,—·			
All technical holding times were met.			ļ	
Cooler temperature criteria was met.		[		
II. ICP/MS Tune			<del> </del>	
Were all isotopes in the tuning solution mass resolution within 0.1 amu?		<b>.</b>		
Were %RSD of isotopes in the tuning solution ≤5%?				
III. Calibration				
Were all instruments calibrated daily, each set-up time?				
Were the proper number of standards used?			<u> </u>	
Were all initial and continuing calibration verification %Rs within the 90-110% (80-120% for mercury) QC limits?				
Were all initial calibration correlation coefficients > 0.995?				
IV. Blanks				
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.	/	<u> </u>		,
V. ICP Interference Check Sample				
Were ICP interference check samples performed daily?				
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?		<u> </u>		
VI. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.	/	h		
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq$ 20% for waters and $\leq$ 35% for soil samples? A control limit of +/- RL(+/-2X RL for soil) was used for samples that were $\leq$ 5X the RL, including when only one of the duplicate sample values were $\leq$ 5X the RL.				
VII. Laboratory control samples	.,	«; <u> </u>		•
Was an LCS anaylzed for this SDG?		<u> </u>	<u> </u>	
Was an LCS analyzed per extraction batch?		<u> </u>	<u> </u>	
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?				



### **VALIDATION FINDINGS CHECKLIST**

Page: Zof Z Reviewer: \_C 2nd Reviewer: \_\_\_\_

Validation Area	Yes	No	NA	Findings/Comments
VIII. Furnace Atomic Absorption QC				
If MSA was performed, was the correlation coefficients > 0.995?				
Do all applicable analysies have duplicate injections? (Level IV only)				
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)				
Were analytical spike recoveries within the 85-115% QC limits?		i		
IX. ICP Serial Dilution	<del></del>		<del></del>	
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the MDL (ICP)/>100X the MDL(ICP/MS)?	_			
Were all percent differences (%Ds) < 10%?		-		
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.				
X. Internal Standards (EPA SW 846 Method 6020/EPA 200.8)			· · · · · ·	
Were all the percent recoveries (%R) within the 30-120% (6020)/60-125% (200.8) of the intensity of the internal standard in the associated initial calibration?				
if the %Rs were outside the criteria, was a reanalysis performed?				
XI. Regional Quality Assurance and Quality Control	···	, <u>.</u>		
Were performance evaluation (PE) samples performed?		_		
Were the performance evaluation (PE) samples within the acceptance limits?	L	<u> </u>		1
XII. Sample Result Verification			<del></del>	
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/	<u></u>		
XIII. Overall assessment of data		-		
Overall assessment of data was found to be acceptable.	/			
XIV. Field duplicates	· ·	<del></del>	<del></del>	
Field duplicate pairs were identified in this SDG.	/			
Target analytes were detected in the field duplicates.			<u> </u>	
XV. Field blanks				
Field blanks were identified in this SDG.	/			
Target analytes were detected in the field blanks.	/	1_	<u> </u>	

LDC#: UMM9BY

### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

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

All circled elements are applicable to each sample.

	<del></del> 1	
Sample ID	Matrix	Target Analyte List (TAL)
1-23		Al, Sb, 🙉, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, 🦚, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN.
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN.
Q:24-29		Al, Sb, Ag, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mh, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> .
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> .
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN',
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN',
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	,	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		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,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		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.
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN.
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN
		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,
	<del></del>	Analysis Method.
ICP	ļ	Al, Sb, As, Ba, Bé, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN
ICP-MS		Al, Sb, 🔊 Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn) Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN.
GFAA	<u></u>	Al Sh As Ba Be Cd Ca Cr Co Cu Fe Ph Mg Mn Hg Ni K Se Ag Na Tl V Zn Mo B Si CN

Comments:	Mercury by CVAA if performed	

LDC #: 24449B4

METHOD: Trace metals (EPA SW 864 Method 6010B/6020/7000) Sample Concentration units, unless otherwise noted: mg/kg

VALIDATION FINDINGS WORKSHEET PB/ICB/CCB QUALIFIED SAMPLES

Associated Samples:

Soil preparation factor applied: 100x x 5x dil

Reviewer: 2nd Reviewer:

Page:\_

Analyte	Maximum PB³ (mg/Kg)	Maximum PB <sup>a</sup> (ug/L)	Maximum ICB/CCB* (ug/L)	Action Limit	No Qualifiers	
Mn	0.151					
Sample Cor	Sample Concentration units, unless otherwise noted: mg/Kg	nits, unless o	therwise not	ed: mg/Kg		Associated Samples: 13-22
Analyte	Maximum PB* (mg/Kg)	Maximum PB³ (ug/L)	Maximum ICB/CCB <sup>a</sup> (ug/L)	Action Limit	No Qualifiers	
Mn	0.0464					
Sample Cor	Sample Concentration units, unless otherwise noted: mg/Kg	nits, unless c	therwise not	ed: mg/Kg	Assoc	Associated Samples: 8-12
Analyte	Maximum PB* (mg/Kg)	Maximum PB <sup>a</sup> (ug/L)	Maximum ICB/CCB <sup>a</sup> (ug/L)	Action Limit	No Qualifiers	
Mn			1.45	0.725		

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".

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

LDC #: 24449B4

## VALIDATION FINDINGS WORKSHEET Field Blanks

2nd Reviewer:

Page:

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

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

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

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

/EB Soil factor applied 100x Sampling date: 8/13/10

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

1-9, 13-22

Associated Samples: Sample Identification No Qualifiers Action Level EB-08132010 Blank 1D 2.5 Analyte 퇻

10-12 Associated Samples: E) 100x Field blank type: (circle one) Field Blank / Rinsate / Other: Soil factor applied \_ Sampling date: 8/12/10

Sample Identification No Qualifiers Action Level Blank ID 1.0 23 Analyte ž

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#: 24449B4

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

2nd Reviewer:

METHOD: Metals (EPA Method 6020/7000)

<u>AN N N</u> Y N NA Were field duplicate pairs identified in this SDG? Were target analytes detected in the field duplicate pairs?

	Concentration	on (mg/Kg) -	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Compound	13	14	RPD	Difference	Limits	(Parent Only)
Arsenic	3,9	3.8	3			
Manganese	690	760	10			

V:\FIELD DUPLICATES\FD\_inorganic\24449B4.wpd

4064472 :# DOT

## Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

	S	7
Page:	Reviewer:_	and Reviewer:

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

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

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

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

					Recalculated	Reported	· · · · · · · · · · · · · · · · · · ·
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
TEN	ICP (Initial calibration)	Mn	6'017	Qoh	201	701	<i>&gt;</i> -
	ICP/MS (Initial calibration)						
	CVAA (Initial calibration)						
	ICP (Continuing calibration)						
(R. (23,54)	(C3:54) ICP/MS (Continuing calibration)	25	50,4	29	(0)	(0)	7
	CVAA (Continuing calibration)						
	GFAA (Initial calibration)						
	GFAA (Continuing calibation)						

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

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

2nd Reviewer: Reviewer:

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

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

%R = Found x 100 True

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

True = Concentration of each analyte in the source.

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

RPD = [S-D]\_ x 100 (S+D)/2

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

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

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

%D = [I-SDR] x 100

					Recalculated	Reported	
Sample ID	Type of Analysis	Element	Found 1 S / 1 K	True / D / SDR (units)	%R/RPD/%D	%R/RPD/%D	Acceptable (Y/N)
7588	ICP interference check		Mr believed	100 mg/c	101	ρl	<b></b>
537	Laboratory control sample	£	7.31	20	<b>ا</b> (	5	
77	Matrix spike	<u></u> )	(SSR-SR)	[9.1	89.	83	
SZIKZ	Duplicate	Mn	MSW	14200	7	. N	
	ICP serial dilution	M	14000	14300	7	$\sim$	7

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#: 24449BY

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page: of Reviewer: 2nd reviewer: V

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

Concentration = $\frac{(RD)(FV)(Dil)}{(ln. Vol.)}$ RD = Raw data concentration  FV = Final volume (ml)  In. Vol. = Initial volume (ml) or weight (G)  Recalculation: $(IOML)(5) \frac{781.7 \text{ upl}(5)}{1000000} = 35$	ollowing
In. Vol. = Initial volume (ml) or weight (G)  Dil = Dilution factor	83.7'
Reported Calculated Concentration Concentration Accepts # Sample ID Analyte (10016) (10016)	
14 AS 60 6.0 Y	
mo 380 380 J	
Note:	

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

Project/Site Name: Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

Collection Date: August 13 through August 16, 2010

**LDC Report Date:** December 10, 2010

Matrix: Soil/Water

Parameters: Arsenic and Manganese

Validation Level: Stage 2B & 4

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6415-1

### Sample Identification

EB-08162010 BDT-4-N-10-16BPC BDT-4-N-10-18BPC\*\* EB-08132010 BDT-4-N-15-2BPC BDT-4-N-10-2BPC BDT-4-N-15-2BPC FD BDT-4-N-10-4BPC BDT-4-N-15-4BPC BDT-4-N-10-6BPC BDT-4-N-15-6BPC BDT-4-N-10-8BPC BDT-4-S-20-2BPC EB-08162010MS BDT-4-S-20-4BPC EB-08162010MSD BDT-4-S-20-6BPC BDT-4-N-10-2BPCMS BDT-4-S-20-8BPC BDT-4-N-10-2BPCMSD BDT-4-S-20-10BPC BDT-4-S-20-12BPC BDT-4-S-20-14BPC BDT-4-S-20-16BPC BDT-4-S-20-18BPC\*\*

BDT-4-S-20-8BPC\_FD BDT-4-N-10-10BPC BDT-4-N-10-12BPC BDT-4-N-10-14BPC BDT-4-N-10-14BPC FD

<sup>\*\*</sup>Indicates sample underwent Stage 4 review

### Introduction

This data review covers 26 soil samples and 4 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Methods 6020 for Arsenic and Manganese.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004).

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.

Samples indicated by a double asterisk on the front cover underwent a Stage 4 review. A Stage 2B review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Stage 2B criteria since this review is based on QC data.

### The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- 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. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

### III. Calibration

An initial calibration was performed.

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

### IV. Blanks

Method blanks were reviewed for each matrix as applicable. No arsenic or manganese contaminants were found in the initial, continuing and preparation blanks with the following exceptions:

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
PB (prep blank)	Manganese	0.0641 mg/Kg	BDT-4-N-15-2BPC BDT-4-N-15-2BPC_FD BDT-4-N-15-4BPC BDT-4-N-15-6BPC BDT-4-S-20-2BPC BDT-4-S-20-6BPC BDT-4-S-20-6BPC BDT-4-S-20-10BPC BDT-4-S-20-12BPC BDT-4-S-20-14BPC BDT-4-S-20-14BPC BDT-4-S-20-16BPC BDT-4-S-20-16BPC BDT-4-S-20-16BPC BDT-4-N-10-10BPC BDT-4-N-10-10BPC BDT-4-N-10-14BPC BDT-4-N-10-14BPC BDT-4-N-10-14BPC BDT-4-N-10-14BPC BDT-4-N-10-16BPC BDT-4-N-10-16BPC BDT-4-N-10-16BPC BDT-4-N-10-16BPC BDT-4-N-10-16BPC BDT-4-N-10-16BPC BDT-4-N-10-2BPC
PB (prep blank)	Manganese	0.0738 mg/Kg	BDT-4-N-10-18BPC** BDT-4-N-10-4BPC BDT-4-N-10-6BPC BDT-4-N-10-8BPC

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

Samples EB-08162010 and EB-08132010 were identified as equipment blanks. No arsenic or manganese contaminants were found in these blanks with the following exceptions:

Equipment Blank ID	Sampling Date	Analyte	Concentration	Associated Samples
EB-08132010	8/13/10	Manganese	2.5 ug/L	BDT-4-S-20-2BPC BDT-4-S-20-4BPC BDT-4-S-20-6BPC BDT-4-S-20-8BPC BDT-4-S-20-10BPC BDT-4-S-20-12BPC BDT-4-S-20-14BPC BDT-4-S-20-16BPC BDT-4-S-20-18BPC** BDT-4-S-20-8BPC_FD
EB-08162010	8/16/10	Manganese	2.8 ug/L	BDT-4-N-15-2BPC BDT-4-N-15-2BPC_FD BDT-4-N-15-4BPC BDT-4-N-15-6BPC BDT-4-N-10-10BPC BDT-4-N-10-14BPC BDT-4-N-10-14BPC_FD BDT-4-N-10-16BPC BDT-4-N-10-18BPC** BDT-4-N-10-2BPC BDT-4-N-10-4BPC BDT-4-N-10-4BPC BDT-4-N-10-4BPC BDT-4-N-10-4BPC BDT-4-N-10-4BPC BDT-4-N-10-6BPC BDT-4-N-10-6BPC

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

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

The frequency of analysis was met.

The criteria for analysis were met.

### VI. 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.

### VII. Duplicate Sample Analysis

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Internal Standards

All internal standard percent recoveries (%R) were within QC limits.

### X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

### XI. ICP Serial Dilution

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

### XII. Sample Result Verification and Project Quantitation Limit

All sample result verifications were acceptable for samples on which a Stage 4 review was performed.

All analytes reported below the PQL were qualified as follows:

Sample	- Finding	Flag	A or P	
All samples in SDG 280-6415-1	All analytes reported below the PQL.	J (all detects)	Α	

Raw data were not evaluated for the samples reviewed by Stage 2B 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 BDT-4-N-15-2BPC and BDT-4-N-15-2BPC\_FD, samples BDT-4-S-20-8BPC and BDT-4-S-20-8BPC\_FD, and samples BDT-4-N-10-14BPC and BDT-4-N-10-14BPC\_FD were identified as field duplicates. No arsenic and manganese were detected in any of the samples with the following exceptions:

	Concentra	tion (mg/Kg)				
Analyte	BDT-4-N-15-2BPC	BDT-4-N-15-2BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	3.0	2.9	3 (≤50)	-	-	-
Manganese	390	430	10 (≤50)	-	-	-

	Concentra	tion (mg/Kg)				
Analyte	BDT-4-S-20-8BPC	BDT-4-S-20-8BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	3.3	3.2	3 (≤50)	<del>-</del>	-	-
Manganese	520	330	45 (≤50)	-	-	-

	Concentration (mg/Kg)				· · · · · · ·	
Analyte	BDT-4-N-10-14BPC	BDT-4-N-10-14BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	4.1	4.0	2 (≤50)	-	-	-
Manganese	350	420	18 (≤50)	-	-	-

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic and Manganese - Data Qualification Summary - SDG 280-6415-1

SDG	Sample	Analyte	Flag	AorP	Reason
280-6415-1	EB-08162010 EB-08132010 BDT-4-N-15-2BPC BDT-4-N-15-2BPC_FD BDT-4-N-15-6BPC BDT-4-N-15-6BPC BDT-4-S-20-2BPC BDT-4-S-20-6BPC BDT-4-S-20-6BPC BDT-4-S-20-10BPC BDT-4-S-20-10BPC BDT-4-S-20-16BPC BDT-4-S-20-16BPC BDT-4-S-20-18BPC** BDT-4-S-20-18BPCFD BDT-4-N-10-10BPC BDT-4-N-10-10BPC BDT-4-N-10-14BPC BDT-4-N-10-14BPC BDT-4-N-10-16BPC BDT-4-N-10-18BPC** BDT-4-N-10-18BPC** BDT-4-N-10-18BPC** BDT-4-N-10-18BPC BDT-4-N-10-18BPC BDT-4-N-10-18BPC BDT-4-N-10-18BPC BDT-4-N-10-18BPC BDT-4-N-10-18BPC BDT-4-N-10-18BPC BDT-4-N-10-18BPC BDT-4-N-10-6BPC BDT-4-N-10-6BPC BDT-4-N-10-6BPC BDT-4-N-10-6BPC BDT-4-N-10-8BPC	All analytes reported below the PQL.	J (all detects)	A	Sample résult verification (PQL) (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic and Manganese - Laboratory Blank Data Qualification Summary - SDG 280-6415-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic and Manganese – Equipment Blank Data Qualification Summary - SDG 280-6415-1

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson

	Hollox Not digate Heliderson
LDC #: 24449C4	<b>VALIDATION COMPLETENESS WORKSHEET</b>
SDG #: <u>280-6415-1</u>	Stage 2B/4
Laboratory: <u>Test America</u>	

Date: 10
Page:of
Reviewer:
2nd Reviewer:

METHOD: As & Mn (EPA SW 846 Method 6020)

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
Į.	Technical holding times	A	Sampling dates: 8/13-16/10
II.	ICP/MS Tune	A	·
III.	Calibration	A	
IV.	Blanks	SW	
V.	ICP Interference Check Sample (ICS) Analysis	A	
VI.	Matrix Spike Analysis	A	MSD
VII.	Duplicate Sample Analysis	N	
VIII.	Laboratory Control Samples (LCS)	A	LCS LCS
iX.	Internal Standard (ICP-MS)	A	719
X.	Furnace Atomic Absorption QC	Ν	NONULINZED
XI.	ICP Serial Dilution	A	
XII.	Sample Result Verification	B	Not reviewed for Stage 2B validation.
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	5W	(3,4), (10,16), (19,20)
ΧV	Field Blanks	SW	EB=1, 7

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 Stage 4 validation

	Soilling	1400							
1	EB-08162010	W	11	BDT-4-S-20-10BPC <		21	BDT-4-N-10-16BPC 5	31	PBW
2	EB-08132010	4	12	BDT-4-S-20-12BPC		22 22	BDT-4-N-10-18BPC**	32	PB51
3 *	BDT-4-N-15-2BPC	5	13	BDT-4-S-20-14BPC		23	BDT-4-N-10-2BPC	33	PBSZ
4	BDT-4-N-15-2BPC_F	D (	14	BDT-4-S-20-16BPC		24	BDT-4-N-10-4BPC	34	
5	BDT-4-N-15-4BPC		15	BDT-4-S-20-18BPC**		25	BDT-4-N-10-6BPC	35	
6	BDT-4-N-15-6BPC		16	BDT-4-S-20-8BPC_FD		26V	BDT-4-N-10-8BPC	36	
7	BDT-4-S-20-2BPC		17	BDT-4-N-10-10BPC		27	EB-08162010MS W	37	
8	BDT-4-S-20-4BPC		18	BDT-4-N-10-12BPC	:	28	EB-08162010MSD	38	
9	BDT-4-S-20-6BPC		19	BDT-4-N-10-14BPC		29	BDT-4-N-10-2BPCMS 5	39	
10	BDT-4-S-20-8BPC	$\overline{A}$	20	BDT-4-N-10-14BPC_FD		30	BDT-4-N-10-2BPCMSD	40	

Notes:	 			
_				

### **VALIDATION FINDINGS CHECKLIST**

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

Wethod: Metals (EPA SW 846 Method 6010B/7000/6020)			T	
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times	,			
All technical holding times were met.		-		
Cooler temperature criteria was met.			<u> </u>	
II. ICP/MS Tune			,	
Were all isotopes in the tuning solution mass resolution within 0.1 amu?		-		
Were %RSD of isotopes in the tuning solution ≤5%?	/			
III. Calibration				
Were all instruments calibrated daily, each set-up time?		2		
Were the proper number of standards used?	/			
Were all initial and continuing calibration verification %Rs within the 90-110% (80- 120% for mercury) QC limits?				
Were all initial calibration correlation coefficients > 0.995?				
IV. Blanks				
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.	~	, pol	CR	
V. ICP Interference Check Sample				
Were ICP interference check samples performed daily?				
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?	/			
VI. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	/		ļ 	
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.	/	<u></u>		
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq$ 20% for waters and $\leq$ 35% for soil samples? A control limit of +/- RL(+/-2X RL for soil) was used for samples that were $\leq$ 5X the RL, including when only one of the duplicate sample values were $\leq$ 5X the RL.	/			
VII. Laboratory control samples	<del>,</del>	<b></b>	<del></del>	•
Was an LCS anaylzed for this SDG?	/			
Was an LCS analyzed per extraction batch?	Ι,	<u> </u>	ļ	
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?	/			



### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 4

Reviewer: 6

2nd Reviewer: 4

Validation Area	Yes	No	NA	Findings/Comments
VIII. Furnace Atomic Absorption QC			<del>/</del>	
If MSA was performed, was the correlation coefficients > 0.995?				
Do all applicable analysies haye 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?				
IX. ICP Serial Dilution				
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the MDL (ICP)/>100X the MDL(ICP/MS)?				
Were all percent differences (%Ds) < 10%?				
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.				
X. Internal Standards (EPA SW 846 Method 6020/EPA 200.8)			,	
Were all the percent recoveries (%R) within the 30-120% (6020)/60-125% (200.8) of the intensity of the internal standard in the associated initial calibration?	_			
If the %Rs were outside the criteria, was a reanalysis performed?		l		
XI. Regional Quality Assurance and Quality Control	<b></b>	,,	· 	
Were performance evaluation (PE) samples performed?		/	ļ,	
Were the performance evaluation (PE) samples within the acceptance limits?	<u> </u>	<u> </u>		
XII. Sample Result Verification	,	·		
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XIII. Overall assessment of data		<del></del>	<u></u>	
Overall assessment of data was found to be acceptable.				
XIV. Field duplicates	T	1_	<del></del>	
Field duplicate pairs were identified in this SDG.	/	[	<u> </u>	
Target analytes were detected in the field duplicates.	/	<u>L</u>		
XV. Field blanks		,,		
Field blanks were identified in this SDG.		1_		
Target analytes were detected in the field blanks.	1	<u> </u>	<u> </u>	

LDC#: Zermach

### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

All circled elements are applicable to each sample.

<del> </del>	<del></del>	
Sample ID	Matrix	Toward American Line (TAL)
1-26	LIVIALTIX	Target Analyte List (TAL)
ļ	5	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
QC: 27-	$\mathcal{D}$	Al, Sb, (As) Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, (Mn) Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN',
<u> </u>		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN',
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	,	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, 8, Si, CN
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		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.
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Analysis Method
ICB		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.
ICP		
ICP-MS		Al, Sb, As) Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mg, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
GEAA	<u> </u>	Al Sh As Ba Be Cd Ca Cr Co Cu Fe Ph Mg Mn Hg Ni K Se Ag Na Ti V Zn Mo B Si CN

		•
Comments:_	Mercury by CVAA if performed	

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VALIDATION FINDINGS WORKSHEET PB/ICB/CCB QUALIFIED SAMPLES METHOD: Trace metals (EPA SW 864 Method 6010B/6020/7000)

Soil preparation factor applied: 100x x 5x dil Associated Samples: 3-21, 23

Reviewer:\_ 2nd Reviewer:

Sample Co	Sample Concentration units, unless otherwise noted: mg/Kg	nits, unless (	otherwise no	Sample Concentration units, unless otherwise noted: mg/Kg	Associated Samples: 3-21, 23	Zila Keviewei.
		. 0 1.2				
Analyte	Maximum PB* (mg/Kg)	Maximum PB <sup>a</sup> (ug/L)	Maximum ICB/CCB* (ug/L)	Action Limit	No Qualifiers	
Mn	0.0641					
Sample Co	Sample Concentration units, unless otherwise noted: mg/Kg	nits, unless o	otherwise no	ted: mg/Kg	Associated Samples: 22, 24-26	
Analyte	Maximum PB <sup>a</sup> (mg/Kg)	Maximum PB* (ug/L)	Maximum ICB/CCB <sup>a</sup> (ug/L)	Action Limit	No Qualifiers	

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.

0.0738

### LDC #: 24449C4

# VALIDATION FINDINGS WORKSHEET Field Blanks

Page:

2nd Reviewer:

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

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

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

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

Sampling date: 8/13/10 Soil factor applied 100x

7-16 Associated Samples: Sample Identification EBField blank type: (circle one) Field Blank / Rinsate / Other: No Qualifiers Action Level Blank ID 2.5 N Analyte 툴

3-6, 17-26 Associated Samples: Œ E 100x Field blank type: (circle one) Field Blank / Rinsate / Other. Soil factor applied Sampling date: 8/16/10

Sample Identification No Qualifiers Action Level Blank ID Analyte ž

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#: 24449C4

### VALIDATION FINDINGS WORKSHEET \_\_\_\_\_Field Duplicates

Page: of Reviewer: 2nd Reviewer:

METHOD: Metals (EPA Method 6020/7000)

YN NA YN NA Were field duplicate pairs identified in this SDG?

Were target analytes detected in the field duplicate pairs?

	Concentration	on (mg/Kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Compound	3	4	RPD	Difference	Limits	(Parent Only)
Arsenic ·	3.0	2.9	3			
Manganese	390	430	10			

V:\FIELD DUPLICATES\FD\_inorganic\24449C4.wpd

	Concentrati	on (mg/Kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Compound	<i>75</i> \ <sup>0</sup>	16	RPD	Difference	Limits	(Parent Only)
Arsenic	3.3	3.2	3			
Manganese	520	330	45			

	Concentration	on (mg/Kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Compound	19	20	RPD	Difference	Limits	(Parent Only)
Arsenic	4.1	4.0	2			
Manganese	350	420	18			

100 #: 54449CY

# VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

Page: of Beviewer: CS

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 = Found x 100 True

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

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
	ICP (Initial calibration)						
74	ICP/MS (Initial calibration)	AS	40,5	0°9	laı	101	)-
	CVAA (initial calibration)						
	ICP (Continuing calibration)						
3	ICP/MS (Continuing calibration)	WN	8'bh	SOD	(00)		)-
	CVAA (Continuing calibration)					)	
	GFAA (Initial calibration)						
	GFAA (Continuing calibation)						

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#:\_29:449CY

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

Page: 2nd Reviewer: Reviewer:

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

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

%R = Found x 100 True

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

True = Concentration of each analyte in the source.

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

RPD =  $|S-D|_X \times 100$ (S+D)/2

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

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

%D = ||-SDR| × 100

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

			·		Recalculated	Reported		
Sample ID	Type of Analysis	Element	Found / S / I	True / D / SDR (units)	%R/RPD/%D	%R/RPD/%D	Acceptable (Y/N)	
$\sim$	ICP interference check	£	John 9'bb	1004	926	9766	>	<del></del>
537	Laboratory control sample	£	2'61	20	R	96	·	
58	Matrix spike	$\Rightarrow$	(ssr-sr) $ 6/3 $	7.8	92	9		
2/52	Duplicate	Mn	084	hdh	.3	Y		
)	ICP serial dilution	$\rightarrow$	ムと	390	7	7	>	

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 #: VYUUGOM

### VALIDATION FINDINGS WORKSHEET <u>Sample Calculation Verification</u>

Page:_	of
Reviewer:_	a_
2nd reviewer:_	

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

Y N I	N/A Have results N/A Are results w N/A Are all detect ed analyte results for _	ow for all questions answered "N". been reported and calculated corr vithin the calibrated range of the in- tion limits below the CRDL?	rectly? struments an	d within the line	ar range of the IC	
Concent RD FV In. Vol. Dil	tration = \(\frac{(RD)(FV)(Dil)}{(In. Vol.)}\)  = Raw data conce = Final volume (m = Initial volume (m = Dilution factor	entration	(LOOML	75)(0,71 19)(6,80	(wok) = (	5.6mg/kg
#	Sample ID	Analyte	,	Reported Concentration (YX (3)	Calculated Concentration	Acceptable (Y/N)
	27	AS MA		5,6	260	
Note:						

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 9 through August 10, 2010

LDC Report Date:

December 2, 2010

Matrix:

Soil/Water

Parameters:

Chlorate and Perchlorate

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6290-1

### Sample Identification

SSAJ2-06-1BPC

SSAJ2-06-3BPC

SSAJ2-06-5BPC

SSAJ3-05-12BPC

SSAJ3-05-16BPC\*\*

SSAJ3-05-1BPC

SSAJ3-05-5BPC

SSAJ3-05-8BPC

SSAJ3-07-12BPC

SSAJ3-07-17BPC

SSAJ3-07-1BPC

SSAJ3-07-5BPC

SSAJ3-07-8BPC

SB03-24BPC

EB-08092010

EB-08102010

SSAJ3-05-12BPCMS

SSAJ3-05-12BPCMSD

SSAJ3-05-12BPCDUP

<sup>\*\*</sup>Indicates sample underwent Stage 4 review

### Introduction

This data review covers 17 soil samples and 2 water samples listed on the cover sheet. The analyses were per EPA SW 846 Method 9056A for Chlorate and EPA Method 314.0 for Perchlorate.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004).

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.

Samples indicated by a double asterisk on the front cover underwent a Stage 4 review. A Stage 2B review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Stage 2B criteria since this review is based on QC data.

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- 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 were met.

### b. Calibration Verification

Calibration verification frequency and analysis criteria were met.

### III. Blanks

Method blanks were reviewed for each matrix as applicable. No chlorate or perchlorate was found in the initial, continuing and preparation blanks.

Samples EB-08092010 and EB-08102010 were identified as equipment blanks. No chlorate or perchlorate was found in these blanks.

### 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.

### 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 and Project Quantitation Limit

All sample result verifications were acceptable for samples on which a Stage 4 review was performed.

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6290-1	All analytes reported below the PQL.	J (all detects)	Α

Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

### VIII. Overall Assessment

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

### IX. Field Duplicates

No field duplicates were identified in this SDG.

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorate and Perchlorate - Data Qualification Summary - SDG 280-6290-1

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-6290-1	SSAJ2-06-1BPC SSAJ2-06-3BPC SSAJ2-06-5BPC SSAJ3-05-12BPC SSAJ3-05-16BPC** SSAJ3-05-1BPC SSAJ3-05-5BPC SSAJ3-07-12BPC SSAJ3-07-17BPC SSAJ3-07-1BPC SSAJ3-07-5BPC SSAJ3-07-8BPC SSAJ3-07-8BPC SSAJ3-07-8BPC SBOJ-24BPC EB-08092010 EB-08102010	All analytes reported below the PQL.	J (all detects)	A .	Sample result verification (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorate and Perchlorate - Laboratory Blank Data Qualification Summary - SDG 280-6290-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorate and Perchlorate - Equipment Blank Data Qualification Summary - SDG 280-6290-1

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET**

LDC #: 24449A6 SDG #: 280-6290-1 Stage 2B/4 Laboratory: Test America

Date: 12-1-16
Page: <u> </u> t_of <u> </u>
Reviewer: 02
2nd Reviewer:

METHOD: (Analyte) Chlorate (EPA SW846 Method 9056A), Perchlorate (EPA Method 314.0)
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The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
1	Technical holding times	A	Sampling dates: 8/9-10/10
IIa.	Initial calibration	<del> </del>	•
llb.	Calibration verification	A	
III.	Blanks	A	
IV	Matrix Spike/Matrix Spike Duplicates	A	MSID
V	Duplicates	A	O.G.
VI.	Laboratory control samples	A	LCS/D
VII.	Sample result verification	A	Not reviewed for Stage 2B validation.
VIII.	Overall assessment of data	M	
IX.	Field duplicates	N	
x	Field blanks	NO	EB=15,16

Note:

A = Acceptable

SW = See worksheet

N = Not provided/applicable

ND = No compounds detected

R = Rinsate FB = Field blank

D = Duplicate TB = Trip blank

EB = Equipment blank

Validated Samples: \*\* Indicates sample underwent Stage 4 validation

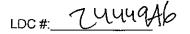
	Soillwa	<del>40</del> C							
1	SSAJ2-06-1BPC	3	11	SSAJ3-07-1BPC	5	21		31	
2	SSAJ2-06-3BPC		12	SSAJ3-07-5BPC	Ī	22		32	
3	SSAJ2-06-5BPC		13	SSAJ3-07-8BPC		23		33	
4	SSAJ3-05-12BPC		14	SB03-24BPC	1	24	•	34	
5	SSAJ3-05-16BPC**		15	EB-08092010	W	25		35	
6	SSAJ3-05-1BPC		16	EB-08102010	<u>&amp;</u>	26		36	
7	SSAJ3-05-5BPC		17	SSAJ3-05-12BPCMS	5	27		37	
8	SSAJ3-05-8BPC	[	18	SSAJ3-05-12BPCMSD		28		38	
9	SSAJ3-07-12BPC		19	SSAJ3-05-12BPCDUP		29		39	
10	SSAJ3-07-17BPC	<u> </u>	20			30		40	

Notes:		
		,

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

Method: Inorganics (EPA Method See Cover)

Method:Inorganics (EPA Method See Cover)				
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times		_		
All technical holding times were met.				
Cooler temperature criteria was met.	/	<u> </u>	<u></u>	
II. Calibration		<b>.</b>		
Were all instruments calibrated daily, each set-up time?		<u></u>		
Were the proper number of standards used?		<u> </u>		
Were all initial calibration correlation coefficients > 0.995?	Ĺ			
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)			/	
III. Blanks				
Was a method blank associated with every sample in this SDG?		<u>.</u> -		
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
IV. Matrix spike/Matrix spike duplicates and Duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.		<u></u>		
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.	/	<u> </u>	,	
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq$ 20% for waters and $\leq$ 35% for soil samples? A control limit of $\leq$ CRDL( $\leq$ 2X CRDL for soil) was used for samples that were $\leq$ 5X the CRDL, including when only one of the duplicate sample values were $\leq$ 5X the CRDL.	/			
V. Laboratory control samples				
Was an LCS anayized 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?				
VI. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				



### **VALIDATION FINDINGS CHECKLIST**

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

LDC#: ZyungAb

### VALIDATION FINDINGS WORKSHEET Sample Specific Analysis Reference

Page:_	of	1
Reviewer:_	Q	
2nd reviewer:_	<u></u>	$\simeq$

All circled methods are applicable to each sample.

Sample ID Matrix	<u>Parameter</u>
-13,15,16	pH TDS CI F NO, NO, SO, PO, ALK CN NH, TKN TOC CR6 CIO,
14	pH TDS CI F NO3 NO, SO4 PO4 ALK CN NH3 TKN TOC CR CIO, CLO3
	pH TDS CI F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> PO <sub>4</sub> ALK CN <sup>-</sup> NH <sub>3</sub> TKN TOC CR <sup>6+</sup> CIO <sub>4</sub>
x:17-19	PH TDS CI F NO, NO, SO, PO, ALK CN NH, TKN TOC CR (CIO,
	pH TDS CLF NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ CIO4
	PH TDS CLF NO3 NO2 SO4 PO4 ALK CN- NH3 TKN TOC CR6+ ClO4
	PH TDS CLF NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR8+ ClO4
	PH TDS CI F NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ CIO4
	PH TDS CI F NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ CIO4
	PH TDS CI F NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ CIO4
	pH TDS CI F NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ CIO4
	PH TDS CI F NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ CIO4
	pH TDS CI F NO3 NO, SO4 PO4 ALK CN NH3 TKN TOC CR6+ CIO4
	ph tds ci f No <sub>3</sub> No <sub>2</sub> So <sub>4</sub> Po <sub>4</sub> Alk CN <sup>-</sup> NH <sub>3</sub> TKN TOC CR <sup>6+</sup> Cio <sub>4</sub>
	ph tds ci f No <sub>3</sub> No <sub>2</sub> So <sub>4</sub> Po <sub>4</sub> Alk Cn <sup>-</sup> Nh <sub>3</sub> TKN TOC CR <sup>6+</sup> Clo <sub>4</sub>
	ph TDS CLF NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ ClO4
	PH TDS CLF NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ ClO4
	PH TDS CLF NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ ClO4
	PH TDS CI F NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ CIO4
	PH TDS CLF NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ ClO4
	ph tds ci f no3 no2 so4 po4 alk cn nh3 tkn toc cr6+ cio4
	ph tds ci f No <sub>3</sub> No <sub>2</sub> So <sub>4</sub> Po <sub>4</sub> Alk Cn <sup>-</sup> Nh <sub>3</sub> Tkn toc CR <sup>6+</sup> Cio <sub>4</sub>
	ph TDS CLF NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ ClO4
	pH TDS CI F NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ CIO4
	ph TDS CI F NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ CIO4
	pH TDS CI F NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ CIO4
	pH TDS CI F NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ CIO4
	ph TDS CI F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> PO <sub>4</sub> ALK CN' NH <sub>3</sub> TKN TOC CR <sup>6+</sup> CIO <sub>4</sub>
	PH TOS CLE NO. NO. SO, PO, ALK CN. NH. TKN TOC CR6+ CIO.

Comments:	

9416hhh # 2017

# Initial and Continuing Calibration Calculation Verification Validation Findings Worksheet

2nd Reviewer:

Method: Inorganics, Method 3140

The correlation coefficient (r) for the calibration of  $\overline{\mathbb{CU}_{\mathcal{U}}}$  was recalculated.Calibration date:\_

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

Where, %R = Found X 100

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

					Recalculated	Reported	Acceptable
Type of analysis	Analyte	Standard	Conc. (mg/l)	Reading	r or r²	r or r²	(V/N)
Initial calibration		s1	1.000	0.00303			
,		s2	2.5	0.00749	0.999410	0.999165	
	<u>3</u>	s3	5	0.02			$\sim$
	<u> </u>	s4	10	0.03			)
		SS	20	0.07			, <del>-</del>
		gs	40	0.15		•	
Calibration verification		TCO	2	Found(18/19   (	96		
Calibration verification	->	CCV	30	22.86	93	)	
Calibration verification	$\rightarrow$						

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

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

2nd Reviewer: Page: Cof Reviewer: (7-2)

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 = Found x 100

Where,

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

True = concentration of each analyte in the source.

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

Where,

S= D=

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

Original sample concentration Duplicate sample concentration

Sample ID	Type of Analysis	Element	Found / S / RS	True / D	Recalculated 900 / DDD	Reported	Acceptable
S27	Laboratory control sample	POTO	P180,0	(C)	2	hb	(NA)
7	Matrix spike sample		(SSR-SR)	5.106	90	3	-
101	Duplicate sample		6,15	0.165		121	)

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# YUU9Ab

### **VALIDATION FINDINGS WORKSHEET**

Sample Calculation Verification

	2nd reviewer:
METHOD: Inorganics, Method Sec Cover	<del></del>
Please see qualifications below for all questions answ Y N N/A Have results been reported and calculated Y N N/A Are results within the calibrated range Y/N N/A Are all detection limits below the CRC Compound (analyte) results for recalculated and verified using the following equation:	e of the instruments? QL? Cu reported with a positive detect were
Concentration = Reca	alculation:
-0,0037x-0,001	$\frac{(0.00317)(10)}{(0.899)(1000)} = 0.0125 \text{ mg/k}$
# Sample ID Analyte	Reported Calculated Concentration Concentration Acceptable (MC 100) (MC 129/ (Y/N)
5 Cloy	0.012 0.013 7
,	
Note:	

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 12, 2010

LDC Report Date:

December 2, 2010

Matrix:

Water

Parameters:

Perchlorate

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6385-1

Sample Identification

EB-08122010

### Introduction

This data review covers one water sample listed on the cover sheet. The analyses were per EPA Method 314.0 for Perchlorate.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004).

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.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- 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(a). Initial Calibration

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

### II(b). Calibration

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 perchlorate was found in the initial, continuing and preparation blanks.

Sample EB-08122010 was identified as an equipment blank. No perchlorate was found in this blank.

### IV. 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.

### V. Duplicates

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

### 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 analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6385-1	All analytes reported below the PQL.	J (all detects)	Α

Raw data were not reviewed for this SDG.

### VIII. Overall Assessment

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

### IX. Field Duplicates

No field duplicates were identified in this SDG.

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Perchlorate - Data Qualification Summary - SDG 280-6385-1

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-6385-1	EB-08122010	All analytes reported below the PQL.	J (all detects)	Α	Sample result verification (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Perchlorate - Laboratory Blank Data Qualification Summary - SDG 280-6385-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Perchlorate - Field Blank Data Qualification Summary - SDG 280-6385-1

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET**

LDC #: 24449B6 SDG #: 280-6385-1 Stage 2B Laboratory: Test America

Date: 12-1-10
Page: <u>\</u> of <u>\</u>
Reviewer: 2
2nd Reviewer:

<b>METHOD:</b> (Analyti	te) Perchlorate	(EPA Method 314.0	)

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: 8/12/10
IIa.	Initial calibration	0	
IIb.	Calibration verification	A	
111.	Blanks	17	
ΙV	Matrix Spike/Matrix Spike Duplicates	$\mathcal{N}$	Clientspecified
V	Duplicates	N N	1
VI.	Laboratory control samples	A	LCS/D
VII.	Sample result verification	N	•
VIII.	Overall assessment of data	A	
IX.	Field duplicates	$\mathcal{N}$	
_x_	Field blanks	NO.	EB=1

Note:

A = Acceptable

N = Not provided/applicable SW = See worksheet

ND = No compounds detected

R = Rinsate FB = Field blank D = Duplicate

TB = Trip blank

EB = Equipment blank

Validated Samples:

1	EB-08122010	11	BBW	21	31	
2		12		22	32	
3		13		23	33	
4		14		24	34	
5		15		25	 35	
6		16		26	36	
7		17		27	 37	
8		18		28	 38	
9		19		29	39	
10		20		30	40	

Notes:	