

## LABORATORY DATA CONSULTANTS, INC.

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

Northgate Environmental Management, Inc.

October 19, 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 fraction listed below. These SDGs were received on September 30, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

## **LDC Project # 24047:**

## SDG # Fraction 280-2280-10, 280-6223-1 Semivolatiles, Chlorinated Pesticides, Metals, Wet Chemistry 280-6290-2, 280-6290-3 280-6345-1/ITH1781 280-6535-1, 280-6583-1 280-6639-1, 280-6674-1 280-6741-1, 280-6783-1 280-6818-1,280-6851-1 280-6886-1

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** 

24047ST.wpd

Attachment 1

DL 09/30/10

Henderson NV / Tronox PCS Additional Sampling)   Henderson NV / Tronox PCS Additional Sampling   Henderson NV / Tronox	0 2 0 3 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0
Tronox PCS Additional Sampling	2 0 3 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0
Tronox PCS Additional Sampling    10	2 0 3 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0
Tronox PCS Additional Sampling    Chlorate	2 0 3 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0
Tronox PCS Additional Sampling    S	2 0 3 0 0 0 0 0 0 0 0 0 0 0 0 0
Tronox PCS Additional Sampling    1990   1995   1	2 0 3 0 0 0 0 0 0 0 0 0 0 0 0
Tronox PCS Additional Sampling   Chlorate   1990   9056A     S	2 0 3 0 0 0 0 0 0 0 0 0 0
Tronox PCS Additional Sampling   S   W   S	2 0 3 0 0 0 0 0 0 0 0 0
Tronox PCS Additional	2 0 3 0 0 0 0 0 0 0 0
Tronox PCS Additional	2 0 3 0 0 0 0 0 0
Tronox PCS Additional	2 0 3 0 0 0 0 0
Tronox PCS Additional	2 0 3 0 0 0 0
Chlorate	2 0 3 0 0 0 0
Chlorate	2 0 3 0 0 0
Chlorate	2 0 3 0 0
Chlorate	2 0 3 0
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CLO <sub>4</sub> Cr(VI) Chlorate (199) (9056A) (199) (	2 0
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4	
Stage 28/4   1-10   1-1	
SDG# Stage:  SDG#  C. Water/Soil  280-2280-10  280-6223-1  280-6223-1  280-6290-2  280-6290-3  280-6290-3  280-6535-1  280-6535-1  280-6535-1  280-6535-1  280-6535-1  280-6531-1  280-674-1  280-674-1  280-674-1  280-674-1  280-674-1  280-678-1  280-678-1  280-678-1  280-678-1  280-678-1  280-6818-1  280-6818-1  280-6886-1  280-6886-1	T/LR
SDG#  **: Water/Soil 280-6223-1 280-6223-1 280-6223-1 280-6290-2 280-6290-3 280-6290-3 280-6535-1 280-6535-1 280-6535-1 280-6533-1 280-6533-1 280-6531-1 280-674-1 280-674-1 280-674-1 280-674-1 280-674-1 280-674-1 280-674-1 280-678-1 280-678-1 280-678-1 280-678-1 280-6818-1 280-6818-1 280-6818-1 280-6818-1 280-6818-1 280-6886-1 280-6886-1	
	Total

## **EDD CHECKLIST**

LDC #: 24047

SDG #: 280-2280-10, 280-6223-1, 280-6290-2, 281-6290-3, 280-6345-1/ITH1781

280-6535-1, 280-6583-1, 280-6639-1, 280-6674-1, 280-6741-1

280-6783-1, 280-6818-1, 280-6851-1, 280-6886-1

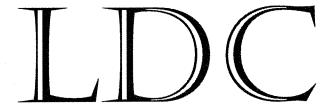
Page: 1 of 1 Reviewer: JE 2nd Reviewer: BC

## Tronox Northgate Henderson Worksheet

EDD Area	Yes	No	NA	Findings/Comments
I. Completeness				
Is there an EDD for the associated Tronox validation report?	X			
II. EDD Qualifier Population				
Were all qualifiers from the validation report populated into the EDD?	X			
III. EDD Lab Anomalies				
Were EDD anomalies identified?	X			
If yes, were they corrected or documented for the client?	X			See EDD_discrepancy_ form_LDC24047_101910.doc
IV. EDD Delivery				
Was the final EDD sent to the client?	Х			

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Data Validation Reports LDC #24047

Semivolatiles



## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 7 through August 9, 2010

LDC Report Date:

October 14, 2010

Matrix:

Soil/Water

Parameters:

Semivolatiles

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6223-1

## Sample Identification

SSAI3-02-5BPC FD SSAJ3-02-12BPC SSAI3-03-11BPC SSAJ3-02-15BPC SSAJ3-02-16BPC **SSAI3-03-14BPC** SSAJ3-02-19BPC\*\* SSAI3-02-5BPC SSAJ3-02-8BPC\*\* SSAI3-02-8BPC SSAI3-03-5BPC\*\* SSAJ3-02-8BPC FD SSAI3-03-8BPC EB-08072010 SSAI3-04-5BPC SSAI3-03-23BPC SSAI3-03-25BPC SSAI3-04-8BPC SSAI3-02-5BPC FDMS SSAI3-04-11BPC SSAI3-02-5BPC FDMSD SSAI3-04-14BPC SSAI3-03-8BPCMS SSAI3-04-14BPC FD SSAI3-03-8BPCMSD SSAI3-04-15BPC SSAI3-04-23BPC SSAI3-04-25BPC SSAI3-02-11BPC

SSAI3-02-14BPC SSAI3-02-15BPC SSAI3-02-19BPC SSAI3-02-25BPC

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

### Introduction

This data review covers 32 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

Samples indicated by a double asterisk on the front cover underwent a 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  $(r^2)$  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 280-26583/1-A	8/12/10	Dimethylphthalate	35,3 ug/Kg	SSAJ3-02-12BPC SSAJ3-02-15BPC SSAJ3-02-16BPC SSAJ3-02-16BPC** SSAJ3-02-8BPC** SSAJ3-02-8BPC_FD SSAI3-03-23BPC SSAI3-03-25BPC SSAI3-04-11BPC SSAI3-04-14BPC_FD SSAI3-04-15BPC SSAI3-04-15BPC SSAI3-04-15BPC SSAI3-02-11BPC SSAI3-02-15BPC SSAI3-02-15BPC SSAI3-02-15BPC SSAI3-02-25BPC SSAI3-02-25BPC_FD

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
SSAJ3-02-16BPC	Dimethylphthalate	43 ug/Kg	43U ug/Kg
SSAJ3-02-8BPC**	Dimethylphthalate	25 ug/Kg	25U ug/Kg
SSAI3-04-14BPC_FD	Dimethylphthalate	29 ug/Kg	29U ug/Kg
SSAI3-04-23BPC	Dimethylphthalate	30 ug/Kg	30U ug/Kg
SSAI3-02-25BPC	Dimethylphthalate	34 ug/Kg	34U ug/Kg

Sample EB-08072010 was identified as an equipment blank. 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.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6223-1	All compounds reported below the PQL.	J (all detects)	Α

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 SSAJ3-02-8BPC\*\* and SSAJ3-02-8BPC\_FD, samples SSAI3-04-14BPC and SSAI3-04-14BPC\_FD, and samples SSAI3-02-5BPC and SSAI3-02-5BPC\_FD were identified as field duplicates. No semivolatiles were detected in any of the samples with the following exceptions:

	Concentra	tion (ug/L)	200	D.11		A or P	
Compound	SSAJ3-02-8BPC**	SSAJ3-02-8BPC_FD	RPD (Limits)	Difference (Limits)	Flags		
Dimethylphthalate	25	340U	-	315 (≤340)	-		

	Concentra	ition (ug/L)		5.77			
Compound	SSAI3-04-14BPC	SSAI3-04-14BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P	
Dimethylphthalate	340U	29	· -	311 (≤340)	-	-	

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-6223-1

	Sample	Compound	Flag	A or P	Reason (Code)
280-6223-1	SSAJ3-02-12BPC	All compounds reported	J (all detects)	А	Project Quantitation Limit
	SSAJ3-02-15BPC	below the PQL.			(sp)
	SSAJ3-02-16BPC				
	SSAJ3-02-19BPC**				
	SSAJ3-02-8BPC**	<b>i</b>			
	SSAJ3-02-8BPC_FD	<u>;</u>			
	EB-08072010	1			ł
	SSAI3-03-23BPC				
	SSAI3-03-25BPC				
	SSAI3-04-11BPC	1			
	SSAI3-04-14BPC			l .	
	SSAI3-04-14BPC_FD				
	SSAI3-04-15BPC				·
	SSAI3-04-23BPC				ł
	SSAI3-04-25BPC				
	SSAI3-02-11BPC				
	SSAI3-02-14BPC				
	SSAI3-02-15BPC				
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	SSAI3-02-25BPC				
	SSAI3-02-5BPC_FD				į
	SSAI3-03-11BPC	•			<u>'</u>
	SSAI3-03-14BPC				[
	SSAI3-02-5BPC				
	SSAI3-02-8BPC				
	SSAI3-03-5BPC**				
	SSAI3-03-8BPC				
	SSAI3-04-5BPC				
	SSAI3-04-8BPC				1

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-6223-1

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
280-6223-1	SSAJ3-02-16BPC	Dimethylphthalate	43U ug/Kg	А	bl
280-6223-1	SSAJ3-02-8BPC**	Dimethylphthalate	25U ug/Kg	А	bl
280-6223-1	SSAI3-04-14BPC_FD	Dimethylphthalate	29U ug/Kg	А	bl
280-6223-1	SSAI3-04-23BPC	Dimethylphthalate	30U ug/Kg	Α	bl
280-6223-1	SSAI3-02-25BPC	Dimethylphthalate	34U ug/Kg	Α	bl

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Equipment Blank Data Qualification Summary - SDG 280-6223-1

No Sample Data Qualified in this SDG

## Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

LDC #:	24047B2a	VALIDATION COMPLETEN
SDG #:	280-6223-1	Stage 2B/
Laborato	ry: Test America	

Date: 10/05/6
Page: \_\_\_\_\_\_of \_\_\_
Reviewer: \_\_\_\_\_\_
2nd Reviewer: \_\_\_\_\_\_

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

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

	Validation Area	,	Comments
l.	Technical holding times	A	Sampling dates: 8/67 _ of /10
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	A	2 KSD r 25 % ca/14 = 25 %
IV.	Continuing calibration/ICV	A	ca/14 = 25?
V.	Blanks	≤n)	
VI.	Surrogate spikes	Α	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	us /b
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	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	Α	<b>Y</b>
XVI.	Field duplicates	SW	$D_1 = 5.6$ $D_2 = 11.12$ $D_3 = 24.21$ * $EB = 7$ $FB = FB = 040/2010 - RZD$
XVII.	Field blanks	MSA	*EB = 7 FB = FB = 040/2010_ RZD

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	_	7	Mer						
1	SSAJ3-02-12BPC	5	11 1	SSAI3-04-14BPC Pr S	21	1	SSAI3-02-5BPC_FD	Dys	31 <b>1</b>	SSAI3-02-5BPC_FDMSD S
2	SSAJ3-02-15BPC		12	SSAI3-04-14BPC_FD DV	<b>∤</b> 22	`	SSAI3-03-11BPC		32	SSAI3-03-8BPCMS
3 1	SSAJ3-02-16BPC		13	SSAI3-04-15BPC	23	2	SSAI3-03-14BPC		33	SSAI3-03-8BPCMSD
4	SSAJ3-02-19BPC**		14	SSAI3-04-23BPC	24	7	SSAI3-02-5BPC	$p_{s_j}$	<del>1</del> 34 1	MB 280 - 26583/1-A
+ 5	SSAJ3-02-8BPC ** VI		15	SSAI3-04-25BPC	25	7	SSAI3-02-8BPC		_ 35 つ	M3 286-26585/1-A
6 1	SSAJ3-02-8BPC_FD= b		16	SSAI3-02-11BPC	<b>≁</b> 26	ר	SSAI3-03-5BPC**		- 36 3	MB 280-26580/1-A
デラ	EB-08072010 W		17	SSAI3-02-14BPC	27	```	SSAI3-03-8BPC		37	
- 1 8	SSAI3-03-23BPC 5		18	SSAI3-02-15BPC	† 28	2	SSAI3-04-5BPC		38	
9 1	SSAI3-03-25BPC		19	SSAI3-02-19BPC	29	2	SSAI3-04-8BPC		39	
10	SSAI3-04-11BPC	И	20 1	SSAI3-02-25BPC	30	7	SSAI3-02-5BPC_FDMS	, ↓	40	

## **VALIDATION FINDINGS CHECKLIST**

Page: \\_\ of \\_2 Reviewer: \\_\ \forall VV 2nd Reviewer: \\_\

Method: Semivolatiles (EPA SW 846 Method 8270C)

Validation Area  1. Technical holding times	Yes	No	NA	Findings/Comments
All technical holding times were met.	/			/
Cooler temperature criteria was met.				
II. GC/MS Instrument performance check	4			An institute of the second of
Were the DFTPP 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) $\leq$ 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?	/			
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 for each matrix and concentration?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
VI Surrogate apikes				
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. Mamx spike/Mamx spike duolicates				
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?	1			
Were the MS/MSD percent recoveries (%R) and the relative percent differences				
(RPD) within the QC limits?				And the state of t
VIII. Laboratory control samples as the state of the stat		7		
Was an LCS analyzed for this SDG?	$\perp$			

## **VALIDATION FINDINGS CHECKLIST**

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

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?	_			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
X. Internal standards				The state of the s
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				<b>中国</b>
Were relative retention times (RRT's) within ± 0.06 RRT units of the standard?	4			
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	_			
Were chromatogram peaks verified and accounted for?				The St. Company of the Company of th
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?	/			
XIII. Tentatively identified compounds (TICs)				
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				the control of the second seco
System performance was found to be acceptable.				
XV. Overall assessment of data as a second of the control of the c				COLUMN TO STATE OF THE STATE OF
Overall assessment of data was found to be acceptable.	/			
XVI Flaid duplicates 1. 15 Sept. 1. 15 Sep				
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.	/			
XVII. Field blanks				And the second s
Field blanks were identified in this SDG.	/			
Target compounds were detected in the field blanks.	1	1/	<u> </u>	

## **VALIDATION FINDINGS WORKSHEET**

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

A. Phenoi**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenol**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	VV. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR, Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA, 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	ກກກ
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	WV.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	WWW.

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

LDC#: 240 47 82c

SDG #: 520 Con-

## VALIDATION FINDINGS WORKSHEET

Page: 1 of

Reviewer:\_ 2nd Reviewer.

Blanks

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". METHOD: GC/MS BNA (EPA SW 846 Method 8270C)

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

Was a method blank analyzed for each concentration preparation level?

Was the blank contaminated? If yes, please see qualification below. Was a method blank associated with every sample?

Blank extraction date: 8/2/10 Blank analysis date: 8/21/10 Conc. units: 1/6 /k.

1-8 9-1 Associated Samples:

-					 		
	Sample Identification	20	34 N				
	S	14	Woe				
Associated Samples.		7	24/4				
ASSOCIE		٦	25 /W				
		MB 280-26 \$83/4- A 3	43/4				
	Blank ID	MB 280-26	35.3				
Conc. units: "3 /kg/	Compound		3				
		Y5	176.5	-			

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

Associated Samples:

Compound	Blank ID	Sample Identification

5x Phthalates 2x all others

BLANKS2tronox.wpd

LDC#: 24047B2a

## VALIDATION FINDINGS WORKSHEET Field Duplicates

Page:	of/
Reviewer:	W6
2nd Reviewer:	<u> </u>

METHOD:	GC/MS	SVOA (EPA SW 846 Method 8270C)
		المنطلة منا للمكنك مصادات مناسسات المناطلة مناسب

YN NA Were

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

Carry a und Nama	Conc	( ug/Kg)	RPD	Diff	Diff Limits	Quais
Compound Name	5	6	(≤50%)			(Parent Only)
Dimethylphthalate	25	340U		315	≤340	

Compound Name	Conc (	ug/Kg)	RPD	Diff	Diff Limits	Quals
Compound Name	11	12	(≤50%)	-		(Parent Only)
Dimethylphthalate	340U	29		311	≤340	

V:\FIELD DUPLICATES\24047B2a.wpd

LDC#: Mo4/B2-

## VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

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,

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

%RSD = 100 \* (S/X)

S= Standard deviation of the RRFs,

X = Mean of the RRFs

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
		Calibration		RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
#	Standard ID Date	Date	Compound (Internal Standard)	( 50 std)	( 50 std)	(Initial)	(Initial)		
_	ICAL	7/21/2010	7/21/2010 1,4-Dioxane (IS1)	0.5607	0.5607	0.5706	0.5707	4.1	4.13
	MSS K		Naphthalene (IS2)	1.0611	1.0611	1.0093	1.0093	5.7	5.70
			Dimethylphthalate (1S3)	1.2729	1.2729	1.2330	1.2330	4.5	4.54
			9	0.2418	0.2418	0.2300	0.2300	3.8	3.81
			Chrysene (IS5)	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	570548	161541	784054	774079	
nc IS/Cpd	40/20	40/20	40/20	40/20	40/20	40/20	

0.6209					
0.5673	1.0632	1.2104		1.1443	0.8934
2	1.0390	1.2650	0.2339	1.1045	0.9948
0.5842	1.0490	1.2667	0.2330	1.1007	1.0754
0.5607	1.0611	1.2729	0.2418	1.1089	1.1425
0.5523	1.0236	1.3129	0.2310	1.0810	1.1683
0.5455	0.9799	1.2048	0.2266	0.9887	1.1297
0.5731	0.9540	1.1924	0.2305	0.9795	1.1318
0.5612	0.9045	1.1391	0.2131	0.9573	1.0989
0.5707	1.0093	1.2330	0.2300	1.0581	1.0794
0.0236	0.0575	0.0559	0.0088	0.0714	0.0920
	0.5523 0.5523 0.5455 0.5731 0.5612 0.0536		1.0236 0.9799 0.9540 0.9045 1.0093	1.0611     1.2729       1.0236     1.3129       0.9799     1.2048       0.9540     1.1924       0.9045     1.1331       1.0093     1.2330       0.0575     0.0559	1.0611         1.2729         0.2418           1.0236         1.3129         0.2310           0.9799         1.2048         0.2266           0.9540         1.1924         0.2305           0.9045         1.1391         0.2131           1.0093         1.2330         0.2300           0.0575         0.0559         0.0088

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 Results Verification **VALIDATION FINDINGS WORSHEET**

| |-| of Page\_\_ Reviewer:\_ 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:

% 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	

		Calibration			Average RRF	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Date	Compound (Reference IS)		(Initial RRF)	(CC RRF)	(CC RRF)	Q%	Q%
-	K5786	08/21/10	1,4-Dioxane	(IS1)	0.5706	0.5426	0.5426	4.9	4.9
			Naphthalene	(IS2)	1.0093	1.1242	1.1242	11.4	11.4
			Dimethylphthalate	(183)	1.2330	1.3364	1.3364	8.4	8.4
			Hexachlorobenzene	(IS4)	0.2300	0.2605	0.2605	13.3	13.3
			Chrysene	(185)	1.0581	1.0796	1.0796	2.0	2.0
			Benzo(a)pyrene	(186)	1.0793	1.2130	1.2130	12.4	12.4
2	K5828	08/23/10	1,4-Dioxane	(IS1)	0.5706	0.5422	0.5422	5.0	5.0
			Naphthalene	(182)	1.0093	1.1210	1.1210	11.1	11.1
			Dimethylphthalate	(183)	1.2330	1.3646	1.3646	10.7	10.7
			Hexachlorobenzene	(1S4)	0.2300	0.2640	0.2640	14.8	14.8
			Chrysene	(185)	1.0581	1.0684	1.0684	1.0	1.0
			Benzo(a)pyrene	(186)	1.0793	1.2357	1.2357	14.5	14.5

					2000	
Compound (Reference IS)		Concentration	Area Cpd	Area IS	Area Cpd	Area IS
		(IS/Cpd)				
1,4-Dioxane (IS	181)	40/80	202999	187054	206796	190700
Naphthalene (1S	lS2)	40/80	1573122	699655	1621366	723208
Dimethylphthalate (IS	(183)	40/80	1094543	409517	1141305	418177
Hexachlorobenzene (1S	(184)	40/80	349167	670085	372526	705597
Chrysene (1S:	(185)	40/80	1632481	756042	1824855	854050
Benzo(a)pyrene (ISI	(98)	40/80	1580828	651624	1845845	746891

LDC#: 24047 329

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

Page:_	_lof_1_
Reviewer:_	No
2nd reviewer:	11

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:

Sample is.	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	100	77.7	78	78	6
2-Fluorobiphenyl		78.3	78	78	
Terphenyl-d14		93./	93	93	
Phenol-d5	150	122.8	8 2	87	
2-Fluorophenol		119.7	79	79	
2,4,6-Tribromophenol		117-8	79	79	V
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol		·			
2-Chlorophenol-d4				·	
1,2-Dichlorobenzene-d4					

SDG #: See Care

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

Page: lof l.
Reviewer: \( \text{Lof l.} \)

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

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

using the following calculation:

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

Where: SSC = Spiked sample concentration SA = Spike added

SC = Sample concentation

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

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples:

s: 30/31

	Sp	ke	Sample	Spiked (	Sample	Matrix Spike	Spike	Matrix Spike Duplicate	- Duplicate	MS/MSD	SD
Compound	Added (*/C, /k	Jed /L )	Concentration (ルタ/ヒ)	Concentration ( 45 / F()	itration (Fc)	Percent Recovery	ecovery	Percent Recovery	всочегу	RPD	0
	MS	O MSD	ρ	MS	MSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenol											
N-Nitroso-di-n-propylamine											
4-Chloro-3-methylphenol											
Acenaphthene	2810	2790	0	2270	2522	8	B	Ø	(8)	0	0
Pentachlorophenol											
Pyrene	0 132	2790	\	2362	2350	n 8	84	λs	84	٥	٥
					-						

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#: 240 47 b 22

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

Reviewer: カル

2nd Reviewer: Page: lof 1

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

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 \* (SC/SA

Where: SSC = Spike concentration SA = Spike added

LCSC = Laboraotry control sample concentration LCSDC = Laboratory control sample duplicate concentration

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

LCS 280-LCS/LCSD samples:

	S	ike	S	ike	31	CS	Ü	CSD	I/SDI	CS/LCSD
Compound	Added ( w) /E	ded /E,	Concer (UK)	Concentration (4s. /ex.)	Percent Recovery	lecovery	Percent Recovery	ecovery	R	RPD
	1.08	O I CSD	) I GS	) I CSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Pheno										
N-Nitroso-di-n-propylamine										
4-Chloro-3-methylphenol										
Acenaphthene	2600	\$	2260	\$	48	87				
Pentachlorophenol										
Pyrene	24.60	>	abrz		88	83				
							\.			

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#: 24047 BZa

## VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	lot1_
Reviewer:	W
2nd reviewer:	ん

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

(Y)	N	N/A
V	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 =  $(A_{\downarrow})(I_{\downarrow})(V_{\downarrow})(DF)(2.0)$   $(A_{\downarrow a})(RRF)(V_{o})(V_{\downarrow})(\%S)$   $A_{\chi}$  = Area of the characteristic ion (EICP) for the compound to be measured  $A_{ia}$  = Area of the characteristic ion (EICP) for the specific internal standard  $I_{\chi}$  = Amount of internal standard added in nanograms (ng)  $V_{o}$  = Volume or weight of sample extract in milliliters (ml) or

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

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

V<sub>t</sub> = Volume of the concentrated extract in microliters (ui)

Df = Dilution Factor.

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

Example:

Sample I.D. <u>4</u> 26 , CC

= 28.16

2 28 mg/kg

2.0	= Factor of 2 to account	t for GPC cleanup			
		Commound	Reported Concentration	Calculated Concentration	Qualification
#	Sample ID	Compound			
<u> </u>	<u> </u>				

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 10 through August 11, 2010

LDC Report Date:

October 15, 2010

Matrix:

Soil

Parameters:

Semivolatiles

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6345-1

## Sample Identification

SSAJ3-07-SW-E-1BPC\*\*

SSAJ3-02-SW-W-1BPCMSD

SSAJ3-05-SW-E-1BPC

SSAJ3-02-SW-E-1BPC

00/100 02 01/ E /DF 0

SSAI3-04-SW-E-1BPC

SSAI3-03-SW-E-1BPC

SSAI3-02-SW-E-1BPC FD

SSAI3-02-SW-E-1BPC

RSAJ03-0BPC

SSAI3-02-SW-W-1BPC

SSAI3-03-SW-W-1BPC

SSAI3-04-SW-W-1BPC

SSAJ3-02-SW-W-1BPC

SSAJ3-05-SW-W-1BPC

SSAJ3-07-SW-W-1BPC\*\*

SSAJ3-07-SW-W-1BPC FD

SSAQ3-03-0BPC

SB01-24BPC

SB02-24BPC

SB01-24BPC FD

SSAJ3-02-SW-W-1BPCMS

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

## Introduction

This data review covers 21 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

Samples indicated by a double asterisk on the front cover underwent a 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.

No field blanks were identified in this SDG.

## VI. Surrogate Spikes

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

Sample	Surrogate	%R (Limits)	Compound	Flag	A or P
SB01-24BPC_FD	Nitrobenzene-d5 2-Fluorobiphenyl	42 (50-120) 49 (50-120)	Hexachlorobenzene	J- (all detects) UJ (all non-detects)	Р

## VII. Matrix Spike/Matrix Spike Duplicates

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

## VIII. Laboratory Control Samples (LCS)

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

## IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. 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 with the following exceptions:

Sample	Compound	Finding	Flag	A or P
SSAI3-04-SW-E-1BPC SSAI3-02-SW-W-1BPC	Benzo(b)fluoranthene	Due to lack of resolution between these compounds in the samples, the laboratory	J (all detects) UJ (all non-detects)	Р
	Benzo(k)fluoranthene	performed the quantitation using the total peak area.	J (all detects) UJ (all non-detects)	

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6345-1	All compounds reported below the PQL.	J (all detects)	Α

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 SSAI3-02-SW-E-1BPC and SSAI3-02-SW-E-1BPC\_FD, samples SSAJ3-07-SW-W-1BPC\*\* and SSAJ3-07-SW-W-1BPC\_FD, and samples SB01-24BPC and SB01-24BPC\_FD were identified as field duplicates. No semivolatiles were detected in any of the samples with the following exceptions:

	Concentra	tion (ug/Kg)		D:#	·		
Compound	SSAI3-02-SW-E-1BPC	SSAI3-02-SW-E-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P	
Octachlorostyrene	3300	3400	3 (≤50)	-	-	-	
Pyrene	12	12	-	0 (≤330)	-	-	
Hexachlorobenzene	24000	25000	4 (≤50)	<u>-</u>	-	-	

	Concentrat	222	5.44				
Compound	SSAJ3-07-SW-W-1BPC**	SSAJ3-07-SW-W-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P	
Hexachlorobenzene	170	160	-	10 (≤340)	•	-	

	Concentrat	ion (ug/Kg)	222	D.W.		A or P	
Compound	SB01-24BPC	SB01-24BPC_FD	RPD (Limits)	Difference (Limits)	Flags		
Hexachlorobenzene	1700	800	-	900 (≤340)	J (all detects)	Α	

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-6345-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6345-1	SB01-24BPC_FD	Hexachlorobenzene	J- (all detects) UJ (all non-detects)	P	Surrogate spikes (%R) (s)
280-6345-1	SSAI3-04-SW-E-1BPC SSAI3-02-SW-W-1BPC	Benzo(b)fluoranthene Benzo(k)fluoranthene	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Compound quantitation and CRQLs (q)
280-6345-1	SSAJ3-07-SW-E-1BPC** SSAJ3-05-SW-E-1BPC SSAJ3-02-SW-E-1BPC SSAI3-04-SW-E-1BPC SSAI3-03-SW-E-1BPC SSAI3-02-SW-E-1BPC FD SSAI3-02-SW-E-1BPC RSAJ03-0BPC SSAI3-02-SW-W-1BPC SSAI3-03-SW-W-1BPC SSAI3-04-SW-W-1BPC SSAJ3-05-SW-W-1BPC SSAJ3-07-SW-W-1BPC SSAJ3-07-SW-W-1BPC SSAJ3-07-SW-W-1BPC SSAJ3-07-SW-W-1BPC SSAJ3-07-SW-W-1BPC SSAJ3-07-SW-W-1BPC SSAJ3-07-SW-W-1BPC FD SSAQ3-03-0BPC SB01-24BPC SB01-24BPC SB01-24BPC FD	All compounds reported below the PQL.	J (all detects)	Ä	Project Quantitation Limit (sp)
280-6345-1	SB01-24BPC SB01-24BPC_FD	Hexachlorobenzene	J (all detects)	А	Field duplicates (Difference) (fd)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-6345-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Field Blank Data Qualification Summary - SDG 280-6345-1

No Sample Data Qualified in this SDG

## Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

LDC #: 24047E2a VALIDATION COMPLETENE
SDG #: 280-6345-1 Stage 2B/4
Laboratory: Test America

Page: 1 of 1
Reviewer: 0VC
2nd Reviewer: 1

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

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

	Validation Area		Comments
l.	Technical holding times	Á	Sampling dates: 8 /10 - 11 /10
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	-À	Ž RSD r~
IV.	Continuing calibration/ICV	A	ca/10 = 25 V
V.	Blanks	Α	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	ics
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	SW	Not reviewed for Stage 2B validation.
XIII.	Tentatively identified compounds (TICs)	N	Not reviewed for Stage 2B validation.
XIV.	System performance	Α	Not reviewed for Stage 2B validation.
XV.	Overall assessment of data	Å	
XVI.	Field duplicates	SW	0, = 7, 6 D2 = 14, 15 P3 = 17,19
XVII.	Field blanks	N	

Note:

A = Acceptable

ND = No compounds detected D = Duplicate

N = Not provided/applicable

R = Rinsate

TB = Trip blank

SW = See worksheet F

FB = Field blank

EB = Equipment blank

Validated Samples:

\*\* Indicates sample underwent Stage 4 validation

	All So	1/5					
1	SSAJ3-07-SW-E-1BPC**	11	SSAI 3-04-SW-W-1BPC	21	SSAJ3-02-SW-W-1BPCMSD	31	MB 280-27168/1-
† 2	SSAJ3-05-SW-E-1BPC	<del>1</del>	SSAJ3-02-SW-W-1BPC	22		32	
3	SSAJ3-02-SW-E-1BPC	<del>1</del> 13	SSAJ3-05-SW-W-1BPC	23		33	
<b>4</b>	SSAI3-04-SW-E-1BPC	<b>∔</b> 14	SSAJ3-07-SW-W-1BPC** Dy	24		34	
<b>∔</b> 5	SSAI3-03-SW-E-1BPC	<del>f</del> 15	SSAJ3-07-SW-W-1BPC FD			35	
<b>+</b> 6	SSAI3-02-SW-E-1BPC FD P	<b>4-</b> 16	SSAQ3-03-0BPC	26		36	
<b>∔</b> 7	SSAI3-02-SW-E-1BPC	† 17	SB01-24BPC <b>0</b> ,	27		37	
<del>1</del> 8	RSAJ03-0BPC	† 18	SB02-24BPC	28		38	
<del>7</del> 9	SSAI3-02-SW-W-1BPC	+ 19	SB01-24BPC_FD D3	29		39	
10	SSAI3-03-SW-W-1BPC	20	SSAJ3-02-SW-W-1BPCMS	30		40	

## **VALIDATION FINDINGS CHECKLIST**

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

Method: Semivolatiles (EPA SW 846 Method 8270C)

Validation Area	Yes	No	NA	Findings/Comments
i. Tachnical holding times				
All technical holding times were met.	/			
Cooler temperature criteria was met.	/			
II Ge/MS nautiment performance enest - 2008   1966   2008   1966   2008   1966   2008			l	
Were the DFTPP performance results reviewed and found to be within the specified criteria?	/			
Were all samples analyzed within the 12 hour clock criteria?		S(**\> 4.45)	orani de la como	
III initial contention				
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 Commung Calibration	ı —			And the state of t
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?				
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?				
Were all percent differences (%D) $\leq$ 25% and relative response factors (RRF) $\geq$ 0.05?	1			
VerBanks in a control of the control				Mark Calendar
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.				
		\\ .ka\.la		
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?				
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?				
(Title Cath, Albay Reality), earlighest research and Cath Cather				Page Charles of Carlos Constitution
Was an LCS analyzed for this SDG?				·

LDC#: 24647 E39

## VALIDATION FINDINGS CHECKLIST

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	/			
D. Regional Quality Assurance and Quality Control All Managements and Control Control All Managements and Control Cont				<b>PROPERTY OF THE PROPERTY OF T</b>
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
Xanend SantentS v				
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 AGIGE COMPONICATION BE				
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?			anni amin	
XII Congonio quantiation (Frois) (Frois) (Frois)				
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.) e italikely identified compounds 1990 (1995)				
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?				
Were relative intensities of the major ions within $\pm$ 20% between the sample and the reference spectra?			/	
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?			_	
System performance was found to be acceptable.		e vet souge en		
Overall assessment of data was found to be acceptable.				
Field duplicate pairs were identified in this SDG.		to a subserver from		
Target compounds were detected in the field duplicates.				
Minero de la companya		1		
Field blanks were identified in this SDG.				
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-Chioroaniline	II. 4-Nitrophenol*	XX. Di-n-butyiphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene⇔	JJ. Dibenzofuran	YY. Fluoranthene**	NNN, Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol⊷	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine⁴	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	OO. 4-Nitroaniline	DDD. Chrysene	SSS, Benzidine
L. Nitrobenzene	AA. 2-Chioronaphthalene	PP, 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	111.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate⊷	ກກກ
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyi-phenylether	GGG. Benzo(b)fluoranthene	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.

## VALIDATION FINDINGS WORKSHEET

Surrogate Recovery

Page: of beviewer:

Reviewer: 2nd Reviewer:

> Please see qualification below for all questions answered "N". Not applicable questions are identified as "N/A". METHOD: GC/MS BNA (EPA SW 846 Method 8270C)

Were percent recoveries (%R) for surrogates 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?

N N N Y.AV.NA S/N/A

Date   Surrogate   WR (Linits)   Qualifications			ī	Т	Г	T	Τ	II -	T	T	T	T	T	1	T	T	T	T	Г			r <del>-</del>	ī	Ţ	<del></del>
Sample ID Surrogate %R (Lints)  \$\frac{1}{4} \tag{7.8P} \tag{8.4 (S1-\frac{1}{4})}{\frac{1}{4}} \tag{5.1}  \$\frac{1}{4} \tag{7.8P} \tag{4.5 (S0-\frac{1}{4})}{\frac{1}{4}} \tag{4.5 (S0-\frac{1}{4})}{\frac{1}{4}} \tag{6.5 (S0-\frac{1}{4})}{\frac{1}	Qualifications	and (mly						<b>→</b>		22) (S) d/ EN/															
S	%R (Limits)	15	( )	ty ( )	( )		( )	) 7¢	( )								( )	( )	( )	( )	( )		( )	( )	( )
J P P P P P P P P P P P P P P P P P P P	Surrogate	787						\ \		N82	FBP														
Date	Sample ID	4		5		$\neg$		М		61															
<u></u>	Date																								
44	*																								

QC Limits (Water) 21-100 10-123 33-110\* 16-110\*

QC Limits (Soil) 25-121 19-122 20-130\*

S5 (2FP)= 2-Fluorophenol S6 (TBP) = 2,4,6-Tribromophenol S7 (2CP) = 2-Chlorophenol-d4 S8 (DCB) = 1,2-Dichlorobenzene-d4

QC Limits (Water) 35-114 43-116 33-141 10-94

LDC# 240 47 F24

## Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Reviewer:

Page: 1 of 1

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

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

Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation? Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound? Y/N N/A

# Date Sample ID Finding Associated Samples Qualifications  4 9 666, 1444 perds uncestived  Let used total perd area for green fitzil on					 							
Date Sample ID	Qualifications											
Date Sample ID	Associated Samples	Wed	for swontitution	4								
Date 4 4	Finding	666, 444 peaks unresd	lab used total peal area									
	Sample ID											
*	Date											
	#											

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24047E2a

### **VALIDATION FINDINGS WORKSHEET**

**Field Duplicates** 

Page:	
Reviewer:_	W
2nd Reviewer:	<u> </u>

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

Y N NA

Were field duplicate pairs identified in this SDG?

Were target analytes detected in the field duplicate pairs?

Commound Nome	Conc	(ug/Kg)	RPD	Diff	Diff Limits	Quals
Compound Name	7	6	(≤50%)	DIII	Dil Lillits	(Parent Only)
Octachiorostyrene	3300	3400	3			
Pyrene	12	12		0	≤330	
Hexachlorobenzene	24000	25000	4			

Compound Name	Conc (	ug/Kg)	RPD	Diff	Diff Limits	Quals
Compound Name	14	15	(≤50%)	Diii	Om Linius	(Parent Only)
Hexachlorobenzene	170	160		10	≤340	

Compound Name	Conc (	ug/Kg)	RPD	Diff	Diff Limits	Quals
Compound Name	17	19	KFD (≤50%)	Dill	Din Limits	(Parent Only)
Hexachlorobenzene	1700	800		900	≤340	Jdets/A (fd)

V:\FIELD DUPLICATES\24047E2a.wpd

LDC# 240 47 F29 SDG#:

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

l of Y Page: Reviewer: 2nd Reviewer:

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

 $A_x = Area of Compound$ 

 $C_x$  = Concentration of compound,

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

%RSD = 100 \* (S/X)

S= Standard deviation of the RRFs,

 $C_{\rm is}$  = 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	Date	Compound (Internal Standard)	( 50 std)	( 50 std)	(Initial)	(Initial)		
_	ICAL	8/27/2010	8/27/2010 1,4-Dioxane (IS1)	0.5926	0.5926	0.5795	0.5795	3.7	3.74
	MSS K		Naphthalene (IS2)	1.0571	1.0571	1.0015	1.0015	8.9	8.92
			Fluorene (IS3)	1.3180	1.3180	1.2421	1.2421	7.9	7.87
			Hexachlorobenzene (IS4)	0.2424	0.2424	0.2313	0.2313	6.1	6.04
			Chrysene (IS5)	1.1257	1.1257	1.0679	1.0679	9.3	9.33
			Benzo(g,h,i)perylene (IS6)	1.1231	1.1231	1.0199	1.0199	7.5	7.53

Area IS	172314	669515	393544	662745	759660	781265	
Area cpd	127636	884641	648342	200827	1068947	1096793	
lc IS/Cpd	40/20	40/20	40/20	40/20	40/20	40/20	

Conc	1,4-Dioxane	Naphthalene	Fluorene	Hexachlorob	Chrysene	Benzo(g,h,i)per
4.00	0.5778	1.1018	1.3240		1.1929	0.9595
10.00	0.6003	1.0722	1.3327	0.2454	1.1472	1.0450
20.00	0.6103	1.0714	1.3075	0.2448	1.1400	1.0900
50.00	0.5926	1.0571	1.3180	0.2424	1.1257	1.1231
80.00	0.5842	1.0008	1.2564	0.2335	1.0651	1.0769
120.00	0.5678	0.9489	1.1901	0.2252	0.9953	1.0108
160.00	0.5547	0.8964	1.1248	0.2168	0.9529	0.9476
200.00	0.5485	0.8636	1.0833	0.2109	0.9244	0.9066
×	0.5795	1.0015	1.2421	0.2313	1.0679	1.0199
S	0.0217	0.0893	0.0977	0.0140	0.0997	0.0768
•						

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#: 24047 Era

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

JVG Page: Yof Reviewer:

2nd Reviewer:

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

%RSD = 100 \* (S/X)

A<sub>x</sub> = Area of Compound

 $C_x$  = Concentration of compound,

A<sub>is</sub> = Area of associated internal standard  $C_{\rm is}$  = Concentration of internal standard

S= Standard deviation of the RRFs,

X = Mean of the RRFs

					Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
		Calibration			RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
#	Standard ID	Date	Compound (Internal Standard)	ırd)	( 50 std)	( 50 std)	(Initial)	(Initial)		
1	ICAL	7/21/2010	7/21/2010 1,4-Dioxane (IS	(181)	0.5607	0.5607	0.5706	0.5707	4.1	4.13
	MSS K		Naphthalene (19	(182)	1.0611	1.0611	1.0093	1.0093	5.7	5.70
			Fluorene (18	(IS3)	1.3101	1.3101	1.2473	1.2473	5.3	5.25
			Hexachlorobenzene (IS	(IS4)	0.2418	0.2418	0.2300	0.2300	3.8	3.81
	æ		Chrysene (IS	(185)	1.1089	1.1089	1.0581	1.0581	6.7	6.75
			Benzo(a)pyrene (IS	(186)	1.1425	1.1425	1.0793	1.0794	8.5	8.53
									-	

				***************************************		
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

616036 358588

160417

112429 817090 587234 161541 784054 774079

Area IS

Area cpd

nc IS/Cpd 40/20 40/20 40/20 40/20 40/20 40/50

565669 542046

534527

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 Results Verification VALIDATION FINDINGS WORSHEET

Page of 1 2nd Reviewer: 1 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:

% 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

		Calibration			Average RRF	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Date	Compound (Reference IS)	erence IS)	(Initial RRF)	(CC RRF)	(CC RRF)	<b>0%</b>	%D
-	K6093	09/02/10	1,4-Dioxane	(IS1)	0.5795	0.5989	0.5989	3.3	3.3
			Naphthalene	(182)	1.0015	1.0241	1.0241	2.3	2.3
			Fluorene	(183)	1.2421	1.2707	1.2707	2.3	2.3
			Hexachlorobenzene	ne (IS4)	0.2313	0.2387	0.2387	3.2	3.2
			Chrysene	(185)	1.0679	1.0864	1.0864	1.7	1.7
			Benzo(g,h,i)perylene	ine (IS6)	1.0199	1.1046	1.1046	8.3	8.3
2	K5874	08/24/10	1,4-Dioxane	(IS1)	0.5706	0.5200	0.5200	8.9	8.9
			Naphthalene	(182)	1.0093	1.1283	1.1283	11.8	11.8
			Fluorene	(183)	1.2470	1.4081	1.4081	12.9	12.9
			Hexachlorobenzene	ne (IS4)	0.2300	0.2632	0.2632	14.5	14.5
			Chrysene	(185)	1.0581	1.0891	1.0891	2.9	2.9
			Benzo(g,h,i)perylene	(IS6) ene	0.9570	1.0899	1.0899	13.9	13.9

Compound (Reference IS)	(S)	Concentration	Area Cpd	Area IS	Area Cpd	Area IS
		(IS/Cpd)				
1,4-Dioxane	(181)	40/80	167425	139782	195412	187881
Naphthalene	(182)	40/80	1107956	540952	1580575	700454
Fluorene	(183)	40/80	806913	317514	1158634	411418
Hexachlorobenzene	(184)	40/80	251814	527560	353417	671295
Chrysene	(185)	40/80	1303161	599747	1707482	783883
Benzo(g,h,i)perylene	(186)	40/80	1411930	639110	1480022	678968

LDC#: 24047 E29

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

Page:	<u>lof 1</u>
Reviewer:	314
2nd reviewer:	1

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found

Sample ID:

Ħ

SS = Surrogate Spiked

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	رف	55.2	2.2	22	6
2-Fluorobiphenyl		57.0	57	57	
Terphenyl-d14		80.5	81	8 /	
Phenol-d5	150	87.9	59	59	
2-Fluorophenol		83.7	56	56	
2,4,6-Tribromophenol	1	90.8	41	61	
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14			,		
Phenol-d5			-		
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5		,			
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					·
2,4,6-Tribromophenol		·			
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

LDC#: 74047 FX SDG #: See Cores

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

Page: Reviewer: 2nd 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:

SSC = Spiked sample concentration SA = Spike added Where:

SC = Sample concentation

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

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

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples:

2

	Spi	Ike	Sample	Spiked	Sample	Matrix Spike	Spike	Matrix Spike Duplicate	Duplicate	MS/MSD	SD
Compound	Added (MS)/F	jed (c. )	Concentration (MS /k_)	Concentration ( 1/2 / /2_)	itration کم)	Percent Recovery	ecovery	Percent Recovery	ecovery	RPD	Q
	MS	) MSD	()	MS	MSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenoi											
N-Nitroso-di-n-propylamine											
4-Chloro-3-methylphenol											
Acenaphthene	2860	2590	0	098)	[890	77	72	23	7.5	λ	ላ
Pentachiorophenoi											
Pyrene	7530	ملك		2 2	2017	<b>۲</b> 8	87	48	۶ ۶	7	>

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

LUU#: 24047 E 25

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

Page: Lof 1 Reviewer:\_\_

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

F-C/ 891/2-082 577 LCS/LCSD samples: \_

	Silke	Spire	1.08	LCSD	1 CS/I CSD
	Added	Concentration			
Compound	(20/62)	( <sup>2</sup> / <sub>2</sub> <sup>2</sup> / <sub>2</sub> )	Percent Recovery	Percent Recovery	RPD

	ď	100		ka Ka	<u> </u>	CS	31	CSD	1 CS/I CSD	CSD
Compound	Add ( 29.0)	Added ( ng /kg. )	Concer Concer	Concentration ( 1/2 //5)	Percent Recovery	ecovery	Percent Recovery	Recovery	RPD	٥
		) I CSD	83	l CSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenol									-	
N-Nitroso-di-n-propylamine								·		
4-Chloro-3-methylphenol										
Acenaphthene	20 %	MA	1940	MA	24	74				
Pentachlorophenol				<		/				
Pyrene	2610		0812	_	18	84				
					,					

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#: 26047 Eng

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

Page:_	lof1_
Reviewer:	W
2nd reviewer:	h

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

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

Concentration = (A,)(I,)(V)(DF)(2.0) (A<sub>s</sub>)(RRF)(V<sub>o</sub>)(V)(%S)

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

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

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

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

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

V<sub>t</sub> = Volume of the concentrated extract in microliters (ul)

Df = Dilution Factor.

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

Example:

Sample I.D. 14 , SS

Conc. =  $(\frac{20746}{1000})(\frac{40}{1000})(\frac{10$ 

7 170 mg/kg

2.0	= Factor of 2 to accoun	t for GPC cleanup			
#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
					·
	·				
				· · · · · · · · · · · · · · · · · · ·	
			_		
1					

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 13, 2010

LDC Report Date:

October 8, 2010

Matrix:

Soil

Parameters:

Semivolatiles

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6535-1

### Sample Identification

SSAL3-07-0BPC

SSAK8-07-0BPCMS SSAK8-07-0BPCMSD

SSAK8-10-0BPC

SSAK8-09-0BPC

SSAK8-07-0BPC\*\*

SSAN6-03-0BPC

SSA07-03-0BPC

SA44-0BPC

SSAP3-01-0BPC\*\*

SSAO4-01-0BPC

SSAK3-05-0BPC

SSAK3-07-0BPC

SSAK3-08-0BPC

SSAL4-04-0BPC

SSAL4-05-0BPC

SSAN6-09-0BPC

SSAN6-08-0BPC

SSAN5-04-0BPC

SSAO3-05-0BPC

SSAO3-04-0BPC

SSAO4-06-0BPC

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

### Introduction

This data review covers 22 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

Samples indicated by a double asterisk on the front cover underwent a 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 with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
9/4/10	Bis(2-ethylhexyl)phthalate	31.3	All samples in SDG 280-6535-1	J+ (all detects)	А

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 280-28701/1-A	8/26/10	Bis(2-ethylhexyl)phthalate Dimethylphthalate	89.5 ug/Kg 39.8 ug/Kg	All samples in SDG 280-6535-1

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
SSAL3-07-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	160 ug/Kg 22 ug/Kg	160U ug/Kg 22U ug/Kg
SSAK8-10-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	92 ug/Kg 25 ug/Kg	92U ug/Kg 25U ug/Kg
SSAK8-09-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	100 ug/Kg 130 ug/Kg	100U ug/Kg 130U ug/Kg
SSAK8-07-0BPC**	Bis(2-ethylhexyl)phthalate Dimethylphthalate	92 ug/Kg 32 ug/Kg	92U ug/Kg 32U ug/Kg
SSAN6-03-0BPC	Bis(2-ethylhexyl)phthalate	390 ug/Kg	390U ug/Kg
SSA07-03-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	95 ug/Kg 22 ug/Kg	95U ug/Kg 22U ug/Kg
SSAP3-01-0BPC**	Bis(2-ethylhexyl)phthalate	370 ug/Kg	370U ug/Kg
SSAO4-01-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	100 ug/Kg 29 ug/Kg	100U ug/Kg 29U ug/Kg
SSAK3-05-0BPC	Dimethylphthalate	150 ug/Kg	150U ug/Kg
SSAK3-07-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	97 ug/Kg 32 ug/Kg	97U ug/Kg 32U ug/Kg
SSAK3-08-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	96 ug/Kg 30 ug/Kg	96U ug/Kg 30U ug/Kg

Sample	Compound	Reported	Modified Final
	TIC (RT in minutes)	Concentration	Concentration
SSAL4-04-0BPC	Bis(2-ethylhexyl)phthalate	95 ug/Kg	95U ug/Kg
	Dimethylphthalate	52 ug/Kg	52U ug/Kg
SSAL4-05-0BPC	Bis(2-ethylhexyl)phthalate	98 ug/Kg	98U ug/Kg
	Dimethylphthalate	45 ug/Kg	45U ug/Kg
SSAN6-09-0BPC	Bis (2-ethylhexyl) phthalate	96 ug/Kg	96U ug/Kg
	Dimethylphthalate	27 ug/Kg	27U ug/Kg
SSAN6-08-0BPC	Bis(2-ethylhexyl)phthalate	100 ug/Kg	100U ug/Kg
SSAN5-04-0BPC	Bis(2-ethylhexyl)phthalate	99 ug/Kg	99U ug/Kg
	Dimethylphthalate	33 ug/Kg	33U ug/Kg
SSA03-05-0BPC	Bis(2-ethylhexyl)phthalate	150 ug/Kg	150U ug/Kg
	Dimethylphthalate	120 ug/Kg	120U ug/Kg
SSAO3-04-0BPC	Bis(2-ethylhexyl)phthalate	94 ug/Kg	94U ug/Kg
SSAO4-06-0BPC	Bis(2-ethylhexyl)phthalate	170 ug/Kg	170U ug/Kg
	Dimethylphthalate	140 ug/Kg	140U 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. Percent recoveries (%R) and relative percent differences (RPD) were within QC limits.

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. 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 with the following exceptions:

Sample	Compound	Finding	Flag	A or P
SSAK8-07-0BPC** SSAO7-03-0BPC SSAO4-01-0BPC SSAO3-04-0BPC SSAO4-06-0BPC	Benzo(b)fluoranthene Benzo(k)fluoranthene	quantitation using the total peak area.	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6535-1	All compounds reported below the PQL.	J (all detects)	Α

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

No field duplicates were identified in this SDG.

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-6535-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6535-1	SSAL3-07-0BPC SSAK8-10-0BPC SSAK8-09-0BPC SSAK8-09-0BPC SSAK8-07-0BPC** SSAN6-03-0BPC SSAO7-03-0BPC SA44-0BPC SSAP3-01-0BPC** SSAO4-01-0BPC SSAK3-05-0BPC SSAK3-05-0BPC SSAK3-08-0BPC SSAL4-04-0BPC SSAN6-09-0BPC SSAN6-09-0BPC SSAN6-09-0BPC SSAN5-04-0BPC SSAO3-05-0BPC SSAO3-05-0BPC SSAO3-05-0BPC SSAO3-04-0BPC SSAO3-04-0BPC	Bis(2-ethylhexyl)phthalate	J+ (all detects)	A	Continuing calibration (%D) (c)
280-6535-1	SSAK8-07-0BPC** SSAO7-03-0BPC SSAO4-01-0BPC SSAO3-04-0BPC SSAO4-06-0BPC	Benzo(b)fluoranthene Benzo(k)fluoranthene	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Compound quantitation and CRQLs (q)
280-6535-1	SSAL3-07-0BPC SSAK8-10-0BPC SSAK8-09-0BPC SSAK8-07-0BPC** SSAN6-03-0BPC SSAO7-03-0BPC SSAO7-03-0BPC SSAO4-01-0BPC SSAK3-05-0BPC SSAK3-07-0BPC SSAK3-07-0BPC SSAK3-08-0BPC SSAL4-04-0BPC SSAL4-04-0BPC SSAN6-09-0BPC SSAN6-09-0BPC SSAN6-09-0BPC SSAN6-08-0BPC SSAN03-04-0BPC SSAO3-04-0BPC SSAO3-04-0BPC SSAO3-04-0BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-6535-1

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
280-6535-1	SSAL3-07-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	160U ug/Kg 22U ug/Kg	А	bl
280-6535-1	SSAK8-10-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	92U ug/Kg 25U ug/Kg	А	bl
280-6535-1	SSAK8-09-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	100U ug/Kg 130U ug/Kg	A	bl
280-6535-1	SSAK8-07-0BPC**	Bis(2-ethylhexyl)phthalate Dimethylphthalate	92U ug/Kg 32U ug/Kg	A	bl
280-6535-1	SSAN6-03-0BPC	Bis(2-ethylhexyl)phthalate	390U ug/Kg	А	bl
280-6535-1	SSAO7-03-0BPC	7-03-0BPC Bis(2-ethylhexyl)phthalate 95U ug/Kg Dimethylphthalate 22U ug/Kg		A	bl
280-6535-1	SSAP3-01-0BPC**	Bis(2-ethylhexyl)phthalate 370U ug/Kg		А	bl
280-6535-1	SSAO4-01-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	100U ug/Kg 29U ug/Kg	A	bl
280-6535-1	SSAK3-05-0BPC	Dimethylphthalate	150U ug/Kg	А	bl
280-6535-1	SSAK3-07-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	97U ug/Kg 32U ug/Kg	A	bl
280-6535-1	SSAK3-08-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	96U ug/Kg 30U ug/Kg	A	bl
280-6535-1	SSAL4-04-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	95U ug/Kg 52U ug/Kg	A	bl
280-6535-1	SSAL4-05-0BPC	Bis(2-ethylhexyl)phthalate 98U ug/Kg Dimethylphthalate 45U ug/Kg		A	bl
280-6535-1	SSAN6-09-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	alate 96U ug/Kg 27U ug/Kg		bl
280-6535-1	SSAN6-08-0BPC	Bis(2-ethylhexyl)phthalate	100U ug/Kg	А	bl

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
280-6535-1	SSAN5-04-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	99U ug/Kg 33U ug/Kg	Α -	bl
280-6535-1	SSAO3-05-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	150U ug/Kg 120U ug/Kg	A	bl ·
280-6535-1	SSAO3-04-0BPC	Bis(2-ethylhexyl)phthalate	94U ug/Kg	Α	bl
280-6535-1	SSAO4-06-0BPC	Bis(2-ethylhexyl)phthalate Dimethylphthalate	170U ug/Kg 140U ug/Kg	А	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Field Blank Data Qualification Summary - SDG 280-6535-1

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

LDC #: 24047F2a VALIDATION COMPLETENE
SDG #: 280-6535-1 Stage 2B/4
Laboratory: Test America

Date: 10/65/10
Page: 1 of 1
Reviewer: 5/6
2nd Reviewer: ---

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 8 /13 /to
II.	GC/MS Instrument performance check	Á	
III.	Initial calibration	A	oh RSD rd
IV.	Continuing calibration/ICV	SW	ca/a = 25 }
V.	Blanks	SW	
VI.	Surrogate spikes	SW	·
VII.	Matrix spike/Matrix spike duplicates	<u> </u>	
VIII.	Laboratory control samples	A	us
IX.	Regional Quality Assurance and Quality Control	N	
Χ.	Internal standards	A	
XI.	Target compound identification	A	Not reviewed for Stage 2B validation.
XII.	Compound quantitation/CRQLs	SW	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	N	
XVII.	Field blanks	STAN	FR - FB - 04072010 - RZC ( from 280 - 2280 ->

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

vanac	All So						
1	SSAL3-07-0BPC	11	SSAK3-07-0BPC	21	SSAK8-07-0BPCMS	31	MB 280_ 28701/1-
2	SSAK8-10-0BPC	12	SSAK3-08-0BPC	22	SSAK8-07-0BPCMSD	32	
3	SSAK8-09-0BPC	13	SSAL4-04-0BPC	23		33	
4	SSAK8-07-0BPC**	14	SSAL4-05-0BPC	24		34	-
5	SSAN6-03-OBPC	15	SSAN6-09-0BPC	25		35	
6	SSAO7-03-0BPC	16_	SSAN6-08-0BPC	26	·	36	
7	SA44-0BPC	17	SSAN5-04-0BPC	27		37	
8	SSAP3-01-0BPC**	18	SSAO3-05-0BPC	28		38	
9	SSAO4-01-0BPC	19	SSAO3-04-0BPC	29		39	
10	SSAK3-05-0BPC	20	SSAO4-06-0BPC	30		40	

Page: \\_of\_2 Reviewer: \_JW 2nd Reviewer: \_V

Method: Semivolatiles (EPA SW 846 Method 8270C)

			-	
Validation Area	Yes	No	NA	Findings/Comments
Technical holding times				
All technical holding times were met.	/			
Cooler temperature criteria was met.		es consultant es	ng ng i ti sainti	
II. GC/MS Instrument performance check				A STATE OF THE STA
Were the DFTPP performance results reviewed and found to be within the specified criteria?	/			
Were all samples analyzed within the 12 hour clock criteria?			ao io 1969.	
III. Initial calibration				
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?	1			
Was a curve fit used for evaluation?	/			
Did the initial calibration meet the curve fit acceptance criteria of <u>&gt;</u> 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			ij.	Marie Carlos Car
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			ar .	(1) 10 10 10 10 10 10 10 10 10 10 10 10 10
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				<b>Ex</b> (10 or 1 or 1)
Was an LCS analyzed for this SDG?			L	

LDC #: 24047 F29

### VALIDATION FINDINGS CHECKLIST

Page: 2 of 2 Reviewer: 16 2nd Reviewer: 2

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX. Regional Quality Assurance and Quality Control.				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?	20 20 15 A			
X Internal standards				All lights of the state of the
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	1			
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?		1		
XIII. Tentatively identified compounds (TICs)			*****	
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.				Med Angle Street
	1			The second of th
XV. Overall assessment of data a superior of the superior of t				
Overall assessment of data was found to be acceptable.				
XVI. Field duplicates			·	
Field duplicate pairs were identified in this SDG.	٠	/		
Target compounds were detected in the field duplicates.			,	
XVII. Field blanks	1	4		
Field blanks were identified in this SDG.	X	/		
Target compounds were detected in the field blanks.	1	<u> </u>		

## **VALIDATION FINDINGS WORKSHEET**

## METHOD: GC/MS BNA (EPA 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. Aniine
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethy/amine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine	Y. 2,4,6-Trichlorophenol	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	OO. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	TTT. 1-Methylnaphthalene
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)	FFF. Di-n-octylphthalate	uuu.
N. 2-Nitrophenol	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	WV.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	WWW.

24047 F22 LDC#:

## VALIDATION FINDINGS WORKSHEET

Continuing Calibration

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

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

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

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

N N/A

Qualifications	J+ duts 1A														
Associated Samples	H 4 II		5												
Finding RRF (Limit: >0.05)										•					
Finding %D (Limit: <25.0%)	31,3														
Compound	(+) =1 =1						·								
Standard ID	D8321	( Pag)													
Date	9/24/10														
#	11														

LDC# 24147 F2a SDG#:

### VALIDATION FINDINGS WORKSHEET **Blanks**

1 of 2 200 Reviewer:\_ 2nd Reviewer:\_\_ Page:

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".

Was a method blank analyzed for each matrix? 

Was a method blank analyzed for each concentration preparation level?

Was a method blank associated with every sample?

 $\frac{1}{2}$  N N/A Was the blank contaminated? If yes, please see qualification below. Blank extraction date:  $\frac{1}{2}$ / $\frac{1}{2}$ 6 / $\frac{1}{2}$ 8 Blank analysis date:  $\frac{1}{2}$ / $\frac{1}{2}$ 6 / $\frac{1}{2}$ 

Associated Samples:

12/2 29/4 370/1 8 12/26 0 390 Sample Identification 1/26 3 = 7 700 130 N/ 22 42/4 22 160 MB 280-78761 Blank ID 89.5 39.8 五年五 ઉ Compound Conc. units: 447.9

651

š

8 Same Blank analysis date: Blank extraction date: Conc. units:

Associated Samples:

above

Sample Identification 286 4 20 7 0 (460 11078c-085 SM Blank ID 89.5 出 Compound

44.2

199

3 25

20

99/4 33/4

E

96

5x Phthalates 2x all others

24047 F2A SDG #: LDC #:

## VALIDATION FINDINGS WORKSHEET

3//6 Page: Yof Reviewer:\_ 2nd Reviewer:

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".

Was a method blank analyzed for each matrix? 

Was a method blank analyzed for each concentration preparation level?

Was a method blank associated with every sample?

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

Blank extraction date: 8/26/9 Blank analysis date: 5/64 //0

Sample Identification Ϋ́ Associated Samples: Z 36 170 /4 45 94 NB 280 - 28 70/4 Blank ID 39.8 81.5 ٥ 医管件 Compound Conc. units:

5240

6

Blank analysis date: Blank extraction date:

Associated Samples:

5x Phthalates 2x all others

BLANKS2tronox.wpd

LDC# 24047 Fra

## VALIDATION FINDINGS WORKSHEET Surrogate Recovery

Reviewer: N6

2nd Reviewer:

o

Page:

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

Please see qualification below for all questions answered "N". Not applicable questions are identified as "N/A" Were percent recoveries (%R) for surrogates 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?

					<del></del>			-	 					 							 ī
Qualifications	No quad (only int)	0	_																		
%R (Limits)	29 (51-120)	( ) )	( 1 ) 27	( )	( )	( )	( )	( )	( )	( )	)	( )	( )	( )	( )	( )	( )	( )	( )	( )	
Surrogate	TBP		<b>-</b>																		
Sample ID	10		(3)																		
Date																					
*																					

QC Limits (Water) 21-100 10-123 33-110\* 16-110\*

QC Limits (Soil) 25-121

19-122 20-130\* 20-130\*

S5 (2FP)= 2-Fluorophenol S6 (TBP) = 2,4,6-Tribromophenol S7 (2CP) = 2-Chlorophenol-44 S8 (DCB) = 1,2-Dichlorobenzene-44

QC Limits (Water) 35-114

QC Limits (Soil)

43-116 33-141 10-94

23-120 30-115 18-137 24-113

\* QC limits are advisory S1 (NBZ) = Nitrobenzene-d5 S2 (FBP) = 2-Fluorobiphenyl S3 (TPH) = Terphenyl-d14 S4 (PHL) = Phenol-d5

LDC# 24047 F2a SDG #: 54

## Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

Page: Reviewer: 2nd Reviewer:

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". N N/A

Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation? Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?

Qualifications	J/43 /	2								
Associated Samples	shed	area for quantitat								
Finding	GGG, HHH Perke UNITS	lab used total peak area for quantitation								
Sample ID	4 6 9 19 20	, , , , , , , , , , , , , , , , , , , ,								
Date										
*										

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24047 Frg SDG #:

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

ō Page: 2nd Reviewer: Reviewer:

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:

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

%RSD = 100 \* (S/X)

 $C_x = Concentration of compound,$  $A_x$  = Area of Compound average RRF = sum of the RRFs/number of standards

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

S= Standard deviation of the RRFs,

X = Mean of the RRFs

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
		Calibration		RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
#	Standard ID Date	Date	Compound (Internal Standard)	( 50 std)	( 50 std)	(Initial)	(Initial)		
1	ICAL	9/3/2010	9/3/2010   1,4-Dioxane (IS1)	0.6430	0.6430	0.6429	0.6429	6.0	5.96
	MSS D		Naphthalene (IS2)	1.0799	1.0799	1.0794	1.0794	1.3	1.30
			Fluorene (IS3)	1.3494	1.3494	1.3276	1.3276	3.1	3.09
			Hexachlorobenzene (IS4)	0.2361	0.2361	0.2444	0.2444	3.9	3.87
		•	Chrysene (IS5)	1.0436	1.0436	1.0676	1.0676	2.4	2.39
			Benzo(g,h,i)perylene (IS6)	1.0554	1.0554	1.0292	1.0292	9.5	9.49

Conc 1,4-Dioxane	4.00 0.6400	10.00	20.00 0.6616	50.00 0.6430	80.00 0.6342	120.00 0.6253	160.00 0.6123	200.00	X = 0.6429	
Ö	4	10	20	20	98	120	160	200	٠	
<u> </u>	53	92	26	74	95	22				
Area IS	279929	1091792	925269	1196474	1364895	1258657				
vrea cpd	225000	1473825	1168084	353133	1780490	1660410				

ic IS/Cpd 40/20 40/20 40/20 40/50 40/50 40/20

Sonc	1,4-Dioxane	Naphthalene	Fluorene	Hexachlorob	Chrysene	Benzo(g,h,i)per
4.00	0.6400	1.0847	1.2767		1.0515	0.8429
0.00	0.7256	1.0641	1.2860	0.2309	1.0670	0.9316
20.00	0.6616	1.0764	1.2870	0.2382	1.0723	1.0000
50.00	0.6430	1.0799	1.3494	0.2361	1.0436	1.0554
80.00	0.6342	1.1063	1.3874	0.2485	1.1110	1.1183
20.00	0.6253	1.0871	1.3636	0.2475	1.0913	1.0941
160.00	0.6123	1.0748	1.3487	0.2535	1.0716	1.0952
200.00	6009:0	1.0620	1.3219	0.2561	1.0325	1.0959
II ×	0.6429	1.0794	1.3276	0.2444	1.0676	1.0292
S	0.0383	0.0140	0.0410	0.0094	0.0255	0.0976

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 # 24047 Fre SDG # See Cover

### Continuing Calibration Results Verification VALIDATION FINDINGS WORSHEET

Page \ of \ Reviewer: 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:

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

ave. RRF = initial calibration average RRF Ax = Area of compound

Ais = Area of associated internal standard RRF = continuing calibration RRF

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

Cx = Concentration of compound

Cis = Concentration of internal standard

Recalculated %D	9.0	7.1	6.5	5.0	6.1	12.9				
Reported %D	9.0	7.1	6.5	5.0	6.1	12.9				
Recalculated (CC RRF)	0.646	1.156	1.414	0.257	1.132	1.162				
Reported (CC RRF)	0.646	1.156	1.414	0.257	1.132	1.162				
Average RRF (Initial RRF)	0.643	1.0794	1.328	0.244	1.068	1.029				
Compound (Reference IS)	1,4-Dioxane (IS1)	Naphthalene (IS2)	Fluorene (IS3)	Hexachlorobenzene (IS4)	Chrysene (1S5)	Benzo(g,h,i)perylene (IS6)				
Calibration Date	08/21/10									
Standard ID	D7712									
#	-						2			

Compound (Reference IS)	Concentration	Area Cpd	Area IS	Area Cpd	Area IS
	(IS/Cpd)				
1,4-Dioxane (IS1)	40/80	377899	292284		
Naphthalene (IS2)	40/80	2581143	1116216		
Fluorene (IS3)	40/80	2023429	715303		
Hexachlorobenzene (IS4)	40/80	634434	1236129		
Chrysene (IS5)	40/80	3164159	1397101		
Benzo(g,h,i)perylene (IS6)	40/80	3098416	1333412		

LDC#: 24047 = 29

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

Page:	lof 1
Reviewer:	W
2nd reviewer:	1~

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found

Sample ID:

SS =	= Surr	ogate	Spik	ed

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	100	72.4	7~	72	0
2-Fluorobiphenyl		74.5	75	75	
Terphenyl-d14	1	90.3	90	90	
Phenol-d5	150	114.4	76	76	
2-Fluorophenol		114.4	76	76	
2,4,6-Tribromophenol		123.4	8~	87	7
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					

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenoi-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

SDG# See Garer

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

Page: lof L

7 Reviewer:\_\_ 2nd Reviewer:

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

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

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

Where:

SC = Sample concentation

SSC = Spiked sample concentration SA = Spike added

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

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples:

	Sp	Ke	Sample	Spiked	Sample	Matrix Spike	Spike	Matrix Spik	Matrix Spika Duplicata	MS/MSD	SD
Compound	Added (Mg)/kg	led ( )	Concentration (かん人)	Concer (۲۸)	Concentration (いく /た.)	Percent Recovery	ecovery	Percent Recovery	Recovery	RPD	
	MS	ø MSD	0	MS	U MSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenol						. 4					
N-Nitroso-di-n-propylamine											
4-Chloro-3-methylphenol											
Acenaphthene	2556	2550	٥	1890	2112	74	74	82	82	1	1/
Pentachlorophenol	١										
Pyrene	<b>→</b>	<del></del> t	30	2050	23/D	79	79	89	84	2	7

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

LDC# 24047 F29

# VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

Page: lof 1

Reviewer: NG 2nd Reviewer:

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

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 \* (SC/SA

SSC = Spike concentration SA = Spike added Where:

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

LCSC = Laboraotry control sample concentration 
LCSDC = Laboratory control sample duplicate concentration

LCS/LCSD samples: LCS 280 ~28761

	S	oike	ďS	ike	71	ı cs	21	csp	I CS/	CS/I CSD
	Ad (VS)	Added ( WS /L)	Concei (%)	Concentration	Percent F	Percent Recovery	Percent F	Percent Recovery	R	RPD
	1.0.8	<i>ا</i> ا دی	ICS	l CSD	Renorted	Recalc	Reported	Recalc.	Reported	Recalculated
N-Nitroso-di-n-propylamine										
	2540	νÆ	1810	MA	17	7.)				
	2540	<b>→</b>	2030		Q8	68				

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#: 24097 F29

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

Page:_	lof1_
Reviewer:	N
2nd reviewer:	V-

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

Υ	N	N/A
y	N	N/A
_	_	

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

Concentration =  $(A_{\bullet})(I_{\bullet})(V_{\bullet})(DF)(2.0)$ 

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

Area of the characteristic ion (EICP) for the compound

Area of the characteristic ion (EICP) for the specific internal standard

Amount of internal standard added in nanograms (ng)

Volume or weight of sample extract in milliliters (ml) or

Volume of extract injected in microliters (ul) ٧,

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

Dilution Factor. Df

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

Sample I.D. # 4 , SS

Conc. = (/65627)(40)(1.70 ml)(100)(1) (1222077)(6.2444)(52.39)(0.979)(1)

~ 700 ug/lex

#.	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		N 10/10/10/10/10/10/10/10/10/10/10/10/10/1			
		The second secon			
			·		
$\neg   \neg$					
_					
			*		

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 16, 2010

LDC Report Date:

October 11, 2010

Matrix:

Soil

Parameters:

Semivolatiles

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6583-1

Sample Identification

SA172-0BPC\*\* SA172-0BPC FD

SA172-0BPCMS

SA172-0BPCMSD

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

### Introduction

This data review covers 4 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, (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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

Samples indicated by a double asterisk on the front cover underwent a 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.
- 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 (r²) 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.

### VI. Surrogate Spikes

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

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. 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.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6583-1	All compounds reported below the PQL.	J (all detects)	A

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 SA172-0BPC\*\* and SA172-0BPC\_FD were identified as field duplicates. No semivolatiles were detected in any of the samples with the following exceptions:

	Concentrat	ion (ug/Kg)	222	D. (4)		
Compound	SA172-0BPC**	SA172-0BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Pyrene	14	17	-	3 (≤330)	-	-

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-6583-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6583-1	SA172-0BPC** SA172-0BPC_FD	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-6583-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Field Blank Data Qualification Summary - SDG 280-6583-1

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson** T

LDC #: 24047G2	aVALIDAT	ION COMPLETENESS WORKSHEE
SDG #: 280-658	3-1	Stage 2B/4
Laboratory: Test An	nerica	•

Reviewer: 2nd Reviewer:

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 8/16/10
11.	GC/MS Instrument performance check	A	,
111.	Initial calibration	A	% RSD ~~
IV.	Continuing calibration/ICV	A	ca/1a = 25 ]
V.	Blanks	A	
VI.	Surrogate spikes	Α	
VII.	Matrix spike/Matrix spike duplicates	Ą	
VIII.	Laboratory control samples	A	lcs
IX.	Regional Quality Assurance and Quality Control	N	·
X.	Internal standards	A	
XI.	Target compound identification	A	Not reviewed for Stage 2B validation.
XII.	Compound quantitation/CRQLs	K	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	SN	p = 1, ~
XVII.	Field blanks	NB	FB = FB 04072010 - RZC (from 280.2280

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

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

	3011						
† 1	SA172-0BPC** 0	11	·	21		31	
† 2	SA172-0BPC_FD D	12		22		32	
3	SA172-0BPCMS	13		23		33	
4	SA172-0BPCMSD	14		24		34	
5	MB 280 - 29198/1-A	15		25		35	
6	,	16		26	e <sup>a</sup>	36	
7		17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	

Page: \\_\ of \\_2 Reviewer: \\_\ \mathcal{JVL} 2nd Reviewer: \\_\ \\_

Method: Semivolatiles (EPA SW 846 Method 8270C)

Validation Area	Yes	No	NA	Findings/Comments
1. Technical holding times				The state of the s
All technical holding times were met.	<			
Cooler temperature criteria was met.			V. 172	
II. GC/MS Instrument performance check *** ********************************	122			
Were the DFTPP performance results reviewed and found to be within the specified criteria?				
Were all samples analyzed within the 12 hour clock criteria?		37×3-3 \$73	16.10 T T T	
(II) Initial calibration				
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				En A Sale Paris N. S.
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?	1			
V Blanks Rasjona (1996) Walter (1996) Alberta Alberta (1996)				
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.				
	2.52			
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?	engalis yaylış <sup>a</sup> eksese	. 1915 ° - 20 - 20 12 15		
vicinalis e de reconstitui disessi				A CONTRACTOR OF THE STATE OF TH
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?		,		
All/A Lebbs, depy organist, earniples as 2004 ill. Market Belle Bolica, 1987 ill. 1987				As a Petrol Control Co.
Was an LCS analyzed for this SDG?				

### **VALIDATION FINDINGS CHECKLIST**

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

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX Regional Quality Assurance and Quality Corpus				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
X-Innerros santanos y 2004 - 100 - 1				interior and the second
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				Manager ed Etype (construction of the
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. Compositio quartination/CRCIES 18 18 18 18 18 18 18 18 18 18 18 18 18				<b>Legislatin</b> and the first of the constitution
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 a enablikely identified compounds (31/39)			-	A North Control of the Control of th
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?			J	
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)				The second of th
System performance was found to be acceptable.				
		+ 2		
Overall assessment of data was found to be acceptable.				·
Field duplicate pairs were identified in this SDG.		i in superioris in the second	2644.00.00	
Target compounds were detected in the field duplicates.				
XVII. 72-0 Familia				
Field blanks were identified in this SDG.				
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-butyiphthalate	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	000. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP, 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	111.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)™	FFF. Di-n-octyiphthalate	ກກກ
N. 2-Nitrophenol**	CC. Dimethyiphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	WW.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS, Hexachlorobenzene	ННН. Benzo(k)fluoranthene	www.

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

LDC#: 24047G2a

## **VALIDATION FINDINGS WORKSHEET**

Field	<b>Duplicates</b>	
	Dupiloutoo	

Page:	lof/
Reviewer:	$\mathfrak{I}_{\mathcal{U}}$
2nd Reviewer:	6

METHOD: GC/MS SVOA (EPA SW 846 Method 8270C)
Y) N NA Were field duplicate pairs identified in this SDG?

Y N NA Y N NA

Compound Name	Conc (	ug/Kg)	RPD	Diff	Diff Limits	Quals
Compound Name	1	2	(≤50%)			(Parent Only)
Pyrene	14	17		3	≤330	

V:\FIELD DUPLICATES\24047G2a.wpd

LDC # 24 0 47

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page:

Reviewer: 2nd Reviewer:

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

 $A_x$  = 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)Standa #

7.5	1.0199	1.0199	1.1231	1.1231	(186)	Benzo(g,h,i)perylene		
8.0	1.1364	1.1364	1.1671	1.1671	(185)	Pyrene		
6.1	0.2313	0.2313	0.2424	0.2424	(184)	Hexachlorobenzene		
7.9	1.2421	1.2421	1.3180	1.3180	(183)	Fluorene		
8.9	1.0015	1.0015	1.0571	1.0571	(182)	Naphthalene		SK
3.7	0.5795	0.5795	0.5926	0.5926	(1S1)	1,4-Dioxane	8/27/2010	AL
	(Initial)	(Initial)	( 50 std)	( 50 std)	Standard)	Compound (Internal	Date	ard ID
%RSD	Average RRF	Average RRF	RRF	RRF			Calibration	
Reported	Recalculated	Reported	Recalculated	Reported				
	%RSD %RSD 3.7 8.9 7.9 6.1 8.0 7.5		Average RRF (Initial) 0.5795 1.0015 1.2421 0.2313 1.1364 1.0199	Reported         Recalculated           Average RRF         Average RRF           (Initial)         (Initial)           0.5795         0.5795           1.0015         1.0015           1.2421         1.2421           0.2313         0.2313           1.1364         1.1364           1.0199         1.0199	Recalculated         Reported         Recalculated           RRF         Average RRF         Average RRF           (50 std)         (Initial)         (Initial)           0.5926         0.5795         0.5795           1.0571         1.0015         1.0015           1.3180         1.2421         1.2421           0.2424         0.2313         0.2313           1.1671         1.1364         1.1364           1.1231         1.0199         1.0199	RRF         Recalculated Reported         Recalculated Reported         Recalculated Recalculated RRF           (50 std)         (50 std)         (Initial)         (Initial)           (50 std)         (50 std)         (Initial)         (Initial)           (1.0571         1.0571         1.0015         1.0015           (2424         0.2424         0.2313         0.2313           (1.1571         1.1671         1.1364         1.1364           (1.1231         1.1231         1.1231         1.0199	Reported         Recalculated Reported         Reported RRF         Recalculated RRF         Reported RRF         Recalculated RRF         Average RRF         Ave	RRF         RRF         Reported         Recalculated         Recalculated         Recalculated           Compound (Internal Standard)         (50 std)         (50 std)         (Initial)         (Initial)           1,4-Dioxane         (IS1)         0.5926         0.5926         0.5795         0.5795           Naphthalene         (IS2)         1.0571         1.0015         1.0015           Fluorene         (IS3)         1.3180         1.2421         1.2421           Hexachlorobenzene         (IS4)         0.2424         0.2313         0.2313           Pyrene         (IS5)         1.1671         1.1671         1.1364         1.1364           Benzo(g,h,i)perylene         (IS6)         1.1231         1.1231         1.0199         1.0199

Area IS	172314	669515	393544	662745	759660	781265
Area cpd	127636	884641	648342	200827	1108295	1096793
inc IS/Cpd	40/50	40/50	40/50	40/20	40/20	40/20

Conc	1,4-Dioxane	Naphthalene	Fluorene	Hexachlorob	Pyrene	Benzo(g,h,i)per
4.00	0.5778	1.1018	1.3240		1.2338	0.9595
10.00	0.6003	1.0722	1.3327	0.2454	1.2475	1.0450
20.00	0.6103	1.0714	1.3075	0.2448	1.2014	1.0900
50.00	0.5926	1.0571	1.3180	0.2424	1.1671	1.1231
80.00	0.5842	1.0008	1.2564	0.2335	1.1244	1.0769
120.00	0.5678	0.9489	1.1901	0.2252	1.0661	1.0108
160.00	0.5547	0.8964	1.1248	0.2168	1.0483	0.9476
200.00	0.5485	0.8636	1.0833	0.2109	1.0025	0.9066
×	0.5795	1.0015	1.2421	0.2313	1.1364	1.0199
S	0.0217	0.0893	0.0977	0.0140	0.0908	0.0768
•						

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 # 240 47 629

# Continuing Calibration Results Verification VALIDATION FINDINGS WORSHEET

Page \_\_\_ of\_ Reviewer: 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:

ave. RRF = initial calibration average RRF % Difference = 100  $^{\star}$  (ave. RRF - RRF)/ave. RRF

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

RRF = continuing calibration RRF

Ax = Area of compound

Ais = Area of associated internal standard

Cis = Concentration of internal standard Cx = Concentration of compound

	1:	<del>- 1</del>		_				Т	 <del>-</del>	 _	
Recalculated	Q%	3.3	2.3	2.3	3.2	1.7	8.3				
Reported	Q%	3.3	2.3	2.3	3.2	1.7	8.3				
Recalculated	(CC RRF)	0.5989	1.0246	1.2707	0.2387	1.1552	1.1046				
Reported	(CC RRF)	0.5989	1.0246	1.2707	0.2387	1.1552	1.1046				
Average RRF	(Initial RRF)	0.5795	1.0015	1.2421	0.2313	1.1364	1.0199				
	3)	(IS1)	(182)	(183)	(184)	(185)	(186)				
	Compound (Reference IS)	1,4-Dioxane	Naphthalene	Fluorene	Hexachlorobenzene	Pyrene	Benzo(g,h,i)perylene				
Calibration	Date	09/02/10									
	Standard ID	K6093									
	#	-									

Compound (Reference IS)	(S)	Concentration	Area Cpd	Area IS
		(IS/Cpd)		
1,4-Dioxane	(IS1)	40/80	167425	139782
Naphthalene	(182)	40/80	1107956	540652
Fluorene	(183)	40/80	806913	317514
Hexachlorobenzene	(IS4)	40/80	251814	527560
Pyrene	(185)	40/80	1385679	599747
Benzo(g,h,i)perylene	(981)	40/80	1411930	639110

LDC#: 24047 G 27

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

Page:	lof 1
Reviewer:	34
2nd reviewer:	7

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:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	100	67.0	67	67	ව
2-Fluorobiphenyl		68.9	69	69	
Terphenyl-d14		81.0	81	81	
Phenol-d5	15.0	110.1	73	73	
2-Fluorophenol		105.4	70	70	
2,4,6-Tribromophenol		114.18	77	77	V
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:					
	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol				·	
2,4,6-Tribromophenol		·			
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

LDC#: ひやのよう らとの SDG #: See Core

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

101 Page: 2nd Reviewer: Reviewer:\_

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

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

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

Where:

SC = Sample concentation

SSC = Spiked sample concentration SA = Spike added

MSDC = Matrix spike duplicate concentration

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

MSC = Matrix spike concentration

MS/MSD samples: \_

	So	ķ.	Sample	Spiked	Sample	Matrix Spike	Spike	Matrix Spik	Matrix Spike Duplicate	MS/MSD	SD
Compound	Added ( NS /K )	19d (K.)	Concentration	Concentration (いっ/に)	itration	Percent Recovery	ecovery	Percent Recovery	Recovery	RPD	0
	MS	O <sub>MSD</sub>	0	MS	/ MSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenol											
N-Nitroso-di-n-propylamine											
4-Chloro-3-methylphenol											
Acenaphthene	25.40	2700	٥	1950	1990	73	93	74	74	۲	~
Pentachiorophenol											
Pyrene	26.90	لم2 ح	þI	2300	2280	85	XE-	73	84	6.7	0.9

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.

100 #: 2404G 2

# Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification VALIDALION FINDINGS WORKSHEEL

Reviewer:\_

Page: Lof 1 2nd 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

SSC = Spike concentration SA = Spike added Where:

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

LCS/LCSD samples: \_

KS 280-29198

	S	ike	Spi	ike	31	CS	: -	csp	1 CS/	CS/I CSD
Compound	Added ( Mg / A	ded	Concentration ( bg / kg)	tration	Percent Recovery	ecovery	Percent F	Percent Recovery	R	RPD
	SUL	LCSD	SUI	UCSD LCSD	Reported	Recalc	Reported	Recalc	Renorted	Receiculated
Phenol										
N-Nitroso-di-n-propylamine										
4-Chioro-3-methylphenol										
Acenaphthene	2580	NA	05.81	N.A	7-7	77				
Pentachlorophenol		V						Ţ		
Pyrene	0220	<b>→</b>	2010		18	(8)				
							\			
	ń*									

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 #: 246 47 6 29

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	_lof_	1_
Reviewer:_	NY	,
2nd reviewer:_	· i	

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

/	Y	N	N/A
	Y	N	N/A
١.	-7	/	

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

Concentration =  $(A_x)(I_x)(V_y)(DF)(2.0)$ (A,)(RRF)(V,)(V,)(%S)

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

Area of the characteristic ion (EICP) for the specific internal standard

Amount of internal standard added in nanograms (ng)

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

Volume of extract injected in microliters (ul)

Volume of the concentrated extract in microliters (ul)

Dilution Factor. Df

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

Example:

Sample I.D. # ZZ

Conc. = (8454)(40)(1ml)(1vn)()

7353/0)(1.1364)(30.34)(0.979)()

= 13.69 ~ 14 ng/kg

2.0	= Factor of 2 to accoun	nt for GPC cleanup				
#	Sample ID	Compound		Reported Concentration ( )	Calculated Concentration ( )	Qualification
			······			
			· ···-			
						·
<b></b>						
					·	
					,	
i	I	1		I	L	L

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 20, 2010

LDC Report Date:

October 11, 2010

Matrix:

Soil

Parameters:

Semivolatiles

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6639-1

Sample Identification

**RSAN7-OBPC** 

### Introduction

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

This review follows 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- 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  $(r^2)$  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.

### 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

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.

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

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

### XI. Target Compound Identifications

Raw data were not reviewed for this SDG.

### XII. Project Quantitation Limit

All compound quantitation and CRQLs were within validation criteria with the following exceptions:

Sample	Compound	Finding	Flag	A or P
RSAN7-OBPC	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

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6639-1	All compounds reported below the PQL.	J (all detects)	Α

Raw data were not reviewed for this SDG.

### XIII. Tentatively Identified Compounds (TICs)

Raw data were not reviewed for this SDG.

### XIV. System Performance

Raw data were not reviewed for this SDG.

### XV. Overall Assessment

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

### XVI. Field Duplicates

No field duplicates were identified in this SDG.

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-6639-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6639-1	RSAN7-OBPC	Benzo(b)fluoranthene Benzo(k)fluoranthene	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Compound quantitation and CRQLs (q)
280-6639-1	RSAN7-OBPC	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-6639-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Field Blank Data Qualification Summary - SDG 280-6639-1

No Sample Data Qualified in this SDG

## **Tronox Northgate Henderson**

_DC #:	24047H2a	VALIDATION COMPLETENESS WORKSHEET	
SDG #:	280-6639-1	Stage 2B	
₋aboratoı	y: Test America		

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

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: ( /20 /ið
II.	GC/MS Instrument performance check	<b>4</b> .	·
111.	Initial calibration	A	% RSD +~ COV/101 = 25 }
IV.	Continuing calibration/ICV	A	COV/101 = 25 }
V.	Blanks	Ą	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	N	client spec
VIII.	Laboratory control samples	A	us
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	N	
XII.	Compound quantitation/CRQLs	SM	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	N	
XV.	Overall assessment of data	Α	
XVI.	Field duplicates	N	
XVII.	Field blanks	NB	FB = FB - 04 6 72010 - RZC (from 260-2280-2

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:

Cail

	5011				
† 1	RSAN7-OBPC	11	21	31	
<u> </u>	MB 280 - 29198 /-A	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	

# 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-Chioro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzyiphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	тт.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	ກກກ
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	w.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	www.

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

LDC#: 24047 HZ SDG#:

# Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

Page: 1 of 1 Reviewer: 2nd Reviewer:

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

Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation? Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?

*	Date	Sample ID	Finding	Associated Samples	Qualifications	
			GGG HHH Deaks uncloding	salved	3/n3/P (4)	<u></u>
			lab weed total peak	are,		
			for quantitation			

Comments: See sample calculation verification worksheet for recalculations

# Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 20 through August 23, 2010

LDC Report Date:

October 12, 2010

Matrix:

Soil/Water

Parameters:

Semivolatiles

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6674-1

### Sample Identification

BDT-3-N-10-10BPC

EB-08202010

BDT-3-N-10-12BPC

**BDT-3-N-10-10BPCMS** 

BDT-3-N-10-14BPC

BDT-3-N-10-10BPCMSD

BDT-3-N-10-16BPC\*\*

BDT-3-N-10-18BPC

BDT-3-N-10-2BPC

BDT-3-N-10-4BPC

BDT-3-N-10-4BPC FD

BDT-3-N-10-6BPC

BDT-3-N-10-8BPC

BDT-3-N-20-10BPC

BDT-3-N-20-12BPC

BDT-3-N-20-14BPC

BDT-3-N-20-16BPC

BDT-3-N-20-18BPC\*\*

BDT-3-N-20-2BPC

BDT-3-N-20-4BPC

BDT-3-N-20-6BPC

BDT-3-N-20-8BPC

BDT-3-N-20-8BPC FD

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

### Introduction

This data review covers 22 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

Samples indicated by a double asterisk on the front cover underwent a 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 (r²) 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 280-29437/1-A	8/31/10	Bis(2-ethylhexyl)phthalate	80.7 ug/Kg	All soil samples in SDG 280-6674-1

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			
BDT-3-N-10-10BPC	Bis(2-ethylhexyl)phthalate	100 ug/Kg	100U ug/Kg			
BDT-3-N-10-12BPC	Bis(2-ethylhexyl)phthalate	89 ug/Kg	89U ug/Kg			
BDT-3-N-10-16BPC**	Bis(2-ethylhexyl)phthalate	97 ug/Kg	97U ug/Kg			
BDT-3-N-10-2BPC	Bis(2-ethylhexyl)phthalate	97 ug/Kg	97U ug/Kg			

Sample EB-08202010 was identified as an equipment blank. No semivolatile contaminants were found in this blank.

### VI. Surrogate Spikes

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

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. 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.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P	
All samples in SDG 280-6674-1	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 BDT-3-N-10-4BPC and BDT-3-N-10-4BPC\_FD and samples BDT-3-N-20-8BPC and BDT-3-N-20-8BPC\_FD were identified as field duplicates. No semivolatiles were detected in any of the samples.

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-6674-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6674-1	BDT-3-N-10-10BPC BDT-3-N-10-12BPC BDT-3-N-10-14BPC BDT-3-N-10-16BPC** BDT-3-N-10-18BPC BDT-3-N-10-2BPC BDT-3-N-10-4BPC_FD BDT-3-N-10-6BPC BDT-3-N-10-6BPC BDT-3-N-20-10BPC BDT-3-N-20-12BPC BDT-3-N-20-14BPC BDT-3-N-20-14BPC BDT-3-N-20-14BPC BDT-3-N-20-16BPC BDT-3-N-20-16BPC BDT-3-N-20-16BPC BDT-3-N-20-16BPC BDT-3-N-20-4BPC BDT-3-N-20-4BPC BDT-3-N-20-4BPC BDT-3-N-20-6BPC BDT-3-N-20-8BPC BDT-3-N-20-8BPC BDT-3-N-20-8BPC_FD EB-08202010	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-6674-1

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
280-6674-1	BDT-3-N-10-10BPC	Bis(2-ethylhexyl)phthalate	100U ug/Kg	А	bl
280-6674-1	BDT-3-N-10-12BPC	Bis(2-ethylhexyl)phthalate	89U ug/Kg	А	bl
280-6674-1	BDT-3-N-10-16BPC**	Bis(2-ethylhexyl)phthalate	97U ug/Kg	А	bl
280-6674-1	BDT-3-N-10-2BPC	Bis(2-ethylhexyl)phthalate	97U ug/Kg	Α	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Equipment Blank Data Qualification Summary - SDG 280-6674-1

No Sample Data Qualified in this SDG

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

24047I2a Stage 2B/4 280-6674-1

Reviewer: 2nd Reviewer:

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 8/20 - 23/10
II.	GC/MS Instrument performance check	A	/
III.	Initial calibration	A	% RSD € 30° ~~ CW/IW € 25° Z
IV.	Continuing calibration/ICV	A	cw/w =252
V.	Blanks	ŚN)	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	Α	
VIII.	Laboratory control samples	A	ics/p
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	*	
XI.	Target compound identification	A	Not reviewed for Stage 2B validation.
XII.	Compound quantitation/CRQLs	<u> </u>	Not reviewed for Stage 2B validation.
XIII.	Tentatively identified compounds (TICs)	Ŋ	Not reviewed for Stage 2B validation.
XIV.	System performance	A	Not reviewed for Stage 2B validation.
XV.	Overall assessment of data	À	
XVI.	Field duplicates	M	$D_1 = 7.8$ $D_2 = 19.20$
XVII.	Field blanks	SAVE	EB = 21 FB = FB - 0413-2010- RIG2 - RZE

Note:

LDC #:

SDG #:

Laboratory: Test America

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

	July 4 Water								
1	BDT-3-N-10-10BPC		_ 11	BDT-3-N-20-10BPC	21 >	EB-08202010 N	Ų:	31 1	MB 280 - 29437/1-A
1 2	BDT-3-N-10-12BPC		12	BDT-3-N-20-12BPC	22	BDT-3-N-10-10BPCMS		- 32 <b>2</b>	MB 280-29014/1-A
3	BDT-3-N-10-14BPC	٠	- 13	BDT-3-N-20-14BPC	23	BDT-3-N-10-10BPCMSD ✓		33	,
۶ 4	BDT-3-N-10-16BPC**		14	BDT-3-N-20-16BPC	24		3	34	
- 5	BDT-3-N-10-18BPC		 15	BDT-3-N-20-18BPC**	25		3	35	
<b>∤</b>	BDT-3-N-10-2BPC		<del> </del> 16	BDT-3-N-20-2BPC	26		Ţ	36	
7	BDT-3-N-10-4BPC D1		- 17	BDT-3-N-20-4BPC	27		ļ	37	
8	BDT-3-N-10-4BPC_FD 0		18	BDT-3-N-20-6BPC	28		ļ	88	
- 9	BDT-3-N-10-6BPC		19	BDT-3-N-20-8BPC	29		ŀ	9	
10	BDT-3-N-10-8BPC		20	BDT-3-N-20-8BPC_FD by	30			Ю	

### **VALIDATION FINDINGS CHECKLIST**

Page: 1 of 2
Reviewer: JV

2nd Reviewer:

Method: Semivolatiles (EPA SW 846 Method 8270C)

Validation Area	Yes	No	NA	Findings/Comments
i. Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.				
ji: GC/MS: insuruneu (pario mareo) chest				
Were the DFTPP performance results reviewed and found to be within the specified criteria?	/			
Were all samples analyzed within the 12 hour clock criteria?		1		
ill stillate alteration				
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?				
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?	/			
Were all percent differences (%D) $\leq$ 25% and relative response factors (RRF) $\geq$ 0.05?	/			
V/Blage				<b>W. Sar Fare Willia</b>
Was a method blank associated with every sample in this SDG?				
Was a method blank analyzed for each matrix and concentration?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.			:	
	4.56			
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?			1	
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?	/		$\dashv$	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	_/			
VII A Laide Caude Action of Samples and all the Caude Ca				
Was an LCS analyzed for this SDG?				

LDC #: 240 47 I 29

### **VALIDATION FINDINGS CHECKLIST**

Validation Area Ye	res	No	NA	Findings/Comments
1 ·				1 mangs/comments
Was an LCS analyzed per extraction batch?	$\Box$			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	/			:
IX Regional Quality Asstrance and Quality Soution				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?		******************************		
X interior and the second control of the sec				<b>经现在经济地域的</b>
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?	/	A. 6 / 100 (100 A	o som miste	
24/3 Cycli Osmpou Ci olemnicanon (1997)				
Were relative retention times (RRT's) within ± 0.06 RRT units of the standard?	4			
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	$\Box$			
Were chromatogram peaks verified and accounted for?	$\Delta$		enderstatic e	
XIII sompoint 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?	/			
XIII Terrialityely kientified compounds [3] (3) (2) (3) (3) (4) (4) (4) (4) (4) (4) (4) (4) (4) (4				
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)?				
System performance was found to be acceptable.	$\overline{\mathcal{A}}$		50 Su. 2 Suk	
Overall assessment of data was found to be acceptable.				
Communication and the following the second communication and the second co				
	$\nearrow$			
Field duplicate pairs were identified in this SDG.	+		$\dashv$	
Target compounds were detected in the field duplicates.	hanga ta			
XVIsFettiens				
Field blanks were identified in this SDG.				
Target compounds were detected in the field blanks.		1		

## 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	LLt. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene∺	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
i. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS, Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	111.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate⊷	ດດດ
N. 2-Nitrophenol*	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	ww.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	www.

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

20	
24047 I	202
LDC #:_	SDG #:

### VALIDATION FINDINGS WORKSHEET Blanks

l of ) Page: Reviewer: 2nd Reviewer:

> Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". METHOD: GC/MS BNA (EPA SW 846 Method 8270C)

Was a method blank analyzed for each matrix?

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

Was a method blank analyzed for each concentration preparation level? Was a method blank associated with every sample?

 $\frac{V/N}{N/A}$  Was the blank contaminated? If yes, please see qualification below. Blank extraction date: Blank analysis date:

(79) Sample Identification 9 7 Z 27 41 Associated Samples: Z 4 8 ζ 00 MB 280-39437 Blank ID 80.7 下でれ Compound Conc. units: №5

2.84 N K

date:	
analysis (	
Blank	
date	
extraction	
lank	

Conc. units:	Compound					
	Blank ID					
Associated						
Associated Samples:						
	Sa					
	Sample Identification					
	tion					
		,	1	1		i

5x Phthalates 2x all others

### LDC# 24047 I 29

## VALIDATION FINDINGS WORKSHEET Surrogate Recovery

1 of 1

Page:

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

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

Were percent recoveries (%R) for surrogates 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?

N/N/N

					Ī				T	<del></del>	T		Г						T-		<u> </u>			П
Qualifications	No qual Conty, i out)	,																						
(1	(55-120)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
%R (Limits)	061																							
Surrogate	Hdi																							
Sample ID	8																							
Date																								
#																						:		

QC Limits (Water) 21-100

QC Limits (Soil) 25-121

10-123 33-110\* 16-110\*

19-122 20-130\* 20-130\*

S5 (2FP)= 2-Fluorophenol S6 (TBP) = 2,4,6-Tribromophenol S7 (2CP) = 2-Chlorophenol-44 S8 (DCB) = 1,2-Dichlorobenzene-44

QC Limits (Water) 35-114 43-116 33-141 10-94

QC Limits (Soil) 23-120

\* QC limits are advisory

30-115 18-137 24-113

S1 (NBZ) = Nitrobenzene-d5 S2 (FBP) = 2-Fluorobiphenyl S3 (TPH) = Terphenyl-d14 S4 (PHL) = Phenol-d5 LDC#: 14047 I 20

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

Page:	<u>lof 1</u>
Reviewer:	Ne
2nd reviewer:	1/2

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

Sample ID: #

SS = Surrogate Spiked

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	In	76.4	76	76	0
2-Fluorobiphenyl		75.7	76	76	
Terphenyl-d14	J.	86.7	87	8.7	
Phenol-d5	150	121.9	اع	8)	
2-Fluorophenol		123.0	6~	87	
2,4,6-Tribromophenol	1	100,4	67	67	
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol				·	
2,4,6-Tribromophenol		·			
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

LDC# 24047 I 24 SDG #: See Core

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

375 Page: lof L Reviewer:\_ 2nd Reviewer:

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

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

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

SSC = Spiked sample concentration SA = Spike added Where:

SC = Sample concentation

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

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples:

42

	ds	Spike	Sample	Spiked	Sample	Matrix	Matrix Spike	Matrix Spike Duplicate	Duplicate	MS/MSD	SD.
Compound	Ad )	ded )	Concentration ( )	Concentration (	itration )	Percent Recovery	ecovery	Percent Recovery	ecovery	RPD	
	MS	MSD		MS	MSD	Reported	Recalc	Reported	Rocain	Correction	Lotelinieno
Phenol											
N-Nitroso-di-n-propylamine											
4-Chloro-3-methylphenol											
Acenaphthene	2876	28.80	٥	2180	220)	79	79	86	CV)	-	-
Pentachlorophenol											
Pyrene	2870	0887	<b>→</b>	an	2450	48	by	85	88		_

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.

LUU#: 24042279

# Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification VALIDALION FINDINGS WORKSHEEL

Reviewer:\_\_

Page: Lof 1

2nd 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

SSC = Spike concentration SA = Spike added Where:

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

LCSC = Laboraotry control sample concentration LCSDC = Laboratory control sample duplicate concentration

29427 K-A -082 2 LCS/LCSD samples:

	ďS	ike	Spi	Ke	SUL	S	Ül	CSD	I CS/I CSD	csp
. Compound	Added (US /ka	ded ka )	Concentration (No /K.)	tration	Percent Recovery	ecovery	Percent Recovery	Recovery	RPD	Q
	SOI		1.05	J CSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenol										
N-Nitroso-di-n-propylamine										
4-Chloro-3-methylphenol										
Acenaphthene	05.57	ΑŻ	2090	N,A	42	82				
Pentachlorophenol										
Pyrene	2550	- 0	21.12		\$8	28				
							\			

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#: 24047 I24

# VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: of Y Reviewer: JVG 2nd Reviewer:

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

 $A_x$  = Area of Compound

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

%RSD = 100 \* (S/X)

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

X = Mean of the RRFs

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
		Calibration		RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
#	Standard ID Date	Date	Compound (Internal Standard)	( 50 std)	( 50 std)	(Initial)	(Initial)		
_	ICAL	9/3/2010	9/3/2010 [1,4-Dioxane (IS1)	0.6430	0.6430	0.6429	0.6429	6.0	5.96
	MSSD		Naphthalene (IS2)	1.0799	1.0799	1.0794	1:0794	1.3	1.30
	******		Fluorene (IS3)	1.3494	1.3494	1.3276	1.3276	3.1	3.09
			Hexachlorobenzene (IS4)	0.2361	0.2361	0.2444	0.2444	3.9	3.87
			Chrysene (IS5)	1.0436	1.0436	1.0676	1.0676	2.4	2.39
			Benzo(g,h,i)perylene (IS6)	1.0554	1.0554	1.0292	1.0292	9.5	9.49

•						
Conc	1,4-Dioxane	Naphthalene	Fluorene	Hexachlorob	Chrysene	Benzo(g,h,i)per
4.00	0.6400	1.0847	1.2767		1.0515	0.8429
10.00	0.7256	1.0641	1.2860	0.2309	1.0670	0.9316
20.00	0.6616	1.0764	1.2870	0.2382	1.0723	1.0000
50.00	0.6430	1.0799	1.3494	0.2361	1.0436	1.0554
80.00	0.6342	1.1063	1.3874	0.2485	1.1110	1.1183
120.00	0.6253	1.0871	1.3636	0.2475	1.0913	1.0941
160.00	0.6123	1.0748	1.3487	0.2535	1.0716	1.0952
200.00	0.6009	1.0620	1.3219	0.2561	1.0325	1.0959
×	0.6429	1.0794	1.3276	0.2444	1.0676	1.0292
S	0.0383	0.0140	0.0410	0.0094	0.0255	0.0976

1364895

1780490 1660410

40/50

40/50

353133

1258657

279929 1091792

1473825

40/50

225000

40/20

Area IS

Area cpd

nc IS/Cpd

692526 1196474

1168084

40/20

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# 26047 LX SDC# Z

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page:  $\sqrt{\phantom{a}}$  of  $\sqrt{\phantom{a}}$ Reviewer:  $\sqrt[3]{6}$  2nd Reviewer:  $\sqrt[3]{6}$ 

METHOD:

GC EPA SW 846 Method 8270C

Bis(2-eh)phthalate Parameter:

Date	Column	Compound	۲ area ratio	X conc ratio	ZvX
09/03/2010	Not specified	Bis(2-eh)phthalate	0.0290	0.100	
			0.1170	0.250	
			0.2768	0.500	
			0.7980	1.250	
			1.3731	2.000	
			2.0628	3.000	The state of the s
	-		2.7364	4.000	- Open op A de level per la lev
			3.3703	5.000	

0.2898

0.4680 0.5537 0.6384 0.6865

0.6876 0.6841 0.6741 0.5853

Regression Output:		Reported	
Constant	-0.04618	= 3	0.061100
Std Err of Y Est	0.03031		
R Squared	0.99951	12=	0.997500
No. of Observations	8.00000		
Degrees of Freedom	000000		
X Coefficient(s) 0.691474	474	= W	0.680400
Std Err of Coef. 0.006254	3254		

LDC # 246 47 I 29 SDG # See Cover

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

Page 1 of Reviewer:\_\_\_\_\_\_\_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:

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

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

ave. RRF = initial calibration average RRF Ax = Area of compound

RRF = continuing calibration RRF

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)	(Initial RRF)	(CC RRF)	(CC RRF)	%D	%D
1	D8366	09/07/10	1,4-Dioxane (IS	(IS1) 0.643	0.603	0.603	6.2	6.2
			Naphthalene (IS	(1S2) 1.079	1.123	1.123	4.0	4.0
			Fluorene (IS	(1S3) 1.328	1.396	1.396	5.1	5.1
			Hexachlorobenzene (IS4)	4) 0.244	0.249	0.249	1.9	1.9
			Bis(2-ethylhexyl)phthalate (IS5)	5) 80.000	89.200	89.180	11.5	11.5
			Benzo(g,h,i)perylene (IS6)	6) 1.029	1.138	1.138	10.6	10.6

Compound (Reference IS)	(	Concentration	Area Cpd	Area IS	Area Cpd	Area IS
		(IS/Cpd)				
1,4-Dioxane	(181)	40/80	329362	273164		
Naphthalene	(182)	40/80	2368897	1055116		
Fluorene	(183)	40/80	1864714	667902		
Hexachlorobenzene	(184)	40/80	572508	1149429		
Bis(2-ethylhexyl)phthalate	(185)	40/80	1887838	1279562		
Benzo(g,h,i)perylene	(186)	40/80	2819169	1238277		

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

Page:	<u> _</u> Ot/
Reviewer:_	No
nd reviewer:	1~

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

Y N N/A Y/N N/A

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

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

Concentration =  $(A_x)(I_x)(V_t)(DF)(2.0)$  $(A_{ls})(RRF)(V_o)(V_i)(%S)$ 

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

Area of the characteristic ion (EICP) for the specific internal standard

Amount of internal standard added in nanograms (ng)

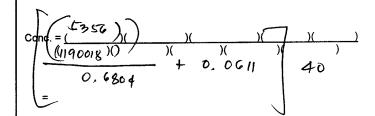
Volume or weight of sample extract in milliliters (ml) or grams (g).

Volume of extract injected in microliters (ul) V,

Volume of the concentrated extract in microliters (ul) V<sub>t</sub>

Dilution Factor. Df

%S Percent solids, applicable to soil and solid matrices only. Example: Sample I.D.



2,7

2.0	= Factor of 2 to accoun	t for GPC cleanup	L			
#	Sample ID	Compound		Reported Concentration ( )	Calculated Concentration ( )	Qualification
			final c	$m_{e.} = (2.7)$	1) (1~1) (1~	~ )
			1. 50	(3		
	<u> </u>	5 t t 4 t t t t t t t t t t t t t t t t			1.00	
	•			= 97.	2 ug lev	
				,	7.7	
						-

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 23 through August 24, 2010

**LDC Report Date:** 

October 11, 2010

Matrix:

Soil/Water

Parameters:

Semivolatiles

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6741-1

### Sample Identification

EB-08232010	BDT-3-N-5-8BPC	BDT-3-S-20-8BPC
BDT-3-N-15-2BPC	BDT-3-S-15-10BPC	BDT-3-N-15-2BPCMS
BDT-3-N-15-4BPC	BDT-3-S-15-12BPC	BDT-3-N-15-2BPCMSD
BDT-3-N-15-6BPC	BDT-3-S-15-14BPC	BDT-3-S-20-10BPCMS
BDT-3-N-15-8BPC	BDT-3-S-15-14BPC_FD	BDT-3-S-20-10BPCMSD
BDT-3-N-15-10BPC	BDT-3-S-15-16BPC	
BDT-3-N-15-12BPC	BDT-3-S-15-18BPC**	
BDT-3-N-15-14BPC	BDT-3-S-15-2BPC	
BDT-3-N-15-16BPC	BDT-3-S-15-4BPC	
BDT-3-N-15-18BPC**	BDT-3-S-15-6BPC	
BDT-3-N-15-6BPC_FD	BDT-3-S-15-8BPC	
BDT-3-N-5-10BPC	BDT-3-S-20-10BPC	
BDT-3-N-5-12BPC	BDT-3-S-20-12BPC	
BDT-3-N-5-14BPC	BDT-3-S-20-14BPC	
BDT-3-N-5-16BPC	BDT-3-S-20-16BPC	
BDT-3-N-5-18BPC**	BDT-3-S-20-18BPC**	
BDT-3-N-5-2BPC	BDT-3-S-20-2BPC	
BDT-3-N-5-4BPC	BDT-3-S-20-4BPC	
BDT-3-N-5-6BPC	BDT-3-S-20-6BPC	
BDT-3-N-5-8BPC_FD	BDT-3-S-20-6BPC_FD	

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

### Introduction

This data review covers 44 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

Samples indicated by a double asterisk on the front cover underwent a 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  $(r^2)$  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.

Sample EB-08232010 was identified as an equipment blank. 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.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6741-1	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 BDT-3-N-15-6BPC and BDT-3-N-15-6BPC\_FD and samples BDT-3-N-5-8BPC and BDT-3-N-5-8BPC\_FD and samples BDT-3-S-15-14BPC\_FD and samples BDT-3-S-20-6BPC and BDT-3-S-20-6BPC\_FD were identified as field duplicates. No semivolatiles were detected in any of the samples with the following exceptions:

	Concentra	ation (ug/Kg)	RPD	Difference		
Compound	BDT-3-N-15-6BPC	BDT-3-N-15-6BPC_FD	(Limits)	(Limits)	Flags	A or P
Bis(2-ethylhexyl)phthalate	96	97	-	1 (≤360)	•	-

	Concentra	ation (ug/Kg)	RPD	Difference			
Compound	BDT-3-N-5-8BPC	BDT-3-N-5-8BPC_FD	(Limits)	(Limits)	Flags	A or P	
Bis(2-ethylhexyl)phthalate	90	360U	-	270 (≤360)	-		

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-6741-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6741-1	EB-08232010	All compounds reported	J (all detects)	А	Project Quantitation Limit
	BDT-3-N-15-2BPC	below the PQL.		,	(sp)
	BDT-3-N-15-4BPC				
	BDT-3-N-15-6BPC				
	BDT-3-N-15-8BPC				
	BDT-3-N-15-10BPC				
	BDT-3-N-15-12BPC				
	BDT-3-N-15-14BPC				
	BDT-3-N-15-16BPC				
	BDT-3-N-15-18BPC**				
	BDT-3-N-15-6BPC_FD				
	BDT-3-N-5-10BPC				
	BDT-3-N-5-12BPC				
	BDT-3-N-5-14BPC				
	BDT-3-N-5-16BPC				
	BDT-3-N-5-18BPC**				
	BDT-3-N-5-2BPC				
	BDT-3-N-5-4BPC				
	BDT-3-N-5-6BPC			Ì	
	BDT-3-N-5-8BPC_FD				
	BDT-3-N-5-8BPC				
	BDT-3-S-15-10BPC				
	BDT-3-S-15-12BPC				
	BDT-3-S-15-14BPC				
	BDT-3-S-15-14BPC_FD		•		
	BDT-3-S-15-16BPC				
	BDT-3-S-15-18BPC**				
	BDT-3-S-15-2BPC				
	BDT-3-S-15-4BPC				
	BDT-3-S-15-6BPC				
	BDT-3-S-15-8BPC				
	BDT-3-S-20-10BPC				
	BDT-3-S-20-12BPC				
	BDT-3-S-20-14BPC				
	BDT-3-S-20-16BPC				
	BDT-3-S-20-18BPC**				
	BDT-3-S-20-2BPC				
	BDT-3-S-20-4BPC				
	BDT-3-S-20-6BPC				
	BDT-3-S-20-6BPC_FD				
	BDT-3-S-20-8BPC			1	

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-6741-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Equipment Blank Data Qualification Summary - SDG 280-6741-1

No Sample Data Qualified in this SDG

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Field Blank Data Qualification Summary - SDG 280-6741-1

No Sample Data Qualified in this SDG

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

LDC #:	24047J2a	VALIDATION COMPLETE
SDG #:_	280-6741-1	Stage 2E
Laborato	ry: Test America	

Date: 10 /06/10 Page: \ of \ \nu Reviewer: 17/6 2nd Reviewer: \_\_\_\_\_\_\_\_

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 8 /23-24 /10
11.	GC/MS Instrument performance check	A	1
111.	Initial calibration	A	2, RSD
IV.	Continuing calibration/ICV	K	CONTO = 25 ]
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	LCS (p
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	_ A	
XI.	Target compound identification	Á	Not reviewed for Stage 2B validation.
XII.	Compound quantitation/CRQLs	<u> </u>	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	<u> </u>
XVI.	Field duplicates	SW	$D_1 = 4, 1$ $D_2 = 21, 20$ $D_3 = 29, 25$ $D_4 = 39, 4$
XVII.	Field blanks	SND	FEB = 1 $FB = FB - 04/3 × 0.10 - R162 - R26$ (from $260 - 2400 - 2$ )  Is detected. $D = Duplicate$

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

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

	> m			WALL				
1 4	EB-08232010	W	11 1	BDT-3-N-15-6BPC_FD 5	21	BDT-3-N-5-8BPC D✓ S	31 3	BDT-3-S-15-8BPC 5
2 )	BDT-3-N-15-2BPC	5	12 }	BDT-3-N-5-10BPC	22 3	BDT-3-S-15-10BPC	32 3	BDT-3-S-20-10BPC
3 1	BDT-3-N-15-4BPC		13	BDT-3-N-5-12BPC	23	BDT-3-S-15-12BPC	33 >	BDT-3-S-20-12BPC
4 1	BDT-3-N-15-6BPC <i>D</i> <sub>1</sub>		14	BDT-3-N-5-14BPC	24 3	BDT-3-S-15-14BPC 03	34	BDT-3-S-20-14BPC
5 1	BDT-3-N-15-8BPC		15	BDT-3-N-5-16BPC	25 3	BDT-3-S-15-14BPC_FD	35	BDT-3-S-20-16BPC
6 1	BDT-3-N-15-10BPC		† 16 \	BDT-3-N-5-18BPC**	26 3	BDT-3-S-15-16BPC	36	BDT-3-S-20-18BPC**
7 1	BDT-3-N-15-12BPC		17 1	BDT-3-N-5-2BPC	- 27 3	BDT-3-S-15-18BPC**	37	BDT-3-S-20-2BPC
8 1	BDT-3-N-15-14BPC		18	BDT-3-N-5-4BPC	28	BDT-3-S-15-2BPC	38	BDT-3-S-20-4BPC
9	BDT-3-N-15-16BPC		19 \	BDT-3-N-5-6BPC	29	BDT-3-S-15-4BPC	39	BDT-3-S-20-6BPC 04
10 (	BDT-3-N-15-18BPC**	1	20 \	BDT-3-N-5-8BPC_FD	30	BDT-3-S-15-6BPC	40	BDT-3-S-20-6BPC_FD 4

### Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

Stage 2B/4

Date:	10/06/1
Page:_	2 of 2
Reviewer:	2/1
2nd Reviewer:	$\sim$

SDG #: 280-6741-1 Laboratory: Test America

LDC #:

24047J2a

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

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

	Validation Area		Comments
l.	Technical holding times		Sampling dates:
II.	GC/MS Instrument performance check		
III.	Initial calibration		Col
IV.	Continuing calibration/ICV		
V.	Blanks		1 ever
VI.	Surrogate spikes		Page
VII.	Matrix spike/Matrix spike duplicates		Y "
VIII.	Laboratory control samples		
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards		
XI.	Target compound identification		Not reviewed for Stage 2B validation.
XII.	Compound quantitation/CRQLs		Not reviewed for Stage 2B validation.
XIII.	Tentatively identified compounds (TICs)		Not reviewed for Stage 2B validation.
XIV.	System performance		Not reviewed for Stage 2B validation.
XV.	Overall assessment of data		
XVI.	Field duplicates		
XVII.	Field blanks		

Note:

A = Acceptable

ND = No compounds detected

D = Duplicate TB = Trip blank

N = Not provided/applicable SW = See worksheet R = Rinsate FB = Field blank

EB = Equipment blank

Validated Samples:

\*\* Indicates sample underwent Stage 4 validation

41	BDT-3-S-20-8BPC \$	51 1	MB 200 -29541/1-A	61	71	
	BDT-3-N-15-2BPCMS	52 7	280-29746/1-2		72	
43	BDT-3-N-15-2BPCMSD	53 °	280-29755/LA		73	
44	₿DT-3-S-20-10BPCMS	- 4 54	280-290/4/-A	l I	74	
45	BDT-3-S-20-10BPCMSD	55	<u> </u>	65	75	
46		56		66	76	
47		57		67	77	
48		58		68	78	
49		59		69	79	
50	-	60		70	80	

LDC #: 29047 Jza

### VALIDATION FINDINGS CHECKLIST

Method: Semivolatiles (EPA SW 846 Method 8270C)

(2.7.6.V 6.6 Mediod 62.766)				
Validation Area	Yes	No	NA	Findings/Comments
1. Technical bolding times	T	L		Total Control of the
All technical holding times were met.	-		<u> </u>	
Cooler temperature criteria was met. If GCMS instrument performance in ⊇t				
Were the DFTPP performance results reviewed and found to be within the specified criteria?				No.
Were all samples analyzed within the 12 hour clock criteria?				
All Animal Californian 2007				
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				TOTAL CASE A SECTION .
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?				
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?				
Were all percent differences (%D) $\leq$ 25% and relative response factors (RRF) $\geq$ 0.05?				
Wellands and the second se				ALSO SELECTION OF THE SECOND O
Was a method blank associated with every sample in this SDG?	1			
Was a method blank analyzed for each matrix and concentration?	4			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
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?				
Me tagina entegración de la companya				
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 A Debo Aliony control complexes				
Was an LCS analyzed for this SDG?				

LDC #: 240 97 5~9

### **VALIDATION FINDINGS CHECKLIST**

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

	T	Г	T	
Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?	-	<del> </del>	<u> </u>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	/			
IX. Regional Quality Assurance and Grainty Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
Xvinieros sancards versiones appearants at the control of the cont				
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?				
KLACICE COLICENSIA COLICENSIA COLICENSIA DE LA COLICENSIA				Angelia or Personal Control
Were relative retention times (RRT's) within $\pm$ 0.06 RRT units of the standard?				
Did compound spectra meet specified EPA "Functional Guidelines" criteria?				
Were chromatogram peaks verified and accounted for?				
XII Composing quantitation/CRC/LS 1998 (1998) 4 1998 (1998)				
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: Tentalively identified compounds (TCs) 3.5				
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)?			/	
Manager and a confidence of the confidence of th				
System performance was found to be acceptable.	/	Volla-Seuse-science		
Outself accompany of data was found to be acceptable				
Overall assessment of data was found to be acceptable.				
Field duplicate pairs were identified in this SDG.	$\perp$	$\perp$		
Target compounds were detected in the field duplicates.				
XVII selvenks				
Field blanks were identified in this SDG.	7			
Target compounds were detected in the field blanks.	M	1		

## VALIDATION FINDINGS WORKSHEET

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

A. Phenol**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachiorophenol™	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phananthrene	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 <sup>th</sup>	T. 4-Chloroanlline	II. 4-Nitrophenol*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol™	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP, Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroanlline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	111.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate™	ກດດ
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	VVV.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	www.

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

LDC#: 24047J2a

### VALIDATION FINDINGS WORKSHEET Field Duplicates

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

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

Y N NA Y N NA Were field duplicate pairs identified in this SDG?

Were target analytes detected in the field duplicate pairs?

Compound Name	Conc (	ug/Kg)	RPD	Diff	Diff Limits	Quals
Oonipouna Numo	4	. 11	(≤50%)			(Parent Only)
Bis(2-ethylhexyl)phthalate	96	97		1	≤360	

Compound Name	Conc (	ug/Kg)	RPD	Diff	Diff Limits	Quals
Compound Numo	21	20	(≤50%)			(Parent Only)
Bis(2-ethylhexyl)phthalate	90	360U		270	≤360	

V:\FIELD DUPLICATES\24047J2a.wpd

LDC#: 24147 Jun

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

οĘ Page: Reviewer: 2nd Reviewer:

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

 $C_x$  = Concentration of compound, A<sub>x</sub> = Area of Compound

A<sub>is</sub> = Area of associated internal standard  $C_{\rm is}$  = Concentration of internal standard

%RSD = 100 \* (S/X)

S= Standard deviation of the RRFs,

X = Mean of the RRFs

					Papartad	Possinologia	Donortod	Postol released	7 0 0 0 0	
					ובלמופח	Necalculated	neholied Lebolied	Recalculated	Reported	Recalculated
<u>පී</u>	ථ	Calibration			RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
Standard ID		Date	Compound (Internal St	nal Standard)	(50 std)	( 50 std)	(Initial)	(Initial)		
ICAL 8	8	3/30/2010	8/30/2010 1,4-Dioxane	(1S1)	0.6494	0.6494	0.6538	0.6538	3.2	3.21
MSS B			Naphthalene	(IS2)	1.0810	1.0810	1.0482	1.0482	11.0	11.01
			Fluorene	(183)	1.3696	1.3696	1.3037	1.3037	11.0	10.98
	_		Hexachlorobenzene	(IS4)	0.2517	0.2517	0.2454	0.2454	3.8	3.76
			Chrysene	(185)	1.1455	1.1455	1.0975	1.0975	8.1	8.10
			Benzo(g,h,i)perylene	(186)	1.1162	1.1162	1.0926	1.0927	2.5	2.49
				-			_	•		•

ı——						
Area IS	246217	961965	558782	945857	1059049	1037135
Area cpd	199862	1299821	956646	297546	1516369	1447002
Inc IS/Cpd	40/20	40/20	40/20	40/20	40/20	40/20

Conc	1,4-Dioxane	Naphthalene	Fluorene	Hexachlorob	Chrysene	Benzo(g,h,i)per
4.00	0.6780	1.1880	1.4403	0.2501	1.1731	1.0576
10.00	0.6510	1.1590	1.4246	0.2516	1.1774	1.0704
20.00	0.6855	1.1306	1.4342	0.2514	1.1786	1.0855
50.00	0.6494	1.0810	1.3696	0.2517	1.1455	1.1162
80.00	0.6655	1.0601	1.3277	0.2522	1.1198	1.1433
120.00	0.6423	0.9839	1.2284	0.2435	1.0281	1.1029
160.00	0.6276	0.9020	1.1307	0.2339	1.0006	1.0822
200.00	0.6313	0.8811	1.0742	0.2287	0.9572	1.0831
×	0.6538	1.0482	1.3037	0.2454	1.0975	1.0927
S	0.0210	0.1154	0.1432	0.0092	0.0889	0.0273
•						

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# マイロインプ

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

> o Page: Reviewer: 2nd Reviewer:

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

 $C_x$  = Concentration of compound, A<sub>x</sub> = Area of Compound

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

S= Standard deviation of the RRFs,

X = Mean of the RRFs

%RSD = 100 \* (S/X)

Reported Recalculated	%RSD %RSD		6.0 5.96	1.3 1.30	3.1 3.09	3.9 3.87	2.4 2.39	9.5 9.49	
Recalculated R	Average RRF	(Initial)	0.6429	1.0794	1.3276	0.2444	1.0676	1.0292	
Reported	Average RRF	(Initial)	0.6429	1.0794	1.3276	0.2444	1.0676	1.0292	
Recalculated	RRF	( 50 std)	0.6430	1.0799	1.3494	0.2361	1.0436	1.0554	
Reported	RRF	( 50 std)	0.6430	1.0799	1.3494	0.2361	1.0436	1.0554	
		Compound (Internal Standard)	9/3/2010 1,4-Dioxane (IS1)	Naphthalene (IS2)	Fluorene (IS3)	Hexachlorobenzene (IS4)	Chrysene (IS5)	Benzo(g,h,i)perylene (IS6)	
	Calibration	Date	9/3/2010						
		Standard ID	ICAL	MSS D					

-						
Conc	1,4-Dioxane	Naphthalene	Fluorene	Hexachlorob	Chrysene	Benzo(g,h,i)per
4.00	0.6400	1.0847	1.2767		1.0515	0.8429
10.00	0.7256	1.0641	1.2860	0.2309	1.0670	0.9316
20.00	0.6616	1.0764	1.2870	0.2382	1.0723	1.0000
50.00	0.6430	1.0799	1.3494	0.2361	1.0436	1.0554
80.00	0.6342	1.1063	1.3874	0.2485	1.1110	1.1183
120.00	0.6253	1.0871	1.3636	0.2475	1.0913	1.0941
160.00	0.6123	1.0748	1.3487	0.2535	1.0716	1.0952
200.00	0.6009	1.0620	1.3219	0.2561	1.0325	1.0959
×	0.6429	1.0794	1.3276	0.2444	1.0676	1.0292
S	0.0383	0.0140	0.0410	0.0094	0.0255	0.0976

1364895

1780490 353133

1258657

1660410

1091792 692526 1196474

1473825 225000

40/20

40/20

1168084

40/20

40/20 40/20 40/20

Area IS

Area cpd

nc IS/Cpd

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 # 240 47 Ju SDG# En Com

# VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: 3 of 3Reviewer: 3 of 32nd Reviewer: 4 color

METHOD: GC E

GC EPA SW 846 Method 8270C

Parameter: Bis

Bis(2-eh)phthalate

	amile)	bailbamo	Y Y orea ratio	X X Sono ratio	X^2
	5		5		
ı	Not specified	Bis(2-eh)phthalate	0.0290	0.100	
			0.1170	0.250	
			0.2768	0.500	
			0.7980	1.250	
	and search dear for sold	,	1.3731	2.000	
		·	2.0628	3.000	
			2.7364	4.000	the second secon
			3.3703	5.000	

0.2898

0.4680

0.5537

0.6865

0.6841

0.5853

Regression Output:		Reported	
Constant	-0.04618	II O	0.061100
Std Err of Y Est	0.03031		
R Squared	0.99951	r2 =	0.997500
No. of Observations	8.00000		
Degrees of Freedom	00000.9		
X Coefficient(s) 0.	0.691474	m =	0.680400
Std Err of Coef. 0.	0.006254		

LDC # 24 047 JrA SDG # See Cover

## Continuing Calibration Results Verification VALIDATION FINDINGS WORSHEET

Page\_\_ Reviewer:\_ 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:

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

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

ave. RRF = initial calibration average RRF Cx = Concentration of compound Ax = Area of compound

Ais = Area of associated internal standard Cis = Concentration of internal standard

RRF = continuing calibration RRF

		Calibration			Average RRF	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Date	Compound (Reference IS)	()	(Initial RRF)	(CC RRF)	(CC RRF)	%D	%D
-	D8366	09/07/10	1,4-Dioxane	(IS1)	0.643	0.603	0.603	6.2	6.2
			Naphthalene	(182)	1.079	1.123	1.123	4.0	4.0
			Fluorene	(183)	1.328	1.396	1.396	5.1	5.1
			Hexachlorobenzene	(184)	0.244	0.249	0.249	1.9	1.9
			Bis(2-ethylhexyl)phthalate	(185)	80.000	89.200	89.180	11.5	11.5
			Benzo(g,h,i)perylene	(9SI)	1.029	1.138	1.138	10.6	10.6
2	D8411	09/08/10	1,4-Dioxane	(181)	0.643	0.638	0.638	0.7	0.7
			Naphthalene	(182)	1.079	1.116	1.116	3.4	3.4
			Fluorene	(183)	1.328	1.371	1.371	3.3	3.3
			Hexachlorobenzene	(184)	0.244	0.250	0.250	2.4	2.4
			Bis(2-ethylhexyl)phthalate	(185)	80.000	84.500	84.480	5.6	5.6
			Benzo(g,h,i)perylene	(981)	1.029	1.164	1.164	13.1	13.1

Compound (Reference IS)		Concentration	Area Cpd	Area IS	Area Cpd	Area IS
		(IS/Cpd)				
1,4-Dioxane	(1S1)	40/80	329362	273164	331786	259818
Naphthalene	(182)	40/80	2368897	1055116	2226266	997033
Fluorene	(IS3)	40/80	1864714	667902	1761648	642587
Hexachlorobenzene	(IS4)	40/80	572508	1149429	547910	1094403
Bis(2-ethylhexyl)phthalate	(IS5)	40/80	1887838	1279562	1774419	1271596
Benzo(g,h,i)perylene	(186)	40/80	2819169	1238277	2855048	1226593

LDC # 74047Jnc SDG # See Cover

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

ېو Page \_\_ Reviewer:\_ 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:

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

ave. RRF = initial calibration average RRF Where:

Ais = Area of associated internal standard RRF = continuing calibration RRF

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

Ax = Area of compound

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)	%D	%D
-	B0064	09/04/10	1,4-Dioxane	(181)	0.654	0.652	0.652	0.3	0.3
			Naphthalene	(182)	1.048	1.064	1.064	1.5	1.5
			Fluorene	(183)	1.304	1.341	1.341	2.9	2.9
			Hexachlorobenzene (	(184)	0.245	0.242	0.242	1.5	1.5
			Chrysene	(185)	1.098	1.095	1.095	0.2	0.2
			Benzo(g,h,i)perylene (	(981)	1.093	1.135	1.135	3.9	3.9
2	B0151	09/08/10	1,4-Dioxane	(181)	0.654	0.626	0.626	4.2	4.2
			Naphthalene	(IS2)	1.029	1.056	1.056	2.6	2.6
			Fluorene	(183)	1.286	1.305	1.305	1.5	1.5
		,	Hexachlorobenzene (	(IS4)	0.245	0.233	0.233	4.8	4.8
			Chrysene	(185)	1.078	1.085	1.085	0.7	0.7
	·		Benzo(g,h,i)perylene (	(186)	1.035	1.131	1.131	9.3	9.3

Compound	Concentration	Area Cpd	Area IS	Area Cpd	Area IS	Area Cpd	Area IS
	(IS/Cpd)						
1,4-Dioxane	40/80	307634	236058	298831	238575	298831	238575
Naphthalene	40/80	1961036	921628	1961939	929068	1961939	929068
Fluorene	40/80	1415785	527712	1398808	535829	1398808	535829
Hexachlorobenzer	40/80	435990	902311	428209	916980	428209	916980
Chrysene	40/80	2155462	983874	2106014	970074	2106014	970074
Benzo(g,h,i)peryle	40/80	2233492	983827	2199377	972419	2199377	972419

LDC#: 24047 J29

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

Page:	_lof_t_
Reviewer:_	NG
2nd reviewer:_	_ <u> </u>

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: # 10

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	100	67.7	68	(8	0
2-Fluorobiphenyl		73.1	73	73	
Terphenyl-d14	1	97.4	97	97	
Phenol-d5	150	104.5	7/	71	
2-Fluorophenol		105,9	71	71	
2,4,6-Tribromophenol	V	112.3	75	75	
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5			•		
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4	·				

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5			·		
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5		·			
2-Fluorophenol				·	
2,4,6-Tribromophenol		·			
2-Chlorophenol-d4				·	
1,2-Dichlorobenzene-d4					

LDC# 240 47 J29 SDG #: See Core

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

Page: 2nd Reviewer:\_ Reviewer:

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

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

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

Where: SSC = Spiked sample concentration SA = Spike added

SC = Sample concentation

MSDC = Matrix spike duplicate concentration

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

MSC = Matrix spike concentration

	es 	ike	Sample	Spiked	Sample	Matrix Spike	Spike	Matrix Spike Duplicate	Duplicate	USW/SW	sp
Compound	Ad Tug	Added (K)	Concentration $(4c/k)$	Concentration $(\mathcal{N}/\mathcal{K})$	ncentration ル/く)	Percent Recovery	есоvегу	Percent Recovery	ecovery	RPD	
	MS	Msp		MS	MSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenol											
N-Nitroso-di-n-propylamine											
4-Chloro-3-methylphenol											
Acenaphthene	2800	2720	0 -	2190	2231	73	28	8~	82	λ	٨
Pentachlorophenol											
Pyrene	09 m	272	7	240	2350	<i>38</i>	18	18	りる	λ	>
			-								

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.

10 #: 24047 Jaa

# VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

Reviewer:\_

Page: Lof 1 2nd Reviewer:\_

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

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 \* (SC/SA

Where: SSC = Spike concentration SA = Spike added

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

3 LCS/LCSD samples: \_

A-2/14262 - 08c

								The second secon		
	Š	ike	ďS	ike	01	CS	31	csn	1001	CS/I CSD
Compound	Ad (us	Added (45 /k )	Concel	Concentration	Percent Recovery	Recovery	Percent Recovery	<b>Зесо</b> very	<u> </u>	RPD
	108	C LCSD	SUL	l CSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenol										
N-Nitroso-di-n-propylamine										
4-Chloro-3-methylphenol										
Acenaphthene	26.70	NA	2120	NA	08	8.0				
Pentachlorophenol										
Pyrene	29.20	<b>→</b>	0461	7	87	&				
								-		

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#: 24047 Jag

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	<u> [01                                   </u>
Reviewer:	22
2nd reviewer:_	h

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

Y N N/A Y N N/A

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

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

Concentration = (A,)(I,)(V,)(DF)(2.0) (A<sub>a</sub>)(RRF)(V<sub>o</sub>)(V,)(%S)

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

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

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

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

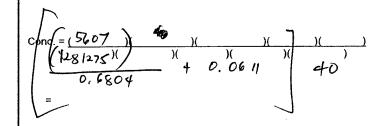
V<sub>i</sub> = Volume of extract injected in microliters (ul)
 V<sub>i</sub> = Volume of the concentrated extract in microliters (ul)

Df = Dilution Factor.

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

Example:

Sample I.D. # 16 , FEE



2 2.70

2.0	= Factor of 2 to accoun	t for GPC cleanup				
#	Sample ID	Compound		Reported Concentration	Calculated Concentration ( )	Qualification
			final cu	$v_{c.} = 2.7$	0 (1M) (	1000)
					30.09) (	.916)
				= 98,	25	
				7 980	19 /10	
					0	
	<del></del>			<u> </u>		<u> </u>

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

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 24, 2010

LDC Report Date:

October 11, 2010

Matrix:

Soil

Parameters:

Semivolatiles

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6783-1

### Sample Identification

BDT-3-S-5-10BPC BDT-3-S-5-2BPCMSD BDT-3-S-5-12BPC BDT-3-S-5-4BPCMSD BDT-3-S-5-4BPCMSD

BDT-3-S-5-16BPC BDT-3-S-5-18BPC\*\*

BDT-3-S-5-2BPC

BDT-3-S-5-4BPC

BDT-3-S-5-6BPC

BDT-3-S-5-8BPC

BDT-3-S-10-10BPC

BDT-3-S-10-12BPC

BDT-3-S-10-14BPC

BDT-3-S-10-16BPC

BDT-3-S-10-18BPC\*\*

BDT-3-S-10-2BPC

BDT-3-S-10-4BPC

BDT-3-S-10-4BPC FD

BDT-3-S-10-6BPC

BDT-3-S-10-8BPC

BDT-3-S-5-2BPCMS

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

### Introduction

This data review covers 23 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, (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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

Samples indicated by a double asterisk on the front cover underwent a 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  $(r^2)$  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.

No field blanks were identified in this SDG.

### VI. Surrogate Spikes

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

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

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

### XI. Target Compound Identifications

All target compound identifications were within validation criteria for samples on which a 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.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6783-1	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 BDT-3-S-10-4BPC and BDT-3-S-10-4BPC\_FD were identified as field duplicates. No semivolatiles were detected in any of the samples.

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-6783-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6783-1	BDT-3-S-5-10BPC BDT-3-S-5-12BPC BDT-3-S-5-14BPC BDT-3-S-5-16BPC BDT-3-S-5-18BPC** BDT-3-S-5-2BPC BDT-3-S-5-6BPC BDT-3-S-5-6BPC BDT-3-S-10-10BPC BDT-3-S-10-12BPC BDT-3-S-10-14BPC BDT-3-S-10-16BPC BDT-3-S-10-16BPC BDT-3-S-10-4BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-6783-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Field Blank Data Qualification Summary - SDG 280-6783-1

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson

LDC #:	24047K2a	VALIDATION COMPLETENESS WORKSHEET
SDG #:	280-6783-1	Stage 2B/4
Laborato	ry: <u>Test America</u>	

Date: 10/66 /co Page:\_\_lof\_/ Reviewer: NC 2nd Reviewer: \_\_\_\_

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 8/24 /10
II.	GC/MS Instrument performance check	A	
111.	Initial calibration	A	2 KD r7
IV.	Continuing calibration/ICV	A	cw/10 = 25 }
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	LCS
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	A	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	KD	D = 16, 17
XVII.	Field blanks	<b>EN</b> ?	FB = FB-04/3 2010 - RI 62 - RZE (from 280

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

		<u> </u>	201	12						
1	BDT-3-S-5-10BPC		. 구 11	BDT-3-S-10-12	3PC	21	<b>め</b> り BDT-3-S-5-2 <del>MSD</del>	c msp	31	MB 280- 29755/1-A
2	BDT-3-S-5-12BPC		ን 12	BDT-3-S-10-14		22 ~	BDT-3-S-5-4 <del>MS</del>	ms	ر 32 ع	MB 286- 29889/-
3 1	BDT-3-S-5-14BPC		13 <b>~</b>	BDT-3-S-10-16		23	BDT-3-S-5-4MSD,	\ Vrc D	33	
4 1	BDT-3-S-5-16BPC		~~ 14	BDT-3-S-10-18**		24			34	
- 1 5	BDT-3-S-5-18BPC*	*	15 <sup>~</sup>	BDT-3-S-10-2		25			35	
6	BDT-3-S-5-2 BPC		7 16	BDT-3-S-10-4	b	26			36	
7	BDT-3-S-5-4		า 17	BDT-3-S-10-4_ <b>ED</b>	FD D	27			37	
8 7	BDT-3-S-5-6	1	18 <b>7</b>	BDT-3-S-10-6		28			38	
9.	BDT-3-S-5-8	1	19	BDT-3-S-10-8		29			39	
10	BDT-3-S-10-10	2	20	BDT-3-S-5-2 <b>M6</b>	/ Ms	30			40	

### **VALIDATION FINDINGS CHECKLIST**

Page: \\_\ of \\_2 Reviewer: \\_\mathcal{JV} 2nd Reviewer: \\_\mathcal{L}

Method: Semivolatiles (EPA SW 846 Method 8270C)

Validation Area	Yes	No	NA	Findings/Comments
1. Technical holding times				Thomas Commons
All technical holding times were met.	/			
Cooler temperature criteria was met.				
III. GCMS Instrument performance cresk = 230 state (120				Appropriate Control of the Control o
Were the DFTPP performance results reviewed and found to be within the specified criteria?	/			
Were all samples analyzed within the 12 hour clock criteria?		NASA N 888		
III Initial calibration:				A PROPERTY OF THE PROPERTY OF
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				And the second second second
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?				
// Blance				AND CONTRACTORS
Was a method blank associated with every sample in this SDG?				
Was a method blank analyzed for each matrix and concentration?	4			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
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?				
vi castellanda este				
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?				
Viide seas a regime Asimbers				
Was an LCS analyzed for this SDG?	1			

LDC#: 24047 1729

### **VALIDATION FINDINGS CHECKLIST**

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

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
DX Regional Quality Assurance and Oxiality Control				
Were performance evaluation (PE) samples performed?		1		
Were the performance evaluation (PE) samples within the acceptance limits?	171.55 FA-5			
Superior Commence of the Comme				
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?				
Al 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?				
XI) is composited quantitation/GRO (2)				
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: Tertalikely identified composition (TAS)				A A Company of the Co
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)?		/	-	
AMAZ Cian oz foruguses				
System performance was found to be acceptable.				·
Overall assessment of data was found to be acceptable.				
Field duplicate pairs were identified in this SDG.		and and an area	talkille.	
	-			
Target compounds were detected in the field duplicates.	] ]\$4%			
KVTE FOR ESTICES	the same of the			
Field blanks were identified in this SDG.	N			
Target compounds were detected in the field blanks.	<u>/</u> /	L		

# VALIDATION FINDINGS WORKSHEET

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

III. Benzo(a)pyrene**	JJJ. Indeno(1,2,3-cd)pyrene	KKK. Dibenz(a,h)anthracene	LLL. Benzo(g,h,i)perylene	MMM. Bis(2-Chloroisopropyl)ether	NNN, Aniline	000. N-Nitrosodimethylamine	PPP. Benzoic Acid	QQQ. Benzyl alcohol	RRR. Pyridine	SSS. Benzidine		ſ	,	W.
TT. Pentachlorophenol**	UU. Phenanthrene	VV. Anthracene	WW. Carbazole	XX. Di-n-butyiphthalate	YY. Fluoranthene**	ZZ. Pyrene 000	AAA. Butylbenzylphthalate	BBB. 3,3'-Dichlorobenzidine QQC	CCC. Benzo(a)anthracene	DDD. Chrysene	EEE. Bis(2-ethylhexyl)phthalate TTT.	FFF. Di-n-octylphthalate**	GGG. Benzo(b)fluoranthene	HHH. Benzo(k)fluoranthene WWW.
EE. 2,6-Dinitrotoluene	FF. 3-Nitroaniline	GG. Acenaphthene⇔	HH. 2,4-Dinitrophenol*	II. 4-Nitrophenol*	JJ. Dibenzofuran	KK. 2,4-Dinitrotoluene	LL. Diethylphthalate	MM. 4-Chlorophenyl-phenyl ether	NN. Fluorene	00. 4-Nitroaniline	PP. 4,6-Dinitro-2-methylphenol	QQ. N-Nitrosodiphenylamine (1)**	RR. 4-Bromophenyl-phenylether	SS. Hexachlorobenzene
P. Bis(2-chloroethoxy)methane	Q. 2,4-Dichlorophenol**	R. 1,2,4-Trichlorobenzene	S. Naphthalene	T. 4-Chloroaniline	U. Hexachlorobutadiene™	V. 4-Chloro-3-methylphenol™	W. 2-Methylnaphthalene	X. Hexachlorocyclopentadiene*	Y. 2,4,6-Trichlorophenoi**	Z. 2,4,5-Trichlorophenol	AA. 2-Chloronaphthalene	BB. 2-Nitroaniline	CC. Dimethylphthalate	DD. Acenaphthylene
A. Phenoi™	B. Bis (2-chloroethyl) ether	C. 2-Chlorophenol	D. 1,3-Dichlorobenzene	E. 1,4-Dichlorobenzene**	F. 1,2-Dichlorobenzene	G. 2-Methylphenol	H. 2,2'-Oxybis(1-chloropropane)	I. 4-Methylphenol	J. N-Nitroso-di-n-propylamine*	K. Hexachloroethane	L. Nitrobenzene	M. Isophorone	N. 2-Nitrophenol**	O. 2,4-Dimethylphenol

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

## LDC#: 24847 KZa

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

JVG Page: Reviewer: 2nd Reviewer:

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:

CON AW ON A

A<sub>x</sub> = Area of Compound

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

 $C_x$  = Concentration of compound,

X = Mean of the RRFs

RRF = $(A_x)(C_{is})(A_{is})(C_x)$ average RRF = sum of the RRFs/number of standards	%KSD = 100 (S/A)
---	------------------

Standard ID

#

MSS Y ICAL

S= Standard deviation of the RRFs,

Recalculated %RSD 4.60 2.14 6.44 2.65 9.82 5.51 Reported %RSD 4.6 5.5 6.4 2.7 9.8 2.1 Average RRF Recalculated 0.6078 1.1045 1.2854 0.2150 1.1025 1.0022 (Initial) Average RRF Reported 0.6078 1.1045 0.2150 1.1025 1.2854 1.0022 Recalculated 50 std) 0.2096 0.9979 1.0905 1.1090 1.2364 RRF 0.6211 Reported 1.0905 0.2096 1.1090 0.9979 50 std) 1.2364 0.6211 RRF (181) (182) (183) (IS4) (185) Compound (Internal Standard) (186) Benzo(g,h,i)perylene Hexachlorobenzene Naphthalene 1,4-Dioxane Chrysene Fluorene Calibration 9/7/2010 Date

Area IS	272113	1065726	671801	1051118	1041159	855935
Area cpd	211270	1452752	1038301	275363	1443358	1067705
onc 1S/Cpd	40/50	40/20	40/20	40/50	40/20	40/50

•						
Conc	1,4-Dioxane	Naphthalene	Fluorene	Hexachlorob	Chrysene	Benzo(g,h,i)per
4.00		1.0765	1.2003		1.0903	0.8090
10.00	0.6455	1.0884	1.2284	0.1920	1.0698	0.9296
20.00	0.5733	1.0785	1.2225	0.2033	1.0870	0.9649
50.00	0.6211	1.0905	1.2364	0.2096	1.1090	0.9979
80.00	0.6367	1.1224	1.3100	0.2235	1.1499	1.0699
120.00	0.5993	1.1231	1.3577	0.2215	1.0926	1.0746
160.00	0.6021	1.1381	1.3567	0.2235	1.1424	1.0836
200.00	0.5769	1.1181	1.3713	0.2314	1.0792	1.0883
×	0.6078	1.1045	1.2854	0.2150	1.1025	1.0022
S	0.0279	0.0236	0.0709	0.0138	0.0292	0.0985
					***************************************	

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#: 24047 KZa

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: Reviewer:

2nd Reviewer:

# 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

 $A_x$  = Area of Compound

A<sub>is</sub> = Area of associated internal standard  $C_{\rm is}$  = Concentration of internal standard

%RSD = 100 \* (S/X)

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

X = Mean of the RRFs

9								
Recalculated %RSD		3.21	11.01	10.98	3.76	8.10	2.49	
Reported %RSD		3.2	11.0	11.0	3.8	8.1	2.5	
Recalculated Average RRF	(Initial)	0.6538	1.0482	1.3037	0.2454	1.0975	1.0927	
Reported Average RRF	(Initial)	0.6538	1.0482	1.3037	0.2454	1.0975	1.0926	
Recalculated RRF	( 50 std)	0.6494	1.0810	1.3696	0.2517	1.1455	1.1162	
Reported RRF	(50 std)	0.6494	1.0810	1.3696	0.2517	1.1455	1.1162	
	nal Standard)	(1S1)	(IS2)	(183)	(184)	(185)	(186)	
	Compound (Internal	8/30/2010 1,4-Dioxane	Naphthalene	Fluorene	Hexachlorobenzene	Chrysene	Benzo(g,h,i)perylene	
Calibration	Date	8/30/2010						
	Standard ID	ICAL	MSS B					
	#	1						

	Hexac	33	46	42	96	2.2	34	27	42	37	32
	Fluorene	1.4403	1.4246	1.4342	1.3696	1.3277	1.2284	1.1307	1.0742	1.3037	0.1432
	Naphthalene	1.1880	1.1590	1.1306	1.0810	1.0601	0.9839	0.9020	0.8811	1.0482	0.1154
	1,4-Dioxane	0.6780	0.6510	0.6855	0.6494	0.6655	0.6423	0.6276	0.6313	0.6538	0.0210
Ľ	Conc	4.00	10.00	20.00	50.00	80.00	120.00	160.00	200.00	X	# S

1059049

1516369 1447002

945857

297546 956646

> 40/20 40/20

40/50

1037135

40/20

961965 558782

40/20

246217 Area IS

> 199862 1299821

40/20

Area cpd

ic IS/Cpd

1.0704 1.0855 1.1162 1.1433 1.1029

1.1774 1.1786 1.1455 1.1198

0.2516 0.2514 0.2517 0.2522 0.2435

0.2501 hlorob

Benzo(g,h,i)per

Chrysene

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.

0.0273

1.0927

1.0975

0.2454

0.0889

0.0092

1.0831

1.0822

1.0006 0.9572

0.2339

0.2287

1.0281

LDC # 240 47 K29

## Continuing Calibration Results Verification VALIDATION FINDINGS WORSHEET

Page 1 of 1 Reviewer: 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:

% 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

		Calibration			Average RRF	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Date	Compound (Reference IS)	ice IS)	(Initial RRF)	(CC RRF)	(CC RRF)	МD	©%
٢	B0151	09/08/10	1,4-Dioxane	(IS1)	0.6538	0.6263	0.6263	4.2	4.2
	MSS B		Naphthalene	(182)	1.0482	1.0559	1.0559	0.7	0.7
			Fluorene	(183)	1.3037	1.3053	1.3053	0.1	0.1
			Hexachlorobenzene	(184)	0.2454	0.2335	0.2335	4.9	4.9
			Chrysene	(185)	1.0975	1.0855	1.0855	1.1	1.1
			Benzo(g,h,i)perylene	(186)	1.0926	1.1309	1.1309	3.5	3.5
2	Y4788	09/09/10	1,4-Dioxane	(1S1)	0.6078	0.6320	0.6320	4.0	4.0
	MSS Y		Naphthalene	(182)	1.1045	1.1416	1.1416	3.4	3.4
			Fluorene	(183)	1.2854	1.3272	1.3272	3.3	3.3
			Hexachlorobenzene	(184)	0.2150	0.2221	0.2221	3.3	3.3
			Chrysene	(185)	1.1025	1.1718	1.1718	6.3	6.3
			Benzo(g,h,i)perylene	(981)	1.0022	1.0318	1.0318	2.9	2.9

Compound (Reference IS)	(SI e	Concentration	Area Cpd	Area IS	Area Cpd	Area IS
:		(IS/Cpd)				
1,4-Dioxane	(181)	40/80	298831	238575	297038	234991
Naphthalenė	(182)	40/80	1961939	929068	2066321	904989
Fluorene	(183)	40/80	1398808	535829	1486157	559884
Hexachlorobenzene	(184)	40/80	428209	916980	398387	896746
Chrysene	(185)	40/80	2106014	970074	1953523	833558
Benzo(g,h,i)perylene	(186)	40/80	2199377	972419	1333281	646123

LDC#: 24047 K29

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

Page:	<u>lof 1</u>
Reviewer:_	34
nd reviewer:	In

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:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	10	72.5	72	72	0
2-Fluorobiphenyl		74.9	75	75	1
Terphenyl-d14	Į.	78.7	78	78	
Phenol-d5	150	113.8	76	7,6	
2-Fluorophenol		111. 2	74	74	
2,4,6-Tribromophenol	+	98.9	66	66	
2-Chiorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5				·	
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5			-		
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4				·	
1,2-Dichlorobenzene-d4					

LDC#: 29047 K29 SDG #: See Cover

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

2nd Reviewer: Page: lof 1 Reviewer:\_

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

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

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

Where:

SC = Sample concentation

SSC = Spiked sample concentration SA = Spike added

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples:

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

7

	Š	ike	Sample	Spiked 5	Sample	Matrix Spike	Spike	Matrix Spike Duplicate	e Duplicate	MS/MSD	ISD
Compound	Added (W)	ded  ()	Concentration (ねぇ /ヒァ)	Concentration (vg /c_)	tration	Percent Recovery	ecovery	Percent Recovery	\ecovery	RPD	D
	MS	OMSD	၁	MS	O <sub>MSD</sub>	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenol											
N-Nitroso-di-n-propylamine											
4-Chloro-3-methylphenol											
Acenaphthene	25 cm	2896	9	2300	27 20	οş	08	73	73	6	6
Pentachlorophenol		-									,
Pyrene	2900	2890	<b>→</b>	2490	3740	98	98	84	82	વ	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.

LUC#: 240 42 1/29

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

Reviewer:\_\_

Page: Lof 1 2nd Reviewer:

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

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 \* (SC/SA

SSC = Spike concentration SA = Spike added Where:

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

LCS/LCSD samples:

2975 -086 Z

	<u></u>	oike oike	<b>が</b>	oike .		CS	9	CSD	TSO T	CS/I CSD
Compound	Ad (MS)	Added (145/F)	Conce (Mg	Concentration (Mg /た)	Percent Recovery	Secovery	Percent	Percent Recovery	RPD	ď
	1 CS	O LCSD	SS	) I CSD	Reported	Book	Potroug O	) oleved	7	Latel colored
Phenol										
N-Nitroso-di-n-propylamine										
4-Chloro-3-methylphenol										
Acenaphthene	26 00	₹N	abl	47	74	7.4				
Pentachlorophenol									$\left  \right $	
Pyrene	26 0)	<b>\</b>	2636		78	26				
							\			

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#: ~4047 K29

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	<u>lof_1</u>
Reviewer:	M
d reviewer.	1 -

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

Υ	N	N/A)
Y	Ν	N/A/
		$ \mathcal{T}$

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?

Conce	entratio	$n = (A_{o})(I_{o})(V_{o})(DF)(2.0)$ $(A_{o})(RRF)(V_{o})(V_{o})(V_{o})$	Example:
A <sub>x</sub>	=	Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D,
$A_{is}$	=	Area of the characteristic ion (EICP) for the specific internal standard	
l <sub>s</sub>	=	Amount of internal standard added in nanograms (ng)	Conc. = ( )( )( )( )( )( )
V <sub>o</sub>	=	Volume or weight of sample extract in milliliters (ml) or grams (g).	
٧,	=	Volume of extract injected in microliters (ul)	=
V,	=	Volume of the concentrated extract in microliters (ul)	
Df	=	Dilution Factor.	
<b>%</b> S	=	Percent solids, applicable to soil and solid matrices only.	
20	_	Factor of 2 to account for CPC cleanup	

2.0	= Factor of 2 to accoun			· · · · · · · · · · · · · · · · · · ·	<del>,</del>
#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration	Qualification
·					

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

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 25, 2010

**LDC Report Date:** 

October 11, 2010

Matrix:

Soil/Water

Parameters:

Semivolatiles

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6818-1

### Sample Identification

BDT-2-N-20-10.0BPC BDT-2-N-5-2BPC BDT-2-N-20-12.0BPC BDT-2-N-5-4BPC BDT-2-N-20-14.0BPC BDT-2-N-5-6BPC BDT-2-N-20-2.0BPC BDT-2-N-10-10BPC BDT-2-N-20-4.0BPC BDT-2-N-10-12BPC BDT-2-N-20-6.0BPC BDT-2-N-10-14BPC\*\* BDT-2-N-20-8.0BPC BDT-2-N-10-2BPC BDT-2-N-20-12.0BPC FD BDT-2-N-10-4BPC BDT-2-N-15-1.0BPC BDT-2-N-10-6BPC BDT-2-N-15-12.0BPC BDT-2-N-10-8BPC BDT-2-N-15-14.0BPC\*\* BDT-2-N-10-6BPC FD BDT-2-N-5-2.0BPC EB-08252010 BDT-2-N-15-4.0BPC BDT-2-N-15-6.0BPCMS BDT-2-N-15-6.0BPC BDT-2-N-15-6.0BPCMSD BDT-2-N-15-8.0BPC BDT-2-N-10-6BPCMS BDT-2-N-10-6BPCMSD BDT-2-N-5-10BPC BDT-2-N-5-12BPC BDT-2-N-5-14BPC\*\*

BDT-2-N-5-12BPC\_FD BDT-2-N-5-8BPC

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

### Introduction

This data review covers 35 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, (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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

Samples indicated by a double asterisk on the front cover underwent a 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 (r²) 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.

Sample EB-08252010 was identified as an equipment blank. No semivolatile contaminants were found in this blank.

### VI. Surrogate Spikes

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

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. 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.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6818-1	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 BDT-2-N-20-12.0BPC and BDT-2-N-20-12.0BPC\_FD and samples BDT-2-N-5-12BPC and BDT-2-N-5-12BPC\_FD and samples BDT-2-N-10-6BPC and BDT-2-N-10-6BPC\_FD were identified as field duplicates. No semivolatiles were detected in any of the samples.

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-6818-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6818-1	BDT-2-N-20-10.0BPC BDT-2-N-20-12.0BPC BDT-2-N-20-14.0BPC BDT-2-N-20-14.0BPC BDT-2-N-20-4.0BPC BDT-2-N-20-6.0BPC BDT-2-N-20-6.0BPC BDT-2-N-20-8.0BPC BDT-2-N-15-1.0BPC BDT-2-N-15-1.0BPC BDT-2-N-15-14.0BPC** BDT-2-N-15-4.0BPC BDT-2-N-15-4.0BPC BDT-2-N-15-4.0BPC BDT-2-N-15-6.0BPC BDT-2-N-15-6.0BPC BDT-2-N-5-12BPC BDT-2-N-5-12BPC BDT-2-N-5-12BPC BDT-2-N-5-14BPC** BDT-2-N-5-4BPC BDT-2-N-5-6BPC BDT-2-N-5-6BPC BDT-2-N-10-10BPC BDT-2-N-10-10BPC BDT-2-N-10-12BPC BDT-2-N-10-14BPC** BDT-2-N-10-4BPC BDT-2-N-10-6BPC	All compounds reported below the PQL.	J (all detects)		Project Quantitation Limit (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-6818-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Equipment Blank Data Qualification Summary - SDG 280-6818-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Field Blank Data Qualification Summary - SDG 280-6818-1

No Sample Data Qualified in this SDG

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

LDC #: 24047L2a SDG #: Stage 2B/4 280-6818-1 Laboratory: Test America

Date: 10 /66 /10 Page: 1 of Reviewer: 516 2nd Reviewer:

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: & /25 /10
11.	GC/MS Instrument performance check	A	
III.	Initial calibration	A	72 RSD rd
IV.	Continuing calibration/ICV	A	cw/w = 25 Z
V.	Blanks	Á	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	Á	
VIII.	Laboratory control samples	A	ies /p
IX.	Regional Quality Assurance and Quality Control	N	
Χ.	Internal standards	A	
XI.	Target compound identification	A	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	ND	$D_1 = 2.8$ $D_2 = 19, 19$ $D_3 = 29.31$ $D_3 = 10$
XVII.	Field blanks	SAND	$D_1 = 2.8$ $D_2 = 19, 19$ $D_3 = 29.31$ $D_2 = 15$ $FB = 72$ $FB = FB - 0413.2010 - RFG2 - RZE$

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 1	BDT-2-N-20-10.0BPC S	- γ 11	BDT-2-N-15-14.0BPC**	ት ጋ 21	BDT-2-N-5-2BPC	ر ع 31	BDT-2-N-10-6BPC_FD りょく
2 1	BDT-2-N-20-12.0BPC D,	* 12 \mathcal{\mathcal{L}}	BDT-2-N-5-2.0BPC	ر 22	BDT-2-N-5-4BPC	- 4 32	EB-08252010 W
3 1	BDT-2-N-20-14.0BPC	13	BDT-2-N-15-4.0BPC	23	BDT-2-N-5-6BPC	33	BDT-2-N-15-6.0BPCMS
† 4 }	BDT-2-N-20-2.0BPC	14	BDT-2-N-15-6.0BPC	- <b>3</b>	BDT-2-N-10-10BPC	34	BDT-2-N-15-6.0BPCMSD
5	BDT-2-N-20-4.0BPC	- > 15	BDT-2-N-15-8.0BPC	ر 25	BDT-2-N-10-12BPC	35 <sup>3</sup>	BDT-2-N-10-6BPCMS
6	BDT-2-N-20-6.0BPC	- <b>*</b> 16	BDT-2-N-5-10BPC	26	BDT-2-N-10-14BPC**	36	BDT-2-N-10-6BPCMSD
7 1	BDT-2-N-20-8.0BPC	- ~ 17	BDT-2-N-5-12BPC <i>D</i> -/	27	BDT-2-N-10-2BPC	_ 37 \	MB 280-29889/1-A
έγ	BDT-2-N-20-12.0BPC_FD	<b>&gt;</b> 18	BDT-2-N-5-14BPC**	28 3	BDT-2-N-10-4BPC	38 Y	280-29898/1-A
9 2	BDT-2-N-15-1.0BPC	- <b>?</b> 19	BDT-2-N-5-12BPC_FD B	- 29 <sup>첫</sup>	BDT-2-N-10-6BPC	39 <b>9</b>	280-29938 /1-X
10 -	BDT-2-N-15-12.0BPC	- 20 20	BDT-2-N-5-8BPC	30 3	BDT-2-N-10-8BPC	40 <b>4</b>	280 29647 /14

Page: \\_\ of \\_2 Reviewer: \\_\mathcal{JVL} 2nd Reviewer: \\_\subseteq

Method: Semivolatiles (EPA SW 846 Method 8270C)

Method: Serrivolatiles (EFA SVV 646 Method 6270C)	T	Ι	T	
Validation Area	Yes	No	NA	Findings/Comments
1 Technical holding times			(4.8.4)	
All technical holding times were met.				
Cooler temperature criteria was met.				
II. GCMS 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?		Mark a straight	√ wassini	
III delikal calibration.				Constitution of the Consti
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 <pre>     0.990? </pre>	_			
Were all percent relative standard deviations (%RSD) $\leq$ 30% and relative response factors (RRF) $\geq$ 0.05?				·
IV Continuing cafforation				e servicing and the service of
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?				
V/Bankson from the control of the co				
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.				
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?				
VILLAGO CONTROLIZAÇÃO CONTRACTOR DE CONTRACTOR DE CONTRACTOR DE CONTRACTOR DE CONTRACTOR DE CONTRACTOR DE CONT				
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?				
7012 Abtratory control samples				
Was an LCS analyzed for this SDG?				

LDC#: 740 47 12a

### **VALIDATION FINDINGS CHECKLIST**

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Reviewer: 10
2nd Reviewer: 10

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
X. Regional Quality Assurance and Quality Lengton.				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?	20 - 12 5 5 2 3	an was species	7075 (2.45	
Zimicrostauricanis (4) - 1 - 2 - 2 - 2 - 2 - 2 - 2 - 2 - 2 - 2				<b>LOCATION CONTRACTOR CONTRACTOR</b>
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 Tarrel compound dentification				pagging a self-transcription and
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 Composite quantitation CRCLs 1998 200 200 200 200 200 200 200 200 200 20				<b>Mark to</b> the second second second
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	/			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
All: Tentalizely identified compounds (TICS) % 2000 1000 1000 1000 1000 1000 1000 10				
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)?				
AV Selement kinger:				
System performance was found to be acceptable.			2000 mg 2 14	
Overall assessment of data was found to be acceptable.		74.500		
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.				
Medical merchanisms				
Field blanks were identified in this SDG.	/	Liet La Lace de	Wat consti	
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. Naphthaiene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitropheno!*	XX. Di-n-butyiphthalate	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	000. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP, Benzoic Acid
i. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenoi**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroanlline	DDD. Chrysene	SSS, Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEÉ. Bis(2-ethylhexyl)phthalate	TTT,
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate™	ດດດ
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	WW.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	www.

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

24047 629 LDC#

## VALIDATION FINDINGS WORKSHEET

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Reviewer: 2nd Reviewer:

Surrogate Recovery

Please see qualification below for all questions answered "N". Not applicable questions are identified as "N/A" METHOD: GC/MS BNA (EPA SW 846 Method 8270C) ANCAX S

Were percent recoveries (%R) for surrogates within QC limits?

¥ N N

N(N/A

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?

								-					$\Box$							_				
Qualifications	to much (only 1 ord)																							
s)	( 5/- 120)	( )	( )	( )	( )	( )	) (	( )	( )	( )	( )	) (	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
%R (Limits)	48																							
Surrogate	7BP														-									and the state of t
Sample ID	92																							
Date																								
*																								

QC Limits (Water) 21-100

10-123 33-110\* 16-110\*

QC Limits (Soil) 25-121 19-122 20-130\*

S5 (2FP)= 2-Fluorophenol S6 (TBP) = 2,4,6-Tribromophenol S7 (2CP) = 2-Chlorophenol-44 S8 (DCB) = 1,2-Dichlorobenzene-44

QC Limits (Water) 35-114 43-116 33-141 10-94

QC Limits (Soil) 23-120

\* QC limits are advisory

30-115 18-137 24-113

S1 (NBZ) = Nitrobenzene-d5 S2 (FBP) = 2-Fluorobipheny S3 (TPH) = Terpheny-d14 S4 (PHL) = Phenol-d5

LDC#: 24097139 SDG #:

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page:

Reviewer: 2nd Reviewer:

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

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

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

%RSD = 100 \* (S/X)

S= Standard deviation of the RRFs,

X = Mean of the RRFs

				Renorted	Porsinisted	Panortad	Poteli plened	Donorted	Potell leted
				parioday	recalculated	non Indox I	Incompared	ויבאחוופת	recalculated
		Calibration		RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
#	Standard ID Date	Date	Compound (Internal Standard)	( 50 std)	( 50 std)	(Initial)	(Initial)		
_	ICAL	8/27/2010	8/27/2010 1,4-Dioxane (IS1)	0.5926	0.5926	0.5795	0.5795	3.7	3.74
	MSSK		Naphthalene (IS2)	1.0571	1.0571	1.0015	1.0015	8.9	8.92
			Fluorene (IS3)	1.3180	1.3180	1.2421	1.2421	6.7	7.87
			Hexachlorobenzene (IS4)	0.2424	0.2424	0.2313	0.2313	6.1	6.04
		-	Chrysene (IS5)	1.1257	1.1257	1.0679	1.0679	9.3	9.33
			Benzo(g,h,i)perylene (IS6)	1.1231	1.1231	1.0199	1.0199	7.5	7.53
			-						

Conc	1,4-Dioxane	Naphthalene	Fluorene	Hexachlorob	Chrysene	Benzo(g,h,i)per
4.00	0.5778	1.1018	1.3240		1.1929	0.9595
10.00	0.6003	1.0722	1.3327	0.2454	1.1472	1.0450
20.00	0.6103	1.0714	1.3075	0.2448	1.1400	1.0900
20.00	0.5926	1.0571	1.3180	0.2424	1.1257	1.1231
80.00	0.5842	1.0008	1.2564	0.2335	1.0651	1.0769
120.00	0.5678	0.9489	1.1901	0.2252	0.9953	1.0108
160.00	0.5547	0.8964	1.1248	0.2168	0.9529	0.9476
200.00	0.5485	0.8636	1.0833	0.2109	0.9244	0.9066
×	0.5795	1.0015	1.2421	0.2313	1.0679	1.0199
S	0.0217	0.0893	0.0977	0.0140	0.0997	0.0768
•			337			

Area IS	172314	669515	393544	662745	759660	781265	
Area cpd	127636	884641	648342	200827	1068947	1096793	
nc IS/Cpd	40/50	40/20	40/20	40/20	40/20	40/20	

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 Results Verification **VALIDATION FINDINGS WORSHEET**

Page \_\_ of\_ Reviewer:\_ 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:

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

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

Ax = Area of compound

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

Ais = Area of associated internal standard

Cis = Concentration of internal standard Cx = Concentration of compound

		Calibration		Average RRF	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Date	Compound (Reference IS)	(Initial RRF)	(CC RRF)	(CC RRF)	%D	Q%
-	K6193	09/09/10	1,4-Dioxane (IS1)	) 0.5795	0.5841	0.5841	8.0	0.8
,			Naphthalene (IS2)	1.0015	1.0152	1.0152	1.4	1.4
			Fluorene (IS3)	1.2421	1.2642	1.2642	1.8	1.8
			Hexachlorobenzene (IS4)	0.2313	0.2327	0.2327	9.0	9.0
			Chrysene (IS5)	1.0679	1.0666	1.0666	0.1	0.1
			Benzo(g,h,i)perylene (IS6)	1.0199	1.0547	1.0547	3.4	3.4
2	K6237	09/10/10	1,4-Dioxane (IS1)	0.5795	0.5753	0.5753	0.7	0.7
			Naphthalene (IS2)	1.0015	1.0208	1.0208	1.9	1.9
			Fluorene (IS3)	1.2421	1.2704	1.2704	2.3	2.3
			Hexachlorobenzene (IS4)	) 0.2313	0.2290	0.2290	1.0	1.0
			Chrysene (IS5)	1.0679	1.1085	1.1085	3.8	3.8
			Benzo(g,h,i)perylene (IS6)	1.0199	1.0470	1.0470	2.7	2.7

Compound (Reference IS)	18)	Concentration	Area Cpd	Area IS	Area Cpd	Area IS
		(IS/Cpd)	:			
1,4-Dioxane	(IS1)	40/80	263848	225842	251284	218375
Naphthalene	(182)	40/80	1781060	877199	1721673	843308
Fluorene	(183)	40/80	1311374	518654	1264005	497473
Hexachlorobenzene	(IS4)	40/80	401614	863117	378514	826274
Chrysene	(185)	40/80	1915436	897927	1928995	870082
Benzo(g,h,i)perylene	(186)	40/80	1979145	938251	1826647	872308

LDC #: 24047 L29

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

Page:	<u>lof 1</u>
Reviewer:_	JV4
2nd reviewer:	1~

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Fo

Sample ID: ± 1

SE	=	Surrogate	e r	-ound
SS	=	Surrogate	e S	Spiked

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	10	67.5	67	(7	0
2-Fluorobiphenyl		72.6	73	73	
Terphenyl-d14	+	85.8	8 (	86	
Phenol-d5	150	106.4	7/	71	
2-Fluorophenol		98.6	65	66	
2,4,6-Tribromophenol	1	95.4	64	64	1
2-Chlorophenol-d4				/	7
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5	·				
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol				·	
2,4,6-Tribromophenol					
2-Chlorophenol-d4				·	
1,2-Dichlorobenzene-d4					

LDC#: >40 47 129 SDG #: See Care

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

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

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

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

using the following calculation:

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

Where: SSC = Spiked sample concentration SA = Spike added

SC = Sample concentation

MSDC = Matrix spike duplicate concentration

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

MSC = Matrix spike concentration

MS/MSD samples: \_

ンチ

Compound	Spi		Sample	Spiked	Sample	Matrix Spike	Spike	Matrix Spike Duplicate	Duplicate	MS/MSD	SD
	Added (MS /E)	( eq	Concentration (レベ /トC )	Concentration	itration	Percent Recovery	есочегу	Percent Recovery	ecovery	RPD	
	y X	MSD	0	MS	) MSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenol											
N-Nitroso-di-n-propylamine											
4-Chloro-3-methylphenol											
Acenaphthene	2740	2 780	ò	2140	2112	28	28	22	36	1	
Pentachlorophenol											
Pyrene	27%	2780		22	2492	83	88	06	90	۶	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.

LUC#: 2 4067 L29

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

Page: 1 of 1 Reviewer:\_

2nd Reviewer:\_

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

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 \* (SC/SA

SSC = Spike concentration SA = Spike added Where:

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

LCS/LCSD samples: \_

29898 2-1 -08c 571

	is	oike	Sp	ike		cs		GSD	1.05/	CS/LCSD
Compound	<b>P</b> 3	Added ( 5/ 5/ )	Concei	Concentration ( MS /(c)	Percent Recovery	Recovery	Percent Recovery	Recovery	. P.	RPD
	108	l csn	LCS	) I CSD	Reported	Recalc	Reported	Recalc	Renorted	Recalculated
Phenol										
N-Nitroso-di-n-propytamine										
4-Chloro-3-methylphenol										
Acenaphthene	2670	なみ	2670	Ńλ	38	28				
Pentachlorophenol										
Pyrene	202	_	2330		89	23				
				A	_					
			-							
		-								

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#: 74047 L 29

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	lof1_
Reviewer:_	W.
d reviewer.	Ĭ'n

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

Y	N	N/A	1
Y	Ν	N/A	
		1	

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?

Concent	tration	= $(A_{s})(I_{s})(V_{s})(DF)(2.0)$ $(A_{s})(RRF)(V_{s})(V_{s})(%S)$	Example:
A <sub>x</sub>	=	Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D,,;
A <sub>is</sub>	=	Area of the characteristic ion (EICP) for the specific internal standard	
1,	=	Amount of internal standard added in nanograms (ng)	Conc. = ( )( )( )( )( )( )
V <sub>o</sub>	=	Volume or weight of sample extract in milliliters (ml) or grams (g).	
$V_i$	=	Volume of extract injected in microliters (ul)	= .
$V_{\iota}$	=	Volume of the concentrated extract in microliters (ul)	
Df	=	Dilution Factor.	
<b>%</b> S	=	Percent solids, applicable to soil and solid matrices only.	

2.0	= Factor of 2 to accoun	t for GPC cleanup				
#	Sample ID	Compound	·	Reported Concentration ( )	Calculated Concentration ( )	Qualification
					· · · · · · · · · · · · · · · · · · ·	
				-		
1						

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

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 26, 2010

LDC Report Date:

October 8, 2010

Matrix:

Soil/Water

Parameters:

Semivolatiles

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6851-1

### Sample Identification

BDT-2-S-20-10BPC BDT-2-S-5-8BPC BDT-2-S-20-12BPC BDT-2-S-5-2BPC BDT-2-S-20-14BPC\*\* BDT-2-S-5-4BPC BDT-2-S-20-2BPC BDT-2-S-5-6BPC BDT-2-S-20-4BPC BDT-2-S-10-10BPC BDT-2-S-20-6BPC BDT-2-S-10-12BPC BDT-2-S-20-6BPC FD BDT-2-S-10-14BPC\*\* BDT-2-S-20-8BPC BDT-2-S-10-2BPC BDT-2-S-15-10BPC BDT-2-S-10-4BPC BDT-2-S-15-12BPC BDT-2-S-10-6BPC BDT-2-S-15-14BPC\*\* BDT-2-S-10-8BPC BDT-2-S-15-2BPC SSAJ3-02-5BPC BDT-2-S-15-4BPC EB-08262010 BDT-2-S-15-6BPC BDT-2-S-5-10BPCMS BDT-2-S-15-8BPC BDT-2-S-5-10BPCMSD BDT-2-S-15-2BPC FD BDT-2-S-5-10BPC

BDT-2-S-5-12BPC BDT-2-S-5-14BPC\*\* BDT-2-S-5-12BPC FD

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

### Introduction

This data review covers 34 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

Samples indicated by a double asterisk on the front cover underwent a 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.

Sample EB-08262010 was identified as an equipment blank. 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 with the following exceptions:

Sample	Compound	Finding	Flag	A or P
BDT-2-S-5-2BPC BDT-2-S-10-2BPC	Benzo(b)fluoranthene Benzo(k)fluoranthene	quantitation using the total peak area.	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6851-1	All compounds reported below the PQL.	J (all detects)	A

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 BDT-2-S-20-6BPC and BDT-2-S-20-6BPC\_FD, samples BDT-2-S-15-2BPC and BDT-2-S-15-2BPC\_FD, and samples BDT-2-S-5-12BPC and BDT-2-S-5-12BPC\_FD were identified as field duplicates. No semivolatiles were detected in any of the samples with the following exceptions:

	Concentrat	tion (ug/Kg)					
Compound	BDT-2-S-20-6BPC	BDT-2-S-20-6BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P	
Hexachlorobenzene	37	79	-	42 (≤350)	-	•	

	Concentra	tion (ug/Kg)	555	Difference		
Compound	BDT-2-S-15-2BPC	BDT-2-S-15-2BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Benzo(a)anthracene	20	340U	-	320 (≤340)	-	-
Chrysene	50	29	-	21 (≤340)	-	-
Hexachlorobenzene	950	510	-	440 (≤340)	J (all detects)	Α
Octachlorostyrene	160	110	-	50 (≤340)	-	-
Pyrene	19	340U	-	321 (≤340)	-	-

	Concentra	tion (ug/Kg)				
Compound	BDT-2-S-5-12BPC	BDT-2-S-5-12BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Hexachlorobenzene	170	200	-	30 (≤370)	-	-

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-6851-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6851-1	BDT-2-S-5-2BPC BDT-2-S-10-2BPC	Benzo(b)fluoranthene Benzo(k)fluoranthene	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Compound quantitation and CRQLs (q)
280-6851-1	BDT-2-S-20-10BPC BDT-2-S-20-12BPC BDT-2-S-20-14BPC** BDT-2-S-20-4BPC BDT-2-S-20-4BPC BDT-2-S-20-6BPC BDT-2-S-20-6BPC BDT-2-S-20-6BPC BDT-2-S-15-10BPC BDT-2-S-15-12BPC BDT-2-S-15-14BPC** BDT-2-S-15-4BPC BDT-2-S-15-4BPC BDT-2-S-15-4BPC BDT-2-S-15-4BPC BDT-2-S-15-4BPC BDT-2-S-15-4BPC BDT-2-S-15-4BPC BDT-2-S-15-4BPC BDT-2-S-5-14BPC** BDT-2-S-5-14BPC** BDT-2-S-5-14BPC** BDT-2-S-5-14BPC BDT-2-S-5-14BPC BDT-2-S-5-14BPC BDT-2-S-5-4BPC BDT-2-S-5-4BPC BDT-2-S-5-4BPC BDT-2-S-10-10BPC BDT-2-S-10-14BPC** BDT-2-S-10-14BPC** BDT-2-S-10-4BPC BDT-2-S-10-4BPC BDT-2-S-10-6BPC BDT-2-S-10-6BPC BDT-2-S-10-6BPC BDT-2-S-10-8BPC SSAJ3-02-5BPC EB-08262010	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
280-6851-1	BDT-2-S-15-2BPC BDT-2-S-15-2BPC_FD	Hexachlorobenzene	J (all detects)	Α	Field duplicates (Difference) (fd)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-6851-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Equipment Blank Data Qualification Summary - SDG 280-6851-1

No Sample Data Qualified in this SDG

# **Tronox Northgate Henderson** EET

LDC #:	24047M2a	VALIDATION COMPLETENESS WORKSHE
SDG #:_	280-6851-1	Stage 2B/4
Laborato	ry: Test America	

Date:\_10/05/10 Reviewer: 2nd Reviewer:

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 8/26/fp
11.	GC/MS Instrument performance check	Α	,
111.	Initial calibration	A	? RSD r~
IV.	Continuing calibration/ICV	A	Ca/10 = 25]
V.	Blanks	A	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	A	·
VIII.	Laboratory control samples	A	us/b
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	A	Not reviewed for Stage 2B validation.
XV.	Overall assessment of data	A	
XVI.	Field duplicates	SW	$D_1 = 6.7$ $D_2 = 12, 16$ $D_3 = 18, 20$
XVII.	Field blanks	Alb	FB = 33 FB = F8 -04132010 RIG2 RZE

Note:

A = Acceptable

N = Not provided/applicable SW = See worksheet

**KND** = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

TB = Trip blank

EB = Equipment blank

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

	Soil +	Wate	<u>r</u>				
- 1 1	BDT-2-S-20-10BPC	11 1	BDT-2-S-15-14BPC**	47 21	BDT-2-S-5-8BPC	31	BDT-2-S-10-8BPC
2 1	BDT-2-S-20-12BPC	12	BDT-2-S-15-2BPC D>	22	[(/⊁ ∕/⊌⊁) BDT-2-S-5-2BPC	32	SSAJ3-02-5BPC
3 1	BDT-2-S-20-14BPC**	13	BDT-2-S-15-4BPC	23	BDT-2-S-5-4BPC	33	EB-08262010
4 1	BDT-2-S-20-2BPC	14	BDT-2-S-15-6BPC	24	BDT-2-S-5-6BPC	34 >	BDT-2-S-5-10BPCMS S
+ 5 1	BDT-2-S-20-4BPC	15	BDT-2-S-15-8BPC	25	BDT-2-S-10-10BPC	35	BDT-2-S-5-10BPCMSD
† 6	BDT-2-S-20-6BPC <b>b</b> !	16	BDT-2-S-15-2BPC_FD D	26	BDT-2-S-10-12BPC	- 36 /	MB 280_ 29938/1-A
7 1	BDT-2-S-20-6BPC_FD b	17 <b>7</b>	BDT-2-S-5-10BPC	27	BDT-2-S-10-14BPC**	- 37 <b>2</b>	MB 280- 29978/1-1
<del>}</del> 1	BDT-2-S-20-8BPC	18	BDT-2-S-5-12BPC 1/3	28	BDT-2-S-10-2BPC	38 3	MB 280 - 29697 1-A
ģ 1	BDT-2-S-15-10BPC	197	BDT-2-S-5-14BPC**	29 29	BDT-2-S-10-4BPC	39	7
† 10	BDT-2-S-15-12BPC	20	BDT-2-S-5-12BPC_FD 3	30	BDT-2-S-10-6BPC	40	

# **VALIDATION FINDINGS CHECKLIST**

Method: Semivolatiles (EPA SW 846 Method 8270C)

	_		_	
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	/			
Cooler temperature criteria was met.				
II. GC/MS Instrument performance check		I		
Were the DFTPP performance results reviewed and found to be within the specified criteria?				
Were all samples analyzed within the 12 hour clock criteria?				
III. Initial calibration	1	J		
Did the laboratory perform a 5 point calibration prior to sample analysis?	1			
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?		ł		
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 for each matrix and concentration?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
VI. Surrogate spikes	7			A second
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?			4	
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				
Vas an LCS analyzed for this SDG?				

# VALIDATION FINDINGS CHECKLIST

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	/			
IX. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?  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?		1		
XI. Target compound identification	1			samilya Samilya
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?		,		
XIII. Tentatively identified compounds (TICs)				
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.	_/			
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. Field blanks	/ 			
Field blanks were identified in this SDG.				·
Target compounds were detected in the field blanks.				

# VALIDATION FINDINGS WORKSHEET

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

		7,000		
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-methyiphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	000. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachiorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	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**	nnn
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	WW.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	WWW.

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

LDC# 24047 M22

# VALIDATION FINDINGS WORKSHEET

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

Surrogate Recovery

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

Please see qualification below for all questions answered "N". Not applicable questions are identified as "N/A" Were percent recoveries (%R) for surrogates 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?

N(N/A)

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Qualifications	No mas (only 1 out)																					
%R (Limits)	45 (51-120)		( )	( )	( )		( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	
Surrogate	7BP	1																				
Sample ID	0°C																					
Date																						
*																						

QC Limits (Water) 21-100 10-123 33-110\* 16-110\*

QC Limits (Soil) 25-121 19-122 20-130\*

> S5 (2FP)= 2-Fluorophenol S6 (TBP) = 2,4,6-Tribromophenol S7 (2CP) = 2-Chlorophenol-d4 S8 (DCB) = 1,2-Dichlorobenzene-d4

QC Limits (Water) 35-114 43-116 33-141 10-94

> 23-120 30-115 18-137 24-113

S1 (NBZ) = Nitrobenzene-d5 S2 (FBP) = 2-Fluorobiphenyl S3 (TPH) = Terphenyl-d14 S4 (PHL) = Phenol-d5

QC Limits (Soil)

\* QC limits are advisory

LDC#: 24047 MZA SDG #: Le Corey

# Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

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

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

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

Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?

Qualifications	3/43/p (9)										
Associated Samples	resolved	k area									
Finding	666 HHH peaks un resolved	lab wed total pea	for mantitation	9							
Sample ID	8× 77										
Date											
#											

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24047M2a

# **VALIDATION FINDINGS WORKSHEET** Field Duplicates

Page	: <u> </u>
Reviewer:	W
2nd Reviewer:	5

METHOD: GC/MS SVOA (EPA SW 846 Method 8270C)
Y N NA
Were field duplicate pairs identified in this SDG?
Were target analytes detected in the field duplicate pairs?

Compound Name	Conc (	ug/Kg)	RPD	Diff	Diff Limits	Quals
Compound Name	6	7	(≤50%)	<b>D</b>		(Parent Only)
Hexachlorobenzene	37	79		42	≤350	

Commound Name	Conc (	ug/Kg)	RPD	Diff	Diff Limits	Quals
Compound Name	12	16	(≤50%)	Dill	Din Linits	(Parent Only)
Benzo(a)anthracene	20	340U		320	≤340	
Chrysene	50	29		21	≤340	
Hexachlorobenzene	950	510		440	≤340	Jdet/A (fd)
Octachlorostyrene	160	110		50	≤340	
Pyrene	19	340U		321	≤340	

Compound Name	Conc (	ug/Kg)	RPD	Diff	Diff Limits	Quals
Compound Nume	18	20	(≤50%)	Dill	Om Linius	(Parent Only)
Hexachlorobenzene	170	200		30	≤370	

V:\FIELD DUPLICATES\24047M2a.wpd

LDC #: 2 4047 M29 SDG #: C. (47.8)

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: Reviewer:

2nd Reviewer:

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

 $C_x = Concentration of compound,$ 

A<sub>x</sub> = Area of Compound

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

%RSD = 100 \* (S/X)

S= Standard deviation of the RRFs,

X = Mean of the RRFs

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
		Calibration		RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
#	Standard ID Date	Date	Compound (Internal Standard)	( 50 std)	( 50 std)	(Initial)	(Initial)		
F	ICAL	9/7/2010	9/7/2010 (1,4-Dioxane (1S1)	0.6211	0.6211	0.6078	0.6078	4.6	4.60
	MSS Y		Naphthalene (IS2)	1.0905	1.0905	1.1045	1.1045	2.1	2.14
			Fluorene (IS3)	1.2364	1.2364	1.2854	1.2854	5.5	5.51
			Hexachlorobenzene (IS4)	0.2096	0.2096	0.2150	0.2150	6.4	6.44
			Chrysene (IS5)	1.1090	1.1090	1.1025	1.1025	2.7	2.65
			Benzo(g,h,i)perylene (IS6)	0.9979	0.9979	1.0022	1.0022	9.8	9.82

	Area IS	272113	1065726	671801	1051118	1041159	855935	
:	Area cpd	211270	1452752	1038301	275363	1443358	1067705	
	nc IS/Cpd	40/20	40/20	40/20	40/20	40/20	40/20	

•						
Conc	1,4-Dioxane	Naphthalene	Fluorene	Hexachlorob	Chrysene	Benzo(g,h,i)per
4.00		1.0765	1.2003		1.0903	0.8090
10.00	0.6455	1.0884	1.2284	0.1920	1.0698	0.9296
20.00	0.5733	1.0785	1.2225	0.2033	1.0870	0.9649
50.00	0.6211	1.0905	1.2364	0.2096	1.1090	0.9979
80.00	0.6367	1.1224	1.3100	0.2235	1.1499	1.0699
120.00	0.5993	1.1231	1.3577	0.2215	1.0926	1.0746
160.00	0.6021	1.1381	1.3567	0.2235	1.1424	1.0836
200.00	0.5769	1.1181	1.3713	0.2314	1.0792	1.0883
×	0.6078	1.1045	1.2854	0.2150	1.1025	1.0022
S	0.0279	0.0236	0.0709	0.0138	0.0292	0.0985

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# 24047 M29

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

4 ζot Page:

Reviewer:

2nd Reviewer:

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

 $C_x$  = Concentration 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,

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
		Calibration		RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
#	Standard ID Date	Date	Compound (Internal Standard)	( 50 std)	( 50 std)	(Initial)	(Initial)		
1	ICAL	8/27/2010	8/27/2010 1,4-Dioxane (IS1)	0.5926	0.5926	0.5795	0.5795	3.7	3.74
	MSS K		Naphthalene (IS2)	1.0571	1.0571	1.0015	1.0015	8.9	8.92
			Fluorene (IS3)	1.3180	1.3180	1.2421	1.2421	7.9	78.7
			Hexachlorobenzene (IS4)	0.2424	0.2424	0.2313	0.2313	6.1	6.04
			Chrysene (IS5)	1.1257	1.1257	1.0679	1.0679	9.3	9.33
			Benzo(g,h,i)perylene (IS6)	1.1231	1.1231	1.0199	1.0199	7.5	7.53

_						
Conc	1,4-Dioxane	Naphthalene	Fluorene	Hexachlorob	Chrysene	Benzo(g,h,i)per
4.00	0.5778	1.1018	1.3240		1.1929	0.959
10.00	0.6003	1.0722	1.3327	0.2454	1.1472	1.0450
20.00	0.6103	1.0714	1.3075	0.2448	1.1400	1.090(
50.00	0.5926	1.0571	1.3180	0.2424	1.1257	1.123
80.00	0.5842	1.0008	1.2564	0.2335	1.0651	1.076
120.00	0.5678	0.9489	1.1901	0.2252	0.9953	1.010
160.00	0.5547	0.8964	1.1248	0.2168	0.9529	0.9476
200.00	0.5485	0.8636	1.0833	0.2109	0.9244	906.0
×	0.5795	1.0015	1.2421	0.2313	1.0679	1.0199
S	0.0217	0.0893	0.0977	0.0140	0.0997	920'0
-						

172314 669515 393544 662745 759660 781265

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# Continuing Calibration Results Verification VALIDATION FINDINGS WORSHEET

ا م ا Page\_ Reviewer: 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:

% 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

		Calibration			Average RRF	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Date	Compound (Reference IS)	eference IS)	(Initial RRF)	(CC RRF)	(CC RRF)	₩D	%D
-	Y4788	09/09/10	1,4-Dioxane	(IS1)	0.6078	0.6320	0.6320	4.0	4.0
			Naphthalene	(182)	1.1045	1.1416	1.1416	3.4	3.4
			Fluorene	(183)	1.2854	1.3272	1.3272	3.3	3.3
			Hexachlorobenzene	zene (IS4)	0.2150	0.2221	0.2221	3.3	3.3
			Chrysene	(185)	1.1025	1.1718	1.1718	6.3	6.3
			Benzo(g,h,i)perylene	ylene (IS6)	1.0022	1.0318	1.0318	2.9	2.9
2	Y4836	09/10/10	1,4-Dioxane	(1S1)	0.6078	0.6535	0.6535	7.5	7.5
			Naphthalene	(182)	1.1045	1.1494	1.1494	4.1	4.1
			Fluorene	(183)	1.2854	1.3478	1.3478	4.9	4.9
			Hexachlorobenzene	zene (IS4)	0.2150	0.2416	0.2416	12.4	12.4
			Chrysene	(185)	1.1025	1.1377	1.1377	3.2	3.2
			Benzo(g,h,i)perylene	ylene (IS6)	1.0022	0.9672	0.9672	3.5	3.5

Compound (Reference IS)	IS)	Concentration	Area Cpd	Area IS	Area Cpd	Area IS
		(IS/Cpd)				
1,4-Dioxane	(IS1)	40/80	297038	234991	293253	224375
Naphthalene	(182)	40/80	2066321	904989	1977504	860218
Fluorene	(183)	40/80	1486157	559884	1512241	561014
Hexachlorobenzene	(IS4)	40/80	398387	896746	442594	915793
Chrysene	(185)	40/80	1953523	833558	1994638	876639
Benzo(g,h,i)perylene	(981)	40/80	1333281	646123	1187355	613808

# LDC #04047 1124

# Continuing Calibration Results Verification VALIDATION FINDINGS WORSHEET

۶ بو Page\_\_\_\_ Reviewer: 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:

% 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

		Calibration		Average RRF	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Date	Compound (Reference IS)	(Initial RRF)	(CC RRF)	(CC RRF)	%D	Q%
1	K6193	09/09/10	1,4-Dioxane (IS1)	0.5795	0.5841	0.5841	0.8	0.8
			Naphthalene (IS2)	1.0015	1.0152	1.0152	1.4	1.4
			Fluorene (IS3)	1.2421	1.2642	1.2642	1.8	1.8
			Hexachlorobenzene (IS4)	0.2313	0.2327	0.2327	9.0	9.0
			Chrysene (IS5)	1.0679	1.0666	1.0666	0.1	0.1
			Benzo(g,h,i)perylene (IS6)	1.0199	1.0547	1.0547	3.4	3.4
		-						

Compound (Reference IS)	IS)	Concentration	Area Cpd	Area IS
		(IS/Cpd)		
1,4-Dioxane	(IS1)	40/80	263848	225842
Naphthalene	(185)	40/80	1781060	877199
Fluorene	(183)	40/80	1311374	518654
Hexachlorobenzene	(184)	40/80	401614	863117
Chrysene	(185)	40/80	1915436	897927
Benzo(g,h,i)perylene	(981)	40/80	1979145	938251

LDC #: 24047 M29

# **VALIDATION FINDINGS WORKSHEET Surrogate Results Verification**

Page:	<u>lof_1</u>
Reviewer:_	J16_
nd reviewer.	10

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

Sample ID:\_

SS = Surrogate Spiked

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	100	66.3	66	66	0
2-Fluorobiphenyl		66.8	67	67	
Terphenyl-d14		97.6	-98	98	
Phenol-d5	150	106.7	7/	71	
2-Fluorophenol		161.8	68	68	
2,4,6-Tribromophenol	1	99.6	66	66	8
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5			·		
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4			£		
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

# LDC# 24047 M29

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

Page: Lof 1 Reviewer:

2nd 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

SSC = Spike concentration SA = Spike added Where:

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

2.999 LCSC = Laboraotry control sample concentration LCSDC = Laboratory control sample duplicate concentration

LCS/LCSD samples:

280 -প্র

:
Added Concentration ( Mg/kg)
KA 2000
2310

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#: 2 4047M29

# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	lof1_
Reviewer:_	NO
2nd reviewer:_	10_

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

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

Concentration =  $(A_{\cdot\cdot})(I_{\cdot\cdot})(V_{\cdot})(DF)(2.0)$   $(A_{\cdot\cdot})(RRF)(V_{\circ})(V_{\cdot})(\%S)$   $A_{\times}$  = Area of the characteristic ion (EICP) for the compound to be measured  $A_{\cdot\cdot}$  = Area of the characteristic ion (EICP) for the specific internal standard  $I_{\cdot\cdot}$  = Amount of internal standard added in nanograms (ng)  $V_{\cdot\cdot}$  = Volume or weight of sample extract in milliliters (ml) or grams (g).  $V_{\cdot\cdot}$  = Volume of extract injected in microliters (ul)  $V_{\cdot\cdot}$  = Volume of the concentrated extract in microliters (ul)  $D_{\cdot\cdot}$  = Dilution Factor.  $C_{\cdot\cdot}$  = Percent solids, applicable to soil and solid matrices only.

Factor of 2 to account for GPC cleanup

Example: Sample I.D.  $\frac{41}{11}$ , SS: Conc. =  $\frac{(16473)(40)(1m)(100)(100)}{(844707)(0.2150)(31.4g)(0.914)(100)}$ = 126.4 $\frac{130}{2}$   $\frac{130}{2}$   $\frac{130}{2}$   $\frac{130}{2}$ 

2.0	= Factor of 2 to accour	it for GPC clearlup				
#	Sample ID	Compound		Reported Concentration ( )	Calculated Concentration ( )	Qualification
						****
						M
			,			
				•		
	, <u>, , , , , , , , , , , , , , , , , , </u>					
		· · · · · · · · · · · · · · · · · · ·				

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 27, 2010

**LDC Report Date:** 

October 8, 2010

Matrix:

Soil/Water

Parameters:

Semivolatiles

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6886-1

# Sample Identification

BDT-1-N-20-10BPC	BDT-1-N-10-4BPC	BDT-1-N-10-2BPCMS
BDT-1-N-20-12BPC**	BDT-1-N-10-6BPC	BDT-1-N-10-2BPCMSD
BDT-1-N-20-14BPC	BDT-1-N-10-8BPC	BDT-1-N-15-4BPCMS
BDT-1-N-20-2BPC	BDT-1-N-10-8BPC FD	BDT-1-N-15-4BPCMSD
BDT-1-N-20-4BPC	BDT-1-N-15-10BPC	BDT-1-S-20-4BPCMS
BDT-1-N-20-6BPC	BDT-1-N-15-12BPC	BDT-1-S-20-4BPCMSD
BDT-1-N-20-8BPC	BDT-1-N-15-14BPC**	BDT-1-S-20-6BPCMS
BDT-1-N-20-10BPC FD	BDT-1-N-15-8BPC	BDT-1-S-20-6BPCMSD
BDT-1-N-5-10BPC	BDT-1-N-15-2BPC	
BDT-1-N-5-12BPC	BDT-1-N-15-4BPC	
BDT-1-N-5-14BPC	BDT-1-N-15-6BPC	
BDT-1-N-5-8BPC_FD	BDT-1-S-20-10BPC	
BDT-1-N-5-8BPC	BDT-1-S-20-12BPC	
BDT-1-N-5-2BPC	BDT-1-S-20-14BPC**	
BDT-1-N-5-4BPC	BDT-1-S-20-2BPC	
BDT-1-N-5-6BPC	BDT-1-S-20-4BPC	
BDT-1-N-10-10BPC	BDT-1-S-20-6BPC	
BDT-1-N-10-12BPC	BDT-1-S-20-8BPC	•
BDT-1-N-10-14BPC	BDT-1-S-20-14BPC_FD	
BDT-1-N-10-2BPC	EB-08272010	

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

### Introduction

This data review covers 47 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

Samples indicated by a double asterisk on the front cover underwent a 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.

Sample EB-08272010 was identified as an equipment blank. 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. Although the MS/MSD percent recoveries (%R) were not within QC limits for one compound, the LCS percent recovery (%R) was within QC limits and no data were qualified.

# VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Although the LCS percent recovery (%R) was not within QC limits for one compound, the MS/MSD percent recoveries (%R) 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 compound quantitation and CRQLs 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 280-6886-1	All compounds reported below the PQL.	J (all detects)	Α

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 BDT-1-N-20-10BPC and BDT-1-N-20-10BPC\_FD, samples BDT-1-N-5-8BPC\_FD and BDT-1-N-5-8BPC, samples BDT-1-N-10-8BPC and BDT-1-N-10-8BPC\_FD, and samples BDT-1-S-20-14BPC\*\* and BDT-1-S-20-14BPC\_FD were identified as field duplicates. No semivolatiles were detected in any of the samples with the following exceptions:

	Concentra	tion (ug/Kg)	222	D:#		
Compound	BDT-1-N-10-8BPC	BDT-1-N-10-8BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Hexachlorobenzene	130	180	-	50 (≤350)	-	-

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-6886-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6886-1	BDT-1-N-20-10BPC BDT-1-N-20-12BPC** BDT-1-N-20-14BPC BDT-1-N-20-4BPC BDT-1-N-20-6BPC BDT-1-N-20-6BPC BDT-1-N-20-6BPC BDT-1-N-20-10BPC_FD BDT-1-N-5-10BPC BDT-1-N-5-10BPC BDT-1-N-5-14BPC BDT-1-N-5-8BPC_FD BDT-1-N-5-8BPC BDT-1-N-5-6BPC BDT-1-N-5-6BPC BDT-1-N-10-10BPC BDT-1-N-10-12BPC BDT-1-N-10-4BPC BDT-1-N-10-8BPC BDT-1-N-10-8BPC BDT-1-N-10-8BPC BDT-1-N-10-8BPC BDT-1-N-10-8BPC BDT-1-N-10-8BPC BDT-1-N-15-14BPC BDT-1-N-15-14BPC BDT-1-N-15-14BPC BDT-1-N-15-14BPC BDT-1-N-15-14BPC BDT-1-N-15-4BPC BDT-1-S-20-14BPC BDT-1-S-20-14BPC** BDT-1-S-20-4BPC BDT-1-S-20-4BPC BDT-1-S-20-8BPC BDT-1-S-20-4BPC BDT-1-S-20-4BPC BDT-1-S-20-4BPC BDT-1-S-20-14BPC_FD EB-08272010	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-6886-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Equipment Blank Data Qualification Summary - SDG 280-6886-1

No Sample Data Qualified in this SDG

# **Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET**

24047N2a LDC #: Stage 2B/4 SDG #: 280-6886-1

Page: | of Reviewer: JV 2nd Reviewer:

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

Laboratory: Test America

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 /27 /10
II.	GC/MS Instrument performance check	A	,
III.	Initial calibration	A	2 RED -
V.	Continuing calibration/ICV	A	ca/10 =25)
<b>/</b> .	Blanks	Á	
/1.	Surrogate spikes	΄A	
11.	Matrix spike/Matrix spike duplicates	Sn)	
III.	Laboratory control samples	SW	ks/p
Χ.	Regional Quality Assurance and Quality Control	N	
ζ.	Internal standards	*	
(I.	Target compound identification	A	Not reviewed for Stage 2B validation.
11.	Compound quantitation/CRQLs	A	Not reviewed for Stage 2B validation.
III.	Tentatively identified compounds (TICs)	N	Not reviewed for Stage 2B validation.
IV.	System performance	A	Not reviewed for Stage 2B validation.
V.	Overall assessment of data	A	
VI.	Field duplicates	SM	$D_1^* = 1.8$ $D_2^* = 12.13$ $D_3 = 23.24$ $D_4^* = 34.$ $EB = {}^*40$ $PB = FB - 0415 2010 - RIGI - RZE -$
VII.	Field blanks	AMP	EB= 40 PB = FB-0413 2010- RIGI-RZE

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

¥ND = No compounds detected D = Duplicate

R = Rinsate FB = Field blank TB = Trip blank

EB = Equipment blank

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

	Seil	_ +	WKter				
1	BDT-1-N-20-10BPC P, S	11	BDT-1-N-5-14BPC S	21	BDT-1-N-10-4BPC S	31°	BDT-1-N-15-6BPC
2 1	BDT-1-N-20-12BPC**	12	BDT-1-N-5-8BPC_FD D	22	BDT-1-N-10-6BPC	<b>+</b> 32.	BDT-1-S-20-10BPC
3 7	BDT-1-N-20-14BPC	13	BDT-1-N-5-8BPC	23	BDT-1-N-10-8BPC 03	33	BDT-1-S-20-12BPC
4 7	BDT-1-N-20-2BPC	14	BDT-1-N-5-2BPC	+ 24	BDT-1-N-10-8BPC_FD	34	BDT-1-S-20-14BPC** <b>D4</b>
- 1 5	BDT-1-N-20-4BPC	<b>1</b> 5	BDT-1-N-5-4BPC	* 25	BDT-1-N-15-10BPC	- າ 35	BDT-1-S-20-2BPC
- 7 6	BDT-1-N-20-6BPC	16	BDT-1-N-5-6BPC	26 F	BDT-1-N-15-12BPC	36	BDT-1-S-20-4BPC
7 )	BDT-1-N-20-8BPC	17	BDT-1-N-10-10BPC	27	BDT-1-N-15-14BPC**	1 3 37	BDT-1-S-20-6BPC
- ) 8	BDT-1-N-20-10BPC_FD D,	18	BDT-1-N-10-12BPC	<b>↑</b> .	BDT-1-N-15-8BPC	† : 38	BDT-1-S-20-8BPC
<b>9</b>	BDT-1-N-5-10BPC	19	BDT-1-N-10-14BPC	† - 29	BDT-1-N-15-2BPC	39	BDT-1-S-20-14BPC_FD 24 J
10	BDT-1-N-5-12BPC	20 1	BDT-1-N-10-2BPC	30	BDT-1-N-15-4BPC	40 4	EB-08272010 W

# Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

LDC #: 24047N2a VALIDATION COMPLETENE
SDG #: 280-6886-1 Stage 2B/4
Laboratory: Test America

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

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

	Validation Area		Comments
1.	Technical holding times		Sampling dates:
II.	GC/MS Instrument performance check		
111.	Initial calibration		
IV.	Continuing calibration/ICV		
V.	Blanks		pase
VI.	Surrogate spikes		\
VII.	Matrix spike/Matrix spike duplicates		Sie
VIII.	Laboratory control samples		
IX.	Regional Quality Assurance and Quality Control	N	
Х.	Internal standards		
XI.	Target compound identification		Not reviewed for Stage 2B validation.
XII.	Compound quantitation/CRQLs		Not reviewed for Stage 2B validation.
XIII.	Tentatively identified compounds (TICs)		Not reviewed for Stage 2B validation.
XIV.	System performance		Not reviewed for Stage 2B validation.
XV.	Overall assessment of data		
XVI.	Field duplicates		
XVII.	Field blanks		

Note:

A = Acceptable

ND = No compounds detected D = Duplicate

N = Not provided/applicable

R = Rinsate

TB = Trip blank

SW = See worksheet FB = Field blank

EB = Equipment blank

Validated Samples:

\*\* Indicates sample underwent Stage 4 validation

,						
41	BDT-1-N-10-2BPCMS S	51 /	MB 280-30061/1-A	61	71	1
42	BDT-1-N-10-2BPCMSD	52 Y	MB 280-30099/1-A	62	72	2
<b>4</b> 3	BDT-1-N-15-4BPCMS	53 ×	MB 280 - 30764 /1-A	63	. 73	3
44	BDT-1-N-15-4BPCMSD	- 4 54	MB 280 - 29647 /-A	64	74	4
45 Y	BDT-1-S-20-4BPCMS	55		65	75	5
46 Y	BDT-1-S-20-4BPCMSD	56		66	76	6
<sub>47</sub> 3	BDT-1-S-20-6BPCMS	57		67	77	7
48 <b>3</b>	BDT-1-S-20-6BPCMSD	58		68	78	В
49	•	59		69	79	9
50		60		70	80	0

Page: 1 of 2
Reviewer: JV

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

Method: Semivolatiles (EPA SW 846 Method 8270C)

Validation Area	Yes	No	NA	Findings/Comments
1. Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.			25/2/2000	
II. GCMS Institutent performance eyes (* 425. ) in the second		ı		
Were the DFTPP performance results reviewed and found to be within the specified criteria?	/			
Were all samples analyzed within the 12 hour clock criteria?		ł		
III. Initial calibration Section 3. Application and the section of		i		
Did the laboratory perform a 5 point calibration prior to sample analysis?	<del>                                     </del>		-	
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?			40.5	
IV Continuing calibration				And the Control of th
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?				
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?				
Were all percent differences (%D) $\leq$ 25% and relative response factors (RRF) $\geq$ 0.05?				
V/Blanks (1982) and a Second s				
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.				
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?				
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 Z.E. Berand Z. Shanis kedingler 2 letter i server en				
Was an LCS analyzed for this SDG?				

LDC#: 24047 Nra

# **VALIDATION FINDINGS CHECKLIST**

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?	/		<u> </u>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX, Regional Quality Assurance and Quality Egypta 2012 2013 2013				
Were performance evaluation (PE) samples performed?	ļ.			
Were the performance evaluation (PE) samples within the acceptance limits?	(C. 201 2.27. 85) ev	27.20 PM 101 - 100	/	
X internal additions (1)				
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. X pel compound Demilication				and the many of the state of the
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?			e year and a	
XII Compound quantization/CRGEs as let 3.				
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. Terratively identified compounds (TACS)				
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)?			·	
AL SOCIETA (100) 23				
System performance was found to be acceptable.		And the Andreas Locking		
Overall assessment of data was found to be acceptable.	1			
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.				
Microsoft and the second of th				
Field blanks were identified in this SDG.		no districtiva		
Target compounds were detected in the field blanks.	A	.	十	, , , , , , , , , , , , , , , , , , ,

# VALIDATION FINDINGS WORKSHEET

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

A. Phenol**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT, Pentachlorophenoi**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	VV. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene⊷	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene™	JJ. Dibenzofuran	YY. Fluoranthene™	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butyibenzyiphthalate	PPP. Benzoic Acid
l. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Ntroso-di-n-propylamine⁴	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-octyiphthalate**	nnn
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	WW.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	www.

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

LDC# 24647 N2A

# VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

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

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

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

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

YN N/A

MS/MSD. Soil / Water.

Y N N/A

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

			***************************************		-				
) #	Date	MS/MSD ID	Compound	MS %R (Limits)	s)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		43/44	Sys	252 (51-12	( 0<1-	284 (51.12)	( )	26	( LCS in)
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	Compound	QC Limits (Soil)	RPD (Soil)	QC Limits (Water)	RPD (Water)		Compound	QC Limits (Soil)	RPD (Soll)	QC Limits (Water)	RPD (Water)
∢	Phenol	78-30%	< 35%	12-110%	< 42%	GG	Acenaphthene	31-137%	< 19%	46-118%	< 31%
O	C. 2-Chlorophenol	25-102%	< 50%	27-123%	< 40%	=	4-Nitrophenol	11-114%	< 50%	10-80%	< 50%
ш	1,4-Dichlorobenzene	28-104%	< 27%	36-97%	< 28%	K.	2,4-Dinitrotoluene	28-89%	< 47%	24-96%	< 38%
-5	N-Nitroso-di-n-propylamine	41-126%	< 38%	41-116%	< 38%	Ľ	Pentachlorophenol	17-109%	< 47%	9-103%	< 50%
œ	1,2,4-Trichlorobenzene	38-107%	< 23%	39-98%	< 28%	72.	Pyrene	35-142%	×9e >	26-127%	< 31%
·	/.   4-Chloro-3-methylphenol	26-103%	< 33%	23-97%	< 42%						

LDC# 240 47 N2a

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

l of 1 Page: 2nd Reviewer: Reviewer:

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".

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

Ne RRR	الق	Date	LCS/LCSD ID	Compound	%R	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications	
		$\Box$	42/19002-08c SM	RAR	ફ	(23-120)	( )	( )		) 24	
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LDC#: 24047N2a

# **VALIDATION FINDINGS WORKSHEET**

Field Duplicates

Page	:of <u>_</u> _
Reviewer:	N.
2nd Reviewer:	<i>i</i> ~

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

Y) N NA N NA

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

Compound Name	Conc (	ug/Kg)	RPD (≤ 50%)	Diff	Diff Limits	Quals (Parent Only)
Compound Name	23	24				
Hexachlorobenzene	130	180		50	≤350	

V:\FIELD DUPLICATES\24047N2a.wpd

LDC#: 24047 N2A SDG#:

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

d of Jo Page: Reviewer:

2nd Reviewer:

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

 $A_x$  = Area of Compound

A<sub>is</sub> = Area of associated internal standard  $C_{\rm is}$  = Concentration of internal standard

%RSD = 100 \* (S/X)

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

X = Mean of the RRFs

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
		Calibration		RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
#	Standard ID Date	Date	Compound (Internal Standard)	( 50 std)	( 50 std)	(Initial)	(Initial)		
-	ICAL	9/7/2010	9/7/2010 1,4-Dioxane (IS1)	0.6211	0.6211	0.6078	0.6078	4.6	4.60
	MSS Y		Naphthalene (IS2)	1.0905	1.0905	1.1045	1.1045	2.1	2.14
			Fluorene (IS3)	1.2364	1.2364	1.2854	1.2854	5.5	5.51
			Hexachlorobenzene (IS4)	0.2096	0.2096	0.2150	0.2150	6.4	6.44
			Chrysene (IS5)	1.1090	1.1090	1.1025	1.1025	2.7	2.65
			Benzo(g,h,i)perylene (IS6)	0.9979	0.9979	1.0022	1.0022	9.8	9.82

7.7	- 00000	A 1	Ĺ		-	
<u>+</u>	1,4-Dioxane	Naphthalene	Fluorene	Hexachlorob	Chrysene	Benzo(g,h,i)per
		1.0765	1,2003		1.0903	0.8090
	0.6455	1.0884	1.2284	0.1920	1.0698	0.9296
	0.5733	1.0785	1.2225	0.2033	1.0870	0.9649
	0.6211	1.0905	1.2364	0.2096	1.1090	0.9979
	0.6367	1.1224	1.3100	0.2235	1.1499	1.0699
	0.5993	1.1231	1.3577	0.2215	1.0926	1.0746
	0.6021	1.1381	1.3567	0.2235	1.1424	1.0836
	0.5769	1.1181	1.3713	0.2314	1.0792	1.0883
	0.6078	1.1045	1.2854	0.2150	1.1025	1.0022
	0.0279	0.0236	0.0709	0.0138	0.0292	0.0985
				7,111		

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LDC#: 24047 Nrg

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

4 JVG ر م Page:

Reviewer:

2nd Reviewer:

## 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

%RSD = 100 \* (S/X)

A<sub>x</sub> = Area of Compound

 $C_x$  = Concentration of compound,

A<sub>is</sub> = Area of associated internal standard  $C_{\text{is}} = \text{Concentration of internal standard}$ 

S= Standard deviation of the RRFs,

X = Mean of the RRFs

					Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
		Calibration			RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
#	Standard ID	Date	Compound (Internal Standard)	andard)	( 50 std)	( 50 std)	(Initial)	(Initial)		
-	ICAL	8/30/2010	8/30/2010 1,4-Dioxane	(IS1)	0.6494	0.6494	0.6538	0.6538	3.2	3.21
1	MSS B		Naphthalene	(IS2)	1.0810	1.0810	1.0482	1.0482	11.0	11.01
			Fluorene	(183)	1.3696	1.3696	1.3037	1.3037	11.0	10.98
			Hexachlorobenzene	(IS4)	0.2517	0.2517	0.2454	0.2454	3.8	3.76
			Chrysene	(IS5)	1.1455	1.1455	1.0975	1.0975	8.1	8.10
			Benzo(g,h,i)perylene	(186)	1.1162	1.1162	1.0926	1.0927	2.5	2.49

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Conc	1,4-Dioxane	Naphthalene	Fluorene	Hexachlorob	Chrysene	Benzo(g,h,i)per
4.00	0.6780	1.1880	1.4403	0.2501	1.1731	1.0576
10.00	0.6510	1.1590	1.4246	0.2516	1.1774	1.0704
20.00	0.6855	1.1306	1.4342	0.2514	1.1786	1.0855
50.00	0.6494	1.0810	1.3696	0.2517	1.1455	1.1162
80.00	0.6655	1.0601	1.3277	0.2522	1.1198	1.1433
120.00	0.6423	0.9839	1.2284	0.2435	1.0281	1.1029
160.00	0.6276	0.9020	1.1307	0.2339	1.0006	1.0822
200.00	0.6313	0.8811	1.0742	0.2287	0.9572	1.0831
×	0.6538	1.0482	1.3037	0.2454	1.0975	1.0927
S)	0.0210	0.1154	0.1432	0.0092	0.0889	0.0273
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## Continuing Calibration Results Verification VALIDATION FINDINGS WORSHEET

JVG Page of Reviewer: 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:

% 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

		dito a dila			100 C	7770			
#	Standard ID	Date	Compound (Reference IS)	ice IS)	Average RRF (Initial RRF)	Reported (CC RRF)	(CC RRF)	Reported %D	Recalculated %D
1	B0206	09/10/10	1,4-Dioxane	(IS1)	0.6538	0.6341	0.6341	3.0	3.0
	MSS B		Naphthalene	(182)	1.0482	1.0565	1.0565	8.0	8.0
			Fluorene	(183)	1.3037	1.3158	1.3158	6.0	6.0
			Hexachlorobenzene	(184)	0.2454	0.2311	0.2311	5.8	5.8
			Chrysene	(185)	1.0975	1.0837	1.0837	1.3	1.3
			Benzo(g,h,i)perylene	(186)	1.0926	1.1305	1.1305	3.5	3.5
2	Y4836	09/10/10	1,4-Dioxane	(IS1)	0.6078	0.6535	0.6535	7.5	7.5
	MSS Y		Naphthalene	(182)	1.1045	1.1494	1.1494	4.1	4.1
			Fluorene	(183)	1.2854	1.3478	1.3478	4.9	4.9
			Hexachlorobenzene	(1S4)	0.2150	0.2416	0.2416	12.4	12.4
			Chrysene	(185)	1.1025	1.1377	1.1377	3.2	3.2
			Benzo(g,h,i)perylene	(981)	1.0022	0.9672	0.9672	3.5	3.5

compound (Reference 15)	<u>(S)</u>	Concentration	Area Cpd	Area IS	Area Cpd	Area IS
		(IS/Cpd)				
1,4-Dioxane	(IS1)	40/80	307194	242234	293253	224375
Naphthalene	(182)	40/80	2005560	949171	1977504	860218
Fluorene	(1S3)	40/80	1455116	552933	1512241	561014
Hexachlorobenzene	(IS4)	40/80	437837	947113	442594	915793
Chrysene	(185)	40/80	2166684	999630	1994638	876639
Benzo(g,h,i)perylene	(186)	08/04	2284745	1010468	1187355	613808

LDC#: 24047 N79

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

Page:_	lof_1_
Reviewer:	JV4
2nd reviewer:	4

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:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	100	64.9	65	45	0
2-Fluorobiphenyl		67.9	68	0 8	
Terphenyl-d14	. 1	75.4	75	75	
Phenol-d5	150	100,6	67	G 7	
2-Fluorophenol		95,3	64	64	
2,4,6-Tribromophenol		95.1	64	64	/
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			·		y
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol				-	
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

SDG# Sec Cores

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

lof	<u>ئ</u>	<b>\</b>
Page:	Reviewer:_	and Reviewer

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

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

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

SSC = Spiked sample concentration SA = Spike added Where:

SC = Sample concentation

MSDC = Matrix spike duplicate concentration

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

MSC = Matrix spike concentration

r MS/MSD samples: \_

	ďS	ike	Sample	Spiked	Spiked Sample	Matrix Spike	Spike	Matrix Spik	Matrix Spike Duplicate	USW/SW	SD
Compound	Added (45 /E	Added	Concentration (ねん/と)	Concer (45	Concentration	Percent Recovery	всочегу	Percent F	Percent Recovery	RPD	0
	MS	Ø MSD	0	MS	O MSD	Reported	Recalc	Reported	Recalc	Renorted	Recalculated
Phenol											
N-Nitroso-di-n-propylamine											
4-Chloro-3-methylphenol											
Acenaphthene	2740	0222	0	1980	1810	70	70	67	70	₽v	۵
Pentachlorophenol			1								
Pyrene	27%	a22	}	al I	290/	8	8.7	83	83	7	e
						•					

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.

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

Page: Lof 1 Reviewer: 1/4

2nd 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

SSC = Spike concentration SA = Spike added Where:

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

										The second secon
	l'S	oike	ďS	ike	<u> </u>	SU	01	l CSD	1 CS/I CSD	csn
Compound	Added (Mg /kg	ided (fr. )	Concentration (MS//a)	ntration	Percent Recovery	Recovery	Percent F	Percent Recovery	RPD	٥٥
	1.08	l CSD	l CS	l CSD	Reported	Recalc	Reported	Recalc	Reported	Receiculated
Phenol										
N-Nitroso-di-n-propylamine										
4-Chioro-3-methylphenol										
Acenaphthene	2510	NA	1640	MZM	57	59				
Pentachlorophenol										
Pyrene	25-10	<b>\</b>	(870		p	74	\			
							\			

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 #: 24047 N29

## VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	lof1_
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2nd reviewer:	<b>\</b>

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

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

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

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

Concentration =  $(A_x)(I_x)(V_t)(DF)(2.0)$  $(A_{ix})(RRF)(V_o)(V_t)(%S)$ 

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

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

I = 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>1</sub> = Volume of extract injected in microliters (ul)

V<sub>1</sub> = Volume of the concentrated extract in microliters (ul)

Df = Dilution Factor.

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

Example:

Sample I.D. # 27 , SS

Conc. =  $\frac{(24510)(40)(1m)}{(768976)(0.2150)(30.19)(0.967)}$ 

= 217. Y

2 220 mg/kg

2.0	<ul> <li>Factor of 2 to account</li> </ul>	t for GPC cleanup				
#	Sample ID	Compound		Reported Concentration ( )	Calculated Concentration ( )	Qualification
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## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Data Validation Reports LDC #24047

**Chlorinated Pesticides** 



## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 7 through August 9, 2010

LDC Report Date:

October 8, 2010

Matrix:

Soil/Water

Parameters:

Chlorinated Pesticides

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6223-1

## Sample Identification

SSAJ3-02-12BPC

SSAJ3-02-15BPC

SSAJ3-02-8BPC\*\*

SSAJ3-02-8BPC FD

EB-08072010

SSAI3-04-14BPC

SSAI3-04-14BPC FD

SSAI3-02-11BPC

SSAI3-02-14BPC

SSAI3-02-5BPC FD

SSAI3-03-11BPC

**SSAI3-03-14BPC** 

SSAI3-02-1BPC

SSAI3-02-5BPC

SSAI3-02-8BPC

SSAI3-03-1BPC

SSAI3-03-5BPC\*\*

SSAI3-03-8BPC

SSAI3-03-8BPCMS

SSAI3-03-8BPCMSD

<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 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Samples indicated by a double asterisk on the front cover underwent a 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.

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-08072010 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
EB-08072010	RTI-XLB RTI-35	Decachlorobiphenyl Decachlorobiphenyl	24 (34-122) 22 (34-122)	All TCL compounds	J- (all detects) UJ (all non-detects)	Р

## VII. Matrix Spike/Matrix Spike Duplicates

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

## VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Percent recoveries (%R) 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 280-6223-1	All compounds reported below the PQL.	J (all detects)	A

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 SSAJ3-02-8BPC\*\* and SSAJ3-02-8BPC\_FD, samples SSAI3-04-14BPC and SSAI3-04-14BPC\_FD, and samples SSAI3-02-5BPC\_FD and SSAI3-02-5BPC were identified as field duplicates. No chlorinated pesticides were detected.

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Data Qualification Summary - SDG 280-6223-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6223-1	EB-08072010	All TCL compounds	J- (all detects) UJ (all non-detects)	Р	Surrogate recovery (%R) (s)
280-6223-1	SSAJ3-02-12BPC SSAJ3-02-15BPC SSAJ3-02-8BPC** SSAJ3-02-8BPC_FD EB-08072010 SSAI3-04-14BPC SSAI3-04-14BPC_FD SSAI3-02-11BPC SSAI3-02-14BPC SSAI3-03-14BPC SSAI3-03-14BPC SSAI3-03-14BPC	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 280-6223-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Equipment Blank Data Qualification Summary - SDG 280-6223-1

No Sample Data Qualified in this SDG

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

LDC #: 24047B3a	VALIDATION COMPLETER
SDG #: 280-6223-1	Stage 2E
Laboratory: Test America	

Reviewer: 2nd Reviewer:

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

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

	Validation Area		Comments
I.	Technical holding times	Á	Sampling dates: 8 /67_ 01 /10
11.	GC/ECD Instrument Performance Check	A	
111.	Initial calibration	A	2 KSD 620 6 r7
IV.	Continuing calibration/ICV	A	2 KSD = 20 6 r7  CW/W = 20 6
V.	Blanks	A	
VI.	Surrogate spikes	sw	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	us /b
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	4
Xb.	GPC Calibration	N	<b>,</b>
XI.	Target compound identification	Α	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	Ŋ	$D_1 = \frac{3}{4}$ $D_2 = \frac{6}{7}$ $D_3 = \frac{14}{10}$ $EB = \frac{5}{7}$ $FB = \frac{18}{10}$ $EB = \frac{14}{10}$
XV.	Field blanks	ND	tb = 5 F8 - +8 04 072010-R2D

Note:

A = Acceptable

N = Not provided/applicable SW = See worksheet

ND = No compounds detected

R = Rinsate

D = Duplicate

TB = Trip blank

FB = Field blank

EB = Equipment blank

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

Valida	· ·	⊶`		to vent otage 4 validation				
		76	il_	t Water				
1	SSAJ3-02-12BPC	5	11	SSAI3-03-11BPC	21 ]	MB 280 -26587/14	31	
2	SSAJ3-02-15BPC		- 12	SSAI3-03-14BPC**	22 7	mb 280-26 58 WA	<b>A</b> 2	
3	SSAJ3-02-8BPC* ≯ D,		_ 13	SSAI3-02-1BPC	23		33	
4	SSAJ3-02-8BPC_FD- P,		- 14	SSAI3-02-5BPC	24		34	
- <b>~</b>	f .	٧	 15	SSAI3-02-8BPC	25		35	
6	SSAI3-04-14BPC	Ş	_ 16	SSAI3-03-1BPC	26	•	36	
- 7	SSAI3-04-14BPC_FD D/			SSAI3-03-5BPC**	27		37	
8	SSAI3-02-11BPC		_ 18	SSAI3-03-8BPC	28		38	
9	SSAI3-02-14BPC		19	SSAI3-03-8BPCMS	29		39	
10	SSAI3-02-5BPC_FD 1/3	И	20	SSAI3-03-8BPCMSD	30		40	

## **VALIDATION FINDINGS CHECKLIST**

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times	<del></del>			
All technical holding times were met.	_	<u> </u>	<u> </u>	
Cooler temperature criteria was met.	_			
II. GC/ECD Instrument performance check	T	1	т	1
Was the instrument performance found to be acceptable?				
III. Initial calibration	Ι		·	I
Did the laboratory perform a 5 point calibration prior to sample analysis?				
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations $(\%RSD) \leq 20\%$ ?		-		
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?			<u> </u>	
Did the initial calibration meet the curve fit acceptance criteria?				
Were the RT windows properly established?				
Were the required standard concentrations analyzed in the initial calibration?		/		
IV. Continuing calibration				· · · · · · · · · · · · · · · · · · ·
What type of continuing calibration calculation was performed?%D or%R				
Were Evaluation mix standards analyzed prior to the initial calibration and sample analysis?				
Were endrin and 4,4'-DDT breakdowns ≤ 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?	1			
/ Blanks				
Nas a method blank associated with every sample in this SDG?	1			
Vas a method blank analyzed for each matrix and concentration?	/			
Vere extract cleanup blanks analyzed with every batch requiring clean-up?				
Vas there contamination in the method blanks or clean-up blanks? If yes, please see ne Blanks validation completeness worksheet.		1		
1 Surrogate spikes				
Vere all surrogate %R within the QC limits?		$\triangle$		
the percent recovery (%R) of one or more surrogates was outside QC limits, was a canalysis performed to confirm %R?				
any %R was less than 10 percent, was a reanalysis performed to confirm %R?				
II. Matrix spike/Matrix spike duplicates	,		,	

LDC #: 240 F7 334

## VALIDATION FINDINGS CHECKLIST

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?	<u> </u>			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII. Laboratory control samples	<del></del>			
Was an LCS analyzed for this SDG?				
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		,		
IX: Regional Quality Assurance and Quality Control	I		2	
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
X. Target compound identification				
Were the retention times of reported detects within the RT windows?				
XI Compound quantitation/CRQLs	1			
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.		1		
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-BHC	1. Dieldrin	Q. 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-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	H.
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	
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	LL.
G. Heptachlor epoxide	O.4,4'-DDT	W. Aroclor-1221	EE. 2,4'-DDT	ММ.
H. Endosulfan 1	P. Methoxychlor	X. Aroclor-1232	FF. Hexachlorobenzene	NN.

Notes:

LDC #: 2 \$ 0 \$ 7 B 32

## **VALIDATION FINDINGS WORKSHEET** Surrogate Spikes

Page:

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

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

Y N N/A

Were surrogates spiked into all samples, standards and blanks?

Y N N/A

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

Qualifications	5- M3 4 (2)																	
%R (Limits)	(36,123)	` <b>\</b>	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
%R (L	24	2,2																
Surrogate Compound	0								The state of the s									
Column	RTI-XLB	RTI-35																
Sample ID	ک																	
Date																		
#																		

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A	Tetrachoro-m-xylene			
В	Decachlorobiphenyl			

LDC # 24647 Bra

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: ) of £

Reviewer: 306 2nd Reviewer: \_\_\_\_\_\_

GC EPA SW 846 Method 8081A METHOD:

Hexachlorobenzene Parameter:

	100.00	953705.00			
100000000000000000000000000000000000000	75.00	730674.00			
	50.00	490208.00			
	25.00	249072.00		GCS_P2	
	10.00	103588.00			
	4.00	44827.00	Hexachlorobenzene	CLP1	08/11/2010
•	Conc	Area	Compound	Column	Date .
1					
X^2	×	>			

Regression Output:	ıt.		Reported	77
Constant		0.00000	<b>=</b> 0	0.00000
Std Err of Y Est		8773.78312	and the second s	
R Squared		0.99941	12=	0.999900
No. of Observations		00000'9		
Degrees of Freedom		5.00000		
X Coefficient(s)	9653.526874	-1.270906	# q	9638.000000
Std Err of Coef.	63.877363	0.79	A CONTRACTOR OF THE PROPERTY O	

10358.80 9962.88 9804.16 9742.32

11206.75

Ave RF

9537.05

10101.99

LDC # 24047 829

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: 7 of 4

GC EPA SW 846 Method 8081A METHOD:

Parameter:

4,4'-DDT

X^2				n de option de la constant de la con		and the second s		
×	Conc	4.00	10.00	25.00	50.00	75.00	100.00	
>	Area	33015.00	78046.00	193282.00	386784.00	581766.00	756268.00	
	Compound	4,4'-DDT						
	Column	CLP1		GCS_P2				
	Date	08/11/2010						

Regression Output:	ţ		Reported	
Constant		0.00000	= 0	0.0000
Std Err of Y Est		5851,33656		
R Squared		0.99959	12 =	0.999700
No. of Observations		000009		
Degrees of Freedom	A LANGUAGO CANCINE CONTRACTOR CON	5.00000	A JALA A ROLL MITTER	
× Ocafficiant(c)	7650 960458	1 270906	= 9	7636 000000
A Coefficient(s)	42 600546	0200	2	
old Err of Coef.	247.000.74	0.13		

7731.28 7735.68 7756.88 7562.68

8253.75 7804.60

Ave RF

7807.48

## LDC# 240 F7 B3 G

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

GC EPA SW 846 Method 8081A METHOD:

Parameter:

4,4'-DDT

Date	Column	Compound	Y Area	× Conc	X,X
08/11/2010	CLP2	4,4'-DDT	59795.00	4.00	16.00
			137046.00	10.00	100.00
	GCS_P2		321682.00	25.00	625.00
			607290.00	50.00	2500.00
			883436.00	75.00	5625.00
			1123921.00	100.00	10000.00

Regression Output:			Reported	
Constant		8705.27176	11 0	NR
Std Err of Y Est		3439.22112		
R Squared		96666.0	r2 =	0.999990
No. of Observations		000000		
Degrees of Freedom		3.00000		
			a	NR
X Coefficient(s)	12932.023828	-17.656945	= q	NR
Std Err of Coef.	164.371396	1.57		

Ave RF

Reviewer: TVz 2nd Reviewer:

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Page:

14949

13705

12867

12146

11779

11239

12781

## LDC # 240 47 Boc

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

GC EPA SW 846 Method 8081A METHOD:

Hexachlorobenzene Parameter:

·							
X^2	16.00	100.00	625.00	2500.00	5625.00	10000.00	
Conc	4.00	10.00	25.00	50.00	75.00	100.00	
Y Area	93334.00	210505.00	481272.00	894649.00	1284080.00	1628971.00	
Compound	Hexachlorobenzene			•		•	
Column	CLP2		GCS_P2				
Date	08/11/2010						

Regression Output:	out:		Reported	
Constant		20708.90229	= 0	AR.
Std Err of Y Est		3835.69679		
R Squared		86666.0	r2 =	0.999990
No. of Observations		00000'9	The state of the s	
Degrees of Freedom		3.00000		
			<i>т</i>	N.
X Coefficient(s)	19034.788783	-29.504222	= q	N.
Std Err of Coef.	183.320239	1.76		

17893 21051 19251 17121 16290

23334

19156 Ave RF

Reviewer: 3

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LDC #240 47 B39

## VALIDATION FINDINGS WORKSHEET Continuing Calibration Calculation Verification

Reviewer: 210 Page: \(\lambda\) of \(\lambda\)

METHOD: GC

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

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

Where:

Initial Calibration Factor or Nominal Amount

Calibration Factor from Continuing Calibration Standard or Calculated Amount " # Z O

Recalculated	۵%		1.5	0.0	2.6	6.8	4.6	7.8	6.0	3,3								
Reported	۵%		0.2	7.0	2.7	6.9	1.8	1.3	6.0	3.4								
Recalculated	Conc		50.8	50.0	48.7	46.6	52.3	53.9	49.6	48.4								
Reported	Conc		50.1	49.7	48.7	46.5	6.03	49.3	49.5	48.3							,	
	CCV Conc		50	50	90	50	50											
		Jq.	CLP1	CLP1	CLP2	CLP2	CLP1	CLP1	CLP2	CLP2								
		Compound	HCB	占	E E	l۶	HCB	4,4'-DDT	왕	15								<u> </u>
	Calibration	Date	8/17/2010	20:42			8/18/2010	3:50										
		Standard ID	018F1801				044F4401											
		#	1				2				3	_	_	4		5		

CCV5				
CCV4				
CCV3				
CCV2	Area	503972	411700	
CCV1	Area	489215	381691	
	Slope	9638	7636	
		HCB CLP1	DDT CLP1	

					(-b+ ())/2a	)^ 1/2 (-b-())/2a	261218047.4 16162.2414 48.6806465 596.478959	127378069.6 11286.1893 46.6057277 685.796379	259530536.6 16109.9515 49.5667964 595.592809	125978646.4 11224.021 48.3661733 684.035934
				Calculation		(b^2-4aT) ( )^1/2	261218047.4	127378069.6	259530536.6	125978646.4
			1			7=Y-c	-856707.098	-564353.728	-871006.098	-584167.728
						final conc				
					Conc.	×	48.681	46.606	49.567	48.366
						v	20708.902	8705.272	20708.902	8705.272
Area	503972	411700		٥		٩	19034.789	12932.024	19034.789	12932.024
Area	489215	381691		$Y = a(X^2) + bX + c$		Ø	-29,504	-17.657	-29.504	-17.657
Slope	9638	7636			Area	>	877416	573059		592873
<i>-</i>	HCB CLP1	DDT CLP1			`		CCV1 HCB CLP2	CCV1 ddt CLP2	CCV2 HCB CLP2	CCV2 ddt CLP2

LDC#: 240 47 \$35

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

Page:	1_of_/	
Reviewer:_	276	
2nd reviewer:	10	7

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

The percent recoveries	s (%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: # 3

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene	CLP 1	20	18.0	90	10	0,
Tetrachloro-m-xylene	1 2		17.4	87	87	
Decachlorobiphenyl			20.1	107	16/	
Decachlorobiphenyl	1 1	- X	18.9	95	95	<del></del>

Sample ID:

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

Sample ID:

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

Notes:	

LDC#: 2 40 47 B3 A

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

Page: \ of \ Reviewer: 376 2nd Reviewer:\_

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

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

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

SSC = Spiked sample concentration SA = Spike added

Where:

SC = Concentration

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

MS = Matrix spike percent recovery

MSD = Matrix spike duplicate percent recovery

MS/MSD samples:

<u>v</u>

	is	pike	Sample	Spiked	Sample	Matri	Matrix Spike	Matrix Spik	Matrix Spike Duplicate	MS	MS/MSD
Compound	 \$ ঐ	Added (५८ /६ )	Concentration (いく/と)	Concer ( NC /	Concentration (ル/上 )	Percent	Percent Recovery	Percent	Percent Recovery		RPD
	MS	Ø MSD	٥.	WS	O MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
gamma-BHC	17.9	17.7	9	58:51	16.17	36	38	ا ا	1 6	h	h
4,4'-DDT		7	Þ	16.00	16.79	96	οş	26	26	у.	S
Aroclor 1260						•					
		·									
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Comments: Refer ot Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

## LDC#: 240 47 339

# VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicate Results Verification

Jof 1 Reviewer: 37/2 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

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

Where:

SC = Concentration

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

SSC = Spiked sample concentration SA = Spike added

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

265276-7 280-Z LCS/LCSD samples;

		-111								
		pike ddod	Spiked	Sample	2	ICS				
Compound		Dann.	Conce	ntration			רכ	CSD	TCS//CSD	<u>,</u>
		A WELL	347	("5/E()	Percent Recovery	Recovery	Q			
	သ	CSD		,			Leicent	reicent Recovery	RPD	
			LCS	LCSD	Reported	Recalc.	Donottod	1	L	
gamma-BHC	2	~	7	,			Del Joday	Kecalc.	Reported	Recalc.
		£	- ` + `	۷ ا	>0 >0	×				
4,4'-DDT	6.3/		1 10		6					
Arodos 10eo		4	9.	1	2	<u>~</u>				Ī
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Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicate findings worksheet for list of qualifications and associated samples when reported

V:\Validation Worksheets\Pesticides\LCSDCLC.wpd

LDC #:	740	47	1 3	34
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## VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	of
Reviewer:	JVG
2nd reviewer:	1. /

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

Υ	N	N/A
Υ	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?

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

#	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 10, 2010

LDC Report Date:

October 13, 2010

Matrix:

Soil

Parameters:

Chlorinated Pesticides

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6345-1

## Sample Identification

SSAJ3-07-SW-E-1BPC\*\*

SSAJ3-05-SW-E-1BPC

SSAJ3-02-SW-E-1BPC

SSAI3-04-SW-E-1BPC

SSAI3-03-SW-E-1BPC

SSAI3-02-SW-E-1BPC FD

SSAI3-02-SW-E-1BPC

SSAI3-02-SW-W-1BPC

SSAI3-03-SW-W-1BPC

SSAI3-04-SW-W-1BPC

SSAJ3-02-SW-W-1BPC

SSAJ3-05-SW-W-1BPC

SSAJ3-07-SW-W-1BPC\*\*

SSAJ3-07-SW-W-1BPC FD

**SB01-24BPC** 

SB02-24BPC

SSAJ3-02-SW-W-1BPCMS

SSAJ3-02-SW-W-1BPCMSD

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

## Introduction

This data review covers 18 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Samples indicated by a double asterisk on the front cover underwent a 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
8/26/10	019F1901	CLP2	gamma-BHC Heptachlor gamma-Chlordane Endosulfan sulfate Endrin ketone	23.1 23.8 20.3 20.4 73.9	SSAI3-04-SW-W-1BPC SSAJ3-02-SW-W-1BPC SSAJ3-05-SW-W-1BPC** SSAJ3-07-SW-W-1BPC_FD SSAJ3-02-SW-W-1BPCMS SSAJ3-02-SW-W-1BPCMSD	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.

No field blanks were identified in this SDG.

## VI. Surrogate Spikes

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

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
SSAJ3-05-SW-E-1BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	386 (63-124) 387 (63-124)	All TCL compounds except 4,4'-DDE beta-BHC Hexachlorobenzene	J+ (all detects)	А
SSAJ3-02-SW-E-1BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	619 (63-124) 624 (63-124)	All TCL compounds except 4,4'-DDE 4,4'-DDT beta-BHC Hexachlorobenzene	J+ (all detects)	A
SSAI3-04-SW-E-1BPC	CLP1 CLP2 CLP2	Decachlorobiphenyl Tetrachloro-m-xylene Decachlorobiphenyl	354000 (63-124) 0 (59-115) 59000 (63-124)	4,4'-DDD Aldrin alpha-Chlordane Chlordane (Technical) delta-BHC Dieldrin Endosulfan I Endosulfan II Endrin Endrin aldehyde gamma-BHC gamma-Chlordane Heptachlor epoxide	J (all detects) UJ (all non-detects)	A
SSAI3-03-SW-E-1BPC	CLP1 CLP2 CLP1 CLP2	Tetrachloro-m-xylene Tetrachloro-m-xylene Decachlorobiphenyl Decachlorobiphenyl	137 (59-115) 0 (59-115) 86700 (63-124) 25100 (63-124)	All TCL compounds except Hexachlorobenzene Toxaphene Endosulfan sulfate 4,4'-DDE 4,4'-DDT	J (all detects) UJ (all non-detects)	А

C1-	0-1			_		
Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
SSAI3-02-SW-E-1BPC_FD	CLP1 CLP2 CLP1 CLP2	Tetrachloro-m-xylene Tetrachloro-m-xylene Decachlorobiphenyl Decachlorobiphenyl	212 (59-115) 0 (59-115) 31900 (63-124) 14100 (63-124)	All TCL compounds except 4,4'-DDT Hexachlorobenzene Methoxychlor Toxaphene	J (all detects) UJ (all non-detects)	A
SSAI3-02-SW-E-1BPC	CLP1 CLP2 CLP1 CLP2	Tetrachloro-m-xylene Tetrachloro-m-xylene Decachlorobiphenyl Decachlorobiphenyl	222 (59-115) 0 (59-115) 29900 (63-124) 12800 (63-124)	All TCL compounds except 4,4'-DDT Endosulfan sulfate Hexachlorobenzene Methoxychlor Toxaphene	J (all detects) UJ (all non-detects)	А
SSAI3-02-SW-W-1BPC	CLP1 CLP2 CLP1 CLP2	Tetrachloro-m-xylene Tetrachloro-m-xylene Decachlorobiphenyl Decachlorobiphenyl	238 (59-115) 0 (59-115) 166000 (63-124) 40300 (63-124)	All TCL compounds except 4,4'-DDE 4,4'-DDT Endosulfan sulfate Hexachlorobenzene Toxaphene	J (all detects) UJ (all non-detects)	А
SSAI3-03-SW-W-1BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	915 (63-124) 933 (63-124)	All TCL compounds except Hexachlorobenzene Toxaphene	J+ (all detects)	A
SSAI3-04-SW-W-1BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	4770 (63-124) 3880 (63-124)	All TCL compounds except 4,4'-DDE 4,4'-DDT beta-BHC Hexachlorobenzene Toxaphene	J+ (all detects)	А
SSAJ3-02-SW-W-1BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	378 (63-124) 406 (63-124)	All TCL compounds except beta-BHC Hexachlorobenzene	J+ (all detects)	Α
SSAJ3-05-SW-W-1BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	259 (63-124) 273 (63-124)	All TCL compounds except Hexachlorobenzene beta-BHC	J+ (all detects)	Α
SSAJ3-07-SW-W-1BPC**		Decachlorobiphenyl Decachlorobiphenyl	553 (63-124) 568 (63-124)	All TCL compounds except Hexachlorobenzene beta-BHC	J+ (all detects)	А
SSAJ3-07-SW-W-1BPC_FD		Decachlorobiphenyl Decachlorobiphenyl	642 (63-124) 660 (63-124)	All TCL compounds except Hexachlorobenzene beta-BHC	J+ (all detects)	A
SB01-24BPC	CLP2 CLP1	Tetrachloro-m-xylene Tetrachloro-m-xylene Decachlorobiphenyl Decachlorobiphenyl	265 (59-115) 255 (59-115) 2170 (63-124) 3040 (63-124)	All TCL compounds except 4,4'-DDE 4,4'-DDT alpha-BHC Hexachlorobenzene	J+ (all detects)	A

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
SB02-24BPC	CLP1 CLP2 CLP1 CLP2	Tetrachloro-m-xylene Tetrachloro-m-xylene Decachlorobiphenyl Decachlorobiphenyl	753 (59-115) 1100 (59-115) 12200 (63-124) 32400 (63-124)	All TCL compounds except 4,4'-DDD 4,4'-DDE 4,4'-DDT beta-BHC Hexachlorobenzene	J+ (all detects)	A

## VII. Matrix Spike/Matrix Spike Duplicates

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

## VIII. Laboratory Control Samples (LCS)

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

## IX. Regional Quality Assurance and Quality Control

Not applicable.

## X. 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
SSAJ3-07-SW-W-1BPC**	alpha-BHC delta-BHC	69.3 142.2	J (all detects) J (all detects)	А

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6345-1	All compounds reported below the PQL.	J (all detects)	A

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 SSAI3-02-SW-E-1BPC and SSAI3-02-SW-E-1BPC\_FD and samples SSAJ3-07-SW-W-1BPC\*\* and SSAJ3-07-SW-W-1BPC\_FD were identified as field duplicates. No chlorinated pesticides were detected in any of the samples with the following exceptions:

	Concentra	tion (ug/Kg)				
Compound	SSAI3-02-SW-E-1BPC	SSAI3-02-SW-E-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
4,4'-DDE	22	27	20 (≤50)	-	-	-
alpha-BHC	16	17	6 (≤50)	-	-	-
Endosulfan sulfate	3400U	33	· -	3367 (≤3400)	-	-
Hexachlorobenzene	36000	42000	15 (≤50)	-	•	

	Concentra	tion (ug/Kg)					
Compound	SSAJ3-07-SW-W-1BPC**	SSAJ3-07-SW-W-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P	
4,4'-DDE	17	18	6 (≤50)	-	-	-	
4,4'-DDT	6.0	8.1	-	2.1 (≤1.7)	J (all detects)	А	
alpha-BHC	0.86	0.62	-	0.24 (≤1.7)	-	-	

	Concentra	tion (ug/Kg)				
Compound	SSAJ3-07-SW-W-1BPC**	SSAJ3-07-SW-W-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
delta-BHC	1.7	1.8	-	0.1 (≤1.7)	-	-
Endosulfan sulfate	1.7U	0.38	•	1.32 (≤1.7)	-	-
beta-BHC	290	260	11 (≤50)	-	-	-
Hexachlorobenzene	260	230	12 (≤50)	-	-	-

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Data Qualification Summary - SDG 280-6345-1

				T***	
SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6345-1	SSAI3-04-SW-W-1 BPC SSAJ3-02-SW-W-1 BPC SSAJ3-05-SW-W-1 BPC SSAJ3-07-SW-W-1 BPC** SSAJ3-07-SW-W-1 BPC_FD	gamma-BHC Heptachlor gamma-Chlordane Endosulfan sulfate  Endrin ketone  J+ (all detects)  J+ (all detects)  J+ (all detects)  J+ (all detects)		A	Continuing calibration (%D) (c)
280-6345-1	SSAJ3-05-SW-E-1BPC	All TCL compounds except 4,4'-DDE beta-BHC Hexachlorobenzene	А	Surrogate recovery (%R) (s)	
280-6345-1	SSAJ3-02-SW-E-1BPC	All TCL compounds except 4,4'-DDE 4,4'-DDT beta-BHC Hexachlorobenzene	А	Surrogate recovery (%R) (s)	
280-6345-1	SSAI3-04-SW-E-1BPC	4,4'-DDD Aldrin alpha-Chlordane Chlordane (Technical) delta-BHC Dieldrin Endosulfan I Endosulfan II Endrin Endrin aldehyde gamma-BHC gamma-Chlordane Heptachlor Heptachlor epoxide	J (all detects) UJ (all non-detects)	A	Surrogate recovery (%R) (s)
280-6345-1	SSAI3-03-SW-E-1BPC SSAI3-02-SW-W-1BPC	All TCL compounds except Hexachlorobenzene Toxaphene Endosulfan sulfate 4,4'-DDE 4,4'-DDT	J (all detects) UJ (all non-detects)	Α .	Surrogate recovery (%R) (s)
280-6345-1	SSAI3-02-SW-E-1BPC_FD	All TCL compounds except 4,4'-DDT Hexachlorobenzene Methoxychlor Toxaphene	J (all detects) UJ (all non-detects)	Á	Surrogate recovery (%R) (s)
280-6345-1	SSAI3-02-SW-E-1BPC	All TCL compounds except 4,4'-DDT Endosulfan sulfate Hexachlorobenzene Methoxychlor Toxaphene	J (all detects) UJ (all non-detects)	A	Surrogate recovery (%R) (s)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6345-1	SSAI3-03-SW-W-1BPC	All TCL compounds except Hexachlorobenzene Toxaphene	J+ (all detects)	A	Surrogate recovery (%R) (s)
280-6345-1	SSAI3-04-SW-W-1BPC	All TCL compounds except 4,4'-DDE 4,4'-DDT beta-BHC Hexachlorobenzene Toxaphene	J+ (all detects)	A	Surrogate recovery (%R) (s)
280-6345-1	SSAJ3-02-SW-W-1BPC SSAJ3-05-SW-W-1BPC SSAJ3-07-SW-W-1BPC** SSAJ3-07-SW-W-1BPC_FD	All TCL compounds except beta-BHC Hexachlorobenzene	J+ (all detects)	A	Surrogate recovery (%R) (s)
280-6345-1	SB01-24BPC	All TCL compounds except 4,4'-DDE 4,4'-DDT alpha-BHC Hexachlorobenzene	J+ (all detects)	А	Surrogate recovery (%R) (s)
280-6345-1	SB02-24BPC	All TCL compounds except 4,4'-DDD 4,4'-DDE 4,4'-DDT beta-BHC Hexachlorobenzene	J+ (all detects)	A	Surrogate recovery (%R) (s)
280-6345-1	SSAJ3-07-SW-W-1BPC**	alpha-BHC delta-BHC	J (all detects) J (all detects)	А	Project Quantitation Limit (RPD)(dc)
280-6345-1	SSAJ3-07-SW-E-1BPC** SSAJ3-05-SW-E-1BPC SSAJ3-02-SW-E-1BPC SSAI3-04-SW-E-1BPC SSAI3-03-SW-E-1BPC SSAI3-02-SW-E-1BPC SSAI3-02-SW-E-1BPC SSAI3-02-SW-W-1BPC SSAI3-03-SW-W-1BPC SSAI3-04-SW-W-1BPC SSAJ3-05-SW-W-1BPC SSAJ3-07-SW-W-1BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
280-6345-1	SSAJ3-07-SW-W-1BPC** SSAJ3-07-SW-W-1BPC_FD	4,4'-DDT	J (all detects)	A	Field duplicates (Difference) (fd)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG 280-6345-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG 280-6345-1

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

Stage 2B/4

Date:	10/12/10
Page:_	<u>l</u> of <u>/</u>
Reviewer:	WC
2nd Reviewer:	W_

Laboratory: Test America

LDC #: 24047E3a

SDG #: 280-6345-1

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 10 10
II.	GC/ECD Instrument Performance Check	A	
III.	Initial calibration	A	% RSD = 20 } r ~ CCV /1CV ≤ 20 }
IV.	Continuing calibration/ICV	SM)	Car/100 = 20 ]
V.	Blanks	A	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	A	KZ
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	SH	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	А	
XIV.	Field duplicates	SW	D, = 7,6 D, = 13,14
XV.	Field blanks	N	

Note:

A = Acceptable

N = Not provided/applicable SW = See worksheet ND = No compounds detected D = Duplicate

R = Rinsate

TB = Trip blank

FB = Field blank

EB = Equipment blank

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

	<u>Au</u>	Soils					
1 1	SSAJ3-07-SW-E-1BPC**	11	SSAJ3-02-SW-W-1BPC	21	MB 280-27469/-A	31	
2	SSAJ3-05-SW-E-1BPC	12	SSAJ3-05-SW-W-1BPC	22	MB 280- 28 995/1-A		
3	SSAJ3-02-SW-E-1BPC	13	SSAJ3-07-SW-W-1BPC	23		33	
4	SSAI3-04-SW-E-1BPC	14	SSAJ3-07-SW-W-1BPC_FD	24		34	
5	SSAI3-03-SW-E-1BPC	15	SB01-24BPC	25		35	
6	SSAI3-02-SW-E-1BPC_FD D	16	SB02-24BPC	26		36	
7	SSAI3-02-SW-E-1BPC	17	SSAJ3-02-SW-W-1BPCMS	27		37	
8	SSAI3-02-SW-W-1BPC	18	SSAJ3-02-SW-W-1BPCMSD	28		38	
9	SSAI3-03-SW-W-1BPC	19		29		39	
10	SSAIØ3-04-SW-W-1BPC	20		30		40	

### VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
Reviewer: 176
2nd Reviewer:

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.				
II. GC/ECD instrument performance check				
Was the instrument performance found to be acceptable?		<u> </u>		
III. Initial calibration				
Did the laboratory perform a 5 point calibration prior to sample analysis?				
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations $(\%RSD) \leq 20\%$ ?	/			
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?				
Did the initial calibration meet the curve fit acceptance criteria?				
Were the RT windows properly established?				
Were the required standard concentrations analyzed in the initial calibration?				<i>'</i>
IV. Continuing calibration				
What type of continuing calibration calculation was performed?%D or%R				
Were Evaluation mix standards analyzed prior to the initial calibration and sample analysis?				
Were endrin and 4,4'-DDT breakdowns ≤ 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			<del></del> ,	
Nas a method blank associated with every sample in this SDG?	1			
Nas a method blank analyzed for each matrix and concentration?	4			
Vere extract cleanup blanks analyzed with every batch requiring clean-up?	4			
Vas there contamination in the method blanks or clean-up blanks? If yes, please see he Blanks validation completeness worksheet.				
/I. Surrogate spikes				
Vere all surrogate %R within the QC limits?				
the percent recovery (%R) of one or more surrogates was outside QC limits, was a eanalysis performed to confirm %R?				
any %R was less than 10 percent, was a reanalysis performed to confirm %R?	$\nearrow$			
II. Matrix spike/Matrix spike duplicates				

LDC#: 74047 E39

### **VALIDATION FINDINGS CHECKLIST**

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?	/	1	<u> </u>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII, Laboratory control samples				
Was an LCS analyzed for this SDG?				
Was an LCS analyzed per extraction batch?			<u> </u>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
X. Target compound identification				
Were the retention times of reported detects within the RT windows?				
XI: Compound quantitation/CRQLs				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions, dry weight factors, and clean-up activities applicable to level IV validation?				
XII. System performance				
System performance was found to be acceptable.				
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.				
KIV. Field duplicates				
Field duplicate pairs were identified in this SDG.				•
Farget compounds were detected in the field duplicates.	/			
⟨V: Field blanks				
field blanks were identified in this SDG.		1		
arget compounds were detected in the field blanks.			1	

## **VALIDATION FINDINGS WORKSHEET**

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

A. alpha-BHC	I. Dieldrin	Q. 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-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	н.
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	.لا
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	LL.
G. Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	EE. 2,4'-DDT	ММ.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	FF. Hexachlorobenzene	NN.

V:\Validation Worksheets\Pesticides\COMPLST-3S.wpd

### LDC# 24047 F39

### VALIDATION FINDINGS WORKSHEET Continuing Calibration

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

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". N 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)?

V N N/A V N N/A Level IV/D Only

N N/A

Did the continuing calibration standards meet the percent difference (%D) / relative percent difference (RPD) criteria of <20.0%? Was at least one standard run daily to verify the working curve?

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

##	Date	Standard ID	Column	Compound	%D (Limit ≤ 20.0)	RT (Limits)	Associated Samples	Qualifications
	0/ 2/3	10617610	267	(A)	23.1	)	) 10-14 17.18	J+ det 4 (c)
			-la	F (F)	23.8	)	~	
				T 8,	20.3	).		
				Ä A)	20.4	)		
				(+) Ø	73.9	)		
						)		
						)		
						)		
		-				)		
						)		
						)		
						)		
						)		
						)		
						)		
						)		
-								
						)		
						·		
						)		
						)		
					,			
						)		
A. alp B. bet C. dell D. gar	A. alpha-BHC B. beta-BHC C. delta-BHC D. gamma-BHC	E. Heptachlor F. Aldrin G. Heptachlor epoxide H. Endosulfan I	I. Dieldrin J. 4,4'-DDE K. Endrin L. Endosulfan II	M. 4,4'-DDD N. Endosulfan sulfate O. 4,4'-DDT P. Methoxychlor	lfate	Q. Endrin ketone U. Toxz R. Endrin aldehyde V. Aroc S. alpha-Chlordane W. Aroc T. gamma-Chlordane X. Aroc	U. Toxaphene Y. Aroclor-1242 V. Aroclor-1016 Z. Aroclor-1248 W. Aroclor-1221 AA. Aroclor-1254 X. Aroclor-1232 BB. Aroclor-1260	CC. DB 608 GG. DD. DB 1701 HH. EE. Hexachlobenzene II.

LDC # 240 47 E34

### VALIDATION FINDINGS WORKSHEET Surrogate Spikes

Page: | of 6 Reviewer.\_ 2nd Reviewer.

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

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

| NAN/A | Were surrogates spiked into all samples, standards and blanks?

limits?
neet the QC lin
; (%R) n
Did all surrogate percent recoveries
urrogate per
Did all s
A/A

#	Date	Sample ID	Column	Surrogate Compound	%R (Limits)	Qualifications	
		2	( d72	4	( per 124) 38E	J+ dets 1x (s) (all except J. B	(I)
			٨		387 ( )		\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \
							· ·
		2(×x)			3\$5 ( )	No gral	
			٨		( ) %5 €		31
					( )		
		3			( ) 619	J+ act (5) (all except JOB #	1
			>		624 ( )		\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \
		3(10x)	1		) 055	No my	•
			7		( ) 125		·
					( )		
		7		8	354000 )	5+400 A 3/18/A (S) (*)	ï•
			λ	4	( 311-63) 0		
			λ	В	5900 (69-124)	^	
					( )		-
		4 (5000x)	•	¥	( SII-65 ) Q	No med	
				^			
			7	80	72360 (63-124)		
_			٦		( ) 50276		
							Ţ

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
٧	Tetrachoro-m-xylene			
В	Decachlorobipheny			

\* gue M, F, S, HH, C, I.H, L, K, R, D, T, E, &

LDC# 240 47 F34

### VALIDATION FINDINGS WORKSHEET Surrogate Spikes

Page:  $\chi$  of  $\zeta$ Reviewer:  $\mathcal{W}$ 2nd Reviewer:\_

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

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

N N/A

Were surrogates spiked into all samples, standards and blanks?

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

ations	( anixcut FF 4)	N 7.0)									(5) (All except 0. FA	P, 4 ) '								
Qualifications	(3) A/M/T			· •		No one					3 /AC/A			•		No sour			_	
%R (Limits)	( 311-65 ) 761	( 1 ) 0	86700 (63-124)	_	( )	D (59-115)	( ) 0	( pc1-69 ) pas61	15 to) ( )	( )	217 (59-115)	( 1 ) 0	31900 (63-124)	1410) ( L')	( )	t ( 54-115 )	( ) 0	10600 (63-124)	4 406 9/10 (	_
Surrogate Compound	A		B			Ą		ß	)	•	Ą		В	,		A	₩	*	æ	•
Column	1 db	4	. *	7		1	7	7	٥			7	1	۸.			7	*	7	
Sample ID	S					( X0025) 5					9					( xorox)				
Date																				
#																				

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A	Tetrachoro-m-xylene			
В	Decachlorobiphenyl			

LDC #: 24047 Fag

### VALIDATION FINDINGS WORKSHEET Surrogate Spikes

Page: 3 of 6 Reviewer: NC 2nd Reviewer:

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

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

Were surrogates spiked into all samples, standards and blanks?

Y/N N/A

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

Surrogate Surrogate %R (Limits) Qualifications	CUP 1 4 22 (59-115) 3 ME/A (2) (all 1xust 0, 1) FF	( 1 ) ( 1 )	(63-124)	1 (1) (0.8c) /	1 A 0 (59-115) De grage		1 B 10400 (63-124)	× ( 1 ) 8480 ( 1 ) ×	1 A 238 (59-115) 3/W/A (5) (all except J. D. N		00 (63-124)	$\overline{}$	1 A B (59-115) No great		1 B 39600 (65-174)	7 ( 1 ) (35/6) 7	
			1 8		1 A	\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	d /	7	A	7	/ B	7	)   A	7 /	٦ م	7	_
Sample ID	7				7 (200x)				&				8 (SNON)				
Date																	
#																	

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A	Tetrachoro-m-xylene			
В	Decachlorobiphenyl			

LDC #: 24047 FIR

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

Page: 4 of 6 Reviewer: 306 2nd Reviewer: 6

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

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

Were surrogates spiked into all samples, standards and blanks?

YNNA

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

								_
#	Date	Sample ID	Column	Surrogate Compound	%R (Limits)		Qualifications	
		5	- dz	В	45151 S124	)	3+ dets/A (s) (all except FF	3
			7		J ) 569	)		<u> </u>
					)	)		
		6 (100x)		Ą	511-65) 0	) No grang	2	
			7	-3	0 9			
			-	В	598 (63-124			
			7		5m (			
				•				
		10		В	4170 (	) J+ dets/A	/A (S) (allexunt 30 BFW)	FFW
			٨		1 088€	_		75.
						(		
		10 (SOK)		¥	511-65) 0	) No mal	2	
			^	->	8			
				В	3450 (63-174			
		And the second s	γ		358U ( )			
						)		
				8	) 826	) J+ dets/A	15/ (s) ( all exust B, 1	(L
			メ		406 (			\
		( xax) 11	-	B	) bre	No gre	7	
	W		$\gamma$		<b>え</b> シゼ	7		
Lett	Letter Designation	Surrogate Compound	Re	Recovery QC Limits (Soil)		mits (Water)	Comments	
	Ą	Tetrachoro-m-xylene						
	В	Decachlorobiphenyl						

LDC #: 24 047 F34

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

Page: 5 of 3 Reviewer: The 2nd Reviewer:\_

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

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

Were surrogates spiked into all samples, standards and blanks?

YNNA

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

	0					8						8	\	_	_	_		#	
Qualifications	I+ Acts/A (2) (all excust FF,		No quel			It dets A (S) (PII except FF.		The state of the s	No guel			] + dot /A (S) (all +xunt FF	\ \ \		No mad	- 2		J+Acts/A (5) GA! MANT J,O,	
%R (Limits)	259 (63-124)	( )	238 ( )	239 ( )	( ) )	553 ( )	568 (   )		486 ( )	( ) 405	( )	64v ( )	( ) 0,09	( )	583	594 ( // )	( )	265 2170 ( 59-115)	255 Zeg ( )
Surrogate Compound	4		в	1		В	1		B	<b>→</b>		8	1		4		>	∢	<b>^</b>
Column	CUP 1	^	1	<u> </u>		(	\ \			λ			ン			7			7
Sample ID		editeriological desired in the control of the contr	( xo. 3) ~1			8			12 (2%)			4			14 (26x)	,		15	
Date																			
#																			

Comments		
Recovery QC Limits (Water)		
Recovery QC Limits (Soil)		
Surrogate Compound	Tetrachoro-m-xylene	Decachlorobiphenyl
Letter Designation	A	æ

LDC # 24047 F34

### VALIDATION FINDINGS WORKSHEET Surrogate Spikes

Page: 6 of 6 Reviewer: The 2nd Reviewer:\_

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

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

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

	(H	\						(#	\			 						
Qualifications	(all except JOA		No mul			*		J+ 10ts/A (S) (4) (44 (44) M, J,0, B, FF)	ر			No gue			•			
%R (Limits)	2170 (62,124)	3040 ( 7 )	( )11-65 ) 84	49 ( )	1200 (62-124)	( ) () to	( )	( 511-163) 651	1160 ( 1)	( pc1-69) oocc1	32400 ( 1)	14) (59-115)	( 1 ) (2)	15300 (63-124)	(1) 641	( )	( )	( )
Surrogate Compound	В		¥	_	4	1		Ą	<b>\</b>	45		A		В				
Column	CIP 1	\ \	_	٨		7			٨		\ <u>\</u>		γ		χ			
Sample ID	3/	AND THE PROPERTY OF THE PROPER	15 (MX)					91				( ×00×)						
Date																		
#																		

Comments		
Recovery QC Limits (Water)		
Recovery QC Limits (Soil)		
Surrogate Compound	Tetrachoro-m-xylene	Decachlorobiphenyl
Letter Designation	¥	В

LDC#: 240 47 E29 SDG#:\_

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

2nd Reviewer: 4 Page: Reviewer:

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". N N N

Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? Was a MS/MSD analyzed every 20 samples for each matrix or whenever a sample extraction was performed?

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

#	Date	OIOSW/SW	panoamo		MS */P /! imite)	MSD W (1 imite)	771-17 000			
		81/ 41	Sereck	H	Compound	MATTER A.		1	Associated Samples	Ala en a l
			1.2.14	ł	25	and 2 RPD	99	1		City. MC MC
					( )	1				( A. C. M. O. V. )
					)			î		
						( )	,	,		
								<u> </u>		
					(			<u> </u>		
					( )		)	<u></u>		
					( )	)	_	^		
					)	( )		-		
					( )	( )	)	-		
					( )	)	)	Î		
					)			<u> </u>		
					( )			^		
					J	(	)	^		
					)		)	-		
					)		)	Î,		
					)			-		
					(	(	)	î		
					( )		)	(		
					( )	( )	·	Î î		
					( )	( )	<u> </u>	<u> </u>		
					( )	( )	_	î		
					( )	( )	)	^		
					( )	( )	)	_		
					( )	( )		^		

LDC# 24047 ERG SDG #: See Correy

### Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

Reviewer: JVC 2nd Reviewer: 1 Page: 1 of 1

METHOD: /GC HPLC

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

Level IV/D Only Y N NA Y N NA

Were CRQLs adjusted for sample dilutions, dry weight factors, etc.?

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

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

If no. please see findings bellow.

	יייטייט פעיייטייט טכט אינייטייט, אינייטיט פעיייטייט			
*	Compound Name	Sample ID	(%RPD)%D Between Two Columns/Detectors Limit (≤ 40%)	Qualifications
	, A	[3	69, 3	J dets/A (dc)
	S	1	142,2	,

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24047E3a

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

<u>'</u> of <u>)</u>
NG

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

Y N NA

Were field duplicate pairs identified in this SDG?

Were target analytes detected in the field duplicate pairs?

Compound Name	Conc (	ug/Kg)	RPD	Diff	Diff Limits	Quals
Compodita Name	7	6	(≤50%)			(Parent Only)
4,4'-DDE	22	27	20			
alpha-BHC	16	17	6			
Endosulfan sulfate	3400U	33		3367	≤3400	
Hexachlorobenzene	36000	42000	15			

Compound Name	Conc ( u	ıg/Kg)	RPD	Diff	Diff Limits	Quals
Compound Name	1/25	6/14	(≤50%)	<b>~</b>		(Parent Only)
4,4'-DDE	17	18	6			
4,4'-DDT	6.0	8.1		2.1	≤1.7	Jdets/A (fd)
alpha-BHC	0.86	0.62		0.24	≤1.7	
delta-BHC	1.7	1.8		0.1	≤1.7	
Endosulfan sulfate	1.7U	0.38		1.32	≤1.7	
beta-BHC	290	260	11			
Hexachlorobenzene	260	230	12			

V:\FIELD DUPLICATES\24047E3a.wpd

LDC # 240 47 E34

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

GC EPA SW 846 Method 8081A METHOD:

Hexachlorobenzene Parameter:

Date	Column	Compound	Y Area	Conc	X^2
08/11/2010	CLP1	Hexachlorobenzene	35416.00	4.00	16.00
			79982.00	10.00	100.00
	GCS_P1		186328.00	25.00	625.00
			366503.00	50.00	2500.00
		-	532247.00	75.00	5625.00
			700881.00	100.00	10000.00

			i i	
Regression Output:			Reported	
Constant		6214.81624	II O	NR
Std Err of Y Est		2282.53420		
R Squared		96666.0	12 =	1.000000
No. of Observations		00000'9		
Degrees of Freedom		3.00000		
			a=	N. R.
X Coefficient(s)	7365.983258	-4.269664	= q	NR
Std Err of Coef.	109.089622	1.05		

7097

Ave RF

7623

Reviewer: 小

Page: 1 of 4

7998 7453 7330

LDC # 740 47 E34

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page:  $\frac{1}{2}$  of  $\frac{4}{3\sqrt{2}}$ Reviewer:  $\frac{1}{3\sqrt{2}}$ Reviewer: 3

METHOD:

GC EPA SW 846 Method 8081A

Parameter:

Hexachlorobenzene

X"Z				17.7/2/2014		A SAME AND		
>	Conc	4.00	10.00	25.00	50.00	75.00	100.00	
×	Area	38101.00	87056.00	206854.00	408434.00	593608.00	783179.00	
-	Compound	Hexachlorobenzene						
	Column	CLP		GCS_P1				
	Date	08/11/2010						

Regression Output:	out:		Reported	
Constant		000000	II O	0.0000
Std Err of Y Est		9097.68589		
R Squared		0.99905	r2 =	0.999700
No. of Observations		000000		
Degrees of Freedom		2.00000		
		-		
X Coefficient(s)	7921.897276	-1.270906	= q	7928,000000
Std Err of Coef.	66.235531	0.79		

7831.79

Ave RF

8274.16 8168.68 7914.77

9525.25 8705.60 8403.38

LDC# 240 47 E34

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Reviewer: 1/2 2nd Reviewer:

Page: h of

GC EPA SW 846 Method 8081A METHOD:

Parameter:

4,4'-DDT

	100.00	508/46.00			
	75.00	384225.00			
	50.00	260822.00			
	25.00	129507.00		GCS_P1	
	10.00	54935.00			
	4.00	23760.00	4,4'-DDT	CLP1	08/11/2010
	Conc	Area	Compound	Column	Date
X^2	Å	×			

Regression Output:			Reported	
Constant		000000	<b>≡</b> 0	0.00000
Std Err of Y Est		3488.48800		
R Squared		79666.0	12 =	006666.0
No. of Observations		000000'9		
Degrees of Freedom		5.00000		
X Coefficient(s)	5121.098272	-1.270906	= Q	5090,000000
Std Err of Coef.	25.397871	62'0		

5180.28 5216.44 5123.00 5087.46

5940.00 5493.50

Ave RF

5340.11

LDC #: 1047 634 SDG#:

VALIDATION FINDINGS WORKSHEET

Initial Calibration Calculation Verification

ō Page:

Reviewer: 2nd Reviewer:

METHOD: GC Pesticides (EPA SW 846 Method 8081)

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

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

Where

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

 $\begin{aligned} A_x &= \text{Area of Compound} \\ C_x &= \text{Concentration of compound}, \end{aligned}$ 

X = Mean of the RRFs

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

S= Standard deviation of the RRFs,

					Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
		Calibration					Average CF	Average CF	%RSD	%RSD
#	Standard ID	Date	Š	Compound	(100 std)	(100 std)	(Initial)	(Initial)		
_	ICAL	8/11/2010 4,4'-DDT		(CLP2)	5271	5271	5475	5475	4.8	4.779
	GCS P1									
2										

Response cpd	527096			
Conc	100			
Compound	ddt			

ddt	5948	5611	5336	5386	5298	5271	5475	262	
Conc	4	10	25	20	75	100	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: of

> / HPLC METHOD: GC\_

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

Calibration Factor from Continuing Calibration Standard or Calculated Amount Initial Calibration Factor or Nominal Amount || || || U Where: Percent difference (%D) = 100 \* (N - C)/N

Recalculated	۵%		0.3	8.0	5.6	6.2	1.2	9.7	8.6	17.0						
Reported	O %		0.3	9'9	3.9	6.2	1.2	8.4	8.2	17.0						
Recalculated	Conc		50.1	54.0	52.8	53.1	50.6	54.9	6.42	58.5						
Reported	Conc		50.1	53.3	52.0	53.1	50.6	54.2	54.1	58.5						
	CCV Conc		92	90	20	50	50	90	90	50						
		und	CLP1	CLP1	CLP2	CLP2	CLP1	CLP1	CLP2	CLP2						
		Compound	НСВ	DDT	HCB	DDT	HCB	DDT	HCB	TOO						
	Calibration	Date	8/26/2010	10:34			8/26/2010	4:00	14:35							
		Standard ID	005F0501				019F1901								-	
		#	1				2				ო			4	•	

5000	435408		
2090	279289		
5475 290810	320384		

50.1297378 1675,06064 50,58519 1674,60519 (-b-() // 2a (-b+ ())/2a 48134581.08 6937.90898 48080629.6 6934.01973 ( )^ 1/2 Calculation (b^2 - 4aT) -358525.1838 -361684.1838 7 = Y-c final conc Conc. 50.130 6214.816 6214.816 ပ 7365.983 7365.983  $Y = a(X^2) + bX + c$ 4.270 367899 364740 Area Y CCV1 HCB CLP1 CCV2 HCB CLP1

LDC #: 240 47 F31

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

Page:	of
Reviewer:	276
2nd reviewer:	T/A

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	,
Tetrachloro-m-xylene	CLP 1	20	16.5	8~	8~	0
Tetrachloro-m-xylene	1 7	`	17,0	85	85	
Decachlorobiphenyl			17.9	Iou	10)	
Decachlorobiphenyl	1 7 7	X	70.7	104	164	

Sample ID:\_\_\_\_

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

Sample ID:

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
<u> </u>				Reported	Recalculated	
Tetrachloro-m-xylene				-		
Tetrachioro-m-xylene			7			
Decachlorobiphenyl						
Decachlorobiphenyl						

Sample ID:

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

Notes:	

LDC#: 240 47 E 34

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

2nd Reviewer: Page: of / Reviewer: 376

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

The percent recoveries (%R) and Relative Percent difference (RPD) of the matrix spike and matrix spike duplicate were recalculation:

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

SSC = Spiked sample concentration SA = Spike added Where:

SC = Concentration

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

MSD = Matrix spike duplicate percent recovery

MS/MSD sa

MS = Matrix spike percent recovery

Bed avide vineral o		
, ,	\\ \ \	
	samples:	

rai cua	Spike Added	Sample Concentration	Spiked Sample Concentration	Matrix Spike	ike	Matrix Spil	Matrix Spike Duplicate		Welker
Dipodiio	<b>1</b> 1	(4/54)		Percent Recovery	overy	Percent	Percent Recovery		SMISD
	MS MSD	<i>&gt;</i>		L					KPD
gamma-BHC			OSM SIII	Reported	Recalc.	Reported	Recalc.	Reported	Population
	16.4	0	21.8 10.1			, ;			Necalculated
4,4'-DDT		1 6		120	133	/٥٧	101	18	7
A 1 4 000		3.5	31.5 32.1	2	5	112			9
Arocior 1260				+			2	۲	7
					Ī				
				-					
					<del></del> ,				

Comments: Refer ot Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within

MSDCLC.3S

### LDC#: 740 47 F31

# VALIDATION FINDINGS WORKSHEET

lof 1 Reviewer: J1/2

Page:

2nd Reviewer:

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

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

SSC = Spiked sample concentration SA = Spike added Where:

SC = Concentration

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

LCS/LCSD samples:

LCS = Laboratory control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery 2-80-27469 /2-4

	Spike	0.11.0						
Compound	Added	Spiked Sample Concentration	TCS	Ş	רכ	TCSD	SOT	LCS/I CSD
	( 3//()	(45/6, )	Percent Recovery	BCDVerv				
	rcs 6	2 33			Percent	Percent Recovery	R	RPD
gamma-BHC		CSD CONTROL	Reported	Recalc.	Reported	Recolc.	Panortod	
	16, 7 NA	12,9	2	ક			parodesi	Recalc.
4,4'-DDT				80				,
Aroclor 1260		7 7 7	SK	ملا				
					j			
				_	_		_	

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicate findings worksheet for list of qualifications and associated samples when reported

LDC #: 24047 F 39

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:		
Reviewer:	JVG	
d reviewer	1 4	

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

$(Y)_N$	N/A
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?

Example:

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
			·		
		-			
					<del></del>

Note:	

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 17, 2010

LDC Report Date:

October 11, 2010

Matrix:

Soil

Parameters:

Chlorinated Pesticides

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6583-1

### Sample Identification

BDT-4-N-15-16.0BPC

BDT-4-N-15-18.0BPC\*\*

BDT-4-N-15-12.0BPC

BDT-4-N-15-14.0BPC

BDT-4-N-15-8.0BPC

BDT-4-N-15-10.0BPC

BDT-4-N-20-10.0BPC

BDT-4-N-20-12.0BPC

BDT-4-N-20-14.0BPC

BDT-4-N-20-16.0BPC

BDT-4-N-20-18.0BPC\*\*

BDT-4-N-20-2.0BPC

BDT-4-N-20-4.0BPC

BDT-4-N-20-6.0BPC

BDT-4-N-20-8.0BPC

BDT-4-N-20-6.0BPC FD

BDT-4-N-15-16.0BPCMS

BDT-4-N-15-16.0BPCMSD

BDT-4-N-15-18.0BPCMS

BDT-4-N-15-18.0BPCMSD

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

### Introduction

This data review covers 20 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Samples indicated by a double asterisk on the front cover underwent a 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.

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.

### VI. Surrogate Spikes

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

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Pesticide Cleanup Checks

### a. Florisil Cartridge Check

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

### b. GPC Calibration

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

### XI. Target Compound Identification

All target compound identifications were within validation criteria for samples on which a 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 280-6583-1	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-20-6.0BPC and BDT-4-N-20-6.0BPC\_FD were identified as field duplicates. No chlorinated pesticides were detected in any of the samples with the following exceptions:

	Concentra	ition (ug/Kg)	555	D		
Compound	BDT-4-N-20-6.0BPC	BDT-4-N-20-6.0BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
4,4'-DDE	0.78	1.8U	-	1.02 (≤1.8)	-	-
Hexachiorobenzene	0.46	1.8U	-	1.34 (≤1.8)	-	-

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Data Qualification Summary - SDG 280-6583-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6583-1	BDT-4-N-15-16.0BPC BDT-4-N-15-18.0BPC** BDT-4-N-15-12.0BPC BDT-4-N-15-14.0BPC BDT-4-N-15-8.0BPC BDT-4-N-15-10.0BPC BDT-4-N-20-10.0BPC BDT-4-N-20-12.0BPC BDT-4-N-20-14.0BPC BDT-4-N-20-16.0BPC BDT-4-N-20-18.0BPC** BDT-4-N-20-18.0BPC** BDT-4-N-20-6.0BPC BDT-4-N-20-6.0BPC BDT-4-N-20-6.0BPC BDT-4-N-20-6.0BPC BDT-4-N-20-6.0BPC BDT-4-N-20-6.0BPC BDT-4-N-20-6.0BPC	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 280-6583-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG 280-6583-1

No Sample Data Qualified in this SDG

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

•	•	•	•	•	_	•	•	•	_	••	_		•		••	•
										St	a	qε	9	2	В	:/

Date:	16/06/20
Page:_	1 of <i>)</i>
Reviewer:	JV4
2nd Reviewer:	

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 8 /17 /10
11.	GC/ECD Instrument Performance Check	A	
111.	Initial calibration	À	2 ESD = 20 2 rd  Car/icr = 20 2
IV.	Continuing calibration/ICV	K	ca/10 = 20 Z
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	us
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	SW	b = 14.16
XV.	Field blanks	NB	FB = FB - 04122010- R1 52 - RZE

Note:

LDC #: 24047G3a SDG #: 280-6583-1 Laboratory: Test America

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

AU Smils									
1	BDT-4-N-15-16.0BPC	11	BDT-4-N-20-18.0BPC**	- 21	MB 280 - 28878/1-A	31			
<b>}</b> 2	BDT-4-N-15-18.0BPC**	12	BDT-4-N-20-2.0BPC	22	,	32			
3	BDT-4-N-15-12.0BPC	13	BDT-4-N-20-4.0BPC	23		33			
4	BDT-4-N-15-14.0BPC	† 14	BDT-4-N-20-6.0BPC <i>b</i>	24		34			
5	BDT-4-N-15-8.0BPC	15	BDT-4-N-20-8.0BPC	25		35			
6	BDT-4-N-15-10.0BPC	- 16	BDT-4-N-20-6.0BPC_FD D	26		36			
7	BDT-4-N-20-10.0BPC	17	BDT-4-N-15-16.0BPCMS	27		37			
8	BDT-4-N-20-12.0BPC	18	BDT-4-N-15-16.0BPCMSD	28		38			
9	BDT-4-N-20-14.0BPC	19	BDT-4-N-15-18.0BPCMS	29		39			
10	BDT-4-N-20-16.0BPC	20	BDT-4-N-15-18.0BPCMSD	30		40			

### VALIDATION FINDINGS CHECKLIST

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.				
II. GC/ECD Instrument performance check				
Was the instrument performance found to be acceptable?				
III: Initial calibration				
Did the laboratory perform a 5 point calibration prior to sample analysis?				
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) ≤ 20%?				
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?				
Did the initial calibration meet the curve fit acceptance criteria?				
Were the RT windows properly established?				
Were the required standard concentrations analyzed in the initial calibration?	~			
IV. Continuing calibration				
What type of continuing calibration calculation was performed?%D or%R	_			
Were Evaluation mix standards analyzed prior to the initial calibration and sample analysis?		1		
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	•			
Nas a method blank associated with every sample in this SDG?	4			
Nas a method blank analyzed for each matrix and concentration?	~			
Nere extract cleanup blanks analyzed with every batch requiring clean-up?				
Nas there contamination in the method blanks or clean-up blanks? If yes, please see he Blanks validation completeness worksheet.		1		
/f. Stirrogale spikes				
Vere all surrogate %R within the QC limits?	1			
f the percent recovery (%R) of one or more surrogates was outside QC limits, was a eanalysis performed to confirm %R?			1	
f any %R was less than 10 percent, was a reanalysis performed to confirm %R?			_1	
/II. Matrix spike/Matrix spike duplicates				

### VALIDATION FINDINGS CHECKLIST

Page: > of 2
Reviewer: 17//
2nd Reviewer: \_\_\_\_

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?	/			
VIII. Laboratory control samples				
Was an LCS analyzed for this SDG?				
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
X. Target compound identification				
Were the retention times of reported detects within the RT windows?				
XI Compound 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.				
XV Field blanks				
Field blanks were identified in this SDG.				
Target compounds were detected in the field blanks.				

LDC#: 24047G3a

### VALIDATION FINDINGS WORKSHEET Field Duplicates

Page	:of/
Reviewer:	D/
2nd Reviewer:	المنتخب

M	ETHOD:	GC Chlorinated Pesticides (EPA SW 846 Method 8081A)
<u>Y</u>	N NA	Were field duplicate pairs identified in this SDG?
Y	N NA N NA	Were target analytes detected in the field duplicate pairs?

Compound Name	Conc	( ug/Kg)	RPD	Diff	Diff Limits	Quals
Compound Name	14	16	(≤50%)	DIII .		(Parent Only)
4,4'-DDE	0.78	1.8U		1.02	≤1.8	
Hexachlorobenzene	0.46	1.8Ų		1.34	≤1.8	

V:\FIELD DUPLICATES\24047G3a.wpd

LDC # 24 047 520

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Reviewer: 34

of C

Page:

GC EPA SW 846 Method 8081A METHOD:

b-BHC

Parameter:

		- 1	- 1				
Y	4.00	10.00	25.00	50.00	75.00	100.00	
X Area	23110.00	52056.00	124514.00	245293.00	366609.00	479885.00	
Compound	P-BHC				,		
Column	CLP1		GCS_P1				
Date	08/11/2010						

Regression Output:			Reported	
Constant		0.0000	= 0	0.0000
Std Err of Y Est		3979.11102		
R Squared		0.99952	12 =	1.000000
No. of Observations		6.00000		
Degrees of Freedom		5.00000		
-				
X Coefficient(s)	4848.652338	-1.270906	= q	4813.000000
Std Err of Coef.	28.969843	0.79		

5205.60 4980.56 4905.86 4798.85 4888.12

5777.50

X^2

Ave RF

5092.75

### LDC # 240 47 624

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: 7 of 4
Reviewer: 3VC
2nd Reviewer: 6

GC EPA SW 846 Method 8081A METHOD:

Hexachlorobenzene Parameter:

	100.00	953705.00			
	75.00	730674.00			
	50.00	490208.00			
	25.00	249072.00		GCS_P2	
	10.00	103588.00			
	4.00	44827.00	Hexachlorobenzene	CLP1	08/11/2010
	Conc	Area	Compound	Column	Date
Z <sub>v</sub> X	×	Y			

Regression Output:			Reported	
Constant		00000'0	II 0	0.00000
Std Err of Y Est		8773.78312		
R Squared		0.99941	12=	0.999900
No. of Observations		000000'9		
Degrees of Freedom		5.00000		
X Coefficient(s)	9653.526874	-1.270906	= Q	9638.000000
Std Err of Coef.	63.877363	62.0		

10358.80 9962.88 9804.16 9537.05 9742.32

Ave RF

11206.75

10101.99

LDC# 24047 636

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

Reviewer: The 2nd Reviewer:

Page: 3\_ of\_

GC EPA SW 846 Method 8081A METHOD:

b-BHC Parameter:

			<b>\</b>	×	X^2
Date	Column	Compound	Area	Conc	
08/11/2010	CLP2	p-BHC	46113.00	4.00	16.00
			103650.00	10.00	100.00
	GCS_P2		239958.00	25.00	625.00
			450061.00	50.00	2500.00
			648617.00	75.00	5625.00
			826471.00	100.00	10000.00

Regression Output:	ıt:		Reported	
Constant		9066.02542	= 3	NR R
Std Err of Y Est		1421.92497		
R Squared		0.99999	12=	1.000000
No. of Observations		0000009		
Degrees of Freedom		3.00000		
			a II	NR R
X Coefficient(s)	9529.795818	-13.537170	= q	NR
Std Err of Coef.	67.958350	0.65		

Ave RF

11528

9598

10365

9001

8648

8265

9568

### LDC # 24047 632

## VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

METHOD: GC EPA SW 846 Method 8081A

Parameter: Hexachlorobenzene

Compound

Regression Output:	ند		Reported	
Constant		20708.90229	= 0	NR
Std Err of Y Est		3835.69679		
R Squared		86666.0	12 =	0.999990
No. of Observations		6.00000		THE PROPOSOLATION OF THE PROPO
Degrees of Freedom		3.00000		
			n O	NR.
X Coefficient(s)	19034.788783	-29.504222	= q	NR
Std Err of Coef.	183.320239	1.76	CONTROL OF THE PARTY AND THE P	The state of the s

21051 19251 17893 17121 16290

23334

Ave RF 19156

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100 # 240 pt 639

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

Page: | of | 

METHOD: GC\_

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

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

Initial Calibration Factor or Nominal Amount

Calibration Factor from Continuing Calibration Standard or Calculated Amount Where:

Recalculated %D		4.7	11.1	2.0	3.8	3.2	0.6	4.0	1.0					
Reported % D		3.4	9.5	2.1	3.8	1.8	7.4	4.0	1.0		-			
Recalculated Conc		52.4	9299	49.0	51.9	51.6	54.5	48.0	50.5					
Reported		51.7	54.8	49.0	51.9	50.9	53.7	48.0	50.5					
CCV Conc		50	20	90	90	50	50	50	90					
	pu	CLP1	CLP1	CLP2	CLP2	CLP1	CLP1	CLP2	CLP2					
	Compound	HCB	b-BHC	HCB	b-BHC	HCB	p-BHC	HCB	b-BHC					
Calibration	Date		23:53			9/2/2010								
	Standard ID	043F4301				058F5801								
	#	_				2				 8			4	

		CCV1	CCV2	CCV3	CCV4	CCV5
	Slope	Area	Area	Area		
HCB CLP1	9638	504640	497245			
P-BHC CLP1	4813	267482	262265			

					(-b-())/2a	596.183358	652.08737	597.142481	653,469779
				(-b+ ())/2a		48.9762477	51.8851601	48.017124	50,5027508
					( )^ 1/2	16144.7986 48.9762477	8125.03935	62485185.2 16201.3945 48.017124 597.142481	8162.46717
			Calculation		(b^2 - 4aT) ( )^ 1/2	260654521	66016264.49 8125.03935 51.8851601	262485185.2	66625870.32
-		a			T = Y-c	-861482.098	458011.9746	-845970.098	446753.9746
					final conc				
				Conc.	×	48.976	51.885	48.017	50,503
					O	20708.902	9066,025	20708.902	9066.025
497245	262265		O.		q	19034.789	9529.796	19034.789	9529.796
504640	267482		$Y = a(X^2) + bX + c$			-29.504	-13.537	-29.504	-13.537
, 9638	4813			Area		882191	467078	866679	455820
HCB CLP1	b-BHC CLP1	Terrerore de la company de la		⋖	<b>&gt;</b>	CCV1 HCB CLP2	CCV1 b-BHC 2	CCV2 HCB CLP2	CCV2 b-BHC 2

LDC#: 24047 639

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

Page:	<u> </u>
Reviewer:_	JV6
2nd reviewer:	un

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

The percent recoveries	(%R) of surrogate	s were recalculated for the compo	unds identified below using t	ne following calculation:
------------------------	-------------------	-----------------------------------	-------------------------------	---------------------------

% Recovery: SF/SS \* 100

Sample ID:\_\_\_

女

Where: SF = Surrogate Found SS = Surrogate Spiked

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene	CLPI	20	16.7	83	83	6
Tetrachioro-m-xylene			15.~	76	76	
Decachlorobiphenyl	1		21.1	105	110	
Decachlorobiphenyl	1 1	. }	18.6	92	93	

Sample ID:

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

Sample ID:

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

Sample ID:

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

Notes:	
_	

639 LDC# 24047

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

Page: [ of ] Reviewer: 376 

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

The percent recoveries (%R) and Relative Percent difference (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below

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

SSC = Spiked sample concentration SA = Spike added Where;

SC = Concentration

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

MS/MSD samples:

MS = Matrix spike percent recovery

MSD = Matrix spike duplicate percent recovery

Compound	A Ad	Spike Added	Sample Concentration	Spiked Sample Concentration	ء و	Matrix Spike	Spike	Matrix Spil	Matrix Spike Duplicate	W	MS/MSD
	3		W5/K)	(45 hc)		Percent Recovery	ecovery	Percent	Percent Recovery		Vaa
	MS	MSD	2	MS M	OMSD	1 1					וויי
gamma-BHC	ر م	7 0 1				Neported	Kecalc.	Reported	Recaic.	Reported	Recalculated
	2.5	7 7	0	17.9 7.0	6	× 5	8 &	4	7 6	į	8
4,4'-DDT	_			$\vdash$	/ P /	,			`	7	S
Aroclor 1260		0		<u>.</u>		89	80	77	77	^	
				•						١	4
		ž			1						
					I						
					1						
					+						
					1						
					**						
					+						

Comments: Refer ot Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within

LDC#: 24047 629

# VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicate Results Verification

Page: lof / Reviewer: J1/2

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

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

SSC = Spiked sample concentration SA = Spike added Where:

SC = Concentration

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

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

LCS/LCSD samples:\_

28878 280ž

	rcs/rcsD	uad		Reported Recalc										
CSD		Recovery		Recalc. R	╠	_								
		Percent Recovery		Reported				Ì						
rcs		Percent Recovery		Aecaic.	V V		80							
		Percent	Reported		\display \text{\ti}\text{\texi{\text{\texi{\text{\ti}}\\ \text{\ti}\text{\text{\text{\text{\text{\text{\text{\text{\text{\text{\ti}\text{\text{\text{\text{\text{\text{\texi}\tilit{\tiint{\text{\text{\text{\text{\text{\text{\texi}\tint{\text{\tii}}\t		م 4.×							
Spiked Sample			CSD		5		2						·	
Spike	3		rcs	1.5	4.3	Č								
spike Addęd	5/kg)	0	rcsp	- 5	XX									
	2	32	3	<u> </u>		_								
	punodwoo			gamma-BHC		4,4'-DDT	Aroclar 1260							

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicate findings worksheet for list of qualifications and associated samples when reported

V:/Validation Worksheets/Pesticides/LCSDCLC.wpd

LDC #: 24047 634

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	<u>l_of_1</u>
Reviewer:_	NG
2nd reviewer:_	1~

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

_	$\overline{}$		4
	Υ)	N	N/A
		N	N/A
``	7		

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. 
$$\# 2$$
  $\%$   
Conc. =  $(26328 - 3877)$   
 $(4813)$   
= 4.665  
Final eme. =  $(4,665)(16 \text{ ml})$   
 $(750.39)(0.901)$   
= 1.70 ug  $\%$ 

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

Note: (Intercept values and provided in the summaries)

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

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 24, 2010

LDC Report Date:

October 11, 2010

Matrix:

Soil/Water

Parameters:

Chlorinated Pesticides

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6783-1

### Sample Identification

BDT-4-S-5-10BPCMS EB-08242010 BDT-4-S-5-10BPC BDT-4-S-5-10BPCMSD BDT-4-S-5-12BPC BDT-4-N-5-2BPCMS BDT-4-S-5-14BPC\*\* BDT-4-N-5-2BPCMSD BDT-4-S-5-16BPC BDT-4-N-5-4BPCMS BDT-4-S-5-18BPC BDT-4-N-5-4BPCMSD BDT-4-S-5-2BPC BDT-4-N-5-6BPCMS BDT-4-S-5-4BPC BDT-4-N-5-6BPCMSD BDT-4-S-5-6BPC BDT-4-S-5-8BPC BDT-4-S-5-16BPC FD BDT-4-N-5-10BPC BDT-4-N-5-12BPC BDT-4-N-5-14BPC BDT-4-N-5-16BPC BDT-4-N-5-18BPC\*\* BDT-4-N-5-2BPC BDT-4-N-5-4BPC

BDT-4-N-5-6BPC BDT-4-N-5-8BPC

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

### Introduction

This data review covers 27 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Samples indicated by a double asterisk on the front cover underwent a 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.
- 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.

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-08242010 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-5-2BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	153 (63-124) 151 (63-124)	All TCL compounds except 4,4'-DDE 4,4'-DDT	J+ (all detects)	А
BDT-4-S-5-4BPC	CLP1	Decachlorobiphenyl	261 (63-124)	All TCL compounds except 4,4'-DDE 4,4'-DDT	J+ (all detects)	А
BDT-4-N-5-2BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	202 (63-124) 206 (63-124)	All TCL compounds except 4,4'-DDE	J+ (all detects)	А
BDT-4-N-5-2BPC (2X)	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	198 (63-124) 206 (63-124)	4,4'-DDE	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. Although the MS/MSD percent recoveries (%R) and relative percent difference (RPD) were not within QC limits for some compounds, the LCS percent recoveries (%R) were within QC limits and no data were qualified.

### VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Percent recoveries (%R) 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
LCS280-29004/5-A (EB-08242010)	Toxaphene	128 (63-118)	138 (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
BDT-4-S-5-14BPC**	4,4'-DDT	40.6 (≤40)	J (all detects)	Α
BDT-4-N-5-18BPC**	beta-BHC	97.8 (≤40)	J (all detects)	Α

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6783-1	All compounds reported below the PQL.	J (all detects)	A

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-5-16BPC and BDT-4-S-5-16BPC\_FD were identified as field duplicates. No chlorinated pesticides were detected in any of the samples with the following exceptions:

	Concentra	Concentration (ug/Kg)					
Compound	BDT-4-S-5-16BPC	BDT-4-S-5-16BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P	
4,4'-DDE	100	110	10 (≤50)	-	<u>-</u> .	-	
4,4'-DDT	24	16	40 (≤50)	-	_	-	
beta-BHC	1.3	1.6	<b>-</b>	0.3 (≤1.8)	-	-	
Hexachiorobenzene	2.5	2.6	•	0.1 (≤1.8)	-	-	

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Data Qualification Summary - SDG 280-6783-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6783-1	BDT-4-S-5-2BPC BDT-4-S-5-4BPC	All TCL compounds except 4,4'-DDE 4,4'-DDT	J+ (all detects)	A	Surrogate recovery (%R) (s)
280-6783-1	BDT-4-N-5-2BPC	All TCL compounds except 4,4'-DDE	J+ (all detects)	А	Surrogate recovery (%R) (s)
280-6783-1	BDT-4-N-5-2BPC (2X)	4,4'-DDE	J+ (all detects)	A	Surrogate recovery (%R) (s)
280-6783-1	EB-08242010	Toxaphene	J+ (all detects)	Р	Laboratory control samples (%R) (I)
280-6783-1	BDT-4-S-5-14BPC**	4,4'-DDT	J (all detects)	А	Compound quantitation and CRQLs (RPD) (dc)
280-6783-1	BDT-4-N-5-18BPC**	beta-BHC	J (all detects)	А	Compound quantitation and CRQLs (RPD) (dc)
280-6783-1	EB-08242010 BDT-4-S-5-10BPC BDT-4-S-5-12BPC BDT-4-S-5-14BPC** BDT-4-S-5-16BPC BDT-4-S-5-18BPC BDT-4-S-5-2BPC BDT-4-S-5-4BPC BDT-4-S-5-6BPC BDT-4-S-5-16BPC_FD BDT-4-N-5-10BPC BDT-4-N-5-12BPC BDT-4-N-5-14BPC BDT-4-N-5-16BPC	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 280-6783-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Equipment Blank Data Qualification Summary - SDG 280-6783-1

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

SDG #: 280-6783-1	Stage 2B
Laboratory: Test America	J

Page: 10/66/ho
Page: 1 of 1
Reviewer: 34/
2nd Reviewer: 1

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
<u>l.</u>	Technical holding times	A	Sampling dates: 8/24 /10
11.	GC/ECD Instrument Performance Check	Α	
III.	Initial calibration	A	14 RSD = 20 2 12
IV.	Continuing calibration/ICV	A	14 RSD = 20 2 17 COV/101 = 20 2
V.	Blanks	A	
VI.	Surrogate spikes	SN)	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	SW	LCS /p
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	Ā	
XIV.	Field duplicates	CM	D = 5, 11
XV.	Field blanks	M	EB = 1 FB = FB - 04/32010 = RIG 2 - RZE

Note:

A = Acceptable

LDC #: 24047K3a

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

vanua	teu Samples. Indica	res saut		erwerit Stage 4 validation							
	NA	1er	<u> + \</u>	Soil							_
1	EB-08242010	W	11 /	BDT-4-S-5-16BPC_FD \$	21	BDT-4-S-5-10BPCMS S	31	MB	280-2	9748/1-4	
2	BDT-4-S-5-10BPC	S	12	BDT-4-N-5-10BPC	22	BDT-4-S-5-10BPCMSD	32	MB			<b>A</b>
3 1	BDT-4-S-5-12BPC		13	BDT-4-N-5-12BPC	23	BDT-4-N-5-2BPCMS	33 3	MB	780-	29004 /1-	A
4 1	BDT-4-S-5-14BPC**		14	BDT-4-N-5-14BPC	24	BDT-4-N-5-2BPCMSD	34				
5 1	BDT-4-S-5-16BPC	p	15	BDT-4-N-5-16BPC	25	BDT-4-N-5-4BPCMS	35				
6 1	BDT-4-S-5-18BPC		16	BDT-4-N-5-18BPC**	26	BDT-4-N-5-4BPCMSD	36				
7 1	BDT-4-S-5-2BPC		17	BDT-4-N-5-2BPC	27	BDT-4-N-5-6BPCMS	37				
8	BDT-4-S-5-4BPC		18 <b>7</b>	BDT-4-N-5-4BPC	28	8 BDT-4-N-5-6BPCMSD	38				
9 1	BDT-4-S-5-6BPC		19 7	BDT-4-N-5-6BPC	29	9	39				
10 1	BDT-4-S-5-8BPC	<u> </u>	20	BDT-4-N-5-8BPC	30	0	40				

### VALIDATION FINDINGS CHECKLIST

Page: 1 of 2 Reviewer: JVC 2nd Reviewer: 1

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.	_			
II. GC/ECD instrument performance check				Γ
Was the instrument performance found to be acceptable?				
III. Initial calibration	1		г	
Did the laboratory perform a 5 point calibration prior to sample analysis?				
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations $(\%RSD) \le 20\%$ ?	/			
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?				
Did the initial calibration meet the curve fit acceptance criteria?	/			
Were the RT windows properly established?	_			
Were the required standard concentrations analyzed in the initial calibration?				
IV. Continuing calibration		_		
What type of continuing calibration calculation was performed?%D or%R				
Were Evaluation mix standards analyzed prior to the initial calibration and sample analysis?		-		
Were endrin and 4,4'-DDT breakdowns <u>&lt;</u> 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?				
Was a method blank analyzed for each matrix and concentration?				
Were extract cleanup blanks analyzed with every batch requiring clean-up?	4			
Was there contamination in the method blanks or clean-up blanks? If yes, please see the Blanks validation completeness worksheet.				
VI. Surrogate spikes				
Were all surrogate %R within the QC limits?				
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?				
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?				
VII. Matrix spike/Matrix spike duplicates				

### VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
Reviewer: 11//
2nd Reviewer: \_\_\_\_\_

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?				
VIII. Laboratory control samples				
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?				
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?	1			
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?	1			
KII. System performance				
System performance was found to be acceptable.	1			
GHI. Overall assessment of data				
Overall assessment of data was found to be acceptable.	1			
IV: Field duplicates				
ield duplicate pairs were identified in this SDG.				
arget compounds were detected in the field duplicates.				
V. Field blanks				
ield blanks were identified in this SDG.	7			
arget compounds were detected in the field blanks.				

## **VALIDATION FINDINGS WORKSHEET**

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

A. alpha-BHC	I. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	GG. Chlordane
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH. Chlordane (Technical)
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	11. Arodor 1262
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	W. Arodor 1268
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	٦٦.
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:

V:\Validation Worksheets\Pesticides\COMPLST-3S.wpd

LDC#: 240 F7 K39

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

Page: Reviewer: 2nd Reviewer:

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

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

Were surrogates spiked into all samples, standards and blanks?

YMMA

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

#	Date	Sample ID	Column	Surrogate Compound	%R (Limits)	(	Qui	Qualifications	
		_	1 273	A	3) 25	1 511-65	No qual	(I only	<u></u>
			۸	1	49 (	^			
			-	8	9) 0	(b2-12g)			
			1	-	) څح	[, )	\ <u>\</u>		
					)				
				В	) 651	(	J+ Att A	* (S) *	
			7		) (51	(	~		
					)	(			
		( <01 ) (		b	) 0+1	(	ho pre		
			7		) १६/	(			Ī
					)	Ó			
		8	-	8	)  92		J+ dets /	* (s) *	ı
				100-5V		) (			
		8 ( 25×)	•	¥	47 (5	59-115)	No mee		
			>		) SE	1			
			•	B	$\exists$	63-12d)			
	-		7	-	29 (		-		
					$\smile$	(			
		10 (20x)		Ą		( 511-65			
			4	-> ·	)	(	,		
			( )	ς-	0 18	201-60	Ţ		
Lett	Letter Designation	Surrogate Compound	K.	Recovery QC Limits (Soil)	Recov	V Recovery QC Limits (Water)	/ater)	Comments	
	Ą	Tetrachoro-m-xylene							
	В	Decachlorobiphenyl							

LDC#: 240 47 K3A

### VALIDATION FINDINGS WORKSHEET Surrogate Spikes

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

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

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

Were surrogates spiked into all samples, standards and blanks?

Y(N)N/A

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

		T	<del>                                     </del>	T		T	Г	T T	li .	T-	l		II .	ī		1		T		<u> </u>
Qualifications	5+ +cts/A (5) +x	J		(s)/T only)							The state of the s									
ts)	63-124	( )	(	(		(			(	)		)	)	)		) (		)	(	(
%R (Limits)	267	) 900		158 (	902	)	)	)	)		)	)	)	)	)		)		)	
Surrogate Compound	8			В	<b>~</b>							- All All All All All All All All All Al								
Column	1 dr	λ			7															
Sample ID	17			17 (2.0X)																
. Date																				
#																				

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A	Tetrachoro-m-xylene			
В	Decachlorobiphenyl			

LDC# 24047 K3A SDG #: St. Com

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

Page: 2nd Reviewer: Reviewer:

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

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

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

	Qualifications	No ace (LCS S.				No gurl (US.																					
C limits?	Associated Samples	λ				7																					
S (KPD) Within the C	RPD (Limits)	( )	( )	( )	( )	( )	( )	( )	( )	)	( )	( )	)	( )	( )	( )	( )	)	( )	( )	( )	)	( )	( )	( )	( )	
iive percent dimerence	MSD %R (Limits)	(S11-12) br25-	•	( )	_	(241-85) 969	_	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	
les (70K) and the rela	MS %R (Limits)	1 ( 511-19) 25 25-	(×1-85) L9.		( )	(23 (54-12m)	( , )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )		( )	( )	( )	( )	( )	
celli recovel	Compound		Q	A		Л																					_
Were the Monwoo percent recoveries (76K) and the relative percent differences (KPD) within the QC limits?	MS/MSD ID	21/2				23/24																					
C/N/AII)	Date																										
	#																										=

LDC#: 24047 k34 SDG#: 54 Gran

### VALIDATION FINDINGS WORKSHEET Laboratory Control Samples

Page: 1 of / Reviewer:\_

2nd Reviewer.\_

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

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

Were a laboratory control samples (LCS) and laboratory control sample duplicate (LCSD) analyzed for each matrix in this SDG? Were the LCS percent recoveries (%R) and relative percent differences (RPD) within the QC limits? Were a laboratory control samples (LCS) and laboratory control so Y N N/A

Were the LCS percent recoveries (%R) and relative percent differ Level IV/D Only

Y N N/A

Was a LCS analyzed every 20 samples for each matrix or wheney

		R			Π		_	Π	Π	T	T	Г	Ī	Г	T	Π		Т	T					<u> </u>		Ι	Т
	Qualifications	1 3+det/p (	1																								
ormed?	Associated Samples	All W	1																								
extraction was perfo	RPD (Limits)	( )	( )	( )	( )	)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(
or whenever a sample	LCSD %R (Limits)	136 (63-118)		( )	( )		( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	•	( )	( )	( )	( )	( )	( )	( )	( )	
ples for each matrix or	LCS %R (Limits)	128 (63-118)	( )	( )	( )	)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(
every 20 san	Compound	A U																									
Was a LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed?	TCS/LCSD ID	4.5/ 60062 -086 SI																		-							
Y N N/A	# Date																										
X	#																										=

LDC # 24047 K34 SDG # C. C.

# VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

Page: 1 of 1 Reviewer: 5VZ 2nd Reviewer:

METHOD: \_\_GC\_\_ HPLC

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

Level IV/D Only
N N/A WE
N N/A Did
Y N N/A Did

Were CRQLs adjusted for sample dilutions, dry weight factors, etc.?

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

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

If no, please see findings bellow.

	(40)	_	-							
Qualifications	J dets /A (								0	
	J 16									
(ARPD)%D Between Two Columns/Detectors	40.6		47.8							
Sample ID	4		16							
Compound Name	0		<del>e</del> Ω							
#										

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24047K3a

### **VALIDATION FINDINGS WORKSHEET**

**Field Duplicates** 

Page:	lof <i>l</i>
Reviewer:_	WC
2nd Reviewer:_	i^i^

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

Y/N NA

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

Compound Name	Conc (	ug/Kg)	RPD >	Diff	Diff Limits	Quals
Compound Name	5	11	(≤50%)	Dill	Dill Ellilles	(Parent Only)
4,4'-DDE	100	110	10			
4,4'-DDT	24	16	40		,	
beta-BHC	1.3	1.6		0.3	≤1.8	
Hexachlorobenzene	2.5	2.6		0.1	≤1.8	

V:\FIELD DUPLICATES\24047K3a.wpd

LDC # 240 47 K32

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: 1 of 4 Reviewer: NC 2nd Reviewer:

> GC EPA SW 846 Method 8081A METHOD:

Hexachlorobenzene

Parameter:

X^2								
×	Conc	4.00	10.00	25.00	50.00	75.00	100.00	
>	Area	44827.00	103588.00	249072.00	490208.00	730674.00	953705.00	
	Compound	Hexachlorobenzene						
	Column	CLP1		GCS_P2				
-	Date	08/11/2010						

Regression Output:		,	Reported	
Constant		0.00000	= 3	0.00000
Std Err of Y Est		8773.78312		
R Squared		0.99941	r2 =	0.999900
No. of Observations		000000		
Degrees of Freedom		2.00000		
X Coefficient(s)	9653.526874	-1.270906	# Q	9638.000000
Std Err of Coef.	63.877363	0.79		

10358.80 9962.88 9804.16 9742.32 9537.05

11206.75

10101.99 Ave RF

### LDC # 240 47 K24

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Reviewer: NC 2nd Reviewer:

Page: \_\_\_ of\_\_

GC EPA SW 846 Method 8081A METHOD:

4,4'-DDT Parameter:

	00 4.00	10.00		00 20.00		00 100.00	
Area	33015.00	78046.00	193282.00	386784.00	581766.00	756268.00	
Compound	4,4'-DDT		notore				
Column	CLP1		GCS_P2				
Date	08/11/2010						

Regression Output:			Reported	
Constant		0.00000	<b>=</b> 0	0.0000
Std Err of Y Est		5851.33656		
R Squared		0.99959	12 =	0.999700
No. of Observations		00000.9	Additional formation and the second	
Degrees of Freedom		5.00000		
X Coefficient(s)	7650.960458	-1.270906	# Q	7636.000000
Std Err of Coef.	42.600546	62.0		

7804.60 7735.68 7562.68 7731.28 7756.88

8253.75

7807.48 Ave RF

### LDC # 240 47 K 39

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

> of 4

Page:

GC EPA SW 846 Method 8081A METHOD:

Hexachlorobenzene Parameter:

10000.00	100.00	1628971.00			
5625.00	75.00	1284080.00			
2500.00	50.00	894649.00			
625.00	25.00	481272.00		GCS_P2	
100.00	10.00	210505.00			
16.00	4.00	93334.00	Hexachlorobenzene	CLP2	08/11/2010
	Conc	Area	Compound	Column	Date
X^2	×	<b>&gt;</b>			

Regression Output:			Reported	
Constant		20708.90229	<b>ا</b> ا	N.
Std Err of Y Est		3835.69679		
R Squared		86666.0	12 =	0.999990
No. of Observations		000000		
Degrees of Freedom		3.00000		
			es II	N N
X Coefficient(s)	19034.788783	-29.504222	= q	NR
Std Err of Coef.	183.320239	1.76		

23334 21051

19156 Ave RF

### LDC # 24647 K34

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

ひ

Page:

GC EPA SW 846 Method 8081A METHOD:

4,4'-DDT Parameter:

X^2	16.00	100.00	625.00	2500.00	5625.00	10000.00	
Conc	4.00	10.00	25.00	50.00	75.00	100.00	
Y Area	59795.00	137046.00	321682.00	607290.00	883436.00	1123921.00	
Compound	4,4'-DDT						
Column	CLP2		GCS_P2				
Date	08/11/2010						

Regression Output:			Reported	
Constant		8705.27176	11 0	NR
Std Err of Y Est		3439.22112		NAVARIAN PARIAN
R Squared		96666.0	12=	0.999990
No. of Observations		6.00000		
Degrees of Freedom		3.00000		
			ın II	NR.
X Coefficient(s)	12932.023828	-17.656945	= q	NR
Std Err of Coef.	164.371396	1.57		

12867

Ave RF

14949

13705

12146

11779

11239

12781

### LDC # 260 47 K34

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

Reviewer: 2nd Reviewer: 2

METHOD: GC\_\_\_

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

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

Where:

N = Initial Calibration Factor or Nominal Amount
C = Calibration Factor from Continuing Calibration Standard or Calculated Amount

					Reported	Recalculated	Reported	Recalculated
		Calibration		CCV Conc	Conc	Conc	0%	۵%
#	Standard ID	Date	Compound					
-	045F4501	9/8/2010	HCB CLP1	50	52.0	52.6	3.9	5.3
		1:07	4,4'-DDT CLP1	P1 50	52.8	53.1	5.6	6.2
			HCB CLP2	P2 50	9.09	50.6	1.3	1.2
			4,4'-DDT CLP2	P2 50	52.3	52.3	4.6	4.5
2	058F5801	9/8/2010	HCB CLP1	P1 50	52.8	53.5	5.7	7.0
		4:42	4,4'-DDT CLP1	P1 50	52.5	52.8	4.9	5.6
			HCB CLP2	P2 50	51.8	51.8	3.6	3.6
			4,4'-DDT CLP2	P2 50	53.0	52.9	5.9	5.9
ဗ	018F1801	9/8/2010	HCB CLP1	50	51.0	51.7	2.0	3.4
		18:06	4,4'-DDT CLP1	P1 50	50.8	51.1	1.7	2.3
			HCB CLP2	P2 50	50.1	50.1	0.2	0.2
			4,4'-DDT CLP2	P2 50	51.9	51.9	3.8	3.7
4								

200				
CCV4				
cos	Area	498100	390491	
2000	Area	515626	403020	
- ^>	Area	507356	405496	
	Slope	9638	7636	
		HCB CLP1	DDT CLP1	

			(-b-())/2a	594.534828	680.146678	593.376044	679.470002	595.055407	680.544021	709.397426
		(-b+ ())/2a		50.6247775	52.2554288	51,7835619	52.9321046	50.1041984	51.8580858	23.0046807
			( )^1/2	16047,5221	11086.6758	15979.1446	11062.7797	16078.2405	11100.7076	12119.6367
	Calculation		(b^2 - 4aT)	257522966.4	122914380	255333061.5	122385093.8	258509816.2	123225708.3	146885593.9
	1		T = Y-c	-888017.098	-627553.728	-906573.098	-635047.728	-879655.098	-623145.728	-288152.728
			final conc							8.08
		Conc.	×	50.625	52.255	51.784	. 52.932	50.104	51.858	23.005
390491			ပ	20708.902	8705.272	20708.902	8705.272	20708.902	8705.272	8705.272
403020	0		Q	19034.789	12932.024	19034.789	12932.024	19034.789	12932.024	12932.024
405496	Y=a(X^2)+bX+c		æ	-29.504	-17.657	-29.504	-17.657	-29.504	-17.657	-17,657
7636		99		908726	636259	927282	643753	900364	631851	296858
DDT CLP1		Area	>	CCV1 HCB CLP2	CCV1 ddf CLP2	CCV2 HCB CLP2	CCV2 ddf CLP2	CCV3 HCB CLP2	CCV3 ddf CLP2	Sample #4 ddt

LDC#: 240 67 139

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

Page:	1 of 1
Reviewer:_	SYC
nd reviewer:	<b></b>

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

The percent recoveries	(%R) of surrogates were re-	calculated for the compoun-	nds identified below usinc	the following calculation:
------------------------	-----------------------------	-----------------------------	----------------------------	----------------------------

% Recovery: SF/SS \* 100

Sample ID:\_

Where: SF = Surrogate Found SS = Surrogate Spiked

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene	cip,	20	17.7	89	85	0
Tetrachloro-m-xylene	1 7		16.9	85	85	
Decachlorobiphenyl	1		20,4	10 V	16~	
Decachlorobiphenyl	7	J	21.6	105/	108	7

Sample ID:

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

Sample ID:

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

Sample ID:

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

Notes:		· · · · · · · · · · · · · · · · · · ·		 
	<u></u>		 	

LDC#: 24047 K3C

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

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

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

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

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

Where:

SC = Concentration

SSC = Spiked sample concentration SA = Spike added

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

MS/MSD samples:\_

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

	S	Spike	Sample	l	Sample	Matri	Matrix Spike	Matrix Spik	Matrix Spike Duplicate	M	MS/MSD
Compound	Ch		( ns hc )	ı	(VE) /< )	Percent	Percent Recovery	Percent	Percent Recovery		RPD
	MS	Ω	7.	-	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
gamma-BHC	17.7	17.8	0	16.7	17.0	44	44	16	46	۷	7
4,4'-DDT	<u> </u>		૯૬	21.7	23.0	× ) ~	0	v) -	0	-	
Aroclor 1260								3			
									·		
											-

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

LDC# 240 F7 1/34

# VALIDATION FINDINGS WORKSHEET

# Laboratory Control Sample/Laboratory Control Sample Duplicate Results Verification

Page: 10f

Reviewer: 37/2 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:

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

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

2 LCS/LCSD samples:

29748/2-4 180-

	\$ B	Spike	Spiked	Sample		TCS	27	LCSD		400
Compound	(43	(k)		Concentration ( 25 // )	Dorrent	Degreent Document			LCO	EC3/EC3D
		P				Vacovery	Percent	Percent Recovery	R	RPD
	FCS	CCSD	rcs	LCSD	Reported	Recalc.	Reported	Recair	Popular	1
gamma-BHC	6,3	イス	13.95	7	78	28			Deliodev	Kecalc.
4 4'-DDT						Q o				
		-\	13,97	7	√ ∞	\ &				
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#: 29047 13C SDG#: Su Com

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	_\_of_ <i>_</i>
Reviewer:	M.
2nd reviewer:	1/-

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

Υ,	N	N/A
	N	N/A

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

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

Example: Sample I.D.  $\pm \frac{4}{9} = 0$   $y = 4(x^{2}) + 6x + c$   $2\frac{6665}{3} = (17.657) \times^{2} + 12932.624 \times + 8705272$  X = 23.005Ainal cone.  $= \frac{(23.605)(10m)}{(31.25)(0.913)}$  = 8.68 Us /kg $\frac{2}{3} = 8 \text{ Us /kg}$ 

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		:			
		<u> </u>			
-					
					*****
		Ĭ			

Note:	 

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 25, 2010

LDC Report Date:

October 11, 2010

Matrix:

Soil

Parameters:

Chlorinated Pesticides

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6818-1

Sample Identification

SSAJ3-02-1BPC

### Introduction

This data review covers one soil 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- 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  $(r^2)$  was greater than or equal to 0.990.

### 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.

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

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.

### 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
SSAJ3-02-1BPC	CLP1	Decachlorobiphenyl Decachlorobiphenyl	542 (63-124) 565 (63-124)	All TCL compounds except Hexachlorobenzene beta-BHC	J+ (all detects)	А

### VII. 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.

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Pesticide Cleanup Checks

### a. Florisil Cartridge Check

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

### b. GPC Calibration

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

### XI. Target Compound Identification

Raw data were not reviewed for this SDG.

### XII. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6818-1	All compounds reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

No field duplicates were identified in this SDG.

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Data Qualification Summary - SDG 280-6818-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6818-1	SSAJ3-02-1BPC	All TCL compounds except Hexachlorobenzene beta-BHC	J+ (all detects)	А	Surrogate spikes (%R) (s)
280-6818-1	SSAJ3-02-1BPC	All compounds reported below the PQL.	J (all detects)	Α	Project Quantitation Limit (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG 280-6818-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG 280-6818-1

No Sample Data Qualified in this SDG

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

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Date: 10/06/10 Page: \ of \ Reviewer: 2nd Reviewer:

SDG #: 280-6818-1 Laboratory: Test America

LDC #: 24047L3a

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 8/25/10
II.	GC/ECD Instrument Performance Check	A	<i>'</i>
III.	Initial calibration	A	2 RSD = 20 7 r7
IV.	Continuing calibration/ICV	A	cu/w = 20 ]
V.	Blanks	A	
VI.	Surrogate spikes	SW	·
VII.	Matrix spike/Matrix spike duplicates	N	Client spec
VIII.	Laboratory control samples	A	Client spec
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	
Xb.	GPC Calibration	N	
XI.	Target compound identification	N	•
XII.	Compound quantitation and reported CRQLs	N	·
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	7	
XV.	Field blanks	ND	FB = FB-04672010- R2D (from 280-2216-2)

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

TB = Trip blank EB = Equipment blank

Validated Samples:

Soil

	3011						
† 1	SSAJ3-02-1BPC	11		21		31	
2	MB 286-29894/1-A	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	

# VALIDATION FINDINGS WORKSHEET

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

A. alpha-BHC	I. Dieldrin	Q. 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-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	H:
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	JJ.
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	LL.
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.

V:\Validation Worksheets\Pesticides\COMPLST-3S.wpd

LDC #: 240 47 L31

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

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

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

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

Y N N/A

Were surrogates spiked into all samples, standards and blanks?

Y N N/A

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

Qualifications	3+400		No speed											
%R (Limits)	) <b>5</b> \$7 (	(	) 225	> 525	)	)	)	)	)	)	)	)		
Surrogate Compound	B	4	8	8										
Column	•													
Sample ID			(x5)									-		
Date														
#														

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A	Tetrachoro-m-xylene			
8	Decachlorobipheny			

\* All except FF

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

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 26, 2010

**LDC Report Date:** 

October 11, 2010

Matrix:

Soil

Parameters:

Chlorinated Pesticides

**Validation Level:** 

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6851-1

Sample Identification

SSAJ3-02-5BPC

### Introduction

This data review covers one soil 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- 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 (r<sup>2</sup>) was greater than or equal to 0.990.

### 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.

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

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.

No field blanks were identified in this SDG.

### VI. Surrogate Spikes

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

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
SSAJ3-02-5BPC	CLP2	Decachlorobiphenyl	137 (63-124)	All TCL compounds	J+ (all detects)	Р

### VII. 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.

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Pesticide Cleanup Checks

### a. Florisil Cartridge Check

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

### b. GPC Calibration

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

### XI. Target Compound Identification

Raw data were not reviewed for this SDG.

### XII. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6851-1	All compounds reported below the PQL.	J (all detects)	A

Raw data were not reviewed for this SDG.

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

No field duplicates were identified in this SDG.

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Data Qualification Summary - SDG 280-6851-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6851-1	SSAJ3-02-5BPC	All TCL compounds	J+ (all detects)	Р	Surrogate spikes (%R) (s)
280-6851-1	SSAJ3-02-5BPC	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 280-6851-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG 280-6851-1

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

Stage 2B

Date:	10/05/10
Page:_	1 of 1

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

SDG #: 280-6851-1 Laboratory: Test America

LDC #: 24047M3a

poratory: Test America

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

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

		1	
	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 8 /26 /10
II.	GC/ECD Instrument Performance Check	A	,
111.	Initial calibration	A	2 RSD = 20 2 ~~ CW/19 = 20 2
IV.	Continuing calibration/ICV	Ā	ca/100 = 20 }
V.	Blanks	A	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	ú	gient spec
VIII.	Laboratory control samples	A	us to
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	
Xb.	GPC Calibration	N	
XI.	Target compound identification	N	
XII.	Compound quantitation and reported CRQLs	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	K)	
XV.	Field blanks	N	

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

TB = Trip blank

EB = Equipment blank

Validated Samples:

Soil

	3811				
1	SSAJ3-02-5BPC	11	21	31	
2	MB 280 - 29894 /-A	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	

Primary col. =

LDC# >4047 M39

## VALIDATION FINDINGS WORKSHEET Surrogate Spikes

of Page:\_\_\_

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

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

Were surrogates spiked into all samples, standards and blanks?

Y(N) W/A

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

	(5)																		
Qualifications	J+ dus/P	1																	
(	( 62-124)	) (	)	(	)	(	)	(	) (	(	) (	(	(	(	(	(	(	) (	)
%R (Limits)	137 (	)		)	)		)	)	)	)	)	)	)	)	)	)	)	)	)
Surrogate Compound	В	-																	
Column	CLP2																		
Sample ID		-																	
Date																			
#																			

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A Tet	etrachoro-m-xylene			
В	Jecachlorobiphenyl			

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

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

Henderson, Nevada

Collection Date: August 27, 2010

LDC Report Date: October 8, 2010

Matrix: Soil/Water

Parameters: Chlorinated Pesticides

Validation Level: Stage 2B & 4

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6886-1

### Sample Identification

BDT-1-N-20-10BPC BDT-1-N-20-12BPC** BDT-1-N-20-14BPC BDT-1-N-20-2BPC BDT-1-N-20-4BPC BDT-1-N-20-6BPC BDT-1-N-20-6BPC BDT-1-N-5-10BPC BDT-1-N-5-10BPC BDT-1-N-5-14BPC BDT-1-N-5-8BPC_FD BDT-1-N-5-8BPC BDT-1-N-5-8BPC BDT-1-N-5-4BPC BDT-1-N-5-4BPC BDT-1-N-5-4BPC BDT-1-N-5-4BPC BDT-1-N-5-4BPC BDT-1-N-5-6BPC BDT-1-N-10-10BPC BDT-1-N-10-12BPC	BDT-1-N-10-4BPC BDT-1-N-10-6BPC BDT-1-N-10-8BPC BDT-1-N-10-8BPC_FD BDT-1-N-15-10BPC BDT-1-N-15-12BPC BDT-1-N-15-14BPC** BDT-1-N-15-8BPC BDT-1-N-15-8BPC BDT-1-N-15-6BPC BDT-1-N-15-6BPC BDT-1-S-20-10BPC BDT-1-S-20-14BPC** BDT-1-S-20-4BPC BDT-1-S-20-6BPC BDT-1-S-20-6BPC BDT-1-S-20-8BPC	BDT-1-N-20-10BPCMS BDT-1-N-20-10BPCMSD BDT-1-N-15-4BPCMS BDT-1-N-15-4BPCMSD BDT-1-S-20-4BPCMS BDT-1-S-20-4BPCMSD
BDT-1-N-10-14BPC	BDT-1-S-20-14BPC_FD	
BDT-1-N-10-2BPC	EB-08272010	

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

### Introduction

This data review covers 45 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Samples indicated by a double asterisk on the front cover underwent a 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
9/9/10	041F4101.D	CLP2	4,4'-DDD	22.9	BDT-1-S-20-2BPC BDT-1-S-20-4BPC BDT-1-S-20-6BPC BDT-1-S-20-8BPC BDT-1-S-20-14BPC_FD BDT-1-S-20-4BPCMS BDT-1-S-20-4BPCMSD MB 280-30428/1-A	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-08272010 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-1-N-20-10BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	163 (63-124) 160 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	A
BDT-1-N-20-10BPC (2X)	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	156 (63-124) 152 (63-124)	Hexachlorobenzene	J+ (all detects)	А
BDT-1-N-20-12BPC**	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	137 (63-124) 135 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	А
BDT-1-N-20-12BPC** (2X)	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	137 (63-124) 137 (63-124)	Hexachlorobenzene	J+ (all detects)	Α
BDT-1-N-20-14BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	416 (63-124) 398 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	Α
BDT-1-N-20-2BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	248 (63-124) 250 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	Α
BDT-1-N-5-10BPC	CLP1	Tetrachloro-m-xylene	123 (59-115)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	A
BDT-1-N-5-14BPC	CLP1	Tetrachloro-m-xylene	127 (59-115)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	Α
BDT-1-N-5-8BPC (2X)	CLP1	Decachlorobiphenyl	126 (63-124)	Hexachlorobenzene	J+ (all detects)	А

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
BDT-1-N-5-2BPC	CLP1	Decachlorobiphenyl	132 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	Α
BDT-1-N-5-6BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	135 (63-124) 135 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	Α
BDT-1-N-5-6BPC (2X)	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	138 (63-124) 137 (63-124)	Hexachlorobenzene	J+ (all detects)	Α
BDT-1-N-10-10BPC	CLP1 CLP1 CLP2	Tetrachloro-m-xylene Decachlorobiphenyl Decachlorobiphenyl	116 (59-115) 541 (63-124) 582 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	A
BDT-1-N-10-12BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	383 (63-124) 392 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	А
BDT-1-N-10-14BPC	CLP1 CLP1 CLP2	Tetrachloro-m-xylene Decachlorobiphenyl Decachlorobiphenyl	120 (59-115) 433 (63-124) 474 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	А
BDT-1-N-10-2BPC	CLP2 CLP1 CLP2	Tetrachloro-m-xylene Decachlorobiphenyl Decachlorobiphenyl	146 (59-115) 1681 (63-124) 2102 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	A
BDT-1-N-10-4BPC	CLP1 CLP2 CLP1 CLP2	Tetrachloro-m-xylene Tetrachloro-m-xylene Decachlorobiphenyl Decachlorobiphenyl	121 (59-115) 116 (59-115) 1314 (63-124) 1488 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	Α
BDT-1-N-10-6BPC	CLP2 CLP1 CLP2	Tetrachloro-m-xylene Decachlorobiphenyl Decachlorobiphenyl	168 (59-115) 2942 (63-124) 4283 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	А
BDT-1-N-10-8BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	231 (63-124) 474 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	А
BDT-1-N-10-8BPC_FD	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	219 (63-124) 222 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	А
BDT-1-N-15-10BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	544 (63-124) 590 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	Α
BDT-1-N-15-12BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	390 (63-124) 416 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	А
BDT-1-N-15-14BPC**	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	321 (63-124) 341 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	А

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
BDT-1-N-15-8BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	276 (63-124) 301 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	A
BDT-1-N-15-2BPC	CLP1 CLP2 CLP1 CLP2	Tetrachloro-m-xylene Tetrachloro-m-xylene Decachlorobiphenyl Decachlorobiphenyl	117 (59-115) 182 (59-115) 3325 (63-124) 4896 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	Α
BDT-1-N-15-4BPC	CLP1 CLP1 CLP2	Tetrachloro-m-xylene Decachlorobiphenyl Decachlorobiphenyl	189 (59-115) 4523 (63-124) 7532 (63-124)	All TCL compounds except 4,4'-DDE 4,4'-DDT Hexachlorobenzene	J+ (all detects)	А
BDT-1-N-15-6BPC	CLP1 CLP2 CLP1 CLP2	Tetrachloro-m-xylene Tetrachloro-m-xylene Decachlorobiphenyl Decachlorobiphenyl	125 (59-115) 125 (59-115) 1789 (63-124) 2991 (63-124)	All TCL compounds except Hexachlorobenzene	J+ (all detects)	A
BDT-1-S-20-10BPC	CLP1 CLP2	Decachlorobiphenyl Decachlorobiphenyl	130 (63-124) 208 (63-124)	All TCL compounds	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) and relative percent difference (RPD) were not within QC limits for some compounds, the MS, MSD, or 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.

### 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 280-6886-1	All compounds 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-1-N-20-10BPC and BDT-1-N-20-10BPC\_FD, samples BDT-1-N-5-8BPC\_FD and BDT-1-N-5-8BPC, samples BDT-1-N-10-8BPC and BDT-1-N-10-8BPC\_FD, and samples BDT-1-S-20-14BPC\*\* and BDT-1-S-20-14BPC\_FD were identified as field duplicates. No chlorinated pesticides were detected in any of the samples with the following exceptions:

	Concentrat	tion (ug/Kg)	222	Diff		
Compound	BDT-1-N-20-10BPC	BDT-1-N-20-10BPC_FD	RPD Difference (Limits) Flags			A or P
4,4'-DDE	0.53	1.8U	-	1.27 (≤1.8)	-	-
Hexachlorobenzene	46	3.6	-	42.4 (≤1.8)	J (all detects)	А

	Concentrat	ion (ug/Kg)		Difference	Difference	,	
Compound	BDT-1-N-5-8BPC	BDT-1-N-5-8BPC_FD	RPD Difference (Limits) (Limits)		Flags	A or P	
4,4'-DDE	39	51	27 (≤50)	-	-	-	

	Concentrat	ion (ug/Kg)	DDD	D:#		
Compound	BDT-1-N-5-8BPC BDT-1-N-5-8BPC_FD		RPD (Limits)	Difference (Limits)	Flags	A or P
4,4'-DDT	3.1	2.5	-	0.6 (≤1.8)	-	-
beta-BHC	9.7	3,3	-	6.4 (≤1.8)	J (all detects)	Α
Endrin ketone	4.8	5.6	-	0.8 (≤1.8)	-	-
Hexachlorobenzene	13	4.5	-	8.5 (≤1.8)	J (all detects)	А
Methoxychlor	1.2	1.9	-	0.7 (≤3.4)	-	-

	Concentra	tion (ug/Kg)	DDD	Difference		
Compound	BDT-1-N-10-8BPC	BDT-1-N-10-8BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
4,4'-DDE	1.2	0.98	-	0.22 (≤1.8)	-	-
4,4'-DDT	1.1	0.98	-	0.12 (≤1.8)	-	-
Hexachlorobenzene	210	180	15 (≤50)	•	-	-

	Concentral	tion (ug/Kg)	nnn	Difference		
Compound	BDT-1-S-20-14BPC**	BDT-1-S-20-14BPC_FD	RPD (Limits)	(Limits)	Flags	A or P
4,4'-DDD	1.8U	0.70	-	1.1 (≤1.8)	-	-
4,4'-DDE	22	27	20 (≤50)		-	-
4,4'-DDT	10	14	33 (≤50)		-	-
beta-BHC	24	25	4 (≤50)		-	-
Endrin ketone	1.8U	0.78	-	1.02 (≤1.8)	-	-
Hexachlorobenzene	4.2	4.5	· -	0.3 (≤1.8)	-	-

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Data Qualification Summary - SDG 280-6886-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6886-1	BDT-1-S-20-2BPC BDT-1-S-20-4BPC BDT-1-S-20-6BPC BDT-1-S-20-8BPC BDT-1-S-20-14BPC_FD	4,4'-DDD	J+ (all detects)	А	Continuing calibration (%D) (c)
280-6886-1	BDT-1-N-20-10BPC BDT-1-N-20-12BPC** BDT-1-N-5-6BPC BDT-1-S-20-10BPC	All TCL compounds	J+ (all detects)	Α	Surrogate recovery (%R) (s)
280-6886-1	BDT-1-N-5-8BPC	Hexachlorobenzene	J+ (all detects)	A	Surrogate recovery (%R) (s)
280-6886-1	BDT-1-N-20-14BPC BDT-1-N-20-2BPC BDT-1-N-5-10BPC BDT-1-N-5-14BPC BDT-1-N-5-2BPC BDT-1-N-10-10BPC BDT-1-N-10-12BPC BDT-1-N-10-14BPC BDT-1-N-10-4BPC BDT-1-N-10-6BPC BDT-1-N-10-8BPC BDT-1-N-10-8BPC BDT-1-N-15-10BPC BDT-1-N-15-14BPC BDT-1-N-15-14BPC BDT-1-N-15-14BPC BDT-1-N-15-14BPC** BDT-1-N-15-8BPC BDT-1-N-15-8BPC BDT-1-N-15-8BPC BDT-1-N-15-6BPC	All TCL compounds except Hexachlorobenzene	J+ (all detects)	A	Surrogate recovery (%R) (s)
280-6886-1	BDT-1-N-15-4BPC	All TCL compounds except 4,4'-DDE 4,4'-DDT Hexachlorobenzene	J+ (all detects)	A	Surrogate recovery (%R) (s)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-6886-1	BDT-1-N-20-10BPC BDT-1-N-20-12BPC** BDT-1-N-20-14BPC BDT-1-N-20-2BPC BDT-1-N-20-4BPC BDT-1-N-20-6BPC BDT-1-N-20-6BPC BDT-1-N-20-10BPC_FD BDT-1-N-5-10BPC BDT-1-N-5-12BPC BDT-1-N-5-14BPC BDT-1-N-5-8BPC_FD BDT-1-N-5-8BPC BDT-1-N-5-8BPC BDT-1-N-5-6BPC BDT-1-N-5-6BPC BDT-1-N-10-10BPC BDT-1-N-10-12BPC BDT-1-N-10-12BPC BDT-1-N-10-18PC BDT-1-N-10-8BPC BDT-1-N-10-8BPC BDT-1-N-10-8BPC BDT-1-N-15-18BPC BDT-1-N-15-18BPC BDT-1-N-15-18BPC BDT-1-N-15-18BPC BDT-1-N-15-18BPC BDT-1-N-15-10BPC BDT-1-N-15-18BPC BDT-1-N-15-18BPC BDT-1-N-15-18BPC BDT-1-N-15-18BPC BDT-1-N-15-18BPC BDT-1-N-15-18BPC BDT-1-N-15-18BPC BDT-1-N-15-18BPC BDT-1-N-15-2BPC BDT-1-N-15-4BPC BDT-1-N-15-4BPC BDT-1-N-15-4BPC BDT-1-N-15-6BPC BDT-1-S-20-14BPC** BDT-1-S-20-14BPC** BDT-1-S-20-4BPC BDT-1-S-20-8BPC BDT-1-S-20-8BPC BDT-1-S-20-14BPC_FD EB-08272010	All compounds reported below the PQL.	J (all detects)	Α	Project Quantitation Limit (sp)
280-6886-1	BDT-1-N-20-10BPC BDT-1-N-20-10BPC_FD	Hexachlorobenzene	J (all detects)	A	Field duplicates (Difference) (fd)
280-6886-1	BDT-1-N-5-8BPC BDT-1-N-5-8BPC_FD	beta-BHC Hexachlorobenzene	J (all detects) J (all detects)	A	Field duplicates (Difference) (fd)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG 280-6886-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Equipment Blank Data Qualification Summary - SDG 280-6886-1

No Sample Data Qualified in this SDG

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

Stage 2B/4

Date: 10/05/1	c
Page: 1_of_2	

Reviewer: 2nd Reviewer:

SDG #: 280-6886-1 Laboratory: Test America

LDC #: 24047N3a

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 /27 /10
II.	GC/ECD Instrument Performance Check	A	/
111.	Initial calibration	A	70 RSD = 20 \ r \ car/161 \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \
IV.	Continuing calibration/ICV	SH	car/10 & 20 Z
V.	Blanks	A	
VI.	Surrogate spikes	Sn)	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	A	LCS /b
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	Á	
XIV.	Field duplicates	SW	$D_1 = 1.8$ $D_2 = 12, 13$ $D_3 = 23, 24$ $D_4 = 34,39$ $EB = 40$ $FB = FB = 6413 2010$ RTG2 = RZE
XV.	Field blanks	ND	EB = 40 TB - FB-04132010 RTG2-RZE

Note:

A = Acceptable

N = Not provided/applicable SW = See worksheet

R = Rinsate FB = Field blank

ND = No compounds detected D = Duplicate

TB = Trip blank

EB = Equipment blank

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

	Soil	_		Noter				
1	BDT-1-N-20-10BPC •	S	11	BDT-1-N-5-14BPC S	21	BDT-1-N-10-4BPC -	3	1 BDT-1-N-15-6BPC S
2 1	BDT-1-N-20-12BPC**		12 12	BDT-1-N-5-8BPC_FD <b>1</b> 2_	22	BDT-1-N-10-6BPC	3:	2 BDT-1-S-20-10BPC
3	BDT-1-N-20-14BPC		13	BDT-1-N-5-8BPC ⊅ <sub>&gt;</sub>	23	BDT-1-N-10-8BPC <b>₽</b> >	3	3 BDT-1-S-20-12BPC
4 1	BDT-1-N-20-2BPC		14	BDT-1-N-5-2BPC	24	BDT-1-N-10-8BPC_FD 03	3.	4 BDT-1-S-20-14BPC** <b>D4</b>
5 <sup>1</sup>	BDT-1-N-20-4BPC		15	BDT-1-N-5-4BPC	25	BDT-1-N-15-10BPC	3	5 BDT-1-S-20-2BPC
6	BDT-1-N-20-6BPC		16 <sup>1</sup>	BDT-1-N-5-6BPC	26	BDT-1-N-15-12BPC	3	6 BDT-1-S-20-4BPC
7 1	BDT-1-N-20-8BPC		17 <sup>1</sup>	BDT-1-N-10-10BPC	27	BDT-1-N-15-14BPC**	3	7 BDT-1-S-20-6BPC
8	BDT-1-N-20-10BPC_FD 0,		18	BDT-1-N-10-12BPC	28	BDT-1-N-15-8BPC	3	8 BDT-1-S-20-8BPC
9	BDT-1-N-5-10BPC		19 1	BDT-1-N-10-14BPC	29	BDT-1-N-15-2BPC	3	9 BDT-1-S-20-14BPC_FD 1
10 \	BDT-1-N-5-12BPC		20 \	BDT-1-N-10-2BPC	30	BDT-1-N-15-4BPC	4	EB-08272010 W

### Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

Stage 2B/4

Date:	10/05/1
Page:_	2012
Reviewer:	JVL
2nd Reviewer:	

SDG #: 280-6886-1 Laboratory: Test America

LDC #: 24047N3a

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		Sampling dates:
11.	GC/ECD Instrument Performance Check	-	\
III.	Initial calibration		
IV.	Continuing calibration/ICV		h - 8
V.	Blanks		
VI.	Surrogate spikes		<i>&gt;</i> ~
VII.	Matrix spike/Matrix spike duplicates		
VIII.	Laboratory control samples		
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	
Xb.	GPC Calibration	N	
XI.	Target compound identification		Not reviewed for Stage 2B validation.
XII.	Compound quantitation and reported CRQLs		Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data		·
XIV.	Field duplicates		
XV.	Field blanks		

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

41	BDT-1-N-20-10BPCMS	S	- 1 51	MB 280- 30108/1-A	61	71	
42	BDT-1-N-20-10BPCMSD		- 52 プ	MB 280- 30428/1-A	62	 72	
43	BDT-1-N-15-4BPCMS		- 3 53	MB 280- 30010/1-A	63	73	
44	BDT-1-N-15-4BPCMSD	Ц	54		64	74	
45	BDT-1-S-20-4BPCMS	Ц	55		65	75	
46	BDT-1-S-20-4BPCMSD	И	56		66	76	
47			57	•	67	77	
48			58		68	78	
49			59		69	79	
50			60		70	 80	

### VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
Reviewer: TV6
2nd Reviewer:

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

MCthod: 1 canadcan aba (El A avv e40 Method aba 1780	7	<del>, -</del>		<b>-</b>
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times		<u>,                                     </u>		
All technical holding times were met.	1	1		
Cooler temperature criteria was met.		1		
II. GC/ECD instrument performance check				
Was the instrument performance found to be acceptable?	-	<u>t</u>		
III. Initial calibration				
Did the laboratory perform a 5 point calibration prior to sample analysis?	/			
Was a linear fit used for evaluation? If yes, were all percent relative standard deviation (%RSD) ≤ 20%?	ns			
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?	/			
Did the initial calibration meet the curve fit acceptance criteria?	/			
Were the RT windows properly established?	/			
Were the required standard concentrations analyzed in the initial calibration?				
IV. Continuing calibration				
What type of continuing calibration calculation was performed?%D or%R				
Were Evaluation mix standards analyzed prior to the initial calibration and sample analysis?				
Were endrin and 4,4'-DDT breakdowns ≤ 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%?	v			
Were all the retention times within the acceptance windows?				
/ Blanks				
Vas a method blank associated with every sample in this SDG?				
Vas a method blank analyzed for each matrix and concentration?				
Vere extract cleanup blanks analyzed with every batch requiring clean-up?				
Vas there contamination in the method blanks or clean-up blanks? If yes, please see ne Blanks validation completeness worksheet.		1		
: Surrogate spikes				
/ere all surrogate %R within the QC limits?				· · · · · · · · · · · · · · · · · · ·
the percent recovery (%R) of one or more surrogates was outside QC limits, was a analysis performed to confirm %R?				
any %R was less than 10 percent, was a reanalysis performed to confirm %R?		T	w	ĺ
I. Matrix spike/Matrix spike duplicates	,		-	

LDC #: 24047 N39

### VALIDATION FINDINGS CHECKLIST

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

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?				
VIII. Laboratory control samples				
Was an LCS analyzed for this SDG?	/			
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
X. Target compound identification				
Were the retention times of reported detects within the RT windows?				-
XI Compound quantitation/CRQLs				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions, dry weight factors, and clean-up activities applicable to level IV validation?				
XII System performance	I Z I			
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		- 1	- 1	
Field blanks were identified in this SDG.				
Target compounds were detected in the field blanks.		./		

## VALIDATION FINDINGS WORKSHEET

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

A. alpha-BHC	I. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	GG. Chlordane
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH. Chlordane (Technical)
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	1
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	الل.
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	cc. 2,4'-DDD	KK.
F. Aldrin	N. Endosulian sulfate	V. Aroclor-1016	DD. 2,4'-DDE	LL.
G. Heptachlor epoxide	O.4,4'-DDT	W. Aroclor-1221	EE.2,4-DDT	MM.
H. Endosulfan I	P. Methoxychior	X. Arodor-1232	FF. Hexachlorobenzene	NN.

**		
	VVVV-Listerian Markabanta Destinides COMPLST-3S word	V.V. aligation vv. or Astronomy assessment of the contract of

Notes:

LDC# 24047 N34

### VALIDATION FINDINGS WORKSHEET Continuing Calibration

Page: 1 of 1 2nd Reviewer: Reviewer:

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"

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

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

X N N/A Y NJ N/A

KN N/A

Qualifications	J+ aets A			AAWARAN II																				1 1 1 11	1 1 1 11	1 1 1 11	1 1 1 11	1 1 1 11	CC. DB 608 GG. DD. DB 1701 HH. EE. Hexachlobenzene II.	1     1
Associated Samples	36-39 45.96	80- 30 428 K-A																												
	36 (	-08c &M (		(	)	(	(	(	(	) (	(	)	) (	)	(	)	)	)	)	)		) (			) ) ) U. Toxaphene	) ) ) ) ) ) ) ) ) ) ) ) ) ) ) ) ) ) )	) ) ) ) ) ) ) ) ) ) ) ) ) ) ) ) ) ) )	) ) ) ) ) ) ) ) ) ) ) ) ) ) ) ) ) ) )	) ) ) ) ) ) ) ) ) ) ) ) ) ) ) ) ) ) )	) ) U. Toxaphene V. Aroclor-1016 W. Aroclor-1221 X. Aroclor-1232
0) RT (Limits)		)	Ú		)	)	)	)		)		)	)	)	)	_		)	)	)	,				( C Endrin ketone	( C. Endrin ketone R. Endrin aldehyde	( C. Endrin ketone R. Endrin aldehyde S. alpha-Chlordane	Q. Endrin ketone R. Endrin aldehyde S. alpha-Chlordane	( C Endrin ketone C Endrin aldehyde S. alpha-Chlordane T aamma-Chlordane	Q. Endrin ketone R. Endrin aldehyde S. alpha-Chlordane T. gamma-Chlordane
%D (1 imit < 20.0)	<u> </u>					-																								ın sulfate
Compound	Z																													
Column	7 417					-		5																						
Ctenderd II	0417410																								E Hantachlor	E. Heptachlor	E. Heptachlor F. Aldrin G. Hentachlor enoxi	E. Heptachlor F. Aldrin G. Heptachlor epoxi	E. Heptachlor F. Aldrin G. Heptachlor epoxiv	E. Heptachlor F. Aldrin G. Heptachlor epoxide H. Endosulfan I
*	10	OII/ San / S																5							OHA GHU	A. alpha-BHC	A. alpha-BHC B. beta-BHC C. delta-BHC	A. alpha-BHC B. beta-BHC C. delta-BHC	A alpha-BHC B beta-BHC C centra-BHC	A. alpha-BHC B. beta-BHC C. delta-BHC D. gamma-BHC

LDC# 24147 N34

## VALIDATION FINDINGS WORKSHEET Surrogate Spikes

Page: 1 of Z Reviewer: 10/2 2nd Reviewer: 0

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

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

V N/A

Were surrogates spiked into all samples, standards and blanks?

Y N N/A

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

	( #	`				——————————————————————————————————————	_										#	-
Qualifications	) (A exapt		(FF only)			All except		(FF only)			(All except #			No Jule (FF one)			J+ dets 12 (S) (A) excent #F	<b>→</b>
%R (Limits)	163 (63-12d)	) 09(	156 ( )	( ) 75/	( )	137 ( )	( ) 581	( ) 75/	( ) 25/	( )	4,6 ( )	348 ( )	(	371 ( )	418 ( )	( )	248 ( )	1 02%
Surrogate Compound	8																	
Column	CLP 1	12		7			7		2			/		l	7		1 /	1 1
Sample ID			1 (2x)			2		2 (2x)			3			3 (20x)			4	
Date																		
#																		

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
А	Tetrachoro-m-xylene			
a	Decachlorobiphenyl			

LDC #: 24047 N 29

## **VALIDATION FINDINGS WORKSHEET** Surrogate Spikes

Page: 2 of 7

Reviewer: 17/6 2nd Reviewer: \_\_\_\_\_\_

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

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

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

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

	$\bigcirc$	<del>\</del>	上				<u> </u>	<del>\</del>			<del></del>			$ \wedge $			7			
	(FF. ne	9	* #11 prayt		*		(FF only	9	*		*			(FF only	5		*			
Qualifications	No mal	0	J+ dets A (S																	<b>&gt;</b>
%R (Limits)	(63-124)		( 511-65)	( )	( )	( _ )	(421-69)	( ] )	( )	(	( )	) (	. ( )	( )	( / )		( 511-45)	(	( )	( )
) %%	250		123		(27		126		<b>76</b>		581	135		861	137		116	24)	285	
Surrogate Compound	В		A		¥		સ		B		82	->		В	æ	,	¥	8	->	
Column	CUP 2	4	CLPI		40		3		CAP 1		ar 1	12		CUCI	7	7	199	CLP 1	7 2	
Sample ID	4 (10x)	•	0	•	11		(xe) 41		<del>*</del> -		9)			(xe) 91			17			
Date																				
#																				

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A	Tetrachoro-m-xylene			
æ	Decachlorobiphenyl			

\* All except FF

LDC#: 24047 N3A

## **VALIDATION FINDINGS WORKSHEET** Surrogate Spikes

Page:\_\_ Reviewer:\_

2nd Reviewer:

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

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

| V | N | N/A | Were surrogates spiked into all samples, standards and blanks?

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

			only)						FF only	9					ı	FF my				
Qualifications	J+ dets/A * (5)		OLLE ( FF			(5) * V 41)			gral (FF			- dets (A * (S				qual /	7			
%R (Limits)	3-124)		( ) No c	$\theta$ ( )	( )	15 (51-115)	(62-124)		) 20	( ( )		(59-115) J+	(63-124)		(	0 N ( 211-6-5)		(63-124)		
%R	383	347	372	407		120	433	474	7 ph	463		146	1891	2102		D	9	1677	<081	
Compound	W					¥	В		20		•	∢	B	1		<b>∀</b>	A	В	ਧ	
Column	1 200	\ 		λ				٧		4		7		7		)	^		×	
Sample ID	81		(XOK) SI			19			(x0x) 61			56				×901) OC				
# Date																				

\* All exact FF

LDC#: 24067 N&A

## VALIDATION FINDINGS WORKSHEET Surrogate Spikes

Page: 4 of 7 Reviewer: 370 2nd Reviewer:

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

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

Were surrogates spiked into all samples, standards and blanks?

Y \( \bar{N} \) \( \bar{N} \) \( \bar{N} \) Did all surrogate percent recoveries (%R) meet the QC limits?

#	Date	Sample ID	Column	Surrogate Compound	%R (Limits)	Qualifications
		2/	1 25	А	511-bs) /21	) J+ 11th /4 + (2)
			7	1	( ) 911	
				ঘ	13)4 (63-124	
			ン	1		) /
					)	
		( xex) 12	,	Ą	511-65) 0	No gral (FF ny)
			2			
			-	B.	1263 (63-124	
			ト		J. ) Loe!	<b>1</b>
				۸	)	
		22	2	¥	511-65) 891	) J+ Acts (4 x (3)
				B		
			7		)	1
				^	)	
		22 (200x)		A	S11-65) 0	No gral
			7			
				B	3149 (63-124	
	,		4		3.48 (	7
					)	
		23	1	8	23) (	) J+ Ath (5)
			7	<b>,</b>	474	
Let	Letter Designation	Surrogate Compound	Re	Recovery QC Limits (Soil)	ii) Recovery QC Limits (Water)	nits (Water) Comments
	Ą	Tetrachoro-m-xylene				
	Φ	Decachlorobipheny				

\* All exupt FF

LDC# 240 47 N39

## VALIDATION FINDINGS WORKSHEET Surrogate Spikes

Page: 5 of 7 Reviewer. 76

2nd Reviewer:\_

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

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

Were surrogates spiked into all samples, standards and blanks?

V N N/A

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

			_	Т	il	_		1	1		_	_	1		_	<del></del>	1	T	i T	Г
Qualifications	No oned (FF only)			J+1015 4 4 (S)			No grad (FF ort.)			5+ dets 4 × (5)			No gual (FF orm.)			J+dets/A *			No sture (FF out)	
%R (Limits)	249 (63-124)	262 (   )		( ) 612	( ) \( \cc	( )	( ) SEC	( ) {}c		544 ( )	590 ( )	( )	( ) 509	604 ( )	(	390 ( )	4.6 ( )	( )	( ) 914	408
Surrogate Compound	В	7		8			æ			9			22			В			В	
Column	24-)	٨			ン		•	٧			λ		,	2		,	٨			<i>&gt;</i>
Sample ID	23 (167)			24			( x01 ) the	ا		25		,	25 (25x)			. 92			26 (20x)	- <del>-</del>
Date					·															
*																				

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A	Tetrachoro-m-xylene			
В	Decachlorobiphenyl			

\*

LDC # 24047 N 24

## **VALIDATION FINDINGS WORKSHEET** Surrogate Spikes

Page: 6 of 7
Reviewer: 3/4
2nd Reviewer: 1

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

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

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

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

				40000		A CONTRACTOR OF THE CONTRACTOR
*	Date	Sample ID	Column	Surrogate	%R (Limits)	Qualifications
		27	CLP 1	8	321 ( \$3-124 )	J+1045/A # (3)
		,	2		34 ( )	
		27 (lox)	•	В	>6€ ( )	No jud +x
			7		360 (	
				7	( )	
		28		8	276 ( )	1+ 1ct (3)
			2	->	301 (	1 1
		28 (10x)		8	318 ( )	No such **
			٨	<b>├</b>	1 828	•
					( )	
		5-9	(	Ą	( ) (1	J+ Cotty *
			7	٧	( ) 281	
		The state of the s	, /	8	3326	
			7		4896 ( 1)	<b>\</b>
		29 (200x)	,	Ą	(511-65) 0	No. mal **
			メ			
			-	a	3597 (63-124)	
			X		3764	
Le	Letter Designation	Surrogate Compound		Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Water) Comments
	Ą	Tetrachoro-m-xylene				- Continue of the Continue of
	В	Decachlorobiphenyl				

All except FF me 11 \*\*

SUR.wpd

LDC# 24047 Nag

## **VALIDATION FINDINGS WORKSHEET** Surrogate Spikes

Page: 7 of Reviewer: N6

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

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

| V | N/A | Were surrogates spiked into all samples, standards and blanks?
| V | N/N/A | Did all surrogate percent recoveries (%R) meet the QC limits?

	(FF	\		,		_															
Qualifications	3+ Acts /2 (S) (ail except 50)				10 que (JOFFAG)			•	J+ dets/A * (5)			-	No stral x x			*	3+ 10ts/A (M TCL) #(C)		Comments		
%R (Limits)	1 51-15	3 (63-124)	( ) /654	( )	N ( S11-65) 9		5290 (63-124)	5315 ( 1 )	( 54-12) 251	( ) 521	1789 (63-124)	( ) 1664	0 (511-65)		( 63-124 ) Stoc	( ) ) {8/2	130 ( )	208 J	Recovery QC Limits (Water)		
Surrogate Compound		8			A		8	/	4		. 6	7	$\forall$		2	2	þ		Recovery QC Limits (Soil)		
ID Column	2		٨		۲) ا ز	7		7		2		$\lambda$	1 (x00/	۲	(	<b>&gt;</b>		4	Surrogate Compound	n-xylene	inhenv
Date Sample	-xase-) 0E				30 (200x)				k				9) K				37		Letter Designation Surrog	A Tetrachoro-m-xylene	Decachlorobinhenv
#																			Letter D		

SUR.wpd

Page: 1 of 1

VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Reviewer: 016 2nd Reviewer:

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

LDC #: 24047 N32 SDG #: St Con Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A", X N N/A Y N N/A

Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG?

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

) *	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
Ш		41/47	FF	-141 (50-130)	139 (50-130)	( )		No mal (LCS in)
			и	( )	( )	(23)	-	( " OSW'SW)
					( )	( )		
		43/44	Seneral	comp dunds	outside limits	( ) 74.	30	No mul (LCS in
			1 AN %	oR and 2 R		(		,
				( )	( )	( )		
		45/46	8	134 (8-115)	124 (58-115)	( )	36	No may (45 m
				( )	( )	( )		
						( )		
						( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
					( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				(	( )	( )		

LDC#: 24047N3a

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

Page	:of/
Reviewer:	JV⁄6
2nd Reviewer:	<u> </u>

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

YN NA

Were field duplicate pairs identified in this SDG?

Were target analytes detected in the field duplicate pairs?

Compound Name	Conc (	ug/Kg)	RPD	Diff	Diff Limits	Ouals
Compound Name	1	8	(≤50%)	DIII	Diricuits	<b>Quals</b> (Parent Only)
4,4'-DDE	0.53	1.8U		1.27	≤1.8	
Hexachlorobenzene	46	3.6		42.4	≤1.8	Jdet/A (fd)

Compound Name	Conc (	ug/Kg)	RPD	Diff	Diff Limits	Quals
Compound Name	13	12	(≤50%)	Dill	Jin Limits	(Parent Only)
4,4'-DDE	39	51	27			
4,4'-DDT	3.1	2.5		0.6	≤1.8	
beta-BHC	9.7	3.3		6.4	≤1.8	Jdet/A (fd)
Endrin ketone	4.8	5.6		0.8	≤1.8	
Hexachlorobenzene	13	4.5		8.5	≤1.8	Jdet/A (fd)
Methoxychlor	1.2	1.9		0.7	≤3.4	

Compound Name	Conc (	ug/Kg)	RPD	Diff	Diff Limits	Quals
	23	24	(≤50%)			(Parent Only)
4,4'-DDE	1.2	0.98		0.22	≤1.8	
4,4'-DDT	1.1	0.98		0.12	≤1.8	
Hexachlorobenzene	210	180	15			

Compound Name	Conc (	ug/Kg)	RPD	Diff	Diff Limits	Quals
•	34	39	(≤50%)		·	(Parent Only)
4,4'-DDD	1.8U	0.70		1.1	≤1.8	
4,4'-DDE	22	27	20			
4,4'-DDT	10	14	33			
beta-BHC	24	25	4			
Endrin ketone	1.8U	0.78		1.02	≤1.8	
Hexachlorobenzene	4.2	4.5		0.3	≤1.8	

## LDC #240 47 N 32

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Reviewer: The 2nd Reviewer:

1 of 4

Page:

GC EPA SW 846 Method 8081A METHOD:

Parameter:

Hexachlorobenzene

			and the second s	manuschillechale (Annaha)	· ·	The state of the s		
×	Conc	4.00	10.00	25.00	50.00	75.00	100.00	-
<b>≻</b>	Area	44827.00	103588.00	249072.00	490208.00	730674.00	953705.00	
	Compound	Hexachlorobenzene				· · · · · · · · · · · · · · · · · · ·		
	Column	CLP1		GCS_P2				
	Date	08/11/2010						

		-		
Regression Output:			Reported	
Constant		0.00000	<b>"</b> 2	0.0000
Std Err of Y Est		8773.78312		
R Squared		0.99941	r2 =	0.999900
No. of Observations		6.00000		
Degrees of Freedom		5.00000		
				readonne e e e e e e e e e e e e e e e e e e
X Coefficient(s)	9653.526874	-1.270906	11 Q	9638,000000
Std Err of Coef.	63.877363	0.79		

10358.80 9962.88 9804.16 9537.05 9742.32

11206.75

Ave RF

10101.99

## LDC # 240 47 N 3 ~

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

METHOD:

GC EPA SW 846 Method 8081A

Parameter:

4,4'-DDT

Regression Output:			Reported	
Constant		0.00000	11 0	0.0000
Std Err of Y Est	Westernament and the second se	5851.33656	OPPORT AND THE PROPERTY OF THE	The state of the s
R Squared		0.99959	12=	0.999700
No. of Observations		0000009	And the state of t	
Degrees of Freedom		5.00000		
X Coefficient(s)	7650.960458	-1.270906	= q	7636.000000
Std Err of Coef.	42.600546	62.0		

7804.60 7731.28 7735.68 7756.88 7562.68

8253.75

7807.48 Ave RF

4 Page:

LDC # 24647 NAC

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

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Page:

GC EPA SW 846 Method 8081A METHOD:

Hexachlorobenzene Parameter:

Xv2	16.00	100.00	625.00	2500.00	5625.00	10000.00	
Conc	4.00	10.00	25.00	50.00	75.00	100.00	
Y Area	93334.00	210505.00	481272.00	894649.00	1284080.00	1628971.00	
Compound	Hexachlorobenzene						
Column	CLP2		GCS_P2				
Date	08/11/2010						

Regression Output:			Reported	
Constant		20708.90229	٥١	N.R.
Std Err of Y Est		3835.69679		
R Squared		86666.0	12=	0.999990
No. of Observations		6.00000		
Degrees of Freedom		3.00000		Amerika da
			Ων Ú	A'N
X Coefficient(s)	19034.788783	-29.504222	= q	AN AN
Std Err of Coef.	183.320239	1.76		

23334 21051

19156 Ave RF

LDC # 24047 N3Q

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

**√** of \_ ,

Page:

Reviewer: M

METHOD:

GC EPA SW 846 Method 8081A

Parameter:

4,4'-DDT

Date	Column	Compound	Y Area	Conc	X^2
08/11/2010	CLP2	4,4'-DDT	59795.00	4.00	16.00
			137046.00	10.00	100.00
	GCS_P2		321682.00	25.00	625.00
			607290.00	50.00	2500.00
	-		883436.00	75.00	5625.00
		7	1123921.00	100.00	10000.00

Regression Output:	out:		Reported	
Constant		8705.27176	# O	NR
Std Err of Y Est		3439.22112		17 T 100 T 1
R Squared		96666.0	12 =	0.999990
No. of Observations		00000'9		Andrews and Agricultural Agricultura Agricultural Agricultural Agricultura Agricultural Agricultural Agricultural Agricult
Degrees of Freedom		3.00000		Control of the Contro
			n II	NR
X Coefficient(s)	12932.023828	-17.656945	# q	NR
Std Err of Coef.	164.371396	1.57	The state of the s	The state of the s

13705 12867 12146 11779 11239

14949

12781 Ave RF

LDC # 2 60 47 NO9

## Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Reviewer: Page: \ of \

METHOD: GC / HPLC

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

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

Where:

Initial Calibration Factor or Nominal Amount

Calibration Factor from Continuing Calibration Standard or Calculated Amount " " Z ()

Recalculated	۵%		3.4	5.5	2.8	2.9	4.6	7.8	6.0	1.7	6.7	9.9	1.5	8.6	2.7	2'0	2.7	3.6	
Reported	۵%		2.1	4.9	2.8	6.7	3.2	7.2	6:0	7.2	5.3	5.9	1.5	8.6	1.4	0.7	1.4	3.7	
Recalculated	Conc		51.7	52.8	48.6	46.7	52.3	53.9	49.6	53.6	53.3	53.3	50.7	54.3	51.4	49.7	51.4	48.2	
Reported	Conc		51.0	52.5	48.6	46.6	51.6	53.6	49.5	53.6	52.7	53.0	50.8	54.3	50.7	49.7	50.7	48.2	
	CCV Conc		50	50	90	50	50	50	50	50	50	50	90	50	50	50	50	50	
		ē	CLP1	CLP1	CLP2	CLP2	CLP1	CLP1	CLP2	CLP2	CLP1	CLP1	CLP2	CLP2	CLP1	CLP2	CLP1	CLP2	
		Compound	HCB	4,4'-DDT	HCB	4,4'-DDT	HCB	4,4'-DDT	HCB	4,4-DDT	HCB	4,4'-DDT	НСВ	4,4'-DDT	HCB	HCB	HCB	모	
	Calibration	Date	9/7/2010	14:06			9/9/2010	15:50			9/9/2010	19:25		-	9/8/2010	14:31	9/10/2010	11:56	
		Standard ID	005F0501				015F1501				028F2801				005F0501		005F0501		
		#	-				2				9				4		5		

	····		· · · · ·	a
CCV5	Area	494984		
CCV4	Area	495072		
CCV3	Area	513994	406809	
CCV2	Area	503972	411700	
CCV1	Area	498434	402900	
	Slope	9638	7636	
		HCB CLP1	DDT CLP1	

			Calculation	(-b+ ())/2a	(b^2-4aT) ( )^1/2 (-b-())/2a	261355654.1 16166.4979 48.6085126 596.551093	127331172.6 11284.1115 46.6645661 685.737541	259561574.8 16110.9148 49.5504716 595.609134	121899385 11040.8055 53.5543565 678.84775	257295431.6 16040.4312 50.7449472 594.414658	121315220.9 11014.3189 54.3043863 678.09772	259353040.5 16104.4416 49.6601708 595.499435	262156746.7 16191.2553 48.1889531 596.970652
					T = Y-c	-855541.098	-565017.728	-870743.098	-641924.728	-889945.098	-650195.728	-872510.098	-848753,098
Area	494984				final conc								
Area	495072			Conc.	×	48.609	46.665	49.550	53.554	50.745	54.304	49.660	48.189
Area	513994	406809			o	20708.902	8705.272	20708.902	8705.272	20708.902	8705.272	20708.902	20708.902
Area	503972	411700	v		۵	19034.789	12932.024	19034.789	12932.024	19034.789	12932.024	19034.789	19034.789
Area	498434	402900	$Y = a(X^2) + bX + c$		æ	-29.504	-17.657	-29.504	-17,657	-29.504	-17.657	-29.504	-29.504
Slope	9638	7636		Area	>	876250	573723	891452	650630	910654	658901	893219	869462
	HCB CLP1	DDT CLP1				CCV1 HCB CLP2	CCV1 ddt CLP2	CCV2 HCB CLP2	CCV2 ddt CLP2	CCV3 HCB CLP2	CCV3 ddt CLP2	CCV4 HCB CLP2	CCV5 HCB CLP2

LDC #: 34647 N39

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

Page:_	1 of 1
Reviewer:_	576
2nd reviewer:	h-/

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

Column

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

Surrogate

Spiked

% Recovery: SF/SS \* 100

Surrogate

Where: SF = Surrogate Found SS = Surrogate Spiked

Sample ID:

# >

Surrogate	Percent	Percent	Percent
Found	Recovery	Recovery	Difference
	Reported	Recalculated	

				Reported	Recalculated	
Tetrachloro-m-xylene	Up 1	20	16,2	81	8)	8
Tetrachloro-m-xylene	CLP2		14.6	73	72	
Decachlorobiphenyl	.		27.4	137	137	
Decachlorobiphenyl	1 2	Y	27.0	135	132	

Sample ID:

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

Sample ID:

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

Sample ID:

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

Notes:	

LDC #: 24047 NOA

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

Page: (of / Reviewer; 3V& 2nd Reviewer:

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

The percent recoveries (%R) and Relative Percent difference (RPD) of the matrix spike and matrix spike duplicate were recalculation:

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

Where: SSC = Spiked sample concentration SA = Spike added

SC = Concentration

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

MS = Matrix spike percent recovery

MSD = Matrix spike duplicate percent recovery

MS/MSD samples: 4)

4) 4

47

	o Nico							,	
Compound	Added	Sample Concentration	Spiked Sample Concentration	Matrix Spike	Spike	Matrix Spik	Matrix Spike Duplicate	<b>X</b>	MS/MSD
nimodino		(%/4)	(45/E)	Percent Recovery	BCOVERY	Percent	Percent Recovery		
	MS MSD	١.	MS 0 MSD	Reported	Beer			Н	RPU
gamma-BHC				٦г	Aecaic.	Keported	Recalc.	Reported	Recalculated
44"DDT	2-/1 / -/-		17.02 16.78	97	26	26	くご	٨	
100-11	-}				Ţ,		,		7
Aroclor 1260			1/. 08 (6, 14	96	46	26	56	6,6	8 0
									0
					Ī				
					Ī				

Comments: Refer ot Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within

## LDC #24047 Ngh

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

Page: 1of ] Reviewer: 37/2

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

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

SSC = Spiked sample concentration SA = Spike added Where:

SC = Concentration

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

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

LCS/LCSD samples:

280- 30 FX8/2-A

	~ ¥	Spike Added	Spike	Spiked Sample Concentration	, EC	rcs	37	TCSD		490 190
punodwoo	9N)	5/19)	3,3	4					EC2	LCSD
	-				reicent Recovery	(ecovery	Percent	Percent Recovery	2	RPD
	C.C.	LCSD	CS	CSD	Bonosto					2
gamma-BHC	7 21				Dellocati	Kecalc.	Reported	Recalc.	Reported	Rocalc
	1.0,	MA	(6.0	*	27	1				
4,4'-DDT				4.4		۳ /				
	4	7	シ <u>ラ</u>		/ 0	Ī				
Aroclor 1260						/0/				
						,				
(										

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicate findings worksheet for list of qualifications and associated samples when reported

LDC #: 24047 N39

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	l_ofl
Reviewer:_	JVG
2nd reviewer:_	12

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

$(\mathbf{Y})$	N	N/A
V	N	N/A
$\mathcal{T}$		

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. 
$$\frac{1}{7}$$
  $\frac{1}{7}$   $\frac{$ 

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

Note:		

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Data Validation Reports LDC #24047

Metals



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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

April 8, 2010

LDC Report Date:

October 15, 2010

Matrix:

Soil

Parameters:

Arsenic

Validation Level:

Stage 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-2280-10

Sample Identification

SSAO6-05-6BPC

### Introduction

This data review covers one soil sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Arsenic.

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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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 was found in the initial, continuing and preparation blanks.

No field blanks were identified 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

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

### VII. Duplicate Sample Analysis

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.

### 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.

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-2280-10	All analytes reported below the PQL.	J (all detects)	А

### 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 Arsenic - Data Qualification Summary - SDG 280-2280-10

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-2280- 10	SSAO6-05-6BPC	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (PQL) (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic - Laboratory Blank Data Qualification Summary - SDG 280-2280-10

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic - Equipment Blank Data Qualification Summary - SDG 280-2280-10

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic - Field Blank Data Qualification Summary - SDG 280-2280-10

No Sample Data Qualified in this SDG

### **Tronox Northqate Henderson**

_DC #:	24047A4	VALIDATION COMPLETENESS WORKSHEET	Date: 10 - 5 - 10
SDG #:	280-2280-10	_ Stage 4	Page:of Reviewer:_ M(
_aborato	ry: Test America		
			2nd Reviewer:
METUAL	N. AC (EDA SIM SAGIN	Nethod 6020)	

METHOD: As (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
I.	Technical holding times	A	Sampling dates: 4-8-10
II.	ICP/MS Tune	Ą	
111.	Calibration	A	
IV.	Blanks	Α	
V.	ICP Interference Check Sample (ICS) Analysis	Α	
VI.	Matrix Spike Analysis	N	client specified
VII.	Duplicate Sample Analysis	N	lt tr
VIII.	Laboratory Control Samples (LCS)	Α	LCS
IX.	Internal Standard (ICP-MS)	A	
X.	Furnace Atomic Absorption QC	7	not utilized
XI.	ICP Serial Dilution	N	not utilized not performed
XII.	Sample Result Verification	A	
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	N	
χv	Field Blanks	7	·

N	ot	_	
IA	U	C	

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	SSA06-05-6BPC	11		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	PBS	20		30	40	

Notes:				

### VALIDATION FINDINGS CHECKLIST

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

	Yes	No	NA	Findings/Comments
Validation Area				,
I. Technical holding times	7			
All technical holding times were met.	<u> </u>			
Cooler temperature criteria was met.	<b>/</b>			
II. ICP/MS Tune	7			-
Were all isotopes in the tuning solution mass resolution within 0.1 amu?	<b>√</b>			
Were %RSD of isotopes in the tuning solution ≤5%?	V			
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.		/		
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?	/			
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?	V			

LDC#: 24047A4

### VALIDATION FINDINGS CHECKLIST

Page: 2-of 2
Reviewer: MG
2nd Reviewer:

Validation Area	Yes	No	NA	Findings/Comments
VIII. Furnace Atomic Absorption QC	<sub>1</sub>		r , 1	
If MSA was performed, was the correlation coefficients > 0.995?			V	
Do all applicable analysies have duplicate injections? (Level IV only)			V	
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)			/	
Were analytical spike recoveries within the 85-115% QC limits?			V	
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%?			1	
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)		T	τ	
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?	<u> </u>			
XI. Regional Quality Assurance and Quality Control		<del>- 7</del>	T	
Were performance evaluation (PE) samples performed?		/	ļ <u></u>	<u> </u>
Were the performance evaluation (PE) samples within the acceptance limits?	L		1	
XII. Sample Result Verification	<del>- 7</del>	т	1	T .
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	1			
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/		<u> </u>	
XIV. Field duplicates				<b>Y</b>
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.		/	<u> </u>	
Target analytes were detected in the field blanks.			\ \ \	

LDC# 34047A4

## Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page:

**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:

 $%R = Found \times 100$ 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	Renorted	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
	ICP (Initial calibration)						
TCV	ICP/MS (Initial calibration)	As	41.52	40.0	h <i>01</i>	h0)	<i>&gt;</i>
	CVAA (Initial calibration)						
	ICP (Continuing calibration)						
0302 CCV	ICP/MS (Continuing calibration)	As	50.37	50.0	101	101	>
	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. 3.

LDC# 34047 A4

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

2nd Reviewer: Page: Lof Lawiewer: 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

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. Where,

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

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

Where,

S = Original sample concentration D = Duplicate sample concentration

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

%D = ||-SDR| × 100

Where, 1 = Initial Sample Result (mg/L)
SDR = Serial Dilution Result (mg/L) (Instrument Reading x 5)

Sample ID         Type of Analysis         Element (units)         Found / S / I         True / D / SDR (units)         True / D / D / D / D / D / D / D / D / D /								
Type of Analysis         Found / S / I         True / D / SDR (units)         True / D / SDR (units)         True / D / SDR (units)         %R / RPD / %D         %R / RPD / %D           I CP interference check         A S         103 .30 (Mg/L)         100 (Mg/L)         103 103         103         103           Laboratory control sample         A S         20 .04 (Mg/L)         20 .0 (Mg/L)         20 .0         100         100         100           Matrix spike         -         (SSR-SR)         -         -         -         -         -           I CP serial dilution         -         -         -         -         -         -						Recalculated	Reported	
ICP interference check   As   103	Sample ID	Type of Analysis	Element	Found / S / I (units)	True / D / SDR (units)	%R/RPD/%D	%R/RPD/%D	Acceptable (Y/N)
Laboratory control sample         As         20.04 (mg/kg)         20.0 (mg/kg)         10.0 <td>1931 ICSAB</td> <td>ICP interference check</td> <td>As</td> <td></td> <td></td> <td></td> <td>103</td> <td>&gt;</td>	1931 ICSAB	ICP interference check	As				103	>
Matrix spike (SSR-SR) ( Duplicate	0248	Laboratory control sample	As	20.04 (mg/kg			001	->
Duplicate CP serial dilution	1	Matrix spike	1			_	(	l
ICP serial dilution	,	Duplicate	ì	- Control of the Cont			ı	
		ICP serial dilution	١	)	ą	1	-	

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

%. :

LDC#: 24047 A4

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	
Reviewer:	MG
2nd reviewer:	4

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

Please V N V N V N	N/A Have results N/A Are results w	ow for all questions answered "N". Not apple been reported and calculated correctly? within the calibrated range of the instrument tion limits below the CRDL?			
Detec equati	ted analyte results for _ on:	# 1, As	were recalcu	lated and verified	using the following
Concen	$tration = \frac{(RD)(FV)(Dil)}{(ID)(Dil)}$	Recalculation:			
RD TV n. Vol. Dil	(In. Vol.)  = Raw data conce  = Final volume (m  = Initial volume (m  = Dilution factor		175) =	= 5.06 Mg	fly or mg/k
#	Sample ID	Analyte	Reported Concentration (Mg/kg)	Calculated Concentration ( mg / kg )	Acceptable (Y/N)
1	1	As	5.9	5.9	Y
		·			
					:
					·
Note:_					

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 7 through August 9, 2010

LDC Report Date:

October 8, 2010

Matrix:

Soil/Water

Parameters:

Arsenic

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6223-1

#### Sample Identification

SSAJ3-02-12BPC SSAJ3-02-15BPC SSAJ3-02-8BPC\*\* SSAJ3-02-8BPC\_FD EB-08072010 SSAI3-04-11BPC SSAI3-04-14BPC\_FD SSAI3-02-11BPC SSAI3-02-14BPC SSAI3-02-5BPC\_FD SSAI3-03-11BPC SSAI3-03-14BPC

SSAI3-04-8BPC SSAJ3-02-12BPCMS SSAJ3-02-12BPCMSD

SSAI3-04-5BPC

SSAI3-03-8BPCMS SSAI3-03-8BPCMSD

SSAI3-02-5BPC

00/110 02 021 0

SSAI3-02-8BPC

SSAI3-03-1BPC

SSAI3-03-5BPC\*\*

SSAI3-03-8BPC

SSAI3-04-1BPC

SSAI3-02-1BPC

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

#### Introduction

This data review covers 25 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 6020 for Arsenic.

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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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 was found in the initial, continuing and preparation blanks.

Sample EB-08072010 was identified as an equipment blank. No arsenic was found in this blank.

#### 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) and relative percent differences (RPD) 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 280-6223-1	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 SSAJ3-02-8BPC\*\* and SSAJ3-02-8BPC\_FD, samples SSAI3-04-14BPC and SSAI3-04-14BPC\_FD, and samples SSAI3-02-5BPC\_FD and SSAI3-02-5BPC were identified as field duplicates. No arsenic was detected in any of the samples with the following exceptions:

	Concentration (mg/Kg)			D://			
Analyte	SSAJ3-02-8BPC**	SSAJ3-02-8BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P	
Arsenic	3.5	2.9	15 (≤50)	•	-	-	

	Concentration (mg/Kg)		222	D''			
Analyte	SSAI3-04-14BPC	SSAI3-04-14BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P	
Arsenic	3.5	3	15 (≤50)	-	-	-	

	Concentration (mg/Kg)				wii 1 / 1 .		
Analyte	SSAI3-02-5BPC_FD	SSAI3-02-5BPC	RPD (Limits)	Difference (Limits)	Flags	A or P	
Arsenic	2.5	3.1	21 (≤50)	•	<b>-</b>	•	

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic - Data Qualification Summary - SDG 280-6223-1

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-6223-1	SSAJ3-02-12BPC SSAJ3-02-15BPC SSAJ3-02-8BPC** SSAJ3-02-8BPC_FD EB-08072010 SSAI3-04-11BPC SSAI3-04-14BPC SSAI3-04-14BPC_FD SSAI3-02-11BPC SSAI3-02-11BPC SSAI3-02-14BPC SSAI3-02-14BPC SSAI3-02-14BPC SSAI3-03-14BPC SSAI3-03-14BPC SSAI3-03-18PC SSAI3-03-18PC SSAI3-03-18PC SSAI3-04-18PC SSAI3-04-8BPC SSAI3-04-8BPC SSAI3-04-8BPC SSAI3-04-8BPC	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (PQL) (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic - Laboratory Blank Data Qualification Summary - SDG 280-6223-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic - Equipment Blank Data Qualification Summary - SDG 280-6223-1

No Sample Data Qualified in this SDG

## **Tronox Northgate Henderson**

LDC #:	24047B4	VALIDATION COMPLETENESS WORKSHEE
SDG #:	280-6223-1	_ Stage 2B/4
Laborator	v. Test America	-

Date: 10-3-10 Page: \_\_\_of\_\_\_\_ Reviewer: MG 2nd Reviewer: \_\_\_\_\_\_

METHOD: As (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
I.	Technical holding times	A	Sampling dates: 8-7-10 through 8-9-10
11.	ICP/MS Tune	A	U
III.	Calibration	Α	
IV.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	A	
VI.	Matrix Spike Analysis	A	MS/MSD
VII.	Duplicate Sample Analysis	7	
VIII.	Laboratory Control Samples (LCS)	Α	LCS/LCS D
IX.	Internal Standard (ICP-MS)	Ä	
X.	Furnace Atomic Absorption QC	7	not utilized
XI.	ICP Serial Dilution	A	
XII.	Sample Result Verification	Α	Not reviewed for Stage 2B validation.
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	5W	D=3+4 D=7+8 D=11+15
xv	Field Blanks	ND	D=3+4, D=7+8, D=11+15 EB=5

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

D = Duplicate TB = Trip blank

FB = Field blank

EB = Equipment blank

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

	1 1	SSAJ3-02-12BPC	11	SSAI3-02-5BPC_FD	218	SSAI3-04-5BPC	31
	2 !	SSAJ3-02-15BPC	12	SSAI3-03-11BPC	22 2	SSAI3-04-8BPC	32
Ŋ	3 1	SSAJ3-02-8BPC <b>★</b> ¥	13 a	SSAI3-03-14BPC WH	23	SSAJ3-02-12BPCMS	33
	4	SSAJ3-02-8BPC_FD	م 14	SSAI3-02-1BPC	24 1	SSAJ3-02-12BPCMSD	34
	<sub>5</sub> 3	EB-08072010 W	15 15	SSAI3-02-5BPC	<sub>25</sub> 2	SSAI3-03-8BPCMS	35
	<sub>6</sub> !	SSAI3-04-11BPC	<b>ہ</b> 16	SSAI3-02-8BPC	<sub>26</sub> 2	SSAI3-03-8BPCMSD	36
	<sub>7</sub>	SSAI3-04-14BPC	17 <b>2</b>	SSAI3-03-1BPC	27		37
	8 l	SSAI3-04-14BPC_FD	<sub>18</sub> 2	SSAI3-03-5BPC**	28	PBS1	38
	9 1	SSAI3-02-11BPC	<sub>19</sub> 2	SSAI3-03-8BPC	29 <sup>2</sup>	PB52	39
	10 l	SSAI3-02-14BPC	202	SSAI3-04-1BPC	30 <sup>3</sup>	PBW	40

Notes:			

Method: Metais (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.	V			
II. ICP/MS Tune		······	<b></b>	
Were all isotopes in the tuning solution mass resolution within 0.1 amu?	<b>V</b>			
Were %RSD of isotopes in the tuning solution ≤5%?	<b>V</b>			
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.		V		
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.	/			
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?				
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 2
Reviewer: MG
2nd Reviewer: \_\_\_\_\_\_

Validation Area	Yes	No	NA	Findings/Comments			
VIII. Furnace Atomic Absorption QC							
If MSA was performed, was the correlation coefficients > 0.995?			1				
Do all applicable analysies have duplicate injections? (Level IV only)		_	1				
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)			/				
Were analytical spike recoveries within the 85-115% QC limits?		L	/				
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%?	V						
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)			r				
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?	1						
If the %Rs were outside the criteria, was a reanalysis performed?			<u> </u>				
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?			<u> </u>				
XIII. Overall assessment of data			<b>y</b>				
Overall assessment of data was found to be acceptable.	/						
XIV. Field duplicates							
Field duplicate pairs were identified in this SDG.	/						
Target analytes were detected in the field duplicates.	/						
XV. Field blanks							
Field blanks were identified in this SDG.	/						
Target analytes were detected in the field blanks.			<u> </u>				

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

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Reviewer:_	- T
2nd Reviewer:	$\overline{}$

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

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

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	3	4	RPD	Difference	Limits	(Parent Only)
Arsenic	3.5	2.9	15			

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	7	8	RPD	Difference	Limits	(Parent Only)
Arsenic	3.5	3.0	15			

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	11	15	RPD	Difference	Limits	(Parent Only)
Arsenic	2.5	3.1	21			

V:\FIELD DUPLICATES\FD\_inorganic\24047B4.wpd

LDC# 24047 B4

# Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: Reviewer:\_ 2nd 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 = 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)						
108 ICV	ICP/MS (Initial calibration)	As	41.61	40.0	104	h01	>
	CVAA (Initial calibration)						
	ICP (Continuing calibration)						
3008° CCV	ICP/MS (Continuing calibration)	As	50.66	50.0	1.0.1	101	
	CVAA (Continuing calibration)			2			
	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 # 2404784

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

2nd Reviewer: Page: / of 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

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 =  $1S-D1_{\odot} \times 100$ (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 = [I-SDR] x 100

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

-	<del></del>	<del></del>	<del>,</del>	<del></del>	1	
	Acceptable (Y/N)	<b>&gt;</b>				->
Reported	%R/RPD/%D	103	001	105	3	5.7
Recalculated	%R/RPD/%D	103	001	105	3	5.4
	(units)	(7) Pm)	(mg/kg)	(67/ Bun)	(mg/kg)	(mg/kg)
	True / D / SDR (units)	001	30.0	P. 4	31.66	3.70
	/1	(mg/1)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)
The control of the co	Found / S /   (units)	103.20	30.00	(SSR-SR)	33.36	3.51
	Element	As	As	As	As	As
	Type of Analysis	ICP interference check	Laboratory control sample	Matrix spike	Duplicate	ICP serial dilution
	Sample ID	1745 ICSAB	1946	1957 23	he/Et 1862/1868	b) 111e/801c

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#: 24047B4

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

Page:_	of
Reviewer:_	MG
2nd reviewer:	K
	<i>T</i>

METH	OD: Trace Metals (EPA	A SW 846 Method 6010/6020/7000)			,
Please YN YN YN	N/A Have results	ow for all questions answered "N". Not app been reported and calculated correctly? within the calibrated range of the instrumen tion limits below the CRDL?			
Detect equation	ed analyte results for _ on:	# 3 , As	were recalcu	llated and verified	using the following
	tration = $\frac{(RD)(FV)(Dil)}{(In. Vol.)}$	Recalculation: $(7.73  \mu g/L) (0.11)$	001)(5)	. 40/	
RD V n. Vol. Dil	<ul> <li>Raw data conce</li> <li>Final volume (m</li> <li>Initial volume (m</li> <li>Dilution factor</li> </ul>		0)	3.49 mg/g	or mg/kg
#	Sample ID	Analyte	Reported Concentration (Mg/kg)	Calculated Concentration ( Mg / kq )	Acceptable (Y/N)
ï	3	As	3,5	3.5	Y
		713		J.J.	
	-				
					•
Note:_					

# 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:

October 13, 2010

Matrix:

Soil

Parameters:

Arsenic

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6290-2

Sample Identification

SSAQ4-10-2BPC

SSAQ4-10-3BPC

SSAQ4-10-4BPC

#### Introduction

This data review covers 3 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Arsenic.

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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- 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 was found in the initial, continuing and preparation blanks.

No field blanks were identified 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

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

#### VII. Duplicate Sample Analysis

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.

#### 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.

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6290-2	All analytes reported below the PQL.	J (all detects)	Α

#### 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 Arsenic - Data Qualification Summary - SDG 280-6290-2

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-6290-2	SSAQ4-10-2BPC SSAQ4-10-3BPC SSAQ4-10-4BPC	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (PQL) (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic - Laboratory Blank Data Qualification Summary - SDG 280-6290-2

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic - Equipment Blank Data Qualification Summary - SDG 280-6290-2

No Sample Data Qualified in this SDG

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

LDC #:	24047C4	VALIDATION COMPLETE
SDG #:_	280-6290-2	_ Stage 2
Laborato	ry: Test America	_

Date:_ <i>[0-</i>	5-10
Page: <u>/</u> of	1
Reviewer: M	G
2nd Reviewer: 1	

METHOD: As (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: 8-9-10
11.	ICP/MS Tune	Α	
111.	Calibration	A	
IV.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	Α	
VI.	Matrix Spike Analysis	7	client specified
VII.	Duplicate Sample Analysis	7	to of
VIII.	Laboratory Control Samples (LCS)	Α	LCS
iX.	Internal Standard (ICP-MS)	Α	·
Χ.	Furnace Atomic Absorption QC	7	not utilized
XI.	ICP Serial Dilution	N	not utilized not performed
XII.	Sample Result Verification	N	
XIII.	Overall Assessment of Data	Ą	
XIV.	Field Duplicates	N	
ΧV	Field Blanks	N	EB= EB-08092010 (SDG: 280-6290-1)

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:

				<u> </u>
1	SSAQ4-10-2BPC	11	21	31
2	SSAQ4-10-3BPC	12	22	32
3	SSAQ4-10-4BPC	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	PBS	20	30	40

Notes:		

# 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:** 

October 8, 2010

Matrix:

Soil

Parameters:

Arsenic

Validation Level:

Stage 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6290-3

Sample Identification

SSAQ4-08-9BPC SSAQ4-08-9BPCMS SSAQ4-08-9BPCMSD

#### Introduction

This data review covers 3 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Arsenic.

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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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 was found in the initial, continuing and preparation blanks.

No field blanks were identified 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 with the following exceptions:

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P	
SSAQ4-08-9BPCMS/MSD (All samples in SDG 280-6290-3)	Arsenic	16 (75-125)	42 (75-125)	-	J- (all detects) UJ (all non-detects)	А	

#### 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.

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-6290-3	All analytes reported below the PQL.	J (all detects)	Α

#### 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 Arsenic - Data Qualification Summary - SDG 280-6290-3

SDG	Sample	Analyte	Flag	A or P	Reason (Code)			
280-6290-3	SSAQ4-08-9BPC	Arsenic	J- (all detects) UJ (all non-detects)	А	Matrix spike/Matrix spike duplicates (%R) (m)			
280-6290-3	SSAQ4-08-9BPC	All analytes reported below the PQL.	J (all detects)	Α	Sample result verification (PQL) (sp)			

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic - Laboratory Blank Data Qualification Summary - SDG 280-6290-3

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic - Equipment Blank Data Qualification Summary - SDG 280-6290-3

No Sample Data Qualified in this SDG

		1/4		N COME				ORKSHEET		Date: <u>l0 - 5 - /</u>
LDC #		VA	LIDATIO					Page: <u>Lof_L</u>		
SDG i Labor	#: 280-6290-3 atory: <u>Test America</u>	•			Stage a	ZD	4			Reviewer: MG
	IOD: As (EPA SW 846 N	- ⁄letho	d 6020)		an H					2nd Reviewer:
	amples listed below were tion findings worksheets		ewed for ea	ch of the f	followin	g va	lidation a	areas. Validatio	on find	dings are noted in attached
	Validation	Area								
<u> </u>	Technical holding times		.,	Α	Samplii	ng da	ates:	8-9-10		
II.	ICP/MS Tune			Α						
III.	Calibration			Α						
IV.	Blanks			A						
V.	ICP Interference Check Sai	nple (I	CS) Analysis	A						
VI.	Matrix Spike Analysis			SW	MS	/~	ISD			
VII.	Duplicate Sample Analysis			N						
VIII.	Laboratory Control Sample	Α	LC	S				·		
IX.				Α	•					
X.	C. Furnace Atomic Absorption QC			N	not	٠ ر	Hiliza	ed		
XI.	ICP Serial Dilution			Α						
XII.	Sample Result Verification			N						
XIII.	Overall Assessment of Data	<u>a</u>		Α						
XIV.	Field Duplicates			N						
ΧV	Field Blanks			M	FB	-	EB-01	010£P08	(	SDG: 280-6290-1)
Note:	A = Acceptable N = Not provided/applicable SW = See worksheet ed Samples: ,	<b>e</b>	R = Rin	o compound sate eld blank	ds detecte	ed	TB	Duplicate = Trip blank = Equipment blar	ık	
·	Soil	1	<u></u>							
1	SSAQ4-08-9BPC	11			2	21			31	
2	SSAQ4-08-9BPCMS	12			2	2			32	
3	SSAQ4-08-9BPCMSD	13			2	23			33	
4		14			2	24			34	
5		15			2	25			35	
6		16			2	26		·	36	
7		17			2	7			37	
8		18			2	28			38	

Notes:		-	

PBS

#### VALIDATION FINDINGS CHECKLIST

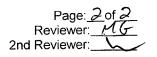
Page: 1 of 2 Reviewer: 4 2nd Reviewer: 4

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.	<u> </u>			
Cooler temperature criteria was met.	/			
II. ICP/MS Tune	<del></del>	<del></del> -	<del> </del>	
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?	1			
Were the proper number of standards used?	1			
Were all initial and continuing calibration verification %Rs within the 90-110% (80- 120% for mercury) QC limits?				
Were all initial calibration correlation coefficients > 0.995?	<b>/</b>		<u> </u>	
IV. Blanks		<u> </u>	т—	
Was a method blank associated with every sample in this SDG?			<b> </b>	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/		
V. ICP Interference Check Sample		<del></del>		
Were ICP interference check samples performed daily?	/		<u> </u>	
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?	/		<u> </u>	
VI. Matrix spike/Matrix spike duplicates			т	
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.		<b>/</b>		
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq$ 20% for waters and $\leq$ 35% for soil samples? A control limit of +/- 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.	1			
VII. Laboratory control samples		т	т—	
Was an LCS anaylzed for this SDG?	1		<del> </del>	
Was an LCS analyzed per extraction batch?	1	ļ	<del> </del>	
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?				

LDC#: 24047D4

#### **VALIDATION FINDINGS CHECKLIST**



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)			v.	
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)			/	
Were analytical spike recoveries within the 85-115% QC limits?	<u></u>		V	
IX. ICP Serial Dilution		<b>,,,,,,</b>	·	
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.		V		A STATE OF THE STA
X. Internal Standards (EPA SW 846 Method 6020/EPA 200.8)		<b></b>		
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?	1			
If the %Rs were outside the criteria, was a reanalysis performed?			/	
XI. Regional Quality Assurance and Quality Control		Y/-	•	
Were performance evaluation (PE) samples performed?		/	ļ,	
Were the performance evaluation (PE) samples within the acceptance limits?			/	
XII. Sample Result Verification	<b>,</b>		·	
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		
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.				

# 1DC#: 24047D4

# Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

Page: Reviewer: 2nd Reviewer:

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

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

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

of 4 or more, no action was taken.

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

N N/A WELEVEL IV ONLY:

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

		<del></del>	Т	T	Ī		Г				Γ				$\Box$
Qualifications	* J-/UJ/A m														
RPD (1 imits)   Associated Samples	a11														
RPD (Limits)															
MSD %Recovery	42 (15-125)					The management of the state of				•					
- 11 - 11	1														
Analyte															
Matrix	1:05														
MS/MSD ID	8/€														
#															

digestion spike is in limit tsod Comments:

LDC# 34047 D4

# Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: / of / 2nd Reviewer.\_ 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 = Found × 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)						
1910 ICV	ICP/MS (Initial calibration)	As	41.31	40.0	(03	(03	<b>\</b>
	CVAA (Initial calibration)						
	ICP (Continuing calibration)						
0842 CCV	ICP/MS (Continuing calibration)	As	51.09	50.0	ر02	(02	>
	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# 34047D4

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

Page: Reviewer: 2nd 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 =  $[S-D]_{\times} \times 100$ (S+D)/2

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

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

 $%D = [I-SDR] \times 100$ 

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 (units)	True / D / SDR (units)	%R/RPD/%D	%R/RPD/%D	Acceptable (Y/N)
3224 ICSAB	ICP interference check	As	101.40 (mg/L)	(7) Bm) 001	101	101	>
0951	Laboratory control sample	As	(mg/kg) H1.81	20.0 (mg/kg)	91	16	
0402 D	Matrix spike	As	(SSR-SR) (may/kg)	(9,3 (mg/k	9)	91	
0402/0405 2/6	Duplicate	As	48.43 (mg/kg)	54.06 (mg/kg)	-		
1580/ 1580	ICP serial dilution	As	45.39 (mg/kg)	45,97 (mg/kg	1.3	1.3	>

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# 34047D4

#### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	of
Reviewer:	MG
2nd reviewer:	

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

IC I II	OD: Trace M	elais (EPA	4 3VV 040 IVIE	11100 60 10/6	020//000)			
lease ON ON	<u>N/A</u> Hav N/A Are	∕e results results w	been reporte	d and calcul rated range	ated correctly? of the instrument		re identified as "N/A ear range of the IC	
etect quati	ed analyte re	sults for _	华1,	As		were recalc	ulated and verified	using the following
oncen D V , Vol.	(In. = Raw = Fina = Initia	o)(FV)(Dil) Vol.) data conce I volume (m Il volume (m ion factor	ntration I) II) or weight (G)	(89.32	Recalculation:  Mg/L)(0  g)(0.	.100 L)(5) 911 )	= 45.39 M	g/g or mg/k
#	Sample	i <b>ID</b>		Analyte		Reported Concentration (mg/kg)	Calculated Concentration ( <sup>MQ</sup> /kq)	Acceptable (Y/N)
,	ı			As		45	45	
ote:_								

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 10, 2010

LDC Report Date:

October 13, 2010

Matrix:

Soil

Parameters:

Metals

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6345-1

#### Sample Identification

SSAJ3-07-SW-E-1BPC\*\*

SB01-24BPCMSD

SSAJ3-05-SW-E-1BPC

SSAJ3-02-SW-E-1BPC

SSAI3-04-SW-E-1BPC

SSAI3-03-SW-E-1BPC

SSAI3-02-SW-E-1BPC FD

SSAI3-02-SW-E-1BPC

SSAI3-02-SW-W-1BPC

SSAI3-03-SW-W-1BPC

SSAI3-04-SW-W-1BPC

SSAJ3-02-SW-W-1BPC

SSAJ3-05-SW-W-1BPC

SSAJ3-07-SW-W-1BPC\*\*

SSAJ3-07-SW-W-1BPC FD

**SB01-24BPC** 

SB02-24BPC\*\*

SB01-24BPC FD

SSAJ3-02-SW-W-1BPCMS

SSAJ3-02-SW-W-1BPCMSD

SB01-24BPCMS

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

#### Introduction

This data review covers 21 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Methods 6010, 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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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.

3

### 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	SB01-24BPC SB02-24BPC**
ICB/CCB	Antimony	3.42 ug/L	SB01-24BPC SB02-24BPC**
ICB/CCB	Selenium	5.55 ug/L	SB01-24BPC SB02-24BPC** SB01-24BPC_FD

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
SB02-24BPC**	Antimony	0.56 mg/Kg	2.0U mg/Kg

No field blanks were identified 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 280-6345-1	All analytes reported below the PQL.	J (all detects)	A

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 SSAI3-02-SW-E-1BPC\_FD and SSAI3-02-SW-E-1BPC, samples SSAJ3-07-SW-W-1BPC\*\* and SSAJ3-07-SW-W-1BPC\_FD, and samples SB01-24BPC and SB01-24BPC\_FD were identified as field duplicates. No metals were detected in any of the samples with the following exceptions:

	Concentration	on (mg/Kg)					
Analyte	SSAI3-02-SW-E-1BPC_FD	SSAI3-02-SW-E-1BPC	RPD (Limits)	Difference (Limits)	Flags	A or P	
Arsenic 2.8		2.7	-	0.1 (≤0.57)	-		

	Concentrati	on (mg/Kg)					
Analyte	SSAJ3-07-SW-W-1BPC**	SSAJ3-07-SW-W-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P	
Arsenic 2.6		3.1	-	0.5 (≤0.61)	-	-	

	Concentrati	RPD	p:#			
Analyte	SB01-24BPC SB01-24BPC_FD			Difference (Limits)	Flags	A or P
Arsenic	6.6	6.6	-	0 (≤1.9)	. <b>-</b>	-
Barium	130	120 8 (≤50)		-	-	-
Cadmium	0.043	0.055	-	0.012 (≤0.48)	-	<u>-</u>
Chromium	12	14	15 (≤50)	-	-	-
Lead	7.4	7.3	1 (≤50)	-	-	-

	Concentrati	on (ug/Kg)				A or P	
Analyte	SB01-24BPC	SB01-24BPC_FD	RPD (Limits)	Difference (Limits)	Flags		
Mercury	6.3	7.2	-	0.9 (≤18)	-	-	

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Metals - Data Qualification Summary - SDG 280-6345-1

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-6345-1	SSAJ3-07-SW-E-1BPC** SSAJ3-05-SW-E-1BPC SSAJ3-02-SW-E-1BPC SSAI3-04-SW-E-1BPC SSAI3-03-SW-E-1BPC SSAI3-02-SW-E-1BPC SSAI3-02-SW-E-1BPC SSAI3-02-SW-W-1BPC SSAI3-03-SW-W-1BPC SSAJ3-05-SW-W-1BPC SSAJ3-07-SW-W-1BPC SSAJ3-07-SW-W-1BPC SSAJ3-07-SW-W-1BPC** SSAJ3-07-SW-W-1BPC-FD SB01-24BPC SB01-24BPC SB01-24BPC_FD	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 280-6345-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Metals - Equipment Blank Data Qualification Summary - SDG 280-6345-1

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson** T

_DC #: <u>24047E4</u>	VALIDATION COMPLETENESS WORKSHEE
SDG #: 280-6345-1	
aboratory: Test America	Stage 2B/4

Date: 10-6-10 Page:\_\_\_of\_\_l\_ Reviewer: MG 2nd Reviewer:

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

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: 8-10-10
11.	ICP/MS Tune	A	
111.	Calibration	Α	
IV.	Blanks	Sw	
V.	ICP Interference Check Sample (ICS) Analysis	Ą	
VI.	Matrix Spike Analysis	A	MS/MSD
VII.	Duplicate Sample Analysis	7	
VIII.	Laboratory Control Samples (LCS)	A	LCS
IX.	Internal Standard (ICP-MS)	A	
Χ.	Furnace Atomic Absorption QC	7	not utilized
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	D=6+7 D=13+14 D=15+17
ΧV	Field Blanks	7	EB= EB-08102010 (from 280-6290-1)

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

	<u> </u>						
1 1	SSAJ3-07-SW-E-1BPC**	11 1	SSAJ3-02-SW-W-1BPC	212	SB01-24BPCMSD	31	
2 1	SSAJ3-05-SW-E-1BPC	12	SSAJ3-05-SW-W-1BPC	22		32	
3 l	SSAJ3-02-SW-E-1BPC	13 t	SSAJ3-07-SW-W-1BPC **	23	an H	33	
4 1	SSAI3-04-SW-E-1BPC	14	SSAJ3-07-SW-W-1BPC_FD	24	1	34	
5	SSAI3-03-SW-E-1BPC	15 2	SB01-24BPC	25		35	
6 1	SSAI3-02-SW-E-1BPC_FD	<sub>16</sub> ユ	SB02-24BPC**	26		36	
7	SSAI3-02-SW-E-1BPC	172	SB01-24BPC_FD	27		37	
8	SSAI3-02-SW-W-1BPC	18 (	SSAJ3-02-SW-W-1BPCMS	28		38	
9 1	SSAI3-03-SW-W-1BPC	19 [	SSAJ3-02-SW-W-1BPCMSD	29 (	PBSI	39	
10	SSAIØ3-04-SW-W-1BPC	202	SB01-24BPCMS	30 ð	PBS2	40	

Notes:	

LDC #: 24047E4

### VALIDATION FINDINGS CHECKLIST

Page: of 2
Reviewer: MG
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.	/			
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?	$\checkmark$			
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?	<b>√</b> .			
Were all initial calibration correlation coefficients > 0.995?	V			
IV. Blanks				
Was a method blank associated with every sample in this SDG?	<b>√</b>			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	/			
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.	<b>√</b>			
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq$ 20% for waters and $\leq$ 35% for soil samples? A control limit of +/- 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.	1			
VII. Laboratory control samples				
Was an LCS anaylzed for this SDG?	/			
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?				

LDC#: 24047E4

### VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
Reviewer: MG
2nd Reviewer: V

Validation Area	Yes	No	NA	Findings/Comments
VIII. Furnace Atomic Absorption QC				
If MSA was performed, was the correlation coefficients > 0.995?			V	
Do all applicable analysies have duplicate injections? (Level IV only)			/	
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)			<b>\</b>	
Were analytical spike recoveries within the 85-115% QC limits?			V	
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%?	<b>✓</b>			
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?			<b>/</b>	
XI. Regional Quality Assurance and Quality Control			·	
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.				
Target analytes were detected in the field duplicates.	/			
XV. Field blanks	,,,,		,	
Field blanks were identified in this SDG.				
Target analytes were detected in the field blanks.				

LDC #: <u>24047</u>E4

### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page:_	of
Reviewer:	MG
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All circled elements are applicable to each sample.

I	1	
Sample ID	Matrix	Target Analyte List (TAL)
1->14	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,
15,16		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',
17		Al, Sb, As, Ba) Be, Cd) Ca, Cr) Co, Cu, Fe, Pb) Mg, Mn, Hg, Ni, K, Ee, Ag) Na, Tl, V, Zn, Mo, B, Si, CN,
QC 18, 19		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> ,
\$ 20,21		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,
		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 <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
		Analysis Method
ICP	S	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg (Ni) K, Se, Ag Na, (1, 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 <sup>-</sup> ,
GFAA		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> ,

Comments: Mercury by CVAA if performed	

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VALIDATION FINDINGS WORKSHEET PB/ICB/CCB QUALIFIED SAMPLES Soil preparation factor applied: 100x Associated Samples: 15, 16 METHOD: Trace metals (EPA SW 864 Method 6010B/6020/7000) Sample Concentration units, unless otherwise noted: mg/Kg

Code: bl

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				0E										
Analyte		Maximum PB <sup>a</sup> (ug/L)	Maximum Maximum Maximum PB³ ICB/CCB³ (mg/Kg) (ug/L) (ug/L)	Action Limit	16									
Sb		-	3.42		0.56/ 2.0U									
Zn	0.430													
METHOD:	<b>METHOD</b> : Trace metals (EPA SW 864 Method 6010B/6020/7000)	(EPA SW 86	64 Method 60	)10B/6020/7(		Soil preparation factor applied: 100x	factor applie	ed: 100x	1	-		2nd Reviewer:	iewer:	

Soil preparation factor applied: 100x METHOD: Trace metals (EPA SW 864 Method 6010B/6020/7000) Sample Concentration units, unless otherwise noted: mg/Kg

Associated Samples: 15-17 (ND)

Analyte		Maximum Maximum Maximum PB³ ICB/CCB³ (ug/L) (ug/L)	Maximum ICB/CCB <sup>a</sup> (ug/L)	Action Limit	Action No Qual's.									
Se			5.55											
Samples with qualified as n	Samples with analyte concent qualified as not detected. "U"	entrations withi	Samples with analyte concentrations within five times the associated ICB, CCB or PB coqualified as not defected. "U".	associated IC	B, CCB or PB	concentration a	re listed above	oncentration are listed above with the identifications from the Validation Completeness Worksheet. These sample results were	fications from	the Validation	Completenes	ss Worksheet.	These sample	e results were

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

### LDC#: 24047E4

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

	Page:_	1	_of	
	Reviewer	:_	MG	·
2nd l	Reviewer	:_	_\_	_

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

**N NA** 

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

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	6	7	RPD	Difference	Limits	(Parent Only)
Arsenic	2.8	2.7		0.1	(≤0.57)	

V:\FIELD DUPLICATES\FD\_inorganic\24047E4.wpd

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	13	14	RPD	Difference	Limits	(Parent Only)
Arsenic	2.6	3.1		0.5	(≤0.61)	

V:\FIELD DUPLICATES\FD\_inorganic\24047E4.wpd

	Concentration (mg/kg)		(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	15	17	RPD	Difference	Limits	(Parent Only)
Arsenic	6.6	6.6		0	(≤1.9)	
Barium	130	120	8			
Cadmium	0.043	0.055		0.012	· (≤0.48)	
Chromium	12	14	15			
Lead	7.4	7.3	1			
Mercury (ug/Kg)	6.3	7.2		0.9	(≤18)	

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LDC# SHOUTEY

# VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

Page: Lof L Reviewer: MG 2nd Reviewer: L

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:

 $%R = Found \times 100$ True

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

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					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
	ICP (Initial calibration)						
1830 ICV	ICP/MS (Initial calibration)	As	40.54	40.0	101	10/	\ \
1134 ICV	CVAA (Initial calibration)	Hg	499.9	7.00	95	36	
	ICP (Continuing calibration)	ס					
1857 CCV	ICP/MS (Continuing calibration)	As	50.13	50.0	00/	00/	
722 0h11	CVAA (Continuing calibration)	Hg	440.3	5.00	101	101	>
	GFAA (Initial calibration)	D					
	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# 34047E4

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

Page: Lof Reviewer: 2nd 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

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).

Concentration of each analyte in the source. True =

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 = [I-SDR] x 100

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

	Acceptable (Y/N)	<b>&gt;</b>				>
Reported	%R/RPD/%D	9.66	007	92	0	4.9
Recalculated	%R/RPD/%D	94.6	(00)	42	0	1.9
	(units)	(7/8m)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)
	True / D / SDR (units)	(7/8m) 001 (7/8m)	30.0	19.3	21.07 (mg/kg	3.34 (mg/kg
	/1	(7/ Bm)	(mg/kg)	R) (mg/kg)	.98 (mg/kg)	.13 (mg/kg)
	Found / S / I (units)	99.61	30.03	(SSR-SR)	30.98	3.13
	Element	As	As	As	As	As
	Type of Analysis	ICP interference check	Laboratory control sample	Matrix spike	Duplicate	ICP serial dilution
	Sample ID	1846 ICSAB	1408 LCS 1	6000	110€/200€	1)

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

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LDC#: 34047E4

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

Page:_	of
Reviewer:_	MG
2nd reviewer:_	$\rightarrow \sim$

METH	OD: Trace Metals (EPA	A SW 846 Method 6010/6020/7000)			•
Please Y)N Y)N Y)N	N/A Have results N/A Are results w	ow for all questions answered "N". Not app been reported and calculated correctly? vithin the calibrated range of the instrument tion limits below the CRDL?			
Detect equati	ed analyte results for _ on:	# 1, As	were recalcu	lated and verified	using the following
•	tration = $\frac{(RD)(FV)(Dil)}{(In. Voi.)}$	Recalculation:	100 /(=)		
RD TV n. Vol. Dil	<ul> <li>Raw data conce</li> <li>Final volume (m</li> <li>Initial volume (m</li> <li>Dilution factor</li> </ul>		950)	= 3.148 M	ig/ or mg/k
#	Sample ID	Analyte	Reported Concentration (mg/kg)	Calculated Concentration ( Mg / K4)	Acceptable (Y/N)
1	l	As	3.1	3.1	Y
2	16	Hg	350 (Mg/kg)	350 (Mg/kg)	<u> </u>
		V	V	V	
				-	
		·			
Note:_					

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 17, 2010

LDC Report Date:

October 8, 2010

Matrix:

Soil

Parameters:

Arsenic & Manganese

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6583-1

### Sample Identification

BDT-4-N-15-16.0BPC

BDT-4-N-15-18.0BPC\*\*

BDT-4-N-15-12.0BPC

BDT-4-N-15-14.0BPC

BDT-4-N-15-8.0BPC

BDT-4-N-15-10.0BPC

BDT-4-N-20-10.0BPC

BDT-4-N-20-12.0BPC

BDT-4-N-20-14.0BPC

BDT-4-N-20-16.0BPC

BDT-4-N-20-18.0BPC\*\*

BDT-4-N-20-2.0BPC

BDT-4-N-20-4.0BPC

BDT-4-N-20-6.0BPC

BDT-4-N-20-8.0BPC

BDT-4-N-20-6.0BPC FD

BDT-4-N-15-16.0BPCMS

BDT-4-N-15-16.0BPCMSD

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

### Introduction

This data review covers 18 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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 was found in the initial, continuing and preparation blanks with the following exceptions:

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
PB (prep blank)	Manganese	0.0497 mg/Kg	All samples in SDG 280-6583-1

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

No field blanks were identified 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) and relative percent differences (RPD) 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 280-6583-1	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 BDT-4-N-20-6.0BPC and BDT-4-N-20-6.0BPC\_FD were identified as field duplicates. No arsenic or manganese was detected in any of the samples with the following exceptions:

	Concentration (mg/Kg)		555	<b>5</b> :4		
Analyte	BDT-4-N-20-6,0BPC	BDT-4-N-20-6.0BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	2.9	2.9	-	0 (≤0.65)	. <del>-</del>	-
Manganese	330	380	14 (≤50)	-	-	-

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic & Manganese - Data Qualification Summary - SDG 280-6583-1

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-6583-1	BDT-4-N-15-16.0BPC BDT-4-N-15-18.0BPC** BDT-4-N-15-12.0BPC BDT-4-N-15-14.0BPC BDT-4-N-15-10.0BPC BDT-4-N-20-10.0BPC BDT-4-N-20-12.0BPC BDT-4-N-20-16.0BPC BDT-4-N-20-18.0BPC** BDT-4-N-20-18.0BPC** BDT-4-N-20-18.0BPC** BDT-4-N-20-4.0BPC BDT-4-N-20-4.0BPC BDT-4-N-20-6.0BPC BDT-4-N-20-6.0BPC BDT-4-N-20-8.0BPC	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (PQL) (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic & Manganese - Laboratory Blank Data Qualification Summary - SDG 280-6583-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic & Manganese - Field Blank Data Qualification Summary - SDG 280-6583-1

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson** SHEET

LDC #:_	24047G4	VALIDATION COMPLETENESS WORKS
SDG #:_	280-6583-1	_ Stage 2B/4
Laborato	ory: Test America	

Date: 10- 1- 1	(
Page: <u>1_</u> of_ <u>1</u>	
Page: 1 of 1 Reviewer: MG	
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
I.	Technical holding times	Α	Sampling dates: 8-17-10
11.	ICP/MS Tune	A	
111.	Calibration	Α	
IV.	Blanks	SW	
V.	ICP Interference Check Sample (ICS) Analysis	Α	
VI.	Matrix Spike Analysis	Α	MS/MSD (Mn To rec /4x rule, no qual.)
VII.	Duplicate Sample Analysis	N	, , ,
VIII.	Laboratory Control Samples (LCS)	Α	LCS
IX.	Internal Standard (ICP-MS)	A	
X.	Furnace Atomic Absorption QC	7	not utilized
XI.	ICP Serial Dilution	A	
XII.	Sample Result Verification	Α	Not reviewed for Stage 2B validation.
XIII.	Overall Assessment of Data	Α	
XIV.	Field Duplicates	5 W	D=14+16
χV	Field Blanks	N	

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

TB = Trip blank

EB = Equipment blank

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

	all soil	•			
1	BDT-4-N-15-16.0BPC	11	BDT-4-N-20-18.0BPC**	21	31
2	BDT-4-N-15-18.0BPC**	12	BDT-4-N-20-2.0BPC	22	32
3	BDT-4-N-15-12.0BPC	13	BDT-4-N-20-4.0BPC	23	33
4	BDT-4-N-15-14.0BPC	14	BDT-4-N-20-6.0BPC	24	34
5	BDT-4-N-15-8.0BPC	15	BDT-4-N-20-8.0BPC	25	35
6	BDT-4-N-15-10.0BPC	16	BDT-4-N-20-6.0BPC_FD	26	36
7	BDT-4-N-20-10.0BPC	17	BDT-4-N-15-16.0BPCMS	27	37
8	BDT-4-N-20-12.0BPC	18	BDT-4-N-15-16.0BPCMSD	28	38
9	BDT-4-N-20-14.0BPC	19		29	39
10	BDT-4-N-20-16.0BPC	20	PBS	30	40

Notes:				
				_

Page: 1 of 2
Reviewer: MG
2nd Reviewer: L

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

metrodimetals (El 7/ evv e-le metrod de l'estresses)			<u> </u>	
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times	······		·	
All technical holding times were met.	/			
Cooler temperature criteria was met.	/		L	
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%?	/		L	
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?			<u> </u>	
IV. Blanks				
Was a method blank associated with every sample in this SDG?	<b>/</b>			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
V. ICP Interference Check Sample				
Were ICP interference check samples performed daily?	<b>V</b>			
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?	<u> </u>			
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?	/			

LDC#: 2404764

### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 2 Reviewer: MG 2nd Reviewer: V

Validation Area	Yes	No	NA	Findings/Comments		
VIII. Furnace Atomic Absorption QC						
If MSA was performed, was the correlation coefficients > 0.995?			1			
Do all applicable analysies have duplicate injections? (Level IV only)			/			
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)			<b>V</b>			
Were analytical spike recoveries within the 85-115% QC limits?			<u> </u>			
IX. ICP Serial Dilution		, <u></u>	·			
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%?	1					
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.		<b>✓</b>				
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?			V			
XI. Regional Quality Assurance and Quality Control						
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.	/					
Target analytes were detected in the field duplicates.	/			-		
XV. Field blanks						
Field blanks were identified in this SDG.		/				
Target analytes were detected in the field blanks.			V			

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### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page:_	of
Reviewer:	
2nd reviewer:	

All circled elements are applicable to each sample.

Sample ID	Matrix	Target Analyte List (TAL)
1-716	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,
ac 17, 18	↓ I	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 <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 <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 <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 <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> ,
	<del></del>	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 <sup>-</sup> ,
ICP-MS	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,
GEAA		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN

Comments:	Mercury by CVAA if performed	

LDC #: 24047G4

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

Page: 1 of Reviewer: M3

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Soil preparation factor applied: 100x Associated Samples: All (>RL)

No Qual's. Action Limit Maximum ICB/CCB<sup>a</sup> (ng/L) Maximum PB<sup>a</sup> (ug/L) Maximum PB<sup>a</sup> (mg/Kg) 0.0497 Analyte ₹

Samples with analyte concentrations within five times the associated ICB, CCB or PB concentration are listed above with the identifications from the Validation Completeness Worksheet. These sample results were qualified as not detected, "U".

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

LDC#: 24047G4

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

Page:_	_of
Reviewer:	MG
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METHOD: Metals (EPA Method 6010B/6020/7000)

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

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	14	16	RPD	Difference	Limits	(Parent Only)
Arsenic	2.9	2.9		0	(≤0.65)	
Manganese	330	380	14			

V:\FIELD DUPLICATES\FD\_inorganic\24047G4.wpd

LDC# 2404764

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

Page: Reviewer. 2nd 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 = Found x 100

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

Where,

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
	ICP (Initial calibration)						
1857 TCV	ICP/MS (Initial calibration)	As	41.60	40.0	h01	h01	<b>&gt;</b>
	CVAA (Initial calibration)						
	ICP (Continuing calibration)		·				
3336 CC V	ICP/MS (Continuing calibration)	Mn	49.37	50.0	66	99	<b>—</b>
·	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# 3404764

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

Page: Lof Reviewer: 2nd 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

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:

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

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

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

%D = |-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 / S / I (units)	True / D / SDR (units)	%R/RPD/%D	%R/RPD/%D	Acceptable (Y/N)
7303 ICSAB	ICP interference check	AS	(7/8m) OH. EO1	(7/8m) 001 (	(03	103	<u> </u>
2319 LCS	Laboratory control sample	M	19.50 (mg/kg)	) 20.0 (mg/kg)	98	47	
L1	Matrix spike	As	(SSR-SR) (mg/kg)	(px/8m) 7.16 (	16	-	
3830/2333	Duplicate	Mn	315.34 (mg/kg	(mg/kg) 310.17 (mg/kg)	ત	R	
) 3687 / 7080	ICP serial dilution	N N	270.61 (mg/kg	(mg/kg) 275.67 (mg/kg)	6,1	6.1	>

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

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LDC#: 2404764

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:(	of
Reviewer:	MG
2nd reviewer:	1~

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

Y) N Y) N Y) N	N N/A   Have results been reported and calculated correctly?   N N/A   Are results within the calibrated range of the instruments and within the linear range of the ICP?   N N/A   Are all detection limits below the CRDL?							
Detec equati	ted analyte results for _ on:	# 2, As	were recalcu	ulated and verified	using the following			
Concen RD FV In. Vol. Dil	tration = \frac{(RD)(FV)(Dil)}{(ln. Vol.)}  = Raw data conce = Final volume (m = Initial volume (m = Dilution factor	1)	100 L)(5)	_ = 5.87	ng/g or mg,			
#	Sample ID	Analyte	Reported Concentration ( <sup>wn</sup> q / kq)	Calculated Concentration (mg/kg)	Acceptable (Y/N)			
1	2	As	5.9	5.9	Ý			
		Mn	780.	380	. ↓			
Note:_								

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

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 24, 2010

LDC Report Date:

October 8, 2010

Matrix:

Soil/Water

Parameters:

Arsenic & Manganese

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6783-1

### Sample Identification

EB-08242010

BDT-4-N-5-8BPCMS BDT-4-N-5-8BPCMSD

BDT-4-S-5-10BPC

BDT-4-S-5-12BPC

BDT-4-S-5-14BPC

0-1-4-0-0-1-01

BDT-4-S-5-16BPC

BDT-4-S-5-18BPC BDT-4-S-5-2BPC

BDT-4-S-5-4BPC

DD 1-4-3-3-4DF C

BDT-4-S-5-6BPC

BDT-4-S-5-8BPC BDT-4-S-5-16BPC FD

BDT-4-N-5-10BPC

BDT-4-N-5-12BPC

BDT-4-N-5-14BPC

BDT-4-N-5-16BPC

BDT-4-N-5-18BPC\*\*

BDT-4-N-5-2BPC

BDT-4-N-5-4BPC

BDT-4-N-5-6BPC

BDT-4-N-5-8BPC

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

### Introduction

This data review covers 21 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 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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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 was found in the initial, continuing and preparation blanks with the following exceptions:

Method Blank ID	Analyte	Maximum Concentration	Associated Samples	
PB (prep blank)	Manganese	0.0434 mg/Kg	All samples in SDG 280-6783-1	

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

Sample EB-08242010 was identified as an equipment blank. No arsenic or manganese was found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Analyte	Concentration	Associated Samples	
EB-08242010	8/24/10	Manganese	12 ug/L	All samples in SDG 280-6783-1	

Sample concentrations were compared to concentrations detected in the equipment 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) and relative percent differences (RPD) 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 280-6783-1	All analytes reported below the PQL.	J (all detects)	A

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-5-16BPC and BDT-4-S-5-16BPC\_FD were identified as field duplicates. No arsenic or mangnese was detected in any of the samples with the following exceptions:

	Concentration (mg/Kg)					
Analyte	BDT-4-S-5-16BPC	BDT-4-S-5-16BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	4.3	4.2	2 (≤50)	-	. <u>.</u>	-
Manganese	360	340	6 (≤50)	-	•	-

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic & Manganese - Data Qualification Summary - SDG 280-6783-1

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-6783-1	EB-08242010 BDT-4-S-5-10BPC BDT-4-S-5-12BPC BDT-4-S-5-14BPC BDT-4-S-5-16BPC BDT-4-S-5-18BPC BDT-4-S-5-2BPC BDT-4-S-5-8BPC BDT-4-S-5-8BPC BDT-4-S-5-16BPC_FD BDT-4-N-5-10BPC BDT-4-N-5-14BPC BDT-4-N-5-16BPC BDT-4-N-5-16BPC BDT-4-N-5-18BPC** BDT-4-N-5-18BPC** BDT-4-N-5-18BPC BDT-4-N-5-8BPC BDT-4-N-5-8BPC BDT-4-N-5-8BPC BDT-4-N-5-8BPC BDT-4-N-5-8BPC	All analytes reported below the PQL.	J (all detects)	<b>A</b>	Sample result verification (PQL) (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic & Manganese - Laboratory Blank Data Qualification Summary - SDG 280-6783-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic & Manganese - Equipment Blank Data Qualification Summary - SDG 280-6783-1

No Sample Data Qualified in this SDG

#### **Tronox Northqate Henderson** T

LDC #:	24047K4	VALIDATION COMPLETENESS WORKSHEE
SDG #:	280-6783-1	Stage 2B/4
Laborator	y: Test America	<del>-</del>

Date: 10-8-10 Page: 1 of 1 Reviewer: MG 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
I.	Technical holding times	A	Sampling dates: 8-24-10
11.	ICP/MS Tune	A	
111.	Calibration	Α	
IV.	Blanks	SW	
V.	ICP Interference Check Sample (ICS) Analysis	Α	
VI.	Matrix Spike Analysis	A	MS/MSD (Mn% rec/4x, ox)
VII.	Duplicate Sample Analysis	7	
VIII.	Laboratory Control Samples (LCS)	Α	LCS/LCSD
IX.	Internal Standard (ICP-MS)	Α	
X.	Furnace Atomic Absorption QC	7	not utilized
XI.	ICP Serial Dilution	A	
XII.	Sample Result Verification	Α	Not reviewed for Stage 2B validation.
XIII.	Overall Assessment of Data	Α	
XIV.	Field Duplicates	SW	D = 5 + 11
×	Field Blanks	SW	EB=1

Note:

A = Acceptable

N = Not provided/applicable SW = See worksheet

ND = No compounds detected

R = Rinsate

D = Duplicate

TB = Trip blank

FB = Field blank

EB = Equipment blank

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

	<sub>1</sub>	EB-08242010	W	11	BDT-4-S-5-16BPC_FD	21	1 B	BDT-4-N-5-8BPCMS	s	31
	2	BDT-4-S-5-10BPC	S	12	BDT-4-N-5-10BPC	22	2 B	BDT-4-N-5-8BPCMSD	J	32
	3	BDT-4-S-5-12BPC	1	13	BDT-4-N-5-12BPC	23	3			33
8	4	BDT-4-S-5-14BPC***		14	BDT-4-N-5-14BPC	24	4			34
	5	BDT-4-S-5-16BPC		15	BDT-4-N-5-16BPC	25	5			35
	6	BDT-4-S-5-18BPC		16	BDT-4-N-5-18BPC**	26	3			36
	7	BDT-4-S-5-2BPC		17	BDT-4-N-5-2BPC	27	7			37
	88	BDT-4-S-5-4BPC		18	BDT-4-N-5-4BPC	28	3			38
	9	BDT-4-S-5-6BPC		19	BDT-4-N-5-6BPC	29	91	PBW		39
l	10	BDT-4-S-5-8BPC	l	20	BDT-4-N-5-8BPC	30	2	PBS		40

Notes:					
_					

#### VALIDATION FINDINGS CHECKLIST

Page: L of 2
Reviewer: MG
2nd Reviewer: V

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

IVIETIOU: IVIETAIS (EPA SVV 646 IVIETIOU 60 100/1000/0020)	Wetifod. Metals (EFA SW 846 Metalod 66 168/1666/3025)								
Validation Area	Yes	No	NA	Findings/Comments					
I. Technical holding times	·	······································							
All technical holding times were met.	/								
Cooler temperature criteria was met.	/								
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%?	/								
	L	L							
III. Calibration	/		<u> </u>						
Were all instruments calibrated daily, each set-up time?									
Were the proper number of standards used?			ļ						
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?	<b>V</b>								
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	/								
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									
	l .	l							
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	1								
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.	/								
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	T		·						
Was an LCS anaylzed for this SDG?	/		<u> </u>						
Was an LCS analyzed per extraction batch?	1								
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?	/		٠	·					

LDC#: 24047K4

#### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 2
Reviewer: MG
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Validation Area	Yes	No	NA	Findings/Comments
VIII. Furnace Atomic Absorption QC			<u> </u>	
If MSA was performed, was the correlation coefficients > 0.995?			/	
Do all applicable analysies have duplicate injections? (Level IV only)			<u>/</u>	
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%?	<u> </u>			
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?			V	
XI. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?		/	ļ	
Were the performance evaluation (PE) samples within the acceptance limits?			/	
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				
Field duplicate pairs were identified in this SDG.	/			
Target analytes were detected in the field duplicates.	/			
XV. Field blanks	····	···		
Field blanks were identified in this SDG.	/			
Target analytes were detected in the field blanks.	/		<u> </u>	

LDC #: 24047K4

#### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page:_	<u>l_</u> of
Reviewer:	MG
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All circled elements are applicable to each sample.

Sample ID	Matrix	Target Analyte List (TAL)
1→20	s/w	Al, Sb, As) Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, (n) Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
OC 21,22	S	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-,
		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, Ti, 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 <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 <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,
		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,
<u> </u>	- II	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	S/W	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,
GFAA		Al Sh As Ba Be Cd Ca Cr Co Cu Fe Ph Mg Mn Hg Ni K Se Ag Na Tl V 7n Mo B Si CN

Comments:	Mercury by CVAA if performed	

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VALIDATION FINDINGS WORKSHEET PB/ICB/CCB QUALIFIED SAMPLES

Page: \_\_\_of\_\_ Reviewer: MG

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Soil preparation factor applied: 100x Associated Samples: All soil (>RL) METHOD: Trace metals (EPA SW 864 Method 6010B/6020/7000)

Sample Concentration units, unless otherwise noted: mg/Kg

No Qual's. Action Limit Maximum ICB/CCB<sup>a</sup> (ng/L) Maximum PB<sup>a</sup> (ng/L) Maximum PB<sup>a</sup> (mg/Kg) 0.0434 Analyte

Samples with analyte concentrations within five times the associated ICB, CCB or PB concentration are listed above with the identifications from the Validation Completeness Worksheet. These sample results were

qualified as not detected, "U". Note: a - The listed analyte concentration is the highest ICB, CCB, or PB detected in the analysis of each element.

LDC #: 24047K4

# VALIDATION FINDINGS WORKSHEET Field Blanks

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

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

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

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

Associated sample units: mg/Kg Blank units: ug/L Sampling date: 8-24-10

All soil (>10x) Associated Samples: Sample Identification EB Sampling date: 8-24-10 Soil factor applied 100x Field blank type: (circle one) Field Blank / Rinsate / Other No Qual's. Action Level 12 Blank ID 7 Analyte Ę

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

LDC#: 24047K4

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

Page:	of <i>_</i> _
Reviewer:	MG
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METHOD: Metals (EPA Method 6010B/6020/7000)

YN NA YN NA

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

	Concentration (mg/kg)		(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	5	11	RPD	Difference	Limits	(Parent Only)
Arsenic	4.3	4.2	2			
Manganese	360	340	6			

V:\FIELD DUPLICATES\FD\_inorganic\24047K4.wpd

LDC# 24047 KY

# Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: Lot L eviewer: MG Reviewer: 2nd 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 = Found x 100 True

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

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
X	ICP (Initial calibration)						
1832 ICV	ICP/MS (Initial calibration)	Mn	41.23	40.0	103	103	>-
	CVAA (Initial calibration)						
	ICP (Continuing calibration)						
1938 CCV	ICP/MS (Continuing calibration)	As	49.98	50.0	001	001	<b>→</b>
	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# 34047 KY

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

Page: | of | 5 Reviewer:\_\_ 2nd 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

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

Where, 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	DR (units) Acceptable Acceptable %R / RPD / %D (Y/N)	\ 86 86 (7) 8m)	66 66 (by/6m)	281 (m) (m)	(mg/lng) 8 8	(m3/kg) 4.2 4.2
Found / S / 1 True / D / SDR (units) %R / RPD		(m2 (r)				
		98.34 (mg/L) 100	74 (mg/kg)	(62/6m) 9 h	(mg/kg)	79 (mg/kg) 377.01 (mg/kg)
	Element	Mn 98	le As 19.	(SSR-SR) Mu 35.	As 21.	MM 361.79
Type of Analysis		ICP interference check	Laboratory control sample	Matrix spike	Duplicate	ICP serial dilution
	Sample ID	1851 ICSAB	1916 LCS	30.8 21	20/16	2006/2002

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#: 24047K4

#### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	of
Reviewer:	MG
2nd reviewer:	

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

WETHOD. Trace Wetais (EF)	A 300 640 Method 60 10/0020/1000)	•		
Y N N/A Have results N N/A Are results w	ow for all questions answered "N". Not appliate the polynomial pol			
Detected analyte results for _equation:	#16, Ma	were recalcu	lated and verified	using the following
Concentration = (RD)(FV)(Dil) (In. Vol.)	Recalculation:			
RD = Raw data conce FV = Final volume (m n. Vol. = Initial volume (m Dil = Dilution factor	1)	0.913)	= 335.84	1 mg/ or mg
# Sample ID	Analyte	Reported Concentration (Mg/kg)	Calculated Concentration ( <sup>vn</sup> g / kq)	Acceptable (Y/N)
1 16	As	6.1	6.1	Y
	Mn	340	340	J.
				Y
				,
	·			
			·	
Note:				

# Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 25, 2010

LDC Report Date:

October 11, 2010

**Matrix:** 

Soil/Water

Parameters:

Arsenic & Manganese

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6818-1

#### Sample Identification

BDT-2-N-20-10.0BPC BDT-2-N-5-2BPC BDT-2-N-20-12.0BPC BDT-2-N-5-4BPC BDT-2-N-5-6BPC BDT-2-N-20-14.0BPC BDT-2-N-10-10BPC BDT-2-N-20-2.0BPC BDT-2-N-10-12BPC BDT-2-N-20-4.0BPC BDT-2-N-20-6.0BPC BDT-2-N-10-14BPC\*\* BDT-2-N-20-8.0BPC BDT-2-N-10-2BPC BDT-2-N-20-12.0BPC FD BDT-2-N-10-4BPC BDT-2-N-15-10.0BPC BDT-2-N-10-6BPC BDT-2-N-15-12.0BPC BDT-2-N-10-8BPC BDT-2-N-10-6BPC FD BDT-2-N-15-14.0BPC\*\* BDT-2-N-15-2.0BPC EB-08252010 BDT-2-N-15-4.0BPC BDT-2-N-15-6.0BPCMS BDT-2-N-15-6.0BPCMSD BDT-2-N-15-6.0BPC BDT-2-N-5-10BPCMS BDT-2-N-15-8.0BPC BDT-2-N-5-10BPC BDT-2-N-5-10BPCMSD BDT-2-N-5-12BPC BDT-2-N-5-14BPC\*\*

BDT-2-N-5-12BPC\_FD BDT-2-N-5-8BPC

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

#### Introduction

This data review covers 35 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 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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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 was found in the initial, continuing and preparation blanks.

Sample EB-08252010 was identified as an equipment blank. No arsenic or manganese was found in this blank.

#### 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) and relative percent differences (RPD) 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 280-6818-1	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 BDT-2-N-20-12.0BPC and BDT-2-N-20-12.0BPC\_FD and samples BDT-2-N-5-12BPC and BDT-2-N-5-12BPC\_FD and samples BDT-2-N-10-6BPC\_and BDT-2-N-10-6BPC\_FD were identified as field duplicates. No arsenic or manganese was detected in any of the samples with the following exceptions:

	Concentra	tion (mg/Kg)	DDD	Difference		
Analyte	BDT-2-N-20-12.0BPC	BDT-2-N-20-12.0BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	4.6	4.2	9 (≤50)	-	<u>-</u> .	-

	Concentra	tion (mg/Kg)				
Analyte	BDT-2-N-5-12BPC	BDT-2-N-5-12BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	5.8	5.4	7 (≤50)	-	-	-

	Concentra	tion (mg/Kg)				
Analyte	BDT-2-N-10-6BPC	BDT-2-N-10-6BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	3.4	3.2	-	0.2 (≤0.66)	-	-

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic & Manganese - Data Qualification Summary - SDG 280-6818-1

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
	- Cumpio	Allalyto	riay	AOIF	Neason (Code)
280-6818-1	BDT-2-N-20-10.0BPC	All analytes reported	J (all detects)	Α	Sample result
	BDT-2-N-20-12.0BPC	below the PQL.			verification (PQL) (sp)
	BDT-2-N-20-14.0BPC				
	BDT-2-N-20-2.0BPC		4		
	BDT-2-N-20-4.0BPC				
	BDT-2-N-20-6.0BPC				
	BDT-2-N-20-8.0BPC				
	BDT-2-N-20-12.0BPC_FD				
	BDT-2-N-15-10.0BPC				
	BDT-2-N-15-12.0BPC				
	BDT-2-N-15-14.0BPC**				
	BDT-2-N-15-2.0BPC				
	BDT-2-N-15-4.0BPC				
	BDT-2-N-15-6.0BPC				
	BDT-2-N-15-8.0BPC	•			
	BDT-2-N-5-10BPC				
	BDT-2-N-5-12BPC				
	BDT-2-N-5-14BPC**				
	BDT-2-N-5-12BPC_FD				
	BDT-2-N-5-8BPC				
	BDT-2-N-5-2BPC				
	BDT-2-N-5-4BPC				
	BDT-2-N-5-6BPC				
	BDT-2-N-10-10BPC				
	BDT-2-N-10-12BPC				
	BDT-2-N-10-14BPC**				
	BDT-2-N-10-2BPC				
	BDT-2-N-10-4BPC	·			
	BDT-2-N-10-6BPC				
	BDT-2-N-10-8BPC				
	BDT-2-N-10-6BPC_FD				
	EB-08252010				

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic & Manganese - Laboratory Blank Data Qualification Summary - SDG 280-6818-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic & Manganese - Equipment Blank Data Qualification Summary - SDG 280-6818-1

No Sample Data Qualified in this SDG

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

Stage 2B/4

Date: <u>(</u>	0-6-1
Page:_	_of_(_
Reviewer:_	MG
2nd Reviewer:	1,

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

24047L4

Laboratory: Test America

280-6818-1

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 - 25-10
II.	ICP/MS Tune	A	
111.	Calibration	Α	
IV.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	A	
VI.	Matrix Spike Analysis	A	MS/MSD
VII.	Duplicate Sample Analysis	2	
VIII.	Laboratory Control Samples (LCS)	A	LCS/LCSD
IX.	Internal Standard (ICP-MS)	Α	
X.	Furnace Atomic Absorption QC	2	not utilized
XI.	ICP Serial Dilution	A	
XII.	Sample Result Verification	Α	Not reviewed for Stage 2B validation.
XIII.	Overall Assessment of Data	Α	
XIV.	Field Duplicates	SW	D=2+8 D=17+19 D=29+31
ΧV	Field Blanks	ND	EB= 32

Note:

LDC #:

SDG #:

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-2-N-20-10.0BPC	S	11	BDT-2-N-15-14.0BPC**	S	21 2	BDT-2-N-5-2BPC	S	312	BDT-2-N-10-6BPC_FD	5
2 1		BDT-2-N-20-12.0BPC	1	12	BDT-2-N-2-2.0BPC 90		22	BDT-2-N-5-4BPC		<sub>32</sub> 3	EB-08252010	W
3	I	BDT-2-N-20-14.0BPC		<sub>13</sub> (	BDT-2-N-15-4.0BPC		23 <b>2</b>	BDT-2-N-5-6BPC		<sub>33</sub>	BDT-2-N-15-6.0BPCMS	S
4	ı	BDT-2-N-20-2.0BPC		14	BDT-2-N-15-6.0BPC		د 24	BDT-2-N-10-10BPC		34	BDT-2-N-15-6.0BPCMSD	
5	1	BDT-2-N-20-4.0BPC		15 l	BDT-2-N-15-8.0BPC		25 <b>2</b>	BDT-2-N-10-12BPC		35 A	BDT-2-N-5-10BPCMS	
6	Π	BDT-2-N-20-6.0BPC		16 <sup>2</sup>	BDT-2-N-5-10BPC		<b>?</b> 26	BDT-2-N-10-14BPC**		36 <b>)</b>	BDT-2-N-5-10BPCMSD	_↓
7	Π	BDT-2-N-20-8.0BPC		17 2	BDT-2-N-5-12BPC		27 27	BDT-2-N-10-2BPC		37		
8	I	BDT-2-N-20-12.0BPC_FD		ر 18	BDT-2-N-5-14BPC**		28 A	BDT-2-N-10-4BPC		38	PBSI	
9	1	10.0 BDT-2-N-15-1.0BPC		ړ 19	BDT-2-N-5-12BPC_FD		ر 29	BDT-2-N-10-6BPC		<sub>39</sub> 2	PBS2	
10	1	BDT-2-N-15-12.0BPC		20 2	BDT-2-N-5-8BPC	<b>*</b>	30 <b>2</b>	BDT-2-N-10-8BPC	V	40 <b>3</b>	PBW	

Notes:		

#### VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
Reviewer: M&
2nd Reviewer: \_\_\_\_

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

Validation Area	Yes	No	NA	Findings/Comments
Validation Area				·
I. Technical holding times				· · · · · · · · · · · · · · · · · · ·
All technical holding times were met.	V			
Cooler temperature criteria was met.	_/_			
II. ICP/MS Tune	1			
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.		/		
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) ≤ 20% for waters and ≤ 35% for soil samples? A control limit of +/- RL(+/-2X RL for soil) was used for samples that were ≤ 5X the RL, including when only one of the duplicate sample values were ≤ 5X the RL.	/			
VII. Laboratory control samples				
Was an LCS anaylzed for this SDG?	/			
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?	/	-		

#### **VALIDATION FINDINGS CHECKLIST**

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

Validation Area	Yes	No	NA	Findings/Comments
VIII. Furnace Atomic Absorption QC	,	,		
If MSA was performed, was the correlation coefficients > 0.995?			1	
Do all applicable analysies have duplicate injections? (Level IV only)			<b>V</b>	
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				444
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)			1	
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?	/		ļ.,	
If the %Rs were outside the criteria, was a reanalysis performed?				
XI. Regional Quality Assurance and Quality Control	<b></b>	·		
Were performance evaluation (PE) samples performed?		/	L.,	
Were the performance evaluation (PE) samples within the acceptance limits?	<u>.</u>	<u> </u>	/	
XII. Sample Result Verification	<del>, ,</del>	1	T	
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.	/			
Target analytes were detected in the field duplicates.	<u> </u>			
XV. Field blanks				
Field blanks were identified in this SDG.	/		<u> </u>	
Target analytes were detected in the field blanks.		/		

LDC#: 24047L4

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

	Page:_	Lof L
	Reviewer:	MG
2nd	Reviewer:	

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

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

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	2	8	RPD	Difference	Limits	(Parent Only)
Arsenic	4.6	4.2	9			

V:\FIELD DUPLICATES\FD\_inorganic\24047L4.wpd

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	17	19	RPD	Difference	Limits	(Parent Only)
Arsenic	5.8	5.4	7		·	

V:\FIELD DUPLICATES\FD\_inorganic\24047L4.wpd

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	29	31	RPD	Difference	Limits	(Parent Only)
Arsenic	3.4	3.2		0.2	(≤0.66)	

V:\FIELD DUPLICATES\FD\_inorganic\24047L4.wpd

LDC# 34047 LY

# Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: Reviewer: 2nd 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 = 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

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
	ICP (Initial calibration)						
1953 ICV	ICP/MS (Initial calibration)	As	41.06	40.0	103	(03	>
	CVAA (Initial calibration)						
	ICP (Continuing calibration)						
2007 CCV	ICP/MS (Continuing calibration)	As	49.68	50.0	66	66	
	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# 34047LY

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

2nd Reviewer. Page: 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

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 =  $1S-D1 \times 100$ (S+D)/2

Where,

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  $\times$  5)

<u></u>	-	i <del>r -</del>		_	:	_		_		_	
	Acceptable (Y/N)	,	>	-	V		············		,,, -		>
Reported	%R/RPD/%D		<b>ゆ</b> の)		6		66		n		0.35
Recalculated	%R/RPD/%D		(0)		ر <i>و</i> م		66		3		0.59
	(units)	\ ' "")	(1/S)	1 1	(mg/kg)	0	(mg/kg)	>	(mg/kg)	0'	(mg/kg)
	True / D / SDR (units)		00)		30.0		1.61		23.62 (mg/kg		3.43
	-	1 / 5 /	(7) Bw) 05	, ,	31 (mg/kg)	0 ,	(mg/kg)	) ' · · ·	.93 (mg/kg)	5	(mg/kg)
Francisco	Found / S / I (units)		101.50		18.31	(SSR-SR)	19.52		33.93		3.41
	Element		As		As		As		As		As
	Type of Analysis	ICD intodococcoccic	וכן ווופוופופויפפ כוופכע		Laboratory control sample	Matrix spike	-		Duplicate		ICP serial dilution
	Sample ID	9009	ICSAB	shee	rcs3	2266	35	bsee/osee	35/36	1see/anee	ما

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#: 24047L4

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

Page:_	of
Reviewer:_	MG
2nd reviewer:_	- h

,			2/10/10	
METHOD: Trace Metals (EF	PA SW 846 Method 6010/6020/7000)			
YN N/A Have results N N/A Are results	low for all questions answered "N". Not app s been reported and calculated correctly? within the calibrated range of the instrumen ction limits below the CRDL?			
Detected analyte results for equation:	#18, As	were recalcu	ulated and verified	using the following
Concentration = \frac{(RD)(FV)(Dil)}{(ln. Vol.)}  RD = Raw data concentration = Final volume (rank) to the concentration = Initial volume (rank) to the concentration = Dilution factor		.100 L) (5) ).929)	= 6.40 M	g/g or mg/k
# Sample ID	Analyte	Reported Concentration ( <sup>MG</sup> /kg)	Calculated Concentration ( Mg / kg)	Acceptable (Y/N)
1 18	As	6.4	6.4	Y
·				
				· · · · · · · · · · · · · · · · · · ·
Note:				

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 26, 2010

LDC Report Date:

October 11, 2010

**Matrix:** 

Soil/Water

Parameters:

Arsenic

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6851-1

#### Sample Identification

BDT-2-S-20-10BPC BDT-2-S-5-8BPC BDT-2-S-20-12BPC BDT-2-S-5-2BPC BDT-2-S-5-4BPC BDT-2-S-20-14BPC\*\* BDT-2-S-5-6BPC BDT-2-S-20-2BPC BDT-2-S-20-4BPC BDT-2-S-10-10BPC BDT-2-S-20-6BPC BDT-2-S-10-12BPC BDT-2-S-20-6BPC FD BDT-2-S-10-14BPC\*\* BDT-2-S-20-8BPC BDT-2-S-10-2BPC BDT-2-S-15-10BPC BDT-2-S-10-4BPC BDT-2-S-10-6BPC BDT-2-S-15-12BPC BDT-2-S-10-8BPC BDT-2-S-15-14BPC\*\* BDT-2-S-15-2BPC EB-08262010 BDT-2-S-15-4BPC BDT-2-S-20-10BPCMS BDT-2-S-15-6BPC BDT-2-S-20-10BPCMSD BDT-2-S-5-10BPCMS BDT-2-S-15-8BPC BDT-2-S-15-2BPC FD BDT-2-S-5-10BPCMSD BDT-2-S-5-10BPC BDT-2-S-5-12BPC

BDT-2-S-5-14BPC\*\* BDT-2-S-5-12BPC FD

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

#### Introduction

This data review covers 35 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 6020 for Arsenic.

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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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 was found in the initial, continuing and preparation blanks.

Sample EB-08262010 was identified as an equipment blank. No arsenic was found in this blank.

#### 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) and relative percent differences (RPD) 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 280-6851-1	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 BDT-2-S-20-6BPC and BDT-2-S-20-6BPC\_FD and samples BDT-2-S-15-2BPC and BDT-2-S-15-2BPC\_FD and samples BDT-2-S-5-12BPC and BDT-2-S-5-12BPC\_FD were identified as field duplicates. No arsenic was detected in any of the samples with the following exceptions:

	Concentrat					
Analyte	BDT-2-S-20-6BPC	BDT-2-S-20-6BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	3.3	3.4	3 (≤50)	-	-	-

	Concentrat	222	D'44				
Analyte	BDT-2-S-15-2BPC BDT-2-S-15-2BPC_FD		RPD (Limits)	Difference (Limits)	Flags	A or P	
Arsenic	4.9	5.7	15 (≤50)	-	-	-	

	Concentrat						
Analyte	BDT-2-S-5-12BPC	BDT-2-S-5-12BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P	
Arsenic	7.7	9.0	16 (≤50)	-	-	٠ -	

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic - Data Qualification Summary - SDG 280-6851-1

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-6851-1	BDT-2-S-20-10BPC BDT-2-S-20-12BPC BDT-2-S-20-14BPC** BDT-2-S-20-4BPC BDT-2-S-20-4BPC BDT-2-S-20-6BPC BDT-2-S-20-6BPC BDT-2-S-20-6BPC BDT-2-S-15-10BPC BDT-2-S-15-12BPC BDT-2-S-15-14BPC** BDT-2-S-15-4BPC BDT-2-S-15-6BPC BDT-2-S-15-8BPC BDT-2-S-15-8BPC BDT-2-S-5-12BPC BDT-2-S-5-14BPC** BDT-2-S-5-14BPC** BDT-2-S-5-14BPC BDT-2-S-5-14BPC BDT-2-S-5-14BPC BDT-2-S-5-14BPC BDT-2-S-5-14BPC BDT-2-S-5-14BPC BDT-2-S-5-14BPC BDT-2-S-5-6BPC BDT-2-S-5-6BPC BDT-2-S-10-10BPC BDT-2-S-10-10BPC BDT-2-S-10-14BPC** BDT-2-S-10-14BPC** BDT-2-S-10-14BPC BDT-2-S-10-6BPC BDT-2-S-10-6BPC BDT-2-S-10-6BPC BDT-2-S-10-8BPC EB-08262010	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (PQL) (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic - Laboratory Blank Data Qualification Summary - SDG 280-6851-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic - Equipment Blank Data Qualification Summary - SDG 280-6851-1

No Sample Data Qualified in this SDG

## Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

LDC #:	24047M4	VALIDATION COMPLETENE
SDG #:	280-6851-1	Stage 2B/4
Laborato	ry: Test America	

Date: 10 - 8 - 10
Page: Lof_L
Reviewer: MG
2nd Reviewer:

METHOD: As (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
I.	Technical holding times	Α	Sampling dates: 8 - 26 - 10
H.	ICP/MS Tune	A	
111.	Calibration	Α	
IV.	Blanks	Α	
V.	ICP Interference Check Sample (ICS) Analysis	Α	
VI.	Matrix Spike Analysis	A	MS/MSD
VII.	Duplicate Sample Analysis	7	
VIII.	Laboratory Control Samples (LCS)	Α	LCS/LCSD
IX.	Internal Standard (ICP-MS)	Ą	
X.	Furnace Atomic Absorption QC	2	not utilized
XI.	ICP Serial Dilution	A	
XII.	Sample Result Verification	A	Not reviewed for Stage 2B validation.
XIII.	Overall Assessment of Data	Α.	
XIV.	Field Duplicates	SW	D=6+7 D=12+16 D=18+20
XV	Field Blanks	ND	EB=32

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

, I	BDT-2-S-20-10BPC	5	11	BDT-2-S-15-14BPC**	212	BDT-2-S-5-8BPC S	3	12	BDT-2-S-10-8BPC	S
2 1	BDT-2-S-20-10BPC	T	- 1	BDT-2-S-15-2BPC	1 1	BDT-2-S-5-2BPC	T	3	EB-08262010	W
3 1	BDT-2-S-20-14BPC**	H	1	BDT-2-S-15-4BPC	2	BDT-2-S-5-4BPC	3	1	BDT-2-S-20-10BPCMS	5
4	BDT-2-S-20-2BPC	П	14	BDT-2-S-15-6BPC	24 2	BDT-2-S-5-6BPC	3.	<sub>4</sub> [	BDT-2-S-20-10BPCMSD	1
<sub>5</sub>	BDT-2-S-20-4BPC	П	15	BDT-2-S-15-8BPC	<sub>25</sub> 2	BDT-2-S-10-10BPC	3	<sub>5</sub> 2	BDT-2-S-5-10BPCMS	
<sub>6</sub> 1	BDT-2-S-20-6BPC		16	BDT-2-S-15-2BPC_FD	26 <sup>2</sup>	BDT-2-S-10-12BPC	3	<sub>6</sub> 2	BDT-2-S-5-10BPCMSD	J
<sub>7</sub> [	BDT-2-S-20-6BPC_FD		<sub>17</sub> 2	BDT-2-S-5-10BPC	27	BDT-2-S-10-14BPC**	3	7		
8 <sup>1</sup>	BDT-2-S-20-8BPC		18 J	BDT-2-S-5-12BPC	28	BDT-2-S-10-2BPC	3	B (	PBSI	
9 1	BDT-2-S-15-10BPC		19 2	BDT-2-S-5-14BPC**	29 2	BDT-2-S-10-4BPC	3	9 <b>ə</b>	PBS2	
10 l	BDT-2-S-15-12BPC	4	<sub>20</sub> 2	BDT-2-S-5-12BPC_FD	30 2	BDT-2-S-10-6BPC	4	03	PBW	

Notes:		

Page: 1 of 2
Reviewer: MG
2nd Reviewer: 1

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.	/			
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?	1			
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?	<b>V</b>			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/		
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.	<b>/</b>			
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.	<b>/</b>			
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq$ 20% for waters and $\leq$ 35% for soil samples? A control limit of +/- 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	·			the second continues of the se
Was an LCS anayized for this SDG?	1			
Was an LCS analyzed per extraction batch?				· · · · · · · · · · · · · · · · · · ·
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?	/			

#### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 3
Reviewer: MG
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?				
IX. ICP Serial Dilution				<b>P</b>
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the MDL (ICP)/>100X the MDL(ICP/MS)?	<b>V</b>			
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		· <del>····</del>	·····	
Were performance evaluation (PE) samples performed?		/		
Were the performance evaluation (PE) samples within the acceptance limits?			\ <u>\</u>	
XII. Sample Result Verification	·	r <del></del>		
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.	/		<u> </u>	
XIV. Field duplicates			,	
Field duplicate pairs were identified in this SDG.	1			
Target analytes were detected in the field duplicates.	<u> </u>	<u> </u>		
XV. Field blanks	·	<b></b>	T	T
Field blanks were identified in this SDG.	/	<u> </u>		
Target analytes were detected in the field blanks.				

LDC#: 24047M4

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

Page: \_ l\_of \_l\_ Reviewer: \_ MG\_ 2nd Reviewer: \_ \\_

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

(V) NA

Were field duplicate pairs identified in this SDG?

Were target analytes detected in the field duplicate pairs?

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	6	7	RPD	Difference	Limits	(Parent Only)
Arsenic	3.3	3.4	3			

V:\FIELD DUPLICATES\FD\_inorganic\24047M4.wpd

	Concentration (mg/kg)		(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	12	16	RPD	Difference	Limits	(Parent Only)
Arsenic	4.9	5.7 ·	15			

V:\FIELD DUPLICATES\FD\_inorganic\24047M4.wpd

	Concentration (mg/kg)		(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	18	20	RPD	Difference	Limits	(Parent Only)
Arsenic	7.7	9.0	16			

V:\FIELD DUPLICATES\FD\_inorganic\24047M4.wpd

LDC# 34047M4

# Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Page: Lof 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 = Found x 100 True

Where, Found = concentration (in ug/L) of each analyte  $\underline{\text{measured}}$  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)						
1740 ICV	ICP/MS (Initial calibration)	As	41.18	40.0	103	(03	>
	CVAA (Initial calibration)						
	ICP (Continuing calibration)						
0,832 CCV	ICP/MS (Continuing calibration)	As	50.43	50.0	10)	101	>
	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.

TWINOTE LDC#:

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

Page: 1 of 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

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 =  $|S-D|_X \times 100$ (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 = [I-SDR] x 100

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

				Γ							
	Acceptable (Y/N)	,	>-								->
Reported	%R/RPD/%D	0	(o3				₩ %		<b>7</b> 0		ص بن
Recalculated	%R / RPD / %D		/03	•	<del>-</del>		ત ઝ		8		3,4
	(nuits)	(Ma. 1)	(S)	/ / yw/	( 3/kg/	- \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	1 0/kg	D/	mg/kg	( m	( all
	True / D / SDR (units)		001		30.0		93.0		33.92	1	4.87
	1/	(/ 57/	(J) (1)	/ Jan/	28 (3/kg)	ø'	(mg/kg)	, ,	( Q/ka)	) / cm)	
	Found / S / I (units)		103.30 (0/L)		18.38	(SSR-SR)	11:81		23.10		4.99
	Element	•	As	•	AS		As		As		Ă
	Type of Analysis	ICP interference check		elamos lostado vacterade		Matrix spike		-	Uuplicate	ICB serial dilution	
	Sample ID	7616	<b>ECSAB</b>	51e0	L <5	1660	33	bee 0 / 1660	33/34	1660 /8160	

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#: 24047 M4

# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	
Reviewer:	MG
2nd reviewer:	

NETH	IOD: Trace Metals (EP.	A SW 846 Method 6010/6020/7000)			
Pleas PN DN DN	N/A Have results N/A Are results w	ow for all questions answered "N". Not applead to been reported and calculated correctly? within the calibrated range of the instrument tion limits below the CRDL?			
Detec equati	ted analyte results for _	#3, As	were recalcu	lated and verified	using the following
·	tration = (RD)(FV)(Dil) (In. Vol.) = Raw data conce	1)	00 L)(5) ===============================	6.85 <b>"</b> 8,	g or mg/kg
#	Sample ID	Analyte	Reported Concentration (mg/kg)	Calculated Concentration (mg/Kg)	Acceptable (Y/N)
(	3	As	6.8	6.8	Y
				-	
					·
ote:_					

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 27, 2010

LDC Report Date:

October 11, 2010

Matrix:

Soil/Water

Parameters:

Lead

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6886-1

#### Sample Identification

BDT-1-N-20-10BPC	BDT-1-N-10-4BPC	BDT-1-N-15-4BPCMS
BDT-1-N-20-10BPC**	BDT-1-N-10-4BPC	BDT-1-N-15-4BPCMSD
BDT-1-N-20-14BPC	BDT-1-N-10-8BPC	BDT-1-S-20-4BPCMS
BDT-1-N-20-2BPC	BDT-1-N-10-8BPC_FD	BDT-1-S-20-4BPCMSD
BDT-1-N-20-4BPC	BDT-1-N-15-10BPC	BDT-1-S-20-14BPC_FDMS
BDT-1-N-20-6BPC	BDT-1-N-15-12BPC	BDT-1-S-20-14BPC_FDMSD
BDT-1-N-20-8BPC	BDT-1-N-15-14BPC**	
BDT-1-N-20-10BPC_FD	BDT-1-N-15-8BPC	
BDT-1-N-5-10BPC	BDT-1-N-15-2BPC	
BDT-1-N-5-12BPC	BDT-1-N-15-4BPC	
BDT-1-N-5-14BPC	BDT-1-N-15-6BPC	
BDT-1-N-5-8BPC_FD	BDT-1-S-20-10BPC	
BDT-1-N-5-8BPC	BDT-1-S-20-12BPC	
BDT-1-N-5-2BPC	BDT-1-S-20-14BPC**	
BDT-1-N-5-4BPC	BDT-1-S-20-2BPC	
BDT-1-N-5-6BPC	BDT-1-S-20-4BPC	
BDT-1-N-10-10BPC	BDT-1-S-20-6BPC	· ·
BDT-1-N-10-12BPC	BDT-1-S-20-8BPC	
BDT-1-N-10-14BPC	BDT-1-S-20-14BPC FD	
BDT-1-N-10-2BPC	EB-08272010	

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

#### Introduction

This data review covers 45 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 6020 for Lead.

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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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 lead was found in the initial, continuing and preparation blanks with the following exceptions:

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
PB (prep blank) Lead		0.0239 mg/Kg	BDT-1-N-20-10BPC BDT-1-N-20-12BPC** BDT-1-N-20-14BPC BDT-1-N-20-4BPC BDT-1-N-20-6BPC BDT-1-N-20-6BPC BDT-1-N-20-10BPC_FD BDT-1-N-5-10BPC BDT-1-N-5-12BPC BDT-1-N-5-14BPC BDT-1-N-5-8BPC_FD BDT-1-N-5-8BPC_FD BDT-1-N-5-8BPC BDT-1-N-5-6BPC BDT-1-N-5-6BPC BDT-1-N-10-10BPC BDT-1-N-10-12BPC BDT-1-N-10-14BPC BDT-1-N-10-14BPC BDT-1-N-10-14BPC BDT-1-N-15-4BPC

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

Sample EB-08272010 was identified as an equipment blank. No lead was found in this blank.

#### 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) and relative percent differences (RPD) 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 280-6886-1	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 BDT-1-N-20-10BPC and BDT-1-N-20-10BPC\_FD and samples BDT-1-N-5-8BPC\_FD and BDT-1-N-5-8BPC and samples BDT-1-N-10-8BPC\_and BDT-1-N-10-8BPC\_FD and samples BDT-1-S-20-14BPC\*\* and BDT-1-S-20-14BPC\_FD were identified as field duplicates. No lead was detected in any of the samples with the following exceptions:

	- Concentra	tion (mg/Kg)	222	D:#		
Analyte	BDT-1-N-20-10BPC	BDT-1-N-20-10BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Lead	7.6	7.6	0 (≤50)	-	-	-

	Concentrati	on (mg/Kg)				
Analyte	BDT-1-N-5-8BPC_FD	BDT-1-N-5-8BPC	RPD (Limits)	Difference (Limits)	Flags	A or P
Lead	7.8	7.9	1 (≤50)	-	-	-

	Concentrati	ion (mg/Kg)				
Analyte	BDT-1-N-10-8BPC	BDT-1-N-10-8BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Lead	8.0	8.3	4 (≤50)	_	-	_

	Concentrat	ion (mg/Kg)	200	Diff		
Analyte	BDT-1-S-20-14BPC**	BDT-1-S-20-14BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Lead	8.7	8.1	7 (≤50)	-	•	-

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Lead - Data Qualification Summary - SDG 280-6886-1

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-6886-1	BDT-1-N-20-10BPC BDT-1-N-20-12BPC** BDT-1-N-20-14BPC BDT-1-N-20-4BPC BDT-1-N-20-4BPC BDT-1-N-20-6BPC BDT-1-N-20-6BPC BDT-1-N-5-10BPC BDT-1-N-5-10BPC BDT-1-N-5-12BPC BDT-1-N-5-14BPC BDT-1-N-5-8BPC BDT-1-N-5-8BPC BDT-1-N-5-8BPC BDT-1-N-5-6BPC BDT-1-N-10-10BPC BDT-1-N-10-10BPC BDT-1-N-10-12BPC BDT-1-N-10-18BPC BDT-1-N-10-18BPC BDT-1-N-10-18BPC BDT-1-N-10-18BPC BDT-1-N-10-8BPC BDT-1-N-10-8BPC BDT-1-N-15-12BPC BDT-1-N-15-12BPC BDT-1-N-15-12BPC BDT-1-N-15-12BPC BDT-1-N-15-14BPC** BDT-1-N-15-14BPC** BDT-1-N-15-14BPC BDT-1-N-15-14BPC BDT-1-N-15-4BPC BDT-1-S-20-14BPC** BDT-1-S-20-14BPC** BDT-1-S-20-4BPC BDT-1-S-20-4BPC BDT-1-S-20-4BPC BDT-1-S-20-4BPC BDT-1-S-20-14BPC_FD EB-08272010	All analytes reported below the PQL.	J (all detects)		Sample result verification (PQL) (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Lead - Laboratory Blank Data Qualification Summary - SDG 280-6886-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Lead - Equipment Blank Data Qualification Summary - SDG 280-6886-1

No Sample Data Qualified in this SDG

## Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

LDC #:	24047N4	_ VALIDATION COMPLETENE
SDG #:	280-6886 <b>/</b> 1	Stage 2B/4
Laborator	y: Test America	<del></del>

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

METHOD: Pb (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
I.	Technical holding times	Α	Sampling dates: 8-27-10
II.	ICP/MS Tune	Α	
III.	Calibration	Ā	
IV.	Blanks	SW	
V.	ICP Interference Check Sample (ICS) Analysis	A	
VI.	Matrix Spike Analysis	Α	MS/MSD
VII.	Duplicate Sample Analysis	7	
VIII.	Laboratory Control Samples (LCS)	Α	LCS/LCSD
IX.	Internal Standard (ICP-MS)	Α	,
X.	Furnace Atomic Absorption QC	2	not utilized
XI.	ICP Serial Dilution	Α	
XII.	Sample Result Verification	A	Not reviewed for Stage 2B validation.
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	SW	D= 1+8 D=12+13 D=23+24 D=34+39
ΧV	Field Blanks	ND	EB=40

Note:

A = Acceptable

N = Not provided/applicable SW = See worksheet ND = No compounds detected

R = Rinsate

D = Duplicate

TB = Trip blank

FB = Field blank EB = Equipment blank

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

1 1	BDT-1-N-20-10BPC S	1	11	BDT-1-N-5-14BPC S	212	BDT-1-N-10-4BPC S		<sub>31</sub> 2	BDT-1-N-15-6BPC	\$
2 1	BDT-1-N-20-12BPC**	1	12	BDT-1-N-5-8BPC_FD	22 2	BDT-1-N-10-6BPC	Ţ	<sub>32</sub> 2	BDT-1-S-20-10BPC	
3 1	BDT-1-N-20-14BPC	1	<sub>13</sub> [	BDT-1-N-5-8BPC	23 2	BDT-1-N-10-8BPC		33 <b>2</b>	BDT-1-S-20-12BPC	$\perp \! \! \perp$
4 1	BDT-1-N-20-2BPC	1	4 (	BDT-1-N-5-2BPC	24	BDT-1-N-10-8BPC_FD		<sub>34</sub> 2	BDT-1-S-20-14BPC**	
<sub>5</sub> 1	BDT-1-N-20-4BPC	1	5 1	BDT-1-N-5-4BPC	25 Z	BDT-1-N-15-10BPC	<u> </u> ;	<sub>35</sub> 2	BDT-1-S-20-2BPC	$\perp \! \! \! \! \! \! \! \! \perp$
6	BDT-1-N-20-6BPC	1	16	BDT-1-N-5-6BPC	26 2	BDT-1-N-15-12BPC		<sub>36</sub> 3	BDT-1-S-20-4BPC	
7	BDT-1-N-20-8BPC	1	17	BDT-1-N-10-10BPC	272	BDT-1-N-15-14BPC**		37 <b>7</b>	BDT-1-S-20-6BPC	
<sub>8</sub> 1	BDT-1-N-20-10BPC_FD	1	18	BDT-1-N-10-12BPC	28 2	BDT-1-N-15-8BPC		<sub>38</sub> J	BDT-1-S-20-8BPC	
9 1	BDT-1-N-5-10BPC	1	ا 19	BDT-1-N-10-14BPC	293	BDT-1-N-15-2BPC		<sub>39</sub> ∂	BDT-1-S-20-14BPC_FD	1
10 [	BDT-1-N-5-12BPC	2	<sub>20</sub> ک	BDT-1-N-10-2BPC	<sub>30</sub> l	BDT-1-N-15-4BPC	ر.	404	EB-08272010	W

Notes:		
		,

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

LDC #: 24047N4 Stage 2B/4 SDG #: 280-6886A1

Reviewer: 2nd Reviewer:

Laboratory: Test America

METHOD: Pb (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
1.	Technical holding times	Sampling dates:
11.	ICP/MS Tune	
III.	Calibration	
IV.	Blanks	
V.	ICP Interference Check Sample (ICS) Analysis	4 %
VI.	Matrix Spike Analysis	6, 9
VII.	Duplicate Sample Analysis	cee ox
VIII.	Laboratory Control Samples (LCS)	9 ( /
IX.	Internal Standard (ICP-MS)	
X.	Furnace Atomic Absorption QC	
XI.	ICP Serial Dilution	
XII.	Sample Result Verification	Not reviewed for Stage 2B validation.
XIII.	Overall Assessment of Data	
XIV.	Field Duplicates	
ΧV	Field Blanks	

Note:

A = Acceptable

ND = No compounds detected

D = Duplicate

N = Not provided/applicable SW = See worksheet

R = Rinsate FB = Field blank

TB = Trip blank EB = Equipment blank

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

41	BDT-1-N-15-4BPCMS	51 l	PBSI	61	71	
42 1	BDT-1-N-15-4BPCMSD	<sub>52</sub> 2	<i>P                                    </i>	62	72	
43 <b>3</b>	BDT-1-S-20-4BPCMS	<sub>53</sub> 3	PB53	63	. 73	
44 3	BDT-1-S-20-4BPCMSD	<sub>54</sub> 4	PBW	64	74	
<sub>45</sub> 2	BDT-1-S-20-14BPC_FDMS	55		65	75	
<sub>46</sub> 2	BDT-1-S-20-14BPC_FDMSD	56		66	76	
47		57		67	77	
48		58		68	78	
49		59		69	79	
50		60		70	80	

Notes:		

#### **VALIDATION FINDINGS CHECKLIST**

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

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

Metriod. Metals (LI A OVV 040 Metriod 00 10 Di 1 00 010 020)	1		·	
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	/			
Cooler temperature criteria was met.	/			
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.	/			
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?	1			
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?	/			
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?	/			

LDC #: 24047 N4

#### **VALIDATION FINDINGS CHECKLIST**

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

Validation Area	Yes	No	NA	Findings/Comments
VIII. Furnace Atomic Absorption QC	r		•	
If MSA was performed, was the correlation coefficients > 0.995?			1	
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?		L	<u> </u>	
IX. ICP Serial Dilution				
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the MDL (ICP)/>100X the MDL(ICP/MS)?	1			
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	r		·	
Were performance evaluation (PE) samples performed?		/	Ĺ.,	
Were the performance evaluation (PE) samples within the acceptance limits?	<u> </u>		/	
XII. Sample Result Verification			т	
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				
Field duplicate pairs were identified in this SDG.	1			
Target analytes were detected in the field duplicates.	/			
XV. Field blanks			<b>,</b>	
Field blanks were identified in this SDG.	/	ļ,		
Target analytes were detected in the field blanks.		/	<u> </u>	

LDC #: 24047N4

VALIDATION FINDINGS WORKSHEET PB/ICB/CCB QUALIFIED SAMPLES

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

> Soil preparation factor applied: 100x, 5x dill Associated Samples: 1-19, 30 (>RL) METHOD: Trace metals (EPA SW 864 Method 6010B/6020/7000)

Sample Concentration units, unless otherwise noted: mg/Kg

	Action No Qual's.	Maximim		
ıal's.		ICB/CCB <sup>a</sup> (ug/L)	Maximum Action ICB/CCB <sup>a</sup> Limit (ug/L)	Maximum Action ICB/CCB <sup>a</sup> Limit (ug/L)
				0.0239

Samples with analyte concentrations within five times the associated ICB, CCB or PB concentration are listed above with the identifications from the Validation Completeness Worksheet. These sample results were qualified as not detected, "U".

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

LDC#:\_24047N4

### VALIDATION FINDINGS WORKSHEET Field Duplicates

	Page:_	[ of [
	Reviewer:	MG
2nd	Reviewer:	

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

(Y)N NA (Y)N NA Were field duplicate pairs identified in this SDG?

Were target analytes detected in the field duplicate pairs?

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	1	8	RPD	Difference	Limits	(Parent Only)
Lead	7.6	7.6	0			

V:\FIELD DUPLICATES\FD\_inorganic\24047N4.wpd

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	12	13	RPD	Difference	Limits	(Parent Only)
Lead	7.8	7.9	1 .			

V:\FIELD DUPLICATES\FD\_inorganic\24047N4.wpd

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	23	24	RPD	Difference	Limits	(Parent Only)
Lead	8.0	8.3	4			

V:\FIELD DUPLICATES\FD\_inorganic\24047N4.wpd

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	34	39	RPD	Difference	Limits	(Parent Only)
Lead	8.7	8.1	7			

V:\FIELD DUPLICATES\FD\_inorganic\24047N4.wpd

LDC# 34047 NY

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

Page: Lof 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 = 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

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
	ICP (Initial calibration)						
1953 ICV	ICP/MS (Initial calibration)	Pb	40.69	40.0	(0x	(02	>
	CVAA (Initial calibration)						
	ICP (Continuing calibration)						
7108 CCV	ICP/MS (Continuing calibration)	Pb	49,49	50.0	66	66	<b>&gt;</b>
	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# 24047 NH

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

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

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:

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

Where, Found = Concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation,

Found = SSR (spiked sample result) - SR (sample result).

True = Concentration of each analyte in the source.

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

RPD =  $|S-D|_X \times 100$ (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 = [I-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 (units)	True / D / SDR (units)	nits)	%R/RPD/%D	%R/RPD/%D	Acceptable (Y/N)
ICSAR ICE	ICP interference check	4 C	(1/8m) 18.46	() bm) OO)	(1) bm	36	36	<u> </u>
	Laboratory control sample	100	19.16 (mg/kg)	30.0	(mg/kg)	96	96	-
	Matrix spike	9	٥, ر	) h.oe	mag (Ka)	87	87	
11/19 Pu	Duplicate	90	27.95 (mg/kg)	27.21	ma (kg)	3	8	
30 30 ICF	ICP serial dilution	Pb	10.30 (mg/kg)	10.68 (mg/kg	12/kg	3.7	3.6	-3

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#: 24047 N4

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	
Reviewer:_	MG
2nd reviewer:_	$\sim$

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000) Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". (Y) N N/A Have results been reported and calculated correctly? Are results within the calibrated range of the instruments and within the linear range of the ICP? (Y) N N/A Are all detection limits below the CRDL? (Y) N N/A Detected analyte results for # 2were recalculated and verified using the following equation: Concentration = (RD)(FV)(Dil) Recalculation: (in. Vol.) RD Raw data concentration FV Final volume (ml) Initial volume (ml) or weight (G) In. Vol. Dilution factor Dil Reported Calculated Acceptable Concentration Concentration Sample ID Analyte (Y/N) Ph 2

Note:

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Data Validation Reports LDC #24047

Wet Chemistry



# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 7 through August 9, 2010

**LDC Report Date:** 

October 8, 2010

Matrix:

Soil/Water

Parameters:

Perchlorate

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6223-1

#### Sample Identification

SSAJ3-02-12BPC SSAI3-04-5BPC
SSAJ3-02-15BPC SSAI3-04-8BPC
SSAJ3-02-8BPC\*\* SSAI3-03-8BPCMS
SSAJ3-02-8BPC\_FD SSAI3-03-8BPCMSD
EB-08072010 SSAI3-03-8BPCDUP
SSAI3-04-11BPC SSAI3-04-1BPCMS
SSAI3-04-14BPC SSAI3-04-1BPCMSD
SSAI3-04-14BPC\_FD SSAI3-04-1BPCDUP
SSAI3-02-11BPC
SSAI3-02-11BPC

SSAI3-04-11BPC SSAI3-04-14BPC SSAI3-04-14BPC\_FI SSAI3-02-11BPC SSAI3-02-14BPC SSAI3-02-5BPC\_FD SSAI3-03-11BPC SSAI3-03-14BPC SSAI3-02-1BPC SSAI3-02-5BPC SSAI3-02-5BPC SSAI3-03-1BPC SSAI3-03-1BPC SSAI3-03-1BPC SSAI3-03-1BPC SSAI3-03-1BPC SSAI3-03-1BPC SSAI3-03-1BPC

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

#### Introduction

This data review covers 27 soil samples and 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.

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

Samples indicated by a double asterisk on the front cover underwent a 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 perchlorate was found in the initial, continuing and preparation blanks.

Sample EB-08072010 was identified as an equipment blank. No perchlorate was found in this blank.

#### 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 280-6223-1	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

Samples SSAJ3-02-8BPC\*\* and SSAJ3-02-8BPC\_FD, samples SSAI3-04-14BPC and SSAI3-04-14BPC\_FD, and samples SSAI3-02-5BPC\_FD and SSAI3-02-5BPC were identified as field duplicates. No perchlorate was detected in any of the samples with the following exceptions:

	Concentra	tion (mg/Kg)				
Analyte	SSAJ3-02-8BPC**	SSAJ3-02-8BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Perchlorate	0.085	0.095	11 (≤50)	-	<u>-</u>	-

	Concentrati	ion (mg/Kg)				
Analyte	SSAI3-02-5BPC_FD	SSAI3-02-5BPC	RPD (Limits)	Difference (Limits)	Flags	A or P
Perchlorate	1.7	1.8	6 (≤50)	-	-	•

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Perchlorate - Data Qualification Summary - SDG 280-6223-1

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-6223-1	SSAJ3-02-12BPC SSAJ3-02-15BPC SSAJ3-02-8BPC** SSAJ3-02-8BPC_FD EB-08072010 SSAI3-04-11BPC SSAI3-04-14BPC SSAI3-04-14BPC SSAI3-02-11BPC SSAI3-02-11BPC SSAI3-02-14BPC SSAI3-02-14BPC SSAI3-02-5BPC_FD SSAI3-03-14BPC SSAI3-02-5BPC SSAI3-03-1BPC SSAI3-03-1BPC SSAI3-03-1BPC SSAI3-03-5BPC** SSAI3-03-5BPC** SSAI3-04-8BPC SSAI3-04-8BPC SSAI3-04-8BPC SSAI3-04-8BPC	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Perchlorate - Laboratory Blank Data Qualification Summary - SDG 280-6223-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Perchlorate - Equipment Blank Data Qualification Summary - SDG 280-6223-1

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

Stage 2B/4

Date: <u>/// - 6 -</u>	I(
Page: 1 of 1	
Reviewer: MG	

SDG #: 280-6223-1 Laboratory: Test America

LDC #: 24047B6

Reviewer: MG
2nd Reviewer:

METHOD:	(Analyte)	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
1.	Technical holding times		A	Sampling dates: 8-7-10 through 8-9-10
IIa.	Initial calibration		Ą	Ü
IIb.	Calibration verification		А	
111.	Blanks		Α	
IV	Matrix Spike/Matrix Spike Duplicates		Α	MS/MSD
V	Duplicates		A	DUP
VI.	Laboratory control samples		Α	LCS/LCSD
VII.	Sample result verification		A	Not reviewed for Stage 2B validation.
VIII.	Overall assessment of data	gn.y	SWA	
IX.	Field duplicates		SW	D=3+4 D=7*+8* D=11+15
Lx	Field blanks		ND	EB = 5

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	SSAJ3-02-12BPC	૬	11	SSAI3-02-5BPC_FD S	21	SSAI3-04-5BPC	31	,
<sub>2</sub>	SSAJ3-02-15BPC		12	SSAI3-03-11BPC	22 2	SSAI3-04-8BPC	32	
3	SSAJ3-02-8BPC ¥ €		13 l	SSAI3-03-14BPC	23	SSAI3-03-8BPCMS	33	
, I	SSAJ3-02-8BPC_FD***	4	14	SSAI3-02-1BPC	24	SSAI3-03-8BPCMSD	34	
ر ا	EB-08072010	W	15	SSAI3-02-5BPC	25	SSAI3-03-8BPCDUP	35	
٦,	SSAI3-04-11BPC	5	<sub>16</sub> l	SSAI3-02-8BPC	26	SSAI3-04-1BPCMS	36	
, [	SSAI3-04-14BPC		17	SSAI3-03-1BPC	27	SSAI3-04-1BPCMSD	37	
3	SSAI3-04-14BPC_FD		<sub>18</sub> l	SSAI3-03-5BPC**	28	SSAI3-04-1BPCDUP	38 1	PBS I
, (	SSAI3-02-11BPC		19 <sup>l</sup>	SSAI3-03-8BPC	29		39 <b>2</b>	PBS2
101	SSAI3-02-14BPC	J	20 1	SSAI3-04-1BPC	30		40 3	PBW

Notes:	

Page: \_(\_of\_2 Reviewer: \_ MG-2nd Reviewer: \_ \_\_\_

Method:Inorganics (EPA Method 314.0 )

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	/			
Cooler temperature criteria was met.				
II. Calibration				
Were all instruments calibrated daily, each set-up time?				
Were the proper number of standards used?				
Were all initial calibration correlation coefficients ≥ 0.995?	<b>V</b>			
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?	/		ļ	
Were titrant checks performed as required? (Level IV only)			V	
Were balance checks performed as required? (Level IV only)			/	
III. 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.		/		
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.	<b>/</b>			23
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.	/			91
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq$ 20% for waters and $\leq$ 35% for soil samples? A control limit of $\leq$ CRDL( $\leq$ 2X CRDL for soil) was used for samples that were $\leq$ 5X the CRDL, including when only one of the duplicate sample values were $\leq$ 5X the CRDL.	/		:	
V. Laboratory control samples				
Was an LCS anaylzed for this SDG?	<b>V</b>			
Was an LCS analyzed per extraction batch?	V			
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			<u></u>	
Were performance evaluation (PE) samples performed?		<b>V</b>	·	
Were the performance evaluation (PE) samples within the acceptance limits?			/	

LDC#: 2404786

#### VALIDATION FINDINGS CHECKLIST

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

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#: 24047B6

#### **VALIDATION FINDINGS WORKSHEET**

**Field Duplicates** 

Page:	1 of 1
Reviewer:	MG
2nd Reviewer:	

Inorganics, Method: See Cover

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

	Concentrati	on (mg/Kg)				
Analyte	3	4	RPD (≤50)	Difference	Limits	Qualification (Parent only)
Perchlorate	0.085	0.095	11			·

V:\FIELD DUPLICATES\FD\_inorganic\24047B6.wpd

	Concentrati	on (mg/Kg)			<del></del>	
Analyte	11	15	RPD (≤50)	Difference	Limits	Qualification (Parent only)
Perchlorate	1.7	1.8	6			

V:\FIELD DUPLICATES\FD\_inorganic\24047B6.wpd

98Lhohe LDC #:

# Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

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

> 3/4.0 METHOD: Inorganics, Method

8-10-20 \_ was recalculated. Calibration date:\_ The correlation coefficient (r) for the calibration of CIO 4

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

 $%R = Found \times 100$ True

Where, Found = concentration of each analyte  $\underline{\text{measured}}$  in the analysis of the ICV or CCV solution True = concentration of each analyte in the ICV or CCV source

Type of Analysis         Analyte         Standard ID         Found (units)         True (units)           Initial calibration         Blank         —         —         —           Standard 1         1.0         (Mg/L)         0.00303           Standard 2         3.5         (         0.00749           Standard 3         5.0         (         0.01734           Standard 5         20.0         (         0.05417           Standard 5         20.0         (         0.06935           Standard 6         40.0         (         0.14931           Standard 7         —         —         —           C1Ou         (         0.0         (         0.0           Calibration verification         (         0.0         (         0.0         0.0           C1Ou         (         0.0         0.0         0.0         0.0         0.0         0.0           C1Ou			Recalculated	Donortod	
Standard ID  Standard 2  Standard 2  Standard 3  CIOU  Standard 5  Standard 6  U436  CIOU  CLOU  CLOU					Acceptable
Standard 1   ClOy   Standard 5   Standard 5   Standard 5   Standard 6   ClOy   Standard 6   ClOy   CCV3O   CCV3O   ClOy   Cloy   CCV3O   Cloy   Cloy   Cloy   Cloy   CCV3O   Cloy   Clo		True (units)	r or %R	ror %R	(Y/N)
Standard 1   Standard 2   Standard 2   Standard 3   Standard 5   Standard 6   U Standard 6   U Standard 7   U 36	-	1			
Standard 2  Standard 3  C104  Standard 5  Standard 6  4  Standard 6  4  C104  CCV30  (438	0.0	0.00303			
Standard 3  C   Ou Standard 4 (10  Standard 5 3  Standard 6 4  Standard 7  (436  C   Ou CCV30 6  (438		0.00749			
C104 Standard 4 (6 Standard 5 3 Standard 6 4 Standard 6 4 (436 C104 CCV30 6 (438 (438 C10)		0.01734		: : : : :	`
Standard 5  Standard 6  U Standard 7  U U U U U U U U U U U U U U U U U U	2)	0.03417	0.1414.0	1 =0.744165	>
Standard 6 4 Standard 7  (436  C104  CCV30  (438		0.06135			
C104 CCV30 6	7	0.14931			
(436 C104 CCV30 6 (438 (438 (438 (438 (438 (438 (438 (438					
C104 CCV30 6					
(438) (438)		30.0 (49/2)	66	66	-
	38				
\ \ \ \		(7 km) 0.0e	96	96	
Calibration verification	)	i	a.comp		

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# 2404786

# VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

Page: Lof L Reviewer: MG 2nd Reviewer:

METHOD: Inorganics, Method 314.0

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

 $%R = \frac{Found}{T} \times 100$  Where,

Found =

concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation,

Found = SSR (spiked sample result) - SR (sample result).

True = concentration of each analyte in the source.

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

RPD = <u>IS-DI</u> x 100 Where, (S+D)/2

Original sample concentration

S ...

Duplicate sample concentration

					Recalculated	Reported	
Sample ID	Type of Analysis	Element	Found / S (units)	True / D (units)	%R/RPD	%R/RPD	Acceptable (Y/N)
1003	Laboratory control sample						
1527		C104	0.0948 (mg/kg) 0.100 (mg/kg)	0.100 (mg/kg)	26	25	>
9081	Matrix spike sample		(SSR-SR)				
23		6010	0.1006 (mg/kg) 0.104 (mg/kg)	0.104 (mg/ky)	16	16	
0261/5761	Duplicate sample						
35		Cloy	0.0019 U (mg/la) 0.0000 (mg/la)	0.0000 (mg/kg)	0	NC	

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#: 24047B6

# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	Lof_L
Reviewer:_	MG
2nd reviewer:	10

1	METH	IOD: Inorganics, Metho	d_314.0			
(	Y)N Y) N	N/A Have results N/A Are results w	ow for all questions answered "N". Not app been reported and calculated correctly? ithin the calibrated range of the instrumen tion limits below the CRQL?		e identified as "N/	A".
(	Comp	ound (analyte) results fulated and verified usin	or <u>#18 Clのy</u> g the following equation:	repo	orted with a positiv	ve detect were
(		ntration =	Recalculation: 0.00405 = 0.0037 (x	g/L)-0.0010		
w		m= 0.0037 b= -0.0010 dil= 1x	1.365 Mg/L = X (1.365 Mg/L then (10.9 g.)		0.0135 "9	for mg/k
	#	Sample ID	Analyte	Reported Concentration	Calculated Concentration (Mg/Kg)	Acceptable (Y/N)
	1	18	CIOY	0.014	0.014	Y
ſ	Note:_					

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 10, 2010

LDC Report Date:

October 15, 2010

Matrix:

Soil

Parameters:

Wet Chemistry

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-6345-1/ITH1781

#### Sample Identification

SSAJ3-07-SW-W-1BPC FD SSAI3-07-10BPC SSAI3-07-10BPC FD SB01-24BPC SB02-24BPC\*\*\* SSAI3-07-1BPC SB01-24BPC FD SSAI3-07-5BPC SSAJ3-02-SW-W-1BPCMS SSAJ3-06-1BPC SSAJ3-02-SW-W-1BPCMSD SSAJ3-06-5BPC SSAJ3-02-SW-W-1BPCDUP SSAJ3-06-10BPC SSAJ3-07-SW-W-1BPC FDMS SSAJ3-07-SW-E-1BPC\*\* SSAJ3-07-SW-W-1BPC FDMSD SSAJ3-05-SW-E-1BPC SSAJ3-07-SW-W-1BPC FDDUP SSAJ3-02-SW-E-1BPC SSAI3-04-SW-E-1BPC SSAI3-03-SW-E-1BPC SSAI3-02-SW-E-1BPC FD SSAI3-02-SW-E-1BPC SSAI3-02-SW-W-1BPC SSAI3-03-SW-W-1BPC SSAI3-04-SW-W-1BPC SSAJ3-02-SW-W-1BPC SSAJ3-05-SW-W-1BPC

SSAJ3-07-SW-W-1BPC\*\*

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

<sup>\*\*\*</sup>Indicates sample underwent Stage 4 review for hexavalent chromium, chlorate only

#### Introduction

This data review covers 30 soil samples listed on the cover sheet. The analyses were per EPA SW 846 Method 7199 for Hexavalent Chromium, EPA Method 314.0 for Perchlorate, and SW 846 Method 9056A for Chlorate.

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.

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

Samples indicated by a double asterisk on the front cover underwent a 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 contaminant concentrations were found in the initial, continuing and preparation blanks.

No field blanks were identified in this SDG.

#### IV. Matrix Spike/Matrix Spike Duplicates

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

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
SSAJ3-02-SW-W-1BPCMS/MSD (All samples in SDG 280-6345- 1/ITH1781)	Perchlorate	136 (75-125)	142 (75-125)	-	J+ (all detects)	А

#### V. Duplicates

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

#### VI. Laboratory Control Samples

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

#### VII. Sample Result Verification 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 280-6345- 1/ITH1781	All analytes reported below the PQL.	J (all detects)	А

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

Samples SSAl3-07-10BPC and SSAl3-07-10BPC\_FD and samples SSAl3-02-SW-E-1BPC\_FD and SSAl3-02-SW-E-1BPC and samples SSAJ3-07-SW-W-1BPC\*\* and SSAJ3-07-SW-W-1BPC\_FD and samples SB01-24BPC and SB01-24BPC\_FD were identified as field duplicates. No contaminant concentrations was detected in any of the samples with the following exceptions:

	Concentra	tion (mg/Kg)		D:#*			
Analyte	SSAI3-07-10BPC	SSAI3-07-10BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P	
Perchlorate	0.81	0.78	4 (≤50)	-	-	-	

	Concentrat	ion (mg/Kg)	-	D!#			
Analyte	SSAI3-02-SW-E-1BPC_FD	SSAI3-02-SW-E-1BPC	RPD (Limits)	Difference (Limits)	Flags	A or P	
Perchlorate	0.30	0.25	18 (≤50)	-	-	-	

	Concentra	tion (mg/Kg)	222	D://			
Analyte	SSAJ3-07-SW-W-1BPC**	SSAJ3-07-SW-W-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P	
Perchlorate	0.11	0.10	10 (≤50)	-	-	-	

	Concentra	tion (mg/Kg)	= RPD				
Analyte	SB01-24BPC	ABPC SB01-24BPC_FD		Difference (Limits)	Flags	A or P	
Perchlorate	250	85	99 (≤50)	-	J (all detects)	А	
Chlorate	510	920	57 (≤50)	•	J (all detects)	Α	

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Wet Chemistry - Data Qualification Summary - SDG 280-6345-1/ITH1781

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-6345- 1/ITH1781	SSAI3-07-10BPC SSAI3-07-10BPC_FD SSAI3-07-18PC SSAI3-07-5BPC SSAJ3-06-18PC SSAJ3-06-5BPC SSAJ3-06-10BPC SSAJ3-07-SW-E-1BPC** SSAJ3-07-SW-E-1BPC SSAJ3-02-SW-E-1BPC SSAI3-02-SW-E-1BPC SSAI3-02-SW-E-1BPC SSAI3-02-SW-E-1BPC SSAI3-02-SW-E-1BPC SSAI3-02-SW-E-1BPC SSAI3-02-SW-W-1BPC SSAI3-02-SW-W-1BPC SSAI3-03-SW-W-1BPC SSAJ3-07-SW-W-1BPC SSAJ3-07-SW-W-1BPC SSAJ3-07-SW-W-1BPC SSAJ3-07-SW-W-1BPCFD SB01-24BPC SB01-24BPC_FD	Perchiorate	J+ (all detects)	A	Matrix spike/Matrix spike duplicates (%R) (m)
280-6345- 1/ITH1781	SSAI3-07-10BPC SSAI3-07-10BPC_FD SSAI3-07-1BPC SSAI3-07-1BPC SSAI3-07-5BPC SSAJ3-06-1BPC SSAJ3-06-5BPC SSAJ3-06-10BPC SSAJ3-05-SW-E-1BPC** SSAJ3-05-SW-E-1BPC SSAI3-02-SW-E-1BPC SSAI3-02-SW-E-1BPC SSAI3-02-SW-E-1BPC SSAI3-02-SW-E-1BPC SSAI3-02-SW-BPC SSAI3-03-SW-W-1BPC SSAI3-04-SW-W-1BPC SSAI3-04-SW-W-1BPC SSAI3-07-SW-W-1BPC SSAJ3-07-SW-W-1BPC SSAJ3-07-SW-W-1BPC_FD SB01-24BPC SB01-24BPC_FD	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (sp)
280-6345- 1/ITH1781	SB01-24BPC SB01-24BPC_FD	Perchlorate Chlorate	J (all detects) J (all detects)	А	Field duplicates (RPD) (fd)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Wet Chemistry - Laboratory Blank Data Qualification Summary - SDG 280-6345-1/ITH1781

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Wet Chemistry - Equipment Blank Data Qualification Summary - SDG 280-6345-1/ITH1781

No Sample Data Qualified in this SDG

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

LDC #:_	24047E6
SDG #:_	280-6345-1/ITH1781
Laborate	ory: Test America

Stage 2B/4

Date:	10-6-10
Page:	of [
Reviewer:	

2nd Reviewer:

METHOD: (Analyte)	Hexav	alent Chromi	um (EPA	SW846 Me	thod 7199),	Perchlorate	(EPA Method 314.0	))
Chlorate 1-	EPA	SW-846	meth	9056A				

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	Α	Sampling dates: 8 - 10 - 10
lla.	Initial calibration	A	
IIb.	Calibration verification	A	
111.	Blanks	L A	
IV	Matrix Spike/Matrix Spike Duplicates	sw	M5/MSD
V	Duplicates	A	DUP
VI.	Laboratory control samples	A	LC5/LCSD
VII.	Sample result verification	A	Not reviewed for Stage 2B validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	SW	D=1+2 D=13+14 D=20+21 D=22+24
L <sub>x</sub>	Field blanks	N	EB - EB - 08102010 (from 280-6290-1)
ΧI	SULLOGUE		

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

valida	ated Samples: ** Indicates sar	derwent Stage 4 validation	mt				
1	SSAI3-07-10BPC	11	SSAI3-04-SW-E-1BPC	21	SSAJ3-07-SW-W-1BPC_FD	31	
2	SSAI3-07-10BPC_FD	12	SSAI3-03-SW-E-1BPC	22	SB01-24BPC	32	
3	SSAI3-07-1BPC	13	SSAI3-02-SW-E-1BPC_FD	23	SB02-24BP	33	
4	SSAI3-07-5BPC	14	SSAI3-02-SW-E-1BPC	1	SB01-24BPC_FD	34	
5	SSAJ3-06-1BPC	15	SSAI3-02-SW-W-1BPC	25	SSAJ3-02-SW-W-1BPCMS	35	
6	SSAJ3-06-5BPC	16	SSAI3-03-SW-W-1BPC	26	SSAJ3-02-SW-W-1BPCMSD	36	
7	SSAJ3-06-10BPC	17	SSAI03-04-SW-W-1BPC	27	SSAJ3-02-SW-W-1BPCDUP	37	
8	SSAJ3-07-SW-E-1BPC**	18	SSAJ3-02-SW-W-1BPC	28	SSAJ3-07-SW-W-1BPC_FDMS	38	
9	SSAJ3-05-SW-E-1BPC	19	SSAJ3-05-SW-W-1BPC	29	SSAJ3-07-SW-W-1BPC_FDMSD	39	PBSI
10	SSAJ3-02-SW-E-1BPC	20	SSAJ3-07-SW-W-1BPC**	30	SSAJ3-07-SW-W-1BPC_FDDUP	40	PB52

Notes:	<del>**</del> +,	2	foots	stop	4	<u> </u>	Will	deloute	mly	<b>.</b>
				<u> </u>				1		
									_	

#### VALIDATION FINDINGS CHECKLIST

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

Method:Inorganics (EPA Method See Co VCr)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.				
II. Calibration				
Were all instruments calibrated daily, each set-up time?	/			
Were the proper number of standards used?	/			
Were all initial calibration correlation coefficients > 0.995?	/			
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?	/		<u> </u>	
Were titrant checks performed as required? (Level IV only)			V	
Were balance checks performed as required? (Level IV only)		<u> </u>	/	
III. 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.		/		
IV. Matrix spike/Matrix spike duplicates and Duplicates		,	<b></b>	•
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.		<b>V</b>		
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq$ 20% for waters and $\leq$ 35% for soil samples? A control limit of $\leq$ CRDL( $\leq$ 2X CRDL for soil) was used for samples that were $\leq$ 5X the CRDL, including when only one of the duplicate sample values were $\leq$ 5X the CRDL.	1			
V. Laboratory control samples	<del>,</del>		•	
Was an LCS anaylzed for this SDG?	/			
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% (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?			/	

LDC #: 24047E6

#### **VALIDATION FINDINGS CHECKLIST**

Page:_	$\partial_{\text{of}} \partial$
Reviewer:	MG
2nd Reviewer:	كك

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.	1			
X. Field blanks				
Field blanks were identified in this SDG.				
Target analytes were detected in the field blanks.				

LDC#: 24047 E6

## VALIDATION FINDINGS WORKSHEET Sample Specific Analysis Reference

Page:_	
Reviewer:	MG
2nd reviewer:	

All circled methods are applicable to each sample.

Sample ID	Matrix	Parameter
1-701	S	PH TDS CLF NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ CO4
22,23		PH TDS CI F NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC (R6) (CIO4)
24		pH TDS CLF NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6 CIO4 C103
QC 25-30		pH TDS CLF NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ CO4
		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 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 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 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 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 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 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 CLF NO3 NO2 SO4 PO4 ALK CN NH3 TKN TOC CR6+ ClO4
		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 CLE NO, NO, SO, PO, ALK CN NH, TKN TOC CR <sup>6+</sup> CIO,

Comments:		

# LDC# JHOY7E6

## VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Page: 1 of 1 Z S Reviewer:\_ 2nd Reviewer:

METHOD: Inorganics, EPA Method\_

see cover

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

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

of 4 or more, no action was taken.

Were all duplicate sample relative percent differences (RPD) < 20% for water samples and <35% for soil samples? KN N/A Wer LEVEL IV ONLY: N N/A We

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

Qualifications	J+dets/A m													
Associated Samples	DEAT AM	a11									7			
RPD (Limits)	my			,										
MSD %Recovery	(75-13E) CHI													
MS MSD %Recovery %Recovery	136 (25-135)													
Analyte	C104													
Matrix	Soil													
MS/MSD ID	25/26							100 Maria 1100 Maria 1						
*									$\exists$					

Comments:

#### LDC#: 24047E6

## VALIDATION FINDINGS WORKSHEET Field Duplicates

Page:	į	of_	1
Reviewer:_		1 <u>G</u>	
2nd Reviewer:		~	_

Inorganics, Method: See Cover

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

	Concentrati	on (mg/Kg)				Ovelification	
Analyte	1	2	RPD (≤50)	Difference	Limits	Qualification (Parent only)	
Perchlorate	0.81	0.78	4				

V:\FIELD DUPLICATES\FD\_inorganic\24047E6.wpd

	Concentrati	on (mg/Kg)				Ovalification	
Analyte	13 14		RPD (≤50)	Difference	Limits	Qualification (Parent only)	
Perchlorate	0.30	0.25	18				

V:\FIELD DUPLICATES\FD\_inorganic\24047E6.wpd

	Concentrati	on (mg/Kg)				0	
Analyte	20 21		RPD (≤50)	Difference	Limits	Qualification (Parent only)	
Perchlorate	0.11	0.10	10			·	

V:\FIELD DUPLICATES\FD\_inorganic\24047E6.wpd

	Concentrati	on (mg/Kg)				Qualification	
Analyte	22	24	RPD (≤50)	Difference	Limits	(Parent only)	
Perchlorate	250	85	99			J dets/ A fd	
Chlorate	510	920	57			J dets/ A fd	

V:\FIELD DUPLICATES\FD\_inorganic\24047E6.wpd

LDC# 34047E6

# Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: 1 of 1 Reviewer: MG 2nd Reviewer:

METHOD: Inorganics, Method\_

see aver

The correlation coefficient (r) for the calibration of C ( O 3

8-17-10 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,  $%R = Found \times 100$ True

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

			•		Recalculated	Reported	
			0 vo	4 rea			Acceptable
Type of Analysis	Analyte	Standard ID	Found (units)	True (units)	ror %R	r or %R	(Y/N)
Initial calibration		Blank	ì	)			
		Standard 1	0.1 (mg/L)	0.008			
		Standard 2	0.5 (   )	0.038			
		Standard 3	1.0 ( )	0.055		<u>`</u>	•
·	C103	Standard 4	4.0 ( )	0.328	V=0.99974	1 = 0. 99932	<b>&gt;</b>
		Standard 5	8.0	0.482			
		Standard 6	( ) 0.01	9.606			
		Standard 7	1	•			-,
Calibration verification	1	1453					
	C104	ICV	19.7333 (mg/L) 30.	20. (mg/L)	66	66	
Calibration verification		hoLO					
	C103	CCV	4.8861 (m3/2)	1.8861 (mg/L) 5.00 (mg/L)	98	98	
Calibration verification		1643		lux (1)		not,	
	Cr V1	CCV	51.9888 (48/1)	1.9888 (48/2) 50. (43/2)	104	reported	<del>&gt;</del>

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# 24047 EC

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

Reviewer: MG Page: / of 2nd Reviewer:

> See over METHOD: Inorganics, Method \_\_

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

Where, %R = Found × 100

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:

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

S= 0

Duplicate sample concentration Original sample concentration

					Recalculated	Reported	
Sample ID	Type of Analysis	Element	Found / S (units)	True / D (units)	%R / RPD	%R/RPD	Acceptable (Y/N)
1442	Laboratory control sample						The state of the s
527		Cr v?	14.83 (mg/kg)	14.83 (mg/kg) 16.0 (mg/kg)	93	93	>
1895	Matrix spike sample		(SSR-SR)				
38		C104	0.142 (mg/kg) 0.103 (mg/kg	0.103 (mg/kg)	138	136	
4081/ EhL1	Duplicate sample						
27		4012	0.096 (mg/kg) 0.098 (mg/kg)	0.098 (mg/kg)	8	R	>

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#: 24047E6

#### **VALIDATION FINDINGS WORKSHEET**

Sample Calculation Verification

Page:	of
Reviewer:_	MG
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METHO	D: Inorganics, Metho	d See cover			
	see qualifications belo <u>I/A</u> Have results <u>I/A</u> Are results w	ow for all questions answered "N". Not app been reported and calculated correctly? ithin the calibrated range of the instrumention limits below the CRQL?		e identified as "N/.	<b>A</b> ".
Compo recalcu	und (analyte) results f lated and verified usin	or # 33 CIO3	repo	orted with a positiv	e detect were
Concentr y ≃ ₩	ration =	Recalculation: $0.347 = 0.059 \left(\frac{x}{500}\right) + 0.0$	01	·	
nler	e = 0.059 b = 0.001 $dil = 500 \times$	7084.7 mg/L = X then (2084.7 mg/L)	(0.100L) (0.742)=	27277.4	mg/kg
#	Sample ID	Analyte	Reported Concentration (Mg/kg)	Calculated Concentration (Mg / kg)	Acceptable (Y/N)
	8	C104	32	32	Ý
2	23	C103	27000	27000	
		CrvI	140	140	<b>b</b>
-					
-					
Note:_		William State of the Control of the			