

### Laboratory Data Consultants, Inc.

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Northgate Environmental Management, Inc.

December 30, 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 December 6, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

### **LDC Project # 24522:**

### SDG#

### Fraction

280-8906-1/ITJ2616, 280-8912-1 280-9160-1, 280-9188-1 280-9309-1, 280-9309-2 280-9771-1

Semivolatiles, Chlorinated Pesticides, Metals, Perchlorate

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 Inorganic Data Review, October 2004

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

Sincerely,

Erlinda T. Rauto

**Operations Manager/Senior Chemist** 

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Pest. (8081A)

SVOA (8270C)

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SDG# Water/Soil

Matrix: 임

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LDC #: 24522

SDG #: 280-8906-1/ITJ2616, 280-8912-1, 280-9160-1, 280-9188-1 280-9309-1, 280-9309-2, 280-977-1 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		語·德文 公司		
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?			х	See EDD_discrepancy_ form_LDC24522_122810.doc
IV. EDD Delivery				
Was the final EDD sent to the client?	x			

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

October 22, 2010

LDC Report Date:

December 18, 2010

Matrix:

Soil/Water

Parameters:

**Semivolatiles** 

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-8912-1

### Sample Identification

SSAP3-03-1\_01\_BPC SSAP3-03-5 01 BPC

SSAP3-03-9 01 BPC

SSAP3-04-1 01 BPC

SSAP3-04-1\_01\_BPC\_FD

SSAP3-04-5 01 BPC

SSAP3-04-9 01 BPC\*\* EB-10222010-RZC

SSAP3-03-9\_01 BPCMS SSAP3-03-9 01 BPCMSD

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

### Introduction

This data review covers 9 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.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

### III. Initial Calibration

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 30.0% for all compounds.

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

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

### IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

Percent differences (%D) between the initial calibration RRF and the continuing calibration RRF were within the method criteria of less than or equal to 20.0% for calibration check compounds (CCCs) and 25.0% for all other compounds.

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

All of the continuing calibration relative response factors (RRF) were within method and validation criteria.

### V. Blanks

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

Method Blank ID	Extraction Date	Compound TIC (RT in minutes)	Concentration	Associated Samples
MB 280-37601/1-A	10/27/10	Bis(2-ethylhexyl)phthalate	69.3 ug/Kg	All soil samples in SDG 280-8912-1

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
SSAP3-03-1_01_BPC	Bis(2-ethylhexyl)phthalate	78 ug/Kg	78U ug/Kg
SSAP3-03-5_01_BPC	Bis(2-ethylhexyl)phthalate	76 ug/Kg	76U ug/Kg
SSAP3-03-9_01_BPC	Bis(2-ethylhexyl)phthalate	78 ug/Kg	78U ug/Kg
SSAP3-04-1_01_BPC	Bis(2-ethylhexyl)phthalate	76 ug/Kg	76U ug/Kg
SSAP3-04-1_01_BPC_FD	Bis(2-ethylhexyl)phthalate	71 ug/Kg	71U ug/Kg
SSAP3-04-9_01_BPC**	Bis(2-ethylhexyl)phthalate	75 ug/Kg	75U ug/Kg

Sample EB-10222010-RZC 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 280-8912-1	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 SSAP3-04-1\_01\_BPC and SSAP3-04-1\_01\_BPC\_FD were identified as field duplicates. No semivolatiles were detected in any of the samples with the following exceptions:

	Concentr	ration (ug/Kg)				
Compound	SSAP3-04-1_01_BPC	SSAP3-04-1_01_BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Bis(2-ethylhexyl)phthalate	76	71	-	5 (≤350)	-	-

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-8912-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-8912-1	SSAP3-03-1_01_BPC SSAP3-03-5_01_BPC SSAP3-03-9_01_BPC SSAP3-04-1_01_BPC SSAP3-04-1_01_BPC_FD SSAP3-04-5_01_BPC SSAP3-04-5_01_BPC** EB-10222010-RZC	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 280-8912-1

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	_Code
280-8912-1	SSAP3-03-1_01_BPC	Bis(2-ethylhexyl)phthalate	· 78U ug/Kg	А	Ы
280-8912-1	SSAP3-03-5_01_BPC	Bis(2-ethylhexyl)phthalate	76U ug/Kg	А	ы
280-8912-1	SSAP3-03-9_01_BPC	Bis(2-ethylhexyl)phthalate	78U ug/Kg	А	bl
280-8912-1	SSAP3-04-1_01_BPC	Bîs(2-ethylhexyl)phthalate	76U ug/Kg	А	bl
280-8912-1	SSAP3-04-1_01_BPC_FD	Bis(2-ethylhexyl)phthalate	71U ug/Kg	А	bl
280-8912-1	SSAP3-04-9_01_BPC**	Bis(2-ethylhexyl)phthalate	75U ug/Kg	А	ld

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Field Blank Data Qualification Summary - SDG 280-8912-1

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson**

LDC #: <u>24522B2a</u>	_VALIDATION COMPLETENESS WORKSHEET
SDG #: 280-8912-1	Stage 2B/4
Laboratory: Test America	

	Date:	12/15/10
	Page:_	
	Reviewer:	<del></del>
2nd	Reviewer:	<u> </u>
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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: 10 /22/to
II.	GC/MS Instrument performance check	Á	,
III.	Initial calibration	A	? RSD rv
IV.	Continuing calibration/ICV	A	CW/ICV = 25 b
V.	Blanks .	SW	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	Δ	
VIII.	Laboratory control samples	A	les b
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	K	
XI.	Target compound identification	A	Not reviewed for Stage 2B validation.
XII.	Compound quantitation/CRQLs	<i>*</i>	Not reviewed for Stage 2B validation.
XIII.	Tentatively identified compounds (TICs)	Ŋ	Not reviewed for Stage 2B validation.
XIV.	System performance	1	Not reviewed for Stage 2B validation.
XV.	Overall assessment of data	A	
XVI.	Field duplicates	<u>Su</u>	D = 4.5.
XVII.	Field blanks	ND)	th = 8

A = Acceptable Note:

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:

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1	SSAP3-03-1_01_BPC S	打	MB 280-37601/1	-21	31	
2	SSAP3-03-5_01_BPC	12	MB 280-38179 XX	-22	32	
3	SSAP3-03-9_01_BPC	13	./	23	 33	
4	SSAP3-04-1_01_BPC 1	14		24	34	
5	SSAP3-04-1_01_BPC_FD D	15		25	 35	
6	SSAP3-04-5_01_BPC	16		26	 36	
7	SSAP3-04-9_01_BPC**	17		27	37	
8 7	EB-10222010-RZC W	18		28	38	
9	SSAP3-03-9_01_BPCMS 5	19		29	 39	
10	SSAP3-03-9_01_BPCMSD V	20		30	 40	

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Reviewer: \( \frac{1}{2} \)
2nd Reviewer: \( \frac{1}{2} \)

Method: Semivolatiles (EPA SW 846 Method 8270C)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times	344	rigina)		design and the constraint of t
All technical holding times were met.				
Cooler temperature criteria was met.				
II. GC/MS Instrument performance check		***		
Were the DFTPP performance results reviewed and found to be within the specified criteria?				
Were all samples analyzed within the 12 hour clock criteria?				
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?				
Was a curve fit used for evaluation?				
Did the initial calibration meet the curve fit acceptance criteria of ≥ 0.990?	/			
Were all percent relative standard deviations (%RSD) ≤ 30% and relative response factors (RRF) ≥ 0.05?				
IV. Continuing calibration				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?				
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?				
Were all percent differences (%D) ≤ 25% and relative response factors (RRF) ≥ 0.05?				
V. Blanks			17.1	
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	(5)		16-7	1. 1987 (1983) [1] 12 12 12 12 13 14 14 15 15 15 15 15 15 15 15 15 15 15 15 15
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.				
Was a MS/MSD analyzed every 20 samples of each matrix?				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII. Laboratory control samples				
Was an LCS analyzed for this SDG?				

LDC #: 245 m b walldation findings checklist

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

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?	2000			
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?	2		*******	
XI. Target compound identification	Breit of	15 G A		
Were relative retention times (RRT's) within ± 0.06 RRT units of the standard?				
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	<u> </u>			
Were chromatogram peaks verified and accounted for?	$\angle$			
XII. Compound quantitation/CRQLs	rition of	T		
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	faj sejak	(e) (a)	15.8	
System performance was found to be acceptable.	<i>i</i>			
XV. Overall assessment of data			<b>94</b> (1	
Overall assessment of data was found to be acceptable.	1			
XVI: Field:duplicates		14.1		
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.				
XVII. Field blanks				An plant of the Ball of the state of the sta
Field blanks were identified in this SDG.		i/		
Target compounds were detected in the field blanks.			$\Box$	

# VALIDATION FINDINGS WORKSHEET

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

					;										
	III. Benzo(a)pyrene⁴	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	OOO. N-Nitrosodimethylamine	PPP. Benzoic Acid	QQQ. Benzyl alcohol	RRR. Pyridine	SSS. Benzidine	TTT.	ດທາ	VW.	WWW.
	TT. Pentachlorophenoi**	UU. Phenanthrene	VV. Anthracene	WW. Carbazole	XX. Di-n-butylphthalate	YY. Fluoranthene**	ZZ. Pyrene	AAA. Butylbenzylphthalate	BBB. 3,3'-Dichlorobenzidine	CCC. Benzo(a)anthracene	DDD. Chrysene	EEE. Bis(2-ethylhexyl)phthalate	FFF. Di-n-octy/phthalate**	GGG. Benzo(b)fluoranthene	HHH. Benzo(k)fluoranthene
	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	OO. 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-Trichlorophenol**	Z. 2,4,5-Trichlorophenol	AA. 2-Chloronaphthalene	BB. 2-Nitroaniline	CC. Dimethylphthalate	DD. Acenaphthylene
7 3047	A. Phenol**	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-Methylphenoi	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.

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LDC #:_	

## VALIDATION FINDINGS WORKSHEET

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Page:	Reviewer:	2nd Reviewer:

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

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

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

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

Y/N N/A

Was the blank contaminated? If yes, please see qualification below. If  $\sqrt{27/\omega}$  Blank analysis date:  $11/6 > \hbar \sigma$ Blank extraction date: N N/A

(88) . 4 Sample Identification 16 Associated Samples: η/ 78 10075-8843M Blank 1D 6 EFF Compound Conc. units:

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

Common contaminants such as the phthalates and TICs noted above that were detected in samples within ten times the associated method blank concentration were also qualified as not detected, "U". Other contaminants within five times the method blank concentration were also qualified as not detected, "U".

LDC#: 795~ 876

### VALIDATION FINDINGS WORKSHEET Field Duplicates

Page:_	<u></u> of_/
Reviewer:	N/L
2nd reviewer:	0

ETHOD: GC/MS BNA (EPA SW 846 Method 82	70)		
N N/A  Were field duplicate pairs identified  N N/A  Were target compounds identified	ed in this SDG? d in the field duplicate	pairs?	
	Concentration	1 45/kg	
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## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

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:

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

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

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

S= Standard deviation of the RRFs,

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

%RSD

4.82 3.00 1.65 7.64

2.55

Recalculated Reported %RSD 2.5 7.6 4. 8 3.0 1.7 Average RRF Recalculated 0.5399 1.0263 1.2599 0.9702 (Initial) 0.2392 Average RRF Reported 0.5398 1.0263 1.2599 0.9702 0.2392 (Initial) Recalculated (50 std) 0.5276 1.0330 1.2852 0.2387 0.9867 see r2 calculations Reported 50 std) 0.5276 1.0330 1.2852 0.2387 RRF 0.9867 (181) (182) (183) (184) (185) (186) Compound (IS) Benzo(g,h,i)perylene Hexachlorobenzene bis(2eh)phthalate Naphthalene 10/21/2010 1,4-Dioxane Fluorene Calibration Date Standard ID MSS Y ICAL #

c IS/Cpd	Area cpd	Area IS	Conc	1.4-Dioxane
40/50	173342	262843	4 00	0.5556
40/50	1363100	1055622	10.00	0.5952
40/50	1029593	640883	20.00	0.5481
40/50	328282	1100046	50.00	0.5276

1179220 1227402

1620175 1454460

40/20 40/50

Conc	1,4-Dioxane	Naphthalene	Fluorene	Hexachlorob	bis(2eh)phthalat Benzo(g,h,i)per	Benzo(g,h,i)per
4.00	0.5556	1.0630	1.2369		r2	0.8201
10.00	0.5952	1.0515	1.2647	0.2338		0.9115
20.00	0.5481	1.0484	1.2425	0.2301		0.9507
50.00	0.5276	1.0330	1.2852	0.2387		0.9867
80.00	0.5258	1.0388	1.2718	0.2376		1.0052
120.00	0.5245	1.0037	1.2880	0.2459		1.0260
160.00	0.5153	0.9954	1.2542	0.2414		1.0226
200.00	0.5268	0.9765	1.2360	0.2469		1.0388
×	0.5399	1.0263	1.2599	0.2392	0.0000	0.9702
S	0.0260	0.0307	0.0208	0.0061	#DIV/0i	0.0741

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# TOSAN BYA

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

Page:\_\_

· 2nd Reviewer:\_ Reviewer:

METHOD: GCMS Semivolatiles (EPA SW 846 Method 8270C)

Parameter: bis(2-ethylhexyl) phthalate

Order of regression:

Linear

conc ratio 0.500 1.250 2.000 3.000 0.100 0.274694956 0.763439819 1.284374221 1.930453843 0.034710802 3.214338448 area ratio Point 2 Point 3 Point 5 Point 6 Point 4 Point 8 Points Point 1 Point 7 bis(2-ethylhexyl) phthalate Compound Column DB-624 1-Nov-10 Date

0.3471 0.4688 0.5494 0.6108 0.6422 0.6435 0.6435 Ave 0.5685

Regression Output: Regression Output:		Reported WLR	
Constant		11 0	0.04930
Std Err of Y Est	0.04		
R Squared	06666.0	r^2 =	0.99810
No. of Observations	00.9		
Degrees of Freedom	4.00		
		m1 =	0.6407
X Coefficient(s)	0.65305		
Std Err of Coef.	0.01		

## LDC # 24522 829

## Continuing Calibration Results Verification VALIDATION FINDINGS WORSHEET

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

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

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

Where:

% 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

verage RRF         Reported         Recalculated         Recalculated         Recalculated           Initial RRF)         (CC RRF)         (CC RRF)         %D         %D           0.5398         0.5667         5.0         5.0         5.0           1.0263         1.0851         1.0851         5.7         5.7           1.2599         1.3207         4.8         4.8           0.2392         0.2411         0.8         0.8           80.0000         87.3923         9.1         9.1           0.9702         1.1305         1.1305         16.5         16.5					-					
(CC RRF)     (CC RRF)     %D       0.5667     0.5667     5.0       1.0851     1.0851     5.7       1.3207     1.3207     4.8       0.2411     0.2411     0.8       87.3000     87.2923     9.1       1.1305     1.1305     16.5	Calibration	Calibration	_			Average RRF	Reported	Recalculated	Reported	Recalculated
0.5667     0.5667     5.0       1.0851     1.0851     5.7       1.3207     1.3207     4.8       0.2411     0.2411     0.8       87.3000     87.2923     9.1       1.1305     1.1305     16.5       .     .	Standard ID Date Compound (IS)	Compound	- 1			(Initial RRF)	(CC RRF)	(CC RRF)	Q%	Q%
1.0851     1.0851     5.7       1.3207     1.3207     4.8       0.2411     0.2411     0.8       87.3000     87.2923     9.1       1.1305     1.1305     16.5       .     .	Y6516 11/02/10 1,4-Dioxane (IS1)			S1)		0.5398	0.5667	0.5667	5.0	5.0
1.3207 1.3207 4.8 0.2411 0.2411 0.8 87.3000 87.2923 9.1 1.1305 1.1305 16.5	Naphthalene (IS2)			(25)		1.0263	1.0851	1.0851	5.7	5.7
0.2411     0.2411     0.8       87.3000     87.2923     9.1       1.1305     1.1305     16.5       .     .	Fluorene (IS3)			(23)		1.2599	1.3207	1.3207	4.8	4.8
87.3000     87.2923     9.1       1.1305     1.1305     16.5       .     .	Hexachlorobenzene (IS4)			S <del>4</del> )		0.2392	0.2411	0.2411	8.0	0.8
1.1305 1.1305 16.5	bis(2eh)phthalate (IS5)			(92)		80.0000	87.3000	87.2923	9.1	9.1
	Benzo(g,h,i)perylene (IS6)	Benza(g,h,i)perylene (IS6)	Benzo(g,h,i)perylene (IS6)	- (9)	- 1	0.9702	1.1305	1.1305	16.5	16.5
			,							

Compound (Reference IS)	nce IS)	Concentration	Area Cpd	Area IS
		(IS/Cpd)		
1,4-Dioxane	(1S1)	40/80	393857	347510
Naphthalene	(182)	40/80	2968068	1367606
Fluorene	(183)	40/80	2188761	828609
Hexachlorobenzene	(IS4)	40/80	667574	1384246
bis(2eh)phthalate	(185)	40/80	2061130	1508197
Benzo(g,h,i)perylene	(186)	40/80	3223666	1425823

0.6407 bis(2eh)phthala

Ε

0.0493

Response Ratio\*40 1.366618552

87.29234037

Ω.

Conc

LDC#: 24577 32a

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

Page:	<u>l</u> of <u>1</u>
Reviewer:_	T/L
2nd reviewer:_	è
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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	(0)	83.7	84	8 4	9
2-Fluorobiphenyl		82.5	83	83	
Terphenyl-d14	}	100.4	10	100	1
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-Fluorophenoi					***************************************
2,4,6-Tribromophenol					,
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

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

LDC# 24522 p 2a

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

30 Page: lof 1 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 = 1 MSC - MSC 1\* 2/(MSC + MSDC)

MSC = Matrix spike concentration

**o**/ o MS/MSD samples:

	S	Spike	Sample	Spiked Sample	Sample	Matrix Spike	Spike	Matrix Spike Duplicate	- Duplicate	MS/MSD	Q.
Compound	Adde ( MS /	/ kg	Concentration (45 /c.)	Concen	tration )	Percent Recovery	ecovery	Percent Recovery	ecovery	RPD	
	MS	l V	2	MS	MSD	Reported	Recalc	Reported	Recalc	Ronorted	Donalanda
Phenoi											na parameter
N-Nitroso-di-n-propylamine											
4-Chloro-3-methylphenol											
Acenaphthene	2570	2890	٥	. 26 30	2760	2	45	26	96	15	را
Pentachlorophene!											
Pyrene	>	7	8)	2780	26.62	76	94	66	0	9	Q
		>			_						
			4 L								=

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

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)

LCS/LCSD samples: LCS/ 250-78779 /25-4

	aS	ike	5	Spike		93				
	10 V	Added	2	ine		4	9	CSD	103/	LCS/I CSD
Compound	(49/1	九)	(M)	(M/I)	Percent	Percent Recovery	Percent Recovery	Secovery	Ω.	RPD
	l CS	LCSD	168	(CSD)	Reported	Donale	7 7 7 6 6 6		TI.	
Phenol						The last	x eported	Kecalc	Keported	Recalculated
N-Nitroso-di-n-propylamine										
4-Chloro-3-methylphenol										
Acenaphthene	0 X	8	593	621	74	74	7,4	× 7×	p	L,
Pentachierephenol	) <del></del>				-	,	)	2	1	,
Pyrene	7		8.69	199	58	80	23	× ×	4	Þ
		•								
1										

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#: 24 522 \$24

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	<u></u>
Reviewer:	16
2nd reviewer:	
·	

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

	$\overline{}$		
	Υ	<u>N</u>	N/A
(	$\overline{Y}$	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?

/				
	Concer	tration	$n = (A_{s})(I_{s})(V_{s})(DF)(2.0)$ $(A_{s})(RRF)(V_{s})(V_{s})(%S)$	Example:
	A <sub>x</sub>	=	Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D. # 7 , EEE
	$A_{is}$	=	Area of the characteristic ion (EICP) for the specific internal standard	T7
	l <sub>s</sub>	=	Amount of internal standard added in nanograms (ng)	$\begin{bmatrix} \text{Cond} & \frac{1}{2445} & \frac{1}{237} & 1$
	V.	=	Volume or weight of sample extract in milliliters (ml) or grams (g).	(0.6407) + 2.6493
	$V_{i}$	=	Volume of extract injected in microliters (ul)	
	$V_{\iota}$	=	Volume of the concentrated extract in microliters (ul)	
	Df	=	Dilution Factor.	x = 2.088
	%S	=	Percent solids, applicable to soil and solid matrices only.	1 - 2,000
				final cone = (2088) (IM) (

(1000) 2.0 Factor of 2 to account for GPC cleanup Reported ( Concentration Concentration Qualification Sample ID Compound # 58 × 75

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

October 29, 2010

LDC Report Date:

December 18, 2010

Matrix:

Soil

Parameters:

Semivolatiles

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-9188-1

### Sample Identification

SSAM3-02-14\_01\_BPC

SSAM3-02-15 01 BPC

SSAM3-02-16\_01\_BPC

SSAM3-02-17\_01\_BPC

SSAM3-02-18 01 BPC

SSAM3-02-19\_01\_BPC

SSAM3-02-20\_01\_BPC\*\*

SSAM3-02-18 01 BPCMS

SSAM3-02-18 01 BPCMSD

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

### Introduction

This data review covers 9 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.

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.

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 280-9188-1	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

No field duplicates were identified in this SDG.

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-9188-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-9188-1	SSAM3-02-14_01_BPC SSAM3-02-15_01_BPC SSAM3-02-16_01_BPC SSAM3-02-17_01_BPC SSAM3-02-18_01_BPC SSAM3-02-19_01_BPC SSAM3-02-20_01_BPC**	All compounds reported below the PQL.	J (ali detects)	A	Project Quantitation Limit (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-9188-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Field Blank Data Qualification Summary - SDG 280-9188-1

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson** IEET

LDC #: 24522D2a	VALIDATION COMPLETENESS WORKSH
SDG #: 280-9188-1	Stage 2B/4
Laboratory: Test America	<u> </u>

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: lo / 2.9 /ro
II.	GC/MS Instrument performance check	A	, ,
III.	Initial calibration	Ĥ	% RSD
IV.	Continuing calibration/ICV	A	CW/1W 625)
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	ICS
IX.	Regional Quality Assurance and Quality Control	N	电
Χ	Internal standards	A	
XI.	Target compound identification	A A	Not reviewed for Stage 2B validation.
XII.	Compound quantitation/CRQLs	А	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	И	

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:

Can \*\* Indicates sample underwent Stage 4 validation

	2011					
1	SSAM3-02-14_01_BPC	11	MR 280- 31301 /-A	21	31	
2	SSAM3-02-15_01_BPC	12	/	22	32	
3	SSAM3-02-16_01_BPC	13		23	 33	
4	SSAM3-02-17_01_BPC	14		24	34	
5	SSAM3-02-18_01_BPC	15		25	35	
6	SSAM3-02-19_01_BPC	16		26	36	
7	SSAM3-02-20_01_BPC**	17		27_	37	
8	SSAM3-02-18_01_BPCMS	18		28	38	·
9	SSAM3-02-18_01_BPCMSD	19		29	39	
10		20		30	40	

LDC#: 2f522 Dra

### VALIDATION FINDINGS CHECKLIST

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

Method: Semivolatiles (EPA SW 846 Method 8270C)

Method: Semivolatiles (EPA SW 846 Method 8270C)	<del></del>	Γ	f	
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	564			O COMO CONTRA PERMANDIE PRE ENERGY DESE
Were the DFTPP performance results reviewed and found to be within the specified criteria?				
Were all samples analyzed within the 12 hour clock criteria?	i imamum dorasi	Chailean Aleman e co	(7) No. (5) (1) (1)	THE CONTROL OF THE CO
III. Initial calibration				. Ellin er en fram fra vitter fra en er en
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?	<u></u>			
Was a method blank analyzed for each matrix and concentration?	/			· ·
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/	,	
VI. Surrogate spikes				
Were all surrogate %R within QC limits?				
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?				
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?		:		
VII. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.				
Was a MS/MSD analyzed every 20 samples of each matrix?				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII. Laboratory control samples	100	Page.	1	
Was an LCS analyzed for this SDG?				

LDC #: 4522 D16

### 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				
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			11.00	
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?			1	-
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.:Fleld:dupl)cates				
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.			_	
XVII. Field blanks			en E	
Field blanks were identified in this SDG.				
Target compounds were detected in the field blanks.			1	

## LDC # 74 522 D 79

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Reviewer:\_ Page:

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

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

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

%RSD

10.75 10.65 4.39 9.82

3.14

2.02

%RSD = 100 \* (S/X)

			Reported	Recalculated	Reported	Receivitated	Poportod
Calibration			RRF	RRF	Average RRF	Average RRF	%RSD
Standard ID Date		Compound (Internal Standard)	(50 std)	(50 std)	(Initial)	(Initial)	) }
10/26/2010 1,4	4,	1,4-Dioxane (IS1)	0.5482	0.5482	0.5608	0.5608	2.0
Nar	Nag	Naphthalene (IS2)	0.9571	0.9571	0.9636	0.9636	10.8
йШ	副	Fluorene (IS3)	1.2032	1.2032	1.2146	1.2146	10.6
Hex	¥	fexachlorobenzene (IS4)	0.2428	0.2428	0.2399	0.2399	4.4
Chr	Chr	Chrysene (IS5)	1.0446	1.0446	1.0340	1.0340	9.8
Ber	8	Benzo(a)pyrene (IS6)	1.0378	1.0378	1.0351	1.0351	3.1

_	_					
Area IS	211523	829323	488301	822856	961386	965779
Area cpd	144944	992147	734415	249703	1255273	1252892
inc IS/Cpd	40/50	40/50	40/50	40/50	40/50	40/50

_						
Conc	1,4-Dioxane	Naphthalene	Fluorene	Hexachtoro	Chrysene	Benzo(a)py
4.00	0.5705	1.1065	1.3730		1.1650	0.9826
10.00	0.5633	1.0782	1.3549	0.2518	1.1398	1.0026
20.00	0.5694	1.0393	1.3330	0.2500	1.1160	1.0611
50.00	0.5482	0.9571	1.2032	0.2428	1.0446	1.0378
80.00	0.5773	0.9554	1.2098	0.2456	1.0336	1.0802
120.00	0.5528	0.8910	1.1337	0.2360	0.9499	1.0621
160.00	0.5457	0.8514	1.0672	0.2275	0.9110	1.0235
200.002	0.5595	0.8298	1.0417	0.2253	0.9124	1.0306
×	0.5608	0.9636	1.2146	0.2399	1.0340	1.0351
S	0.0113	0.1036	0.1293	0.0105	0.1015	0.0325

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# 29522022

# VALIDATION FINDINGS WORSHEET Continuing Calibration Results Verification

Page of A Reviewer: JVG 2nd Reviewer: &

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

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

% 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

Cx = Concentration of compound, Cis = Concentration of internal standard

Recalculated 12.6 9.0 0. 0.4 ა მ 7.7 Reported ₽% 12.6 9.0 0.4 1.0 7. 3.9 Recalculated (CC RRF) 0.6315 0.2390 1.0516 1.2267 1.0756 0.9697 (CC RRF) Reported 0.6315 0.2390 1.0516 1.0756 0.9697 1.2267 Average RRF (Initial RRF) 1.2146 0.5608 0.9636 0.2399 1.0340 1.0351 (182) (183) (1S1) (IS4) (185) (186) Compound (Reference IS) Hexachlorobenzene Benzo(a)pyrene Naphthalene 1,4-Dioxane Fluorene Chrysene Calibration 11/08/10 Date Standard ID K7783 #

		CCV1		CCV2.	
Compound (Reference IS)	Concentration (IS/Cpd)	Area Cpd	Area IS	Area Cpd	Area IS
1,4-Dioxane (IS1)	40/80	331320	262325		
Naphthalene (IS2)	40/80	1988285	1025242		
Fluorene (1S3)	40/80	1487531	606322		
Hexachlorobenzene (IS4)	40/80	500000	1045965		
Chrysene (IS5)	40/80	2425835	1153403		
Benzo(a)pyrene (IS6)	40/80	2604334	1210627		

LDC#: 24522 DZa

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

Page: lof\_1 Reviewer: 16 2nd reviewer:\_\_

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Sample ID: # 7

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	107	7 2.7	73	73	σ
2-Fluorobiphenyl		77.4	77	77	
Terphenyl-d14	-	97,0	9~	92	8
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					

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# 7957 DX

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

-of  -	174	, 0
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:

Where:

SC = Sample concentation

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

SSC = Spiked sample concentration SA = Spike added

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples:

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

	<i>'</i> \$'.	oike.	Sample	Spiked S	ample	Matrix Spike	Spike	Matrix Spike Duplicate	Duplicate	MS/MSD	SD
Compound	, A	Added (U) /c)	Concentration ( Mg/kg/	Concentration ( ५९) (से )	trațion (व	Percent Recovery	ecovery	Percent Recovery	есоvегу	RPD	
	MS	O MSD	0	MS	MSD	Reported	Recalc	Reported	Pocalc	Topico Co	
Phenol										n <del>a</del> nroday.	Kecalcinated
N-Nitroso-di-n-propylamine											
4-Chloro-3-methylphenol										,	
Acenaphthene	2840	2940	اح	2/20	244D	74	74	ڮ	74	7/	14
Pentachlorophenol			-		-						
Pyrene	>-		0	or72 op25.	2720	4)	15	93	93	6	75
								1		./	,

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

# 

Reviewer: 17/2 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

RPD = 1 LCSC - LCSDC 1\* 2/(LCSC + LCSDC)

1 282 Z LCS/LCSD samples:

F			7.	<del></del>	T	<del></del>	-4	<del></del>	<del></del>	<del></del>	<del></del>	 	T
	CS/LCSD	ر ا		Kecalcillated									
	I CS/I	. Uda	41	керопед									:
	D	SCOVETV	11	The last									
	1GSD	Percent Recovery	Donottod										
		covery	Rocal				79		88				
	103	Percent Recovery	Reported				79		88				
	<u></u>	Concentration ( 45 /1)	l csp.				3	<					
	Spil	Concent	LCS				2112		2350				
		( 24) (A)	usol,				A'A		>-				
	Spi	Add ( Mg	1 CS	,			26.70		4				
		Compound		Phenol	N-Nitroso-di-n-propylamine	4-Chloro-3-methylphenol	Acenaphthene	Pentachlorephenol	Pyrene				

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results. LDC#: >45~ DW

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	
Reviewer:	M
2nd reviewer:_	
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METHOD: GC/MS BNA (EPA SW 846 Method 8270C)

1	$\overline{Y}$	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 =  $(A_s)(L_s)(V_t)(DF)(2.0)$  $(A_{ls})(RRF)(V_o)(V_l)(%S)$ 

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

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

l<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, = 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. # 7, SS:

Conc. = (4745)(40)(1m/)(107)(0)(1027168)(0,2299)(30.29)(0.897)(0)

= 40.4 us/lex

2.0	<ul> <li>Factor of 2 to account</li> </ul>	nt for GPC cleanup			
#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
					•
		·			
		•			
	-				
	·				
	<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: November 12, 2010

LDC Report Date: December 18, 2010

Matrix: Soil

Parameters: Semivolatiles

Validation Level: Stage 2B

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 280-9771-1

Sample Identification

SSAN6-08-2.0\_01\_BPC SSAN6-08-3.0\_01\_BPC SSAN6-08-4.0\_01\_BPC SSAN6-08-4.0\_01\_BPCMS SSAN6-08-4.0\_01\_BPCMSD

### Introduction

This data review covers 5 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.

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.

A curve fit, based on the initial calibration, was established for quantitation. The coefficient of determination (r<sup>2</sup>) was 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 280-41005/1-A	11/15/10	Diethylphthalate	31.9 ug/Kg	All samples in SDG 280-9771-1

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.

### 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

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 280-9771-1	All compounds reported below the PQL.	J (all detects)	А

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 280-9771-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-9771-1	SSAN6-08-2.0_01_BPC SSAN6-08-3.0_01_BPC SSAN6-08-4.0_01_BPC	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 280-9771-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Field Blank Data Qualification Summary - SDG 280-9771-1

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson

DC #: 24522G2a	_VALIDATION COMPLETENESS WORKSHEET
SDG #: 280-9771-1	Stage 2B
_aboratory: <u>Test America</u>	- -

	Date:	12/15/10
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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: 11 /12 /10
<b>I</b> 1.	GC/MS Instrument performance check	Á	,
111.	Initial calibration	A	% KSD +
IV.	Continuing calibration/ICV	À	cω /ιω ≤ 25 2
V.	Blanks	SN	·
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	À	
VIII.	Laboratory control samples	Á	ics
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	·
XI.	Target compound identification	N	
XII.	Compound quantitation/CRQLs	N	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	N	·
XV.	Overall assessment of data	A	
XVI.	Field duplicates	N	
XVII.	Field blanks	Ŋ	

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

	ا ۱۰ ح				 
1	SSAN6-08-2.0_01_BPC	11	MB 280 -41005/1-A	21	31
2	SSAN6-08-3.0_01_BPC	12	<u></u>	22	32
3	SSAN6-08-4.0_01_BPC	13		23	 33
4	SSAN6-08-4.0_01_BPCMS	14		24	34
5	SSAN6-08-4.0_01_BPCMSD	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-chloroethy!) 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-Dichiorobenzene	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. Antline
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-Methylnaphthalane	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP, Benzoic Acid
I. 4-Methylphenol	X. Haxachlorocyclopentediene*	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-ethylhexyf)phthalate	ТТТ.
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	WW.
O. 2,4-Dimethy phenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	www.

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

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### VALIDATION FINDINGS WORKSHEET Blanks

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

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questions are identified as "N/A".
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Was a method blank analyzed for each matrix? Y N N/A

Was a method blank analyzed for each concentration preparation level? N/A

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

Was the blank contaminated? If yes, please see qualification below. n date: 11 /15 /lo Blank analysis date: 11 /17 /lo VN N/A

Associated Samples: Blank extraction date: 11 /15/10 Blank analysis date: 11 Conc. units: ம

Compound	Blank ID				S	Sample Identification	ıtion		
AM	MB 280-4100 5/1-A	4-7							
77	31.9								
		-							
Blank extraction date:	Blank analysis date:	sis date:							
Conc. units:			Associated Samples:	Samples:					

Conc. units:	Compound						
	Blank ID						
	-						
Associa						,	
Associated Samples:							
	Sample Identification		•				
				:			

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

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

October 29, 2010

LDC Report Date:

December 18, 2010

Matrix:

Soil/Water

Parameters:

Chlorinated Pesticides

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-9188-1

### Sample Identification

SA66-11\_01\_BPC

SA66-12 01 BPC

SA66-13 01 BPC

SA66-14 01 BPC

SA66-17 01 BPC

SA66-20 01 BPC\*\*

SA66-20 01 BPC FD

EB-10292010-RZE

SA66-12 01 BPCMS

SA66-12 01 BPCMSD

SA66-20\_01\_BPC\_FDMS

SA66-20 01 BPC FDMSD

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

### Introduction

This data review covers 11 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8081A for Chlorinated Pesticides.

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

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

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/ECD Instrument Performance Check

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

### III. Initial Calibration

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

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.

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

### IV. Continuing Calibration

Continuing calibration was performed at required frequencies.

The percent differences (%D) of calibration factors in continuing standard mixtures were within the 20.0% QC limits for all compounds.

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

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

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

### V. Blanks

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

Sample EB-10292010-RZE 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
SA66-11_01_BPC	CLP 1	Tetrachloro-m-xylene Decachlorobiphenyl	438 (59-115) 284 (63-124)	All TCL compounds except 4,4'-DDE 4,4'-DDT Hexachlorobenzene	J (all detects) UJ (all non-detects)	А
SA66-11_01_BPC	CLP 2	Tetrachloro-m-xylene Decachlorobiphenyl	125 (59-115) 41 (63-124)	All TCL compounds except 4,4'-DDE 4,4'-DDT Hexachlorobenzene	J (all detects) UJ (all non-detects)	А
SA66-12_01_BPC	CLP 1	Tetrachloro-m-xylene Decachlorobiphenyl	397 (59-115) 254 (63-124)	All TCL compounds except 4,4'-DDE 4,4'-DDT	J (all detects) UJ (all non-detects)	Α
SA66-12_01_BPC	CLP 2	Decachlorobiphenyl	39 (63-124)	All TCL compounds except 4,4'-DDE 4,4'-DDT	J (all detects) UJ (all non-detects)	Α
SA66-13_01_BPC	CLP 1	Tetrachloro-m-xylene Decachlorobiphenyl	431 (59-115) 481 (63-124)	All TCL compounds except 4,4'-DDE	J+ (all detects)	A
SA66-13_01_BPC	CLP 2	Decachlorobiphenyl	449 (63-124)	All TCL compounds except 4,4'-DDE	J+ (all detects)	A
SA66-14_01_BPC	CLP 1	Tetrachloro-m-xylene Decachlorobiphenyl	502 (59-115) 731 (63-124)	All TCL compounds except 4,4*-DDE beta-BHC	J (all detects) UJ (all non-detects)	A
SA66-14_01_BPC	CLP 2	Decachlorobiphenyl	0 (63-124)	All TCL compounds except 4,4'-DDE beta-BHC	J (all detects) UJ (all non-detects)	A
SA66-17_01_BPC	CLP 1	Tetrachloro-m-xylene Decachlorobiphenyl	130 (59-115) 289 (63-124)	All TCL compounds except beta-BHC	J (all detects) UJ (all non-detects)	Α
SA66-17_01_BPC	CLP 2	Tetrachloro-m-xylene Decachlorobiphenyl	132 (59-115) 10 (63-124)	All TCL compounds except beta-BHC	J (all detects) UJ (all non-detects)	Α
SA66-20_01_BPC**	CLP 1	Decachlorobiphenyl	138 (63-124)	All TCL compounds	J+ (all detects)	Р

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Percent recoveries (%R) 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 280-9188-1	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 SA66-20\_01\_BPC\*\* and SA66-20\_01\_BPC\_FD were identified as field duplicates. No chlorinated pesticides were detected in any of the samples with the following exceptions:

	Concentr	ation (ug/Kg)				
Compound	\$A66-20_01_BPC**	SA66-20_01_BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
4,4'-DDE	0.46	2.3U	-	1.84 (≤2.3)	-	-
beta-BHC	2.2	1.9	-	0.3 (≤2.3)	-	-

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Data Qualification Summary - SDG 280-9188-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-9188-1	SA66-11_01_BPC	All TCL compounds except 4,4'-DDE 4,4'-DDT Hexachlorobenzene	J (all detects) UJ (all non-detects)	А	Surrogate spikes (%R) (s)
280-9188-1	SA66-12_01_BPC	All TCL compounds except 4,4'-DDE 4,4'-DDT	J (all detects) UJ (all non-detects)	А	Surrogate spikes (%R)(s)
280-9188-1	SA66-13_01_BPC	All TCL compounds except 4,4'-DDE	J+ (all detects)	А	Surrogate spikes (%R) (s)
280-9188-1	SA66-14_01_BPC	All TCL compounds except 4,4'-DDE beta-BHC	J (all detects) UJ (all non-detects)	Α	Surrogate spikes (%R)(s)
280-9188-1	SA66-17_01_BPC	All TCL compounds except beta-BHC	J (all detects) UJ (all non-detects)	А	Surrogate spikes (%R) (s)
280-9188-1	SA66-20_01_BPC**	All TCL compounds	J+ (all detects)	Р	Surrogate spikes (%R) (s)
280-9188-1	SA66-11_01_BPC SA66-12_01_BPC SA66-13_01_BPC SA66-14_01_BPC SA66-17_01_BPC SA66-20_01_BPC** SA66-20_01_BPC_FD EB-10292010-RZE	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG 280-9188-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG 280-9188-1

No Sample Data Qualified in this SDG

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

Stage 2	2B/4
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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
l.	Technical holding times	A	Sampling dates: 10 /24 /to
11.	GC/ECD Instrument Performance Check	A	,
111.	Initial calibration	A	· RSD r
IV.	Continuing calibration/ICV	A	CW/1W =202
V.	Blanks	A	
VI.	Surrogate spikes	TM	
VII.	Matrix spike/Matrix spike duplicates	SN)	
VIII.	Laboratory control samples	A	ics/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	_A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	K	
XIV.	Field duplicates	. SM	D = 6,7
XV.	Field blanks	M	EB = 8

Note:

A = Acceptable

LDC #: 24522D3a SDG #: 280-9188-1 Laboratory: Test America

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>	<del>)</del> -	N'Ater			
1	SA66-11_01_BPC S	11	SA66-20_01_BPC_FDMS		MB 280-39724/-1	<b>.</b> 31
2	SA66-12_01_BPC	12	SA66-20_01_BPC_FDMSD 22	2	MB 280-38773/	32
3	SA66-13_01_BPC	13	23	:3	MB 286- 39241-1	33
4	SA66-14_01_BPC	14	24	4		34
5	SA66-17_01_BPC	15	25	5		35
6	SA66-20_01_BPC** 1	16	26	6		36
7	SA66-20_01_BPC_FD D	17	27	7		37
8 1	EB-10292010-RZE W	18	28	8		38
9	SA66-12_01_BPCMS S	19	29	9		39
10	SA66-12_01_BPCMSD	20	30	0		40

Page: \ of 2
Reviewer: JVG
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	т			
Was the instrument performance found to be acceptable?	<u> </u>			
III, Initial calibration	T	l T		, , , , , , , , , , , , , , , , , , ,
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?			Secretary of the	
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%.0 for individual breakdown in the Evaluation mix standards?		,		
Was a continuing calibration analyzed daily?				
Were all percent differences (%D) ≤ 20%.0 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?	_4			
Were extract cleanup blanks analyzed with every batch requiring clean-up?	_			
Was there contamination in the method blanks or clean-up blanks? If yes, please see the Blanks validation completeness worksheet.		./		
Ⅵ. Surrogäte spikes				
Were all surrogate %R within the QC limits?				
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?		/		
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?			_/	

LDC#: 2/522 D39

### VALIDATION FINDINGS CHECKLIST

Page: 2of 2
Reviewer: TVC
2nd Reviewer:

Validation Area	Yes	No	NA	Findings/Comments
VII. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			
Was a MS/MSD analyzed every 20 samples of each matrix?				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?		/		
Ⅷ. Laboratory control samples				
Was an LCS analyzed for this SDG?	/			
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.				

# 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	II. Arochlor 1262
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	JJ. Arochlor 1268
E. Heptachlor	М. 4,4'-DDD	U. Toxaphene	CC. 2,4'-DDD	KK. oxy Chlordane
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. 2,4'-DDE	LL. trans-Nonachior
G. Heptachlor epoxide	0.4,4'-DDT	W. Aroclor-1221	EE. 2,4'-DDT	MM. cis-Nonachlor
H. Endosulfan i	P. Methoxychlor	X. Aroclor-1232	FF. Hexachlorobenzene	NN.

Notes:

## VALIDATION FINDINGS WORKSHEET Surrogate Spikes

Page: | of 3 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?

Y(N)N/A

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

	( <u>)</u>							0)	<u> </u>			<del>-</del>					(2)	I				
Qualific	* 1/MJ/A (All 4x apt JOFF)		7		No mal			* JMS A (All except J. C				No quel					It dots/A (All +xuat			/ater) Comments		
Limits	135 (59-115)	284 (63-124)		( )	442 (59-115)	56 (63-124)	( ) 0	347 (54-115)	252 (63-124)		(	358 (59-115)	32 ( 4 )	(42-124)	( 1 ) 7	( )	(511-63) 157	_	449	Recovery QC Limits (Water)		
Surr	7 2	20			*	cia —	2 1	1 A	8	2 3		A	2	8	7		1 A	æ   B	\ \ \	Recovery QC Limits (Soil)		
Column	CCC 7	-	12		26	270	\ 	CLP	<del></del>	,									\	nd		
Sample ID					(50×)	)		2		-		2 (50x)	,		-		m			Surrogate Compound	Tetrachoro-m-xylene	Decachlorobiphenyl
Date																				Letter Designation	٩	В
#																				Let		·

\* matrix Interference

LDC#: 2452x D32

## VALIDATION FINDINGS WORKSHEET Surrogate Spikes

Page: 2 of 3 Reviewer: 36 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? N N/A

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

		Document Of Imile Material	Recovery OC Limits (Soil)		Surrogate Compound	Letter Designation
		( )				
		( )				
		( 1 ) (21		7		
		289 (63-124)	<b>-</b>			
\		137 ( 1)		×		
G	* 5/45/A (A) (xccat B)	(311-hs) 0x1	¥		2	
		( )				
		0 ( 1		7		
		147 (63-14)	8			
		127 ( 1 )		٨	,	
	No such	53) (59-115)	Ą	1	4 (10x)	
		_		4	-	
\ 		73) (63-124)		#1		
4	* 5/NS A (All except T B)	(511-65) 2051	¥		7	
			<b>A</b>			
		0 (63-124)	8	- >		
	<i>N</i>	>4 ( ) ) <del> </del>	1	Α		
	No sure	373 (51-115)	Ą	- 33	2 (20x)	
	Qualifications	%R (Limits)	Surrogate Compound	Column	Sample ID	Date
_						

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
Ą	Tetrachoro-m-xylene			
8	Decachlorobipheny:			

LDC#: 24527 Dr.

### VALIDATION FINDINGS WORKSHEET Surrogate Spikes

Page: 3 of 3 

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? X (N) N/A

			<del></del>				<del>,</del>		i—		_	-			<del></del>			-		
Qualifications	No mal			J+ dets/P (All TOL) (S																
nits)	203 (63-124)	^ 	(		_	( )	( )	( )	( )	( )	( )	( )	<u> </u>	( )	( )	( )	( )	( )	( )	( )
%R (Limits)	202	153		1 28																
Surrogate Compound	B	_		2																
Column	- 3	2																		
Sample ID	5 (Iox)	· ·		9																
Date																				
. #																				

_	_	
Comments		
Recovery QC Limits (Water)		
Recovery QC Limits (Soil)		
Surrogate Compound	Tetrachoro-m-xylene	Decachlorobiphenyl
Letter Designation	A	В

LDC#: 24522 D34 SDG#:

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

Page: 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". 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?

Y N N/A

Qualifications	gual	( 12 in )			mag /	\$ \$.																				
	o N	າງ			Aio		,										   									
Associated Samples	7				7																		;			
(Limits)	7	( )	( )	( )	,	( )	( )	( )	_	(	(	(	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
RPD	h'mits			:																						
MSD %R (Limits)	out side)	( d	(	( )	231 (54-135)	( )	( )	( )	( )	,	(	( )	(	( )	] ()	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(
, 8R €		13			231																					
MS %R (Limits)	amp onades	R( and)	^	( )	236 (54-135)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	, ,	( )	( )	( )	( )	( )	[( )
		20 B			236																					
Compound	several	لمحر			И																					
MS/MSD ID	9 PE			, , ,	11/12																					
Date																										
*																										

LDC#:24522D3

### VALIDATION FINDINGS WORKSHEET Field Duplicates

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?

	Concentrati	on (ug/Kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Compound	6	7	RPD	Difference	Limits	(Parent Only)
4,4'-DDE	0.46	2.3U		1.84	(≤2.3)	
beta-BHC	2.2	1.9		0.3	(≤2.3)	

V:\FIELD DUPLICATES\24522D3a.wpd

10C# 2452/ 10 34

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

Page:\_

2nd Reviewer:\_ Reviewer:

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

Parameter: b-BHC

Order of regression:

Linear

	~				,		
y conc	4.000	10.000	25.000	50.000	75.000	100.000	many to
× area	21825	48527	111185	221399	335819	423680	
Points	Point 1	Point 2	Point 3	Point 4	Point 5	Point 6	
Compound	P-BHC						
Column	CLP1						
Date	16-Nov-10		1			-	

RF 5456.2500 4852.7000 4447.4000 4427.9800 4477.5867 4236.8000 Ave 4649.7861

Constant	Output: Regression Output:		Reported WLR	
		-1.49690	= q	-1.07860
Std Err of Y Est		0.04	-	
R Squared		0.99860	r^2 =	0.99950
No. of Observations		9.00		
Degrees of Freedom		4.00		
			#1# #1#	4315.0000
X Coefficient(s)	4252.33517			
Std Err of Coef.	0.01			

LDC# YEary DAR

# VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

2 of S

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

Parameter: 4,4'-DDT

alalicici. 4,4-DD

Order of regression:

Linear

y	4.000	10.000	25.000	50.000	75.000	100.000	
× area	26901	63116	151150	311331	477595	604160	
Points	Point 1	Point 2	Point 3	Point 4	Point 5	Point 6	
Compound	4,4'-DDT						
Column	CLP1						
Date	16-Nov-10		1				

Regression Outp	Output: Regression Output:		Reported WLR	
Constant		-0.39937	= q	-0.37153
Std Err of Y Est		0.04		
R Squared		0.99857	r^2 =	0.99930
No. of Observations		9:00		
Degrees of Freedom		4.00		•
			m1=	6125.0000
X Coefficient(s)	6125.86993			
Std Err of Coef.	0.01			

6311.6000 6311.6000 6046.0000 6226.6200 6367.933 6041.6000 Ave 6286.5006

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

3 of 5

Page: Reviewer: 2nd Reviewer:

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

Parameter: 4,4'-DDT

Order of regression:

Linear

y conc	4.000	10.000	25.000	50.000	75.000	100.000	
area	24700	59507	144805	302113	465980	591865	
Points	Point 1	Point 2	Point 3	Point 4	Point 5	Point 6	
Compound	4,4'-DDT						
Column	CLP2						
Date	16-Nov-10	!					

6175.0000 5950.7000 5792.2000 6042.2600 6213.0667 5918.6500 Ave 6015.3128

Regression Outp	Output: Regression Output:		Reported WLR	
Constant		0.06153	= Q	-0.09770
Std Err of Y Est		0.04		
R Squared		0.99868	r^2 =	0.99930
No. of Observations		00.9		
Degrees of Freedom		4.00		
			m1 =	5976.0000
X Coefficient(s)	6019.30552			
Std Err of Coef.	0.01			

# VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

5 0 5

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

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

Parameter: g-BHC

.

Order of regression:

ssion: Linear

y	4.000	10.000	25.000	50.000	75.000	100.000	
× area	41651	100289	242013	496465	756213	953441	
Points	Point 1	Point 2	Point 3	Point 4	Point 5	Point 6	
Compound	g-BHC						
Column	CLP1						
Date	16-Nov-10		I		<del></del> -J		

10412.7500 10028.9000 9680.5200 9929.3000 10082.8400 9534.4100 Ave 9944.7867

Kegression Outp	Output: Regression Output:		Reported WLR	
Constant		-0.54802	= q	-0.26104
Std Err of Y Est		0.04		
R Squared		0.99838	r^2 =	0.99940
No. of Observations		9.00		
Degrees of Freedom		4.00		
			m1 =	9764.0000
X Coefficient(s)	9674.47945			
Std Err of Coef.	0.01			

LDC# 24547 034

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

2 0 2

Page: Reviewer: 2nd Reviewer:\_

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

Parameter: g-BHC

Linear Order of regression:

				×	^	_
Date	Column	Compound	Points	area	conc	
16-Nov-10	CLP2	g-BHC	Point 1	41460	4.000	т
			Point 2	103531	10.000	_
'			Point 3	252453	25.000	
•			Point 4	519873	50.000	
•			Point 5	790491	75.000	_
,			Point 6	994716	100.000	1
	,					1

Regression Outp	Output: Regression Output:		Reported WLR	
		-0.43829	= q	-0.06438
Std Err of Y Est		0.04		
R Squared	66.0	0.99821	r^2 =	0.99940
No. of Observations		00.9	- t - B	1.012
Degrees of Freedom		4.00		
			m1=	10247.0000
X Coefficient(s)	10117.72331			
Std Err of Coef.	0.01			

10365.0000 10353.1000 10098.1200 10397.4600 10539.8800 9947.1600 Ave 10283.4533

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## Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Reviewer: JVG Page:

> 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:

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

Initial Calibration Factor or Nominal Amount

Calibration Factor from Continuing Calibration Standard or Calculated Amount " " Z O

Compound	Response	Reported m value	Reported b value	Conc
b-BHC (	211575	4315.00	-1.0786	47.95
4,4'-DDT	292192	6125.00	-0.37153	47.33
g-BHC (	486682	10247.00	-0.06438	47.43
4,4'-DDT	275402	5976.00	-0.0977	45.99

LDC #: 24522 D74

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

Page:_	<u>l</u> of <u>1</u>
Reviewer:	_06
2nd reviewer:	
	y

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 calcul	lation:
------------------------	--	---------

% Recovery: SF/SS \* 100

Sample ID:

Where: SF = Surrogate Found

SS = Surrogate Spiked

Surrogate Column Spiked			Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene	CLP 1	20	13, 93	70	70	0
Tetrachloro-m-xylene	\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \		14.32	7~	72	
Decachlorobiphenyl	1		27.54	138	138	<del></del>
Decachlorobiphenyl	1 / 1	X	17.81	89	81	

Sample ID:

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachtoro-m-xylene	_					
Tetrachloro-m-xylene						
Decachlorobiphenyl						<u> </u>
Decachlorobiphenyl				· · · · · · · · · · · · · · · · · · ·	<del> </del>	

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-xyleпе						<del></del>
Decachlorobiphenyl						
Decachlorobiphenyl						<del>-</del>

Notes:		
	· · · · · · · · · · · · · · · · · · ·	

LDC #: 24527 D24

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

Page: 1 of 1 Reviewer: 01/2

2nd Reviewer: 2

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)

MSD = Matrix spike duplicate percent recovery

MS/MSD samples:

MS = Matrix spike percent recovery

2 F

	σ.	pike	Sample	Spiked	Spiked Sample	Matrix	Matrix Spike	Matrix Spil	Matrix Spike Duplicate	SW	MS/MSD
Compound	¥ n)	Added (UA)	Concentration (M)	Conce	Concentration ( $\nu_{\mathcal{O}}/\mathcal{L}$ )	Percent }	Percent Recovery	Percent	Percent Recovery	æ	RPD
	MS	MSD	•	MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
gamma-BHC	18.2	β. \Σ.	٥	0	Q	0	0	0	0	NC	NC
4,4'-DDT	<u>~</u>	\	160	(77	175	\$3 & *	93	* 275	(8	-	75.2
Aroclor 1260		7									

Comments: Refer of 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.

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MSDCLC.3S



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

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

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

Where: SSC = Spiked sample concentration SA = Spike added

SC = Concentration

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

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples:\_

280- 38775/2,3-A ĕ

_						 		 	
TCS/LCSD	RPD	Recalc.	_	-	_				
/SOT	R	Reported							
SD	Recovery	Recalc.	94	96					
TCSD	Percent Recovery	Reported	44	93					
ş	ecovery	Recalc.	43	56					
SOT	Percent Recovery	Reported	93	45					
Sample	Concentration ( 45 /L )	CSD	0.468	b. 47K	•				
Spiked	Concel ( 145	SOT	6,463	6,474					
ike	ded /L)	CSD	. 520						
dS:	Added (Us) /L	SOT	رمحي ٥						
	Compound		gamma-BHC	4,4'-DDT					

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#: 24522 D32

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	of
Reviewer:	W
2nd reviewer:	
•	<del></del>

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

(	K	N	N/A
Ĺ	X	Ņ	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. 6 b. B.HC
Conc. = (25 266 ) + (-1.6786) (43 15.0)
X = 4.7629
final conc = (4.7629) (DMI)
(30.63) (0.713)
= 2.18
2, 2.2 us /kg

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

Note:		•	
Note,	 		

2,1

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

October 21, 2010

LDC Report Date:

December 21, 2010

Matrix:

Soil

Parameters:

Arsenic

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-8906-1/TJ2616

### Sample Identification

SSAN7-04-6 01 BPC

SSAN7-04-7 01 BPC\*\*

SSAN7-04-7 01 BPC FD

SSAN7-04-8 01 BPC

SSAN7-04-9 01 BPC

SSAN7-04-10 01 BPC

SSAM7-07-6 01 BPC

SSAM7-07-7\_01\_BPC

SSAM7-07-8 01 BPC

SSAM7-07-9 01 BPC

SSAM7-07-10 01 BPC

SSAM7-06-6\_01\_BPC

SSAM7-06-7 01 BPC

SSAM7-06-8\_01\_BPC

SSAM7-06-9\_01\_BPC\*\*

SSAM7-06-10\_01\_BPC

SSAM7-06-6 01 BPC FD

SSAM7-06-6 01 BPC FDMS

SSAM7-06-6 01 BPC FDMSD

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

### Introduction

This data review covers 19 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Methods 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.

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.

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

### X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

### XI. ICP Serial Dilution

ICP serial dilution was not performed for this SDG.

### 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 280-8906-1/TJ2616	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 SSAN7-04-7\_01\_BPC\*\* and SSAN7-04-7\_01\_BPC\_FD and samples SSAM7-06-6\_01\_BPC and SSAM7-06-6\_01\_BPC\_FD were identified as field duplicates. No arsenic was detected in any of the samples with the following exceptions:

	Concentra	ation (mg/Kg)				:
Analyte	SSAN7-04-7_01_BPC**	SSAN7-04-7_01_BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	7.0	6.8	3 (≤50)	~	-	-

	Concentr	ation (mg/Kg)		-			
Analyte	SSAM7-06-6_01_BPC	SSAM7-06-6_01_BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P	
Arsenic	11	13	17 (≤50)	*	-	-	

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic - Data Qualification Summary - SDG 280-8906-1/TJ2616

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-8906-1/ TJ2616	SSAN7-04-6_01_BPC SSAN7-04-7_01_BPC** SSAN7-04-7_01_BPC_FD SSAN7-04-8_01_BPC SSAN7-04-9_01_BPC SSAN7-04-10_01_BPC SSAM7-07-6_01_BPC SSAM7-07-8_01_BPC SSAM7-07-8_01_BPC SSAM7-07-10_01_BPC SSAM7-07-10_01_BPC SSAM7-07-10_01_BPC SSAM7-08-6_01_BPC SSAM7-08-9_01_BPC SSAM7-08-9_01_BPC SSAM7-08-9_01_BPC SSAM7-08-9_01_BPC SSAM7-08-9_01_BPC SSAM7-08-9_01_BPC SSAM7-08-9_01_BPC	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 280-8906-1/TJ2616

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic - Field Blank Data Qualification Summary - SDG 280-8906-1/TJ2616

No Sample Data Qualified in this SDG

DC#	: 24522A4	VA		nox Nor N COMP			rson VORKSHE	ET	Date:्।३-।੫-	
DG # abora	t:280-8906-1/ITJ26 atory:_Test_America	<u>16</u>		St	age 2E	3/4			Page: l of l Reviewer: Ms 2nd Reviewer: L	
ETH	OD: Arsenic (EPA SW	846 N	ethod 6020	))					2nd Reviewer:	
ne sa	·	e revi		•	ollowing	validation	n areas. Valid	dation find	lings are noted in attached	
iiuai	lon indings worksneets	•	-	Ī	i .		<u></u>			
	Validation	Area	<u> </u>	Ι	<u> </u>	······································	<del></del>	mments		
<u>l.</u>	Technical holding times		<del></del>	A	Sampling	dates:	10-21-	10		
<u>[].</u>	ICP/MS Tune		<del></del>	A	<u> </u>					
II. 	Calibration			A		<del></del>	<del> </del>		·	
V. V.	Blanks ICP Interference Check Sai	mnio /i	CS) Applyais	A						
<u>v.</u> /l.	Matrix Spike Analysis	ripie (i	Co) Allalysis	A	Me	/MSD				
<del>/1:</del> /11.	Duplicate Sample Analysis			7	, , , ,	7	·			
7111.	Laboratory Control Samples	s (LCS	<u> </u>	Α	LCS					
<u>х.</u>	Internal Standard (ICP-MS)		/ <u></u>	A	not reviewed for level 2B					
<u></u> -	Furnace Atomic Absorption QC		7	not utilized						
<u>.</u>	ICP Serial Dilution			N	not performed					
31.	Sample Result Verification			A	Not reviewed for Stage 2B validation.					
	Overall Assessment of Data			Α						
IV.	Field Duplicates	•		5W	D=2+3 D=12+17					
~~~~ (V	Field Blanks			N	:				<del>'''''                                </del>	
	A = Acceptable N = Not provided/applicable SW = See worksheet d Samples: ** Indicates sam ₹ 1		R = Rin FB = Fi	eld blank	s detected	7	) = Duplicate B = Trip blank B = Equipment	blank		
5	SSAN7-04-6_01_BPC	11	SSAM7-07-10	0_01_BPC	21			31		
	SSAN7-04-7_01_BPC**	12	SSAM7-06-6	01_BPC	22			32		
5	SSAN7-04-7_01_BPC_FD	13	SSAM7-06-7	01_BPC	23			33		
_ 5	SSAN7-04-8_01_BPC	14	SSAM7-06-8	01_BPC	24			34		
5	SSAN7-04-9_01_BPC	15	SSAM7-06-9	01_BPC**	25		<u>.</u>	35		
_ 5	SSAN7-04-10_01_BPC	16	SSAM7-06-10	0_01_BPC	26			36		
_ 5	SSAM7-07-6_01_BPC	17	SSAM7-06-6	01_BPC_FD	27			37		
15	SSAM7-07-7_01_BPC	18	SSAM7-06-6	01_BPC_FE	OMS 28			38		
15	SAM7-07-8_01_BPC	19	SSAM7-06-6	01_BPC_FC	OMSD 29			39		
.   5	SSAM7-07-9_01_BPC	20	PBS		30			40		

LDC#: 24522A4

### **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?	/			
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	<del></del> ,			
Was an LCS anayized for this SDG?	/			
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?	<b>√</b>			

### **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?			/			
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?			/			
IX. ICP Serial Dilution						
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the MDL (ICP)/>100X the MDL(ICP/MS)?		/				
Were all percent differences (%Ds) < 10%?			√			
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.			/			
X. Internal Standards (EPA SW 846 Method 6020/EPA 200.8)						
Were all the percent recoveries (%R) within the 30-120% (6020)/60-125% (200.8) of the intensity of the internal standard in the associated initial calibration?	/					
If the %Rs were outside the criteria, was a reanalysis performed?			/			
XI. Regional Quality Assurance and Quality Control						
Were performance evaluation (PE) samples performed?		<u> </u>	/			
Were the performance evaluation (PE) samples within the acceptance limits?			<u> </u>	· · · · · · · · · · · · · · · · · · ·		
XII. Sample Result Verification	<del></del>	•	1			
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.	/		<u>.</u>			
XIV. Field duplicates	ī					
Field duplicate pairs were identified in this SDG.	/		<u> </u>			
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#: 24522A4

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

Page:_	<u>l_of(</u>
Reviewer:	MG
2nd Reviewer:	

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

YN NA YN NA

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

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	2	3	RPD	Difference	Limits	(Parent Only)
Arsenic	7.0	6.8	3			

V:\FIELD DUPLICATES\FD\_inorganic\24522A4.wpd

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	12	17	RPD	Difference	Limits	(Parent Only)
Arsenic	11	13	17			

V:\FIELD DUPLICATES\FD\_inorganic\24522A4.wpd

LDC# 34532A4

# VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

Page: Lof L Reviewer: MG

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 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)						
1134 TCV	ICP/MS (Initial calibration)	As	25.23934	95.0	101	101	>
	CVAA (Initial calibration)						
	ICP (Continuing calibration)		-				
1403 CC V 2	ICP/MS (Continuing calibration)	As	48.93211	50.0	98	96	
	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#: 34532A4

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

Page: 1 of 1 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:

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

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

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

%D = [I-SDR] x 100

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

							Recalculated	Reported	
Sample ID	Type of Analysis	Element	Found / S / I (units)		True / D / SDR (units)	R (units)	%R/RPD/%D	%R/RPD/%D	Acceptable (Y/N)
1151 ICSAB	ICP interference check	As	As 19.09431 (2	(mg/kg)	30.0 (mg/kg)	(mg/kg)	95	95	>
1246	Laboratory control sample	As	46.593	(mg/kg)	/bm) 0.05	(mg/lug)	93	93	
1956	Matrix spike	As	(SSR-SR)	(mg/kg)	55.0	(mg /kg)	87	87	
1956/1301	Duplicate	As	As 60.920 ("	(mg/kg)	63.856 (my/kg)	(mg//gm)	5	5	>
_	ICP serial dilution	1	]	0	l	)	1	l	ĺ

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 #: 24522A4

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_		of	_
Reviewer:	۲	16_	_
2nd reviewer:			_/
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METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

Please Y)N Y)N Y)N	N/A Are results w	ow for all questions answered "N". Not appl been reported and calculated correctly? within the calibrated range of the instrument tion limits below the CRDL?	icable questions ar	e identified as "N// ear range of the IC	A". :P?
Detect equati	ted analyte results for _ on:	# 2, As	were recalcu	lated and verified	using the following
Concen	tration = <u>(RD)(FV)(Dil)</u> (In. Vol.)	Recalculation:			
RD V n. Vol. Dil	= Raw data conce = Final volume (m = Initial volume (m = Dilution factor	entration $(252.20065  \text{Mg/L})$ (a) or weight (G) $(2.01  \text{g})$ (0.8)	(0.050 L) 910)	= 7.041	ug/g or mg/
#	Sample ID	Analyte	Reported Concentration	Calculated Concentration (Ma/kq)	Acceptable (Y/N)
	2	As	7.0	7.0	Y
		<del></del>			
		·			···
				1	
Note:_					
					,

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

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

Henderson, Nevada

Collection Date: October 27, 2010

LDC Report Date: December 21, 2010

Matrix: Soil/Water

Parameters: Metals

Validation Level: Stage 2B & 4

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 280-9160-1

### Sample Identification

SSAN8-06-1_01_BPC	SSAO8-06-4_01_BPC
SSAN8-06-2_01_BPC	SSAO8-06-5_01_BPC
SSAN8-06-3_01_BPC_FD	SSAO8-09-1_01_BPC
SSAN8-06-3_01_BPC	SSAO8-09-2_01_BPC
SSAN8-06-4_01_BPC	SSAO8-09-3_01_BPC
SSAN8-06-5_01_BPC	SSAO8-09-4_01_BPC
SSAO7-07-1_01_BPC	SSAO8-09-5_01_BPC**
SSAO7-07-2_01_BPC	SSAO8-09-5_01_BPC_FD
SSAO7-07-4_01_BPC	EB-102710-RZC_1
SSAO7-07-3_01_BPC	EB-102710-RZC_2
SSAO7-07-5_01_BPC**	SSAO8-06-3_01_BPCMS
SSAO7-08-1_01_BPC	SSAO8-06-3_01_BPCMSD
SSAO7-08-2_01_BPC	SSAO8-06-4_01_BPCMS
SSAO7-08-3_01_BPC	SSAO8-06-4_01_BPCMSD
SSAO7-08-4_01_BPC	
SSAO7-08-4_01_BPC_FD	
SSAO7-08-5_01_BPC	
SSAO8-06-1_01_BPC**	
SSAO8-06-2_01_BPC	•
SSAO8-06-3_01_BPC	

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

### Introduction :

This data review covers 32 soil samples and 2 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Methods 6020 for Metals. The metals analyzed were Arsenic, Cobalt, and Manganese.

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

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

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. ICPMS Tune

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

### III. Calibration

An initial calibration was performed.

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

### IV. Blanks

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

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
PB (prep blank)	Manganese ,	0.0426 mg/Kg	SSAO8-06-4_01_BPC SSAO8-06-5_01_BPC SSAO8-09-1_01_BPC SSAO8-09-2_01_BPC SSAO8-09-3_01_BPC SSAO8-09-4_01_BPC SSAO8-09-5_01_BPC** SSAO8-09-5_01_BPC_FD
ICB/CCB	Cobalt	0.0106 ug/L	SSAO8-06-4_01_BPC SSAO8-06-5_01_BPC SSAO8-09-1_01_BPC SSAO8-09-2_01_BPC SSAO8-09-3_01_BPC SSAO8-09-4_01_BPC SSAO8-09-5_01_BPC** SSAO8-09-5_01_BPC_FD

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
PB (prep blank)	Manganese	0.0357 mg/Kg	SSAO7-07-1_01_BPC SSAO7-07-2_01_BPC SSAO7-07-4_01_BPC SSAO7-07-3_01_BPC SSAO7-07-5_01_BPC** SSAO7-08-1_01_BPC SSAO7-08-2_01_BPC SSAO7-08-3_01_BPC SSAO7-08-4_01_BPC SSAO7-08-4_01_BPC SSAO7-08-5_01_BPC SSAO7-08-5_01_BPC SSAO8-06-1_01_BPC** SSAO8-06-2_01_BPC SSAO8-06-3_01_BPC
ICB/CCB	Cobalt Manganese	0.0138 ug/L 0.410 ug/L	SSAO7-07-1_01_BPC SSAO7-07-2_01_BPC SSAO7-07-4_01_BPC SSAO7-07-3_01_BPC SSAO7-08-1_01_BPC** SSAO7-08-1_01_BPC SSAO7-08-2_01_BPC SSAO7-08-3_01_BPC SSAO7-08-4_01_BPC SSAO7-08-4_01_BPC_FD SSAO7-08-5_01_BPC SSAO8-06-1_01_BPC** SSAO8-06-1_01_BPC** SSAO8-06-3_01_BPC
ICB/CCB	Cobalt	0.0228 ug/L	All water samples in SDG 280-9160-1

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

Sample	Analyte	Reported Concentration	Modified Final Concentration
EB-102710-RZC_1	Cobalt	0.74 ug/L	1.0U ug/L

Samples EB-102710-RZC\_1 and EB-102710-RZC\_2 were identified as equipment blanks. No metal contaminants were found in these blanks with the following exceptions:

Equipment Blank ID	Sampling Date	Analyte	Concentration	Associated Samples
EB-102710-RZC_1	10/27/10	Cobalt Manganese	0.74 ug/L 46 ug/L	SSAO7-07-1_01_BPC SSAO7-07-2_01_BPC SSAO7-07-4_01_BPC SSAO7-07-3_01_BPC SSAO7-08-1_01_BPC SSAO7-08-1_01_BPC SSAO7-08-2_01_BPC SSAO7-08-4_01_BPC SSAO7-08-4_01_BPC_FD SSAO7-08-5_01_BPC SSAO8-06-1_01_BPC SSAO8-06-1_01_BPC SSAO8-06-3_01_BPC SSAO8-06-3_01_BPC SSAO8-06-5_01_BPC SSAO8-09-1_01_BPC SSAO8-09-1_01_BPC SSAO8-09-1_01_BPC SSAO8-09-1_01_BPC SSAO8-09-1_01_BPC SSAO8-09-1_01_BPC SSAO8-09-1_01_BPC SSAO8-09-1_01_BPC
EB-102710-RZC_2	10/27/10	Cobalt Manganese	2.0 ug/L 110 ug/L	SSA07-07-1_01_BPC SSA07-07-2_01_BPC SSA07-07-4_01_BPC SSA07-07-4_01_BPC SSA07-07-5_01_BPC** SSA07-08-1_01_BPC SSA07-08-2_01_BPC SSA07-08-3_01_BPC SSA07-08-4_01_BPC SSA07-08-4_01_BPC SSA07-08-5_01_BPC SSA08-06-1_01_BPC** SSA08-06-1_01_BPC** SSA08-06-2_01_BPC SSA08-06-3_01_BPC SSA08-06-5_01_BPC SSA08-09-1_01_BPC SSA08-09-2_01_BPC SSA08-09-2_01_BPC SSA08-09-3_01_BPC SSA08-09-3_01_BPC SSA08-09-4_01_BPC SSA08-09-5_01_BPC_TD

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

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

The frequency of analysis was met.

The criteria for analysis were met.

### VI. Matrix Spike Analysis

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

### 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 280-9160-1	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 SSAN8-06-3\_01\_BPC\_FD and SSAN8-06-3\_01\_BPC and samples SSAO7-08-4\_01\_BPC and SSAO7-08-4\_01\_BPC\_FD and samples SSAO8-09-5\_01\_BPC\*\* and SSAO8-09-5\_01\_BPC\_FD were identified as field duplicates. No metals were detected in any of the samples with the following exceptions:

	Concentration (mg/Kg)					
Analyte	SSAN8-06-3_01_BPC_FD	SSAN8-06-3_01_BPC	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	6.4	5.3	19 (≤50)	-	-	-

	Concentra						
Analyte	SSA07-08-4_01_BPC	SSA07-08-4_01_BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P	
Arsenic	2.9	2.9	0 (≤50)	-	-	-	
Cobalt	7.8	7.3	7 (≤50)	-	-	-	
Manganese	360	320	12 (≤50)	-	-	-	

	Concentrat					
Analyte	SSAO8-09-5_01_BPC**	SSAO8-09-5_01_BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	2.6	2.9	11 (≤50)	-	-	-
Cobalt	6.1	6.1	0 (≤50)	-	-	-
Manganese	290	300	3 (≤50)	-	-	-

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Metals - Data Qualification Summary - SDG 280-9160-1

SDG	Sample	Analyte	Flag	AorP	Reason (Code)
280-9160-1	SSAN8-06-1_01_BPC SSAN8-06-2_01_BPC SSAN8-06-3_01_BPC SSAN8-06-3_01_BPC SSAN8-06-4_01_BPC SSAN8-06-5_01_BPC SSAN8-06-5_01_BPC SSAO7-07-1_01_BPC SSAO7-07-2_01_BPC SSAO7-07-3_01_BPC SSAO7-07-3_01_BPC SSAO7-08-1_01_BPC SSAO7-08-1_01_BPC SSAO7-08-4_01_BPC SSAO7-08-4_01_BPC SSAO7-08-4_01_BPC SSAO7-08-5_01_BPC SSAO8-06-1_01_BPC SSAO8-09-1_01_BPC	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 280-9160-1

SDG	Sample	Analyte	Modified Final Concentration	A or P	Code
280-9160-1	EB-102710-RZC_1	Cobalt	1.0U ug/L	А	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Metals - Equipment Blank Data Qualification Summary - SDG 280-9160-1

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson**

LDC #:	_24522C4	VALIDATION COMPLETENESS WORKSHEET	
SDG #:	280-9160-	Stage 2B/4	
Laborator	<u>y: Test America</u>	Laboratories, Inc.	Re

Page:_	l_of_L
Reviewer:	MG
2nd Reviewer:	_ لم

Date: 12-14-10

METHOD: Metals (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	Α	Sampling dates: 10 - 27 - 10
II.	ICP/MS Tune	A	
111.	Calibration	Α	
IV.	Blanks	SW	
V <u>.</u>	ICP Interference Check Sample (ICS) Analysis	Α	
VI.	Matrix Spike Analysis	SW	MS/MSD
VII.	Duplicate Sample Analysis	7	
VIII.	Laboratory Control Samples (LCS)	Д	LCS /LCSD
IX.	Internal Standard (ICP-MS)	Α	
X.	Furnace Atomic Absorption QC	Ž	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=3+4 D=15+16 D=27+28
ΧV	Field Blanks	SW	EB = 29 30

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	SSANS OF 1 OF BDC 5	1.1	20102025412004	ي ۾ ا	6	1	5
<u>  1</u>	SSAN8-06-1_01_BPC 5	11.	SSA07-07-5_01_BPC**	210	SSAO8-06-4_01_BPC 5	31 '	SSAO8-06-3_01_BPCMS
2 1	SSAN8-06-2_01_BPC	12	SSAO7-08-1_01_BPC	22	SSAO8-06-5_01_BPC	32	SSAO8-06-3_01_BPCMSD
3 l	SSAN8-06-3_01_BPC_FD	13 l	SSA07-08-2_01_BPC	23	SSAO8-09-1_01_BPC	33 A	SSAO8-06-4_01_BPCMS
<sub>4</sub>	SSAN8-06-3_01_BPC	14 <sup>]</sup>	SSA07-08-3_01_BPC	24	SSAO8-09-2_01_BPC	34 7	SSAO8-06-4_01_BPCMSD
<sub>5</sub> 1	SSAN8-06-4_01_BPC	15 l	SSAO7-08-4_01_BPC	25	SSAO8-09-3_01_BPC	35	
6	SSAN8-06-5_01_BPC	16 l	SSAO7-08-4_01_BPC_FD	26	SSAO8-09-4_01_BPC	36	
<sub>7</sub>	SSAO7-07-1_01_BPC	17 l	SSAO7-08-5_01_BPC	27	SSAO8-09-5_01_BPC**	37	
8 l	SSAO7-07-2_01_BPC	18	SSAO8-06-1_01_BPC**	28	SSAO8-09-5_01_BPC_FD	38 (	PB51
<sub>9</sub> [	SSAO7-07-4_01_BPC	19 i	SSAO8-06-2_01_BPC	29	EB-102710-RZC_1 W	39 J	PB57
10 1	SSAO7-07-3_01_BPC	20 (	SSAO8-06-3_01_BPC	30	EB-102710-RZC_2	40 3	PBW

Notes:			
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### **VALIDATION FINDINGS CHECKLIST**

Page: 1 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?	/			
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?	1			
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.	/			
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			•	
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?			<b>/</b>					
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	<del></del>							
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%?	$\checkmark$							
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?	<u> </u>		/					
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?	<u></u>		V					
XII. Sample Result Verification		r						
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	V	i	<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.	/	<u> </u>						
XV. Field blanks								
Field blanks were identified in this SDG.								
Target analytes were detected in the field blanks.								

LDC#: 24582C4

### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page:_	of
Reviewer:	MG
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All circled elements are applicable to each sample.

<u></u>	<del></del> -	
Sample ID	Matrix	Target Analyte List (TAL)
(->6	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,
7-> 30	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,
QC31→34	5	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,
	<u> </u>	
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
<u> </u>		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
,		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
<u> </u>		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <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> ,
		At, 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
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Analysis Method
ICP		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
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 <sup>-</sup> ,
GEAA		Al Sb As Ba Be Cd Ca Cr Co Cu Fe Pb 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 METHOD: Trace metals (EPA SW 864 Method 6010B/6020/7000)

Soil preparation factor applied: 100x 21-28 (>RL) Associated Samples:

- 6	Mai	\ -	)
Tage:	Reviewer:	2nd Reviewer:	

No Qual's. Sample Concentration units, unless otherwise noted: mg/Kg Action Limit Maximum ICB/CCB<sup>2</sup> 0.0106 (ng/L) Maximum PB<sup>a</sup> (ug/L) Maximum PB<sup>a</sup> (mg/Kg) 0.0426 Analyte ပိ Ĕ

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

Soil preparation factor applied: 100x Associated Samples: 7-20 (>RL)

Analyte	Maximum PB³ (mg/Kg)	Maximum Maximum PB <sup>a</sup> ICB/CCB <sup>a</sup> (ug/L)	Action Limit	No Qual's.					
ပိ		0.0138							
Mn	0.0357	0.410							

Soil preparation factor applied: NA all water METHOD: Trace metals (EPA SW 864 Method 6010B/6020/7000)

Associated Samples: Sample Concentration units, unless otherwise noted: ug/L

Analyte	Maximum PB <sup>a</sup> (mg/Kg)	Maximum PB <sup>a</sup> (ug/L)	Maximum ICB/CCB <sup>a</sup> (ug/L)	Action Limit	29					
o			0.0228		0.74/ 1.0U				•	

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 #: 24522C4

## VALIDATION FINDINGS WORKSHEET

Page: Lof 1

Reviewer: 2nd Reviewer:

Field Blanks

**МЕТНОD:** Trace Metals (EPA SW846 6010В/7000)

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

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

Blank units: ug/L Associated sample units: mg/Kg Sampling date: 10-27-10 Soil factor applied

EB 100x Sampling date: 10-27-10 Soil factor applied Field blank type: (circle one) Field Blank / Rinsate (Other)

7-28 (>10x) Associated Samples: Sample Identification No Qual's. Action Level 2.0 110 Blank 1D 110 2.0 8 Blank ID 0.74 46 83 Analyte ပိ 툴

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT: Samples with analyte concentrations within five times the associated field blank concentration are listed above, these sample results were qualified as not detected, "U".

### LDC#: 24522C4

## VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

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

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

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

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 NIA

of 4 or more, no action was taken.

Were all duplicate sample relative percent differences (RPD) ≤ 20% for samples?

Was a post digestion spike analyzed for elements that did not meet the required criteria for matrix spike recovery? Y (N NIA) Y N XIA)

EVEL IV ONLY:

Y)N NA

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

(%R)														
Post Spike (%R)														
Qualifications	* No Qual													
Associated	36+17	1:05 11 E									,			
RPD (Limits)		AM6												
MSD %Recovery														о 7
MS %Recovery														2 rec
Analyte	Mn													MsD
Matrix	5011									•	-			* LCS, MS and
MS/MSD ID	33/34								:					
#											-		 -	Comments:

MS-MSD-PS.wpd

:

LDC#: <u>24522C</u>4

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

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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?

	Concentration	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	3	4	RPD	Difference	Limits	(Parent Only)
Arsenic	6.4	5.3	19			

V:\FIELD DUPLICATES\FD\_inorganic\24522C4.wpd

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	15	16	RPD	Difference	Limits	(Parent Only)
Arsenic	2.9	2.9	0			
Cobalt	7.8	7.3	7			
Manganese	360	320	12			

V:\FIELD DUPLICATES\FD\_inorganic\24522C4.wpd

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	27	28	RPD	Difference	Limits	(Parent Only)
Arsenic	2.6	2.9	11			
Cobalt	6.1	6.1	0		,	
Manganese	290	300	3			

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LDC# 3452C4-

# Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: Lof ( Reviewer: んく 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 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)						
1655 ICV	ICP/MS (Initial calibration)	Co	40.95	40.0	102	102	>
	CVAA (Initial calibration)						
	ICP (Continuing calibration)						
1919 CCV	ICP/MS (Continuing calibration)	Mn	50.15	50.0	100	100	
	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. 1

LDC#: 34532C4

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

Page: 2nd Reviewer.\_ Reviewer:

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

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

%R = Found x 100

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

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

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

Where, 1 = Initial Sample Result (mg/L) SDR = Serial Dilution Result (mg/L) (Instrument Reading x 5) %D = II-SDRI x 100

		<b>I</b>	· · · · ·	i <del></del> -	1	
	Acceptable (Y/N)	<u> </u>				>
Reported	%R/RPD/%D	7.6	95	9	3	3.1
Recalculated	%R/RPD/%D	16	95	16	. 3	3. (
	(units)	(Mg/L)	(mg/kg)	(m3/kg)	(mg/kg)	(mg/hg)
	True / D / SDR (units)	001 (78m) bs	0.0%	0.61 (mg/kg) 19.0	(mg/kg) 27.83 (mg/kg)	1747.3 (mg/kg)
	/!	(7/87)	07 (mg/kg)	(mg/kg)	(ga/8m)	3 (mg/kg)
	Found / S / I (units)	96.59	19.07	(SSR-SR)	36.91	1694.3
	Element	Co	Mn	As	00	Mn
	Type of Analysis	ICP interference check	Laboratory control sample	Matrix spike	Duplicate	ICP serial dilution
	Sample ID	1718 ICSAB	1853 1853	3036	31/32	0e 0e0e / he0e

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#: 24522C4

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

Page:_	ofl_
Reviewer:	MG
2nd reviewer:	

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

#	<del>, ''</del>			Analyte	Cor	Reported ncentration	Calculated Concentration	Acceptable (Y/N)	
RD FV In. Vol. Dil	= = =	(In. Vol.)  Raw data concer Final volume (mi Initial volume (mi Dilution factor	)	(21.04	ug/L)(0.10 ig) (0.10	915)	= 10.765	ng/g or mg	/ka
equati		te results for (RD)(FV)(Dil)	<u> </u>	C o	ulation:		lated and verified	-	
(X) N (X) N (X) N	<u>N/A</u> <u>N/A</u> <u>N/A</u>	Have results l Are results wi Are all detecti	been reported a thin the calibrate ion limits below	_	ectly? struments and v	within the line	ar range of the IC	P?	

#_	Sample ID	Analyte	Reported Concentration ( <sup>Mg</sup> /kg)	Calculated Concentration (M4 / K4)	Acceptable (Y/N)
[	[1	As	3.5	3.5	Y
		Co	10	10	
		Mn	2000	3000	
					···
		· · · · · · · · · · · · · · · · · · ·			
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			<u></u>		
<u> </u>		4.44.4		******	
<b> </b>					
<u> </u>					

Note:	 	

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

November 2, 2010

LDC Report Date:

December 19, 2010

Matrix:

Soil

Parameters:

Arsenic

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-9309-1

# Sample Identification

SA142-1 01 BPC

SA142-2 01 BPC

SA142-3 01 BPC

SA142-4 01 BPC

SA142-5 01 BPC

SSAO8-13-1 01 BPC

SSAO8-13-2 01 BPC

SSAO8-13-3 01 BPC

SSAO8-13-4 01 BPC

SSAO8-13-5 01 BPC\*\*

SSAO8-14-1 01 BPC

SSAO8-14-2 01 BPC

SSAO8-14-3 01 BPC

SSAO8-14-3 01 BPC FD

SSA08-14-4 01 BPC

SSAO8-14-5 01 BPC

SSAO8-13-4 01 .BPCMS

SSAO8-13-4\_01\_BPCMSD

<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 Methods 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.

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

# 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 280-9309-1	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 SSAO8-14-3\_01\_BPC and SSAO8-14-3\_01\_BPC\_FD were identified as field duplicates. No metals were detected in any of the samples with the following exceptions:

	Concentra	ation (mg/Kg)				
Analyte	SSAO8-14-3_01_BPC	SSAO8-14-3_01_BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	3.0	3.4	12 (≤50)	-		-

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic - Data Qualification Summary - SDG 280-9309-1

SDG	Sample	Analyte	Flag	АогР	Reason
280-9309-1	SA142-1_01_BPC SA142-2_01_BPC SA142-2_01_BPC SA142-3_01_BPC SA142-4_01_BPC SA142-5_01_BPC SSA08-13-1_01_BPC SSA08-13-2_01_BPC SSA08-13-4_01_BPC SSA08-13-5_01_BPC** SSA08-14-1_01_BPC SSA08-14-2_01_BPC SSA08-14-3_01_BPC SSA08-14-3_01_BPC SSA08-14-3_01_BPC SSA08-14-3_01_BPC SSA08-14-5_01_BPC SSA08-14-5_01_BPC	All analytes reported below the PQL.	J (all detects)	А	Sample result verification (PQL) (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic - Laboratory Blank Data Qualification Summary - SDG 280-9309-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic – Field Blank Data Qualification Summary - SDG 280-9309-1

No Sample Data Qualified in this SDG

# **Tronox Northgate Henderson** VALIDATION COMPLETENESS WORKSHEET

LDC #:	24522E4	VALIDATION CONTLETENESS WOR
SDG #:	280-9309-1	Stage 2B/4
Laborator	v: Test America	Laboratories, Inc.

Date:	10-12-
Page:_	of
Reviewer:	16
2nd Reviewer:	<u>~</u>

METHOD: Arsenic (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	Α	Sampling dates: II- 2-10
11.	ICP/MS Tune	Α	
III.	Calibration	Α	
IV.	Blanks	A	·
V.	ICP Interference Check Sample (ICS) Analysis	_ A	·
VI.	Matrix Spike Analysis	Ą	MS/MSD
VII.	Duplicate Sample Analysis	7	
VIII.	Laboratory Control Samples (LCS)	Α	LCS
IX.	Internal Standard (ICP-MS)	Α	
X.	Furnace Atomic Absorption QC	7	not utilized
XI.	ICP Serial Dilution	А	
XII.	Sample Result Verification	Α	Not reviewed for Stage 2B validation.
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	SW	D= 13+14
ΧV	Field Blanks	7	·

Note: A = Acceptable

N = Not provided/applicable SW = See worksheet

ND = No compounds detected

R = Rinsate

D = Duplicate TB = Trip blank
- EB = Equipment blank

FB = Field blank

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

	all soil				
1	SA142-1_01_BPC	11	SSAO8-14-1_01_BPC	21	31
2	SA142-2_01_BPC	12	SSAO8-14-2_01_BPC	22	32
3	SA142-3_01_BPC	13	SSAO8-14-3_01_BPC	23	33
4	SA142-4_01_BPC	14	SSAO8-14-3_01_BPC_FD	24	34
5	SA142-5_01_BPC	15	SSAO8-14-4_01_BPC	25	35
6	SSAO8-13-1_01_BPC	16	SSAO8-14-5_01_BPC	26	36
7	SSAO8-13-2_01_BPC	17	SSAO8-13-4_01_BPCMS	27	37
88	SSAO8-13-3_01_BPC	18	SSAO8-13-4_01_BPCMSD	28	38
9	SSAO8-13-4_01_BPC	19	PBS	29	39
10	SSAO8-13-5_01_BPC**	20		30	40

Notes:			,	
		-		
			•	

Page: 1 of 2 Reviewer: M& 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?	/			•
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?	/			
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	<u>, , , , , , , , , , , , , , , , , , , </u>		, <del></del> ,	
Was an LCS anaylzed for this SDG?	<b>/</b>			
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?			<u> </u>	
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?			<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)?	<b>/</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?	<u> </u>		/	
XI. Regional Quality Assurance and Quality Control			1	
Were performance evaluation (PE) samples performed?		/	<u> </u>	
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.	<u>/</u>		<u> </u>	
XIV. Field duplicates		,		I
Field duplicate pairs were identified in this SDG.	/			
Target analytes were detected in the field duplicates.	/	<u></u>		
XV. Field blanks			,	
Field blanks were identified in this SDG.		/		
Target analytes were detected in the field blanks.		<u> </u>	<b>/</b>	

LDC#: 34522E4

# **VALIDATION FINDINGS WORKSHEET** Field Duplicates

	Page:_	<u>of</u>
	Reviewer:	MG
2nd	Reviewer:	1

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

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

Analyte	Concentration		(≤50) RPD	(mg/Kg)	(mg/Kg)	Qualifications (Parent Only)
Arsenic	3,0	3.4	12		Linits	(r drent only)

V:\FIELD DUPLICATES\FD\_inorganic\24522E4.wpd

LDC# 24502E4

# VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

Page: Lof L Reviewer: MG 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 = <u>Found</u> 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)						
1333 ICV	ICP/MS (Initial calibration)	As	39.94	40.0	100	001	\ \
	CVAA (Initial calibration)						_
	ICP (Continuing calibration)						
2132 CC V9	ICP/MS (Continuing calibration)	As	50.03	50.0	00/	00/	->
	CVAA (Continuing calibration)						
	GFAA (Initial calibration)						
	GFAA (Continuing calibation)						

Comments: Refer to Calibration Verification findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results. LDC# 24522E4

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

Page: \_\_\_of\_! 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 =  $|S-D|_X \times 100$ (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:

Where, I = Initial Sample Result (mg/L) SDR = Serial Dilution Result (mg/L) (Instrument Reading x 5) %D = [I-SDR] × 100

	Acceptable (Y/N)	>				<b>د</b> ۔۔۔
Reported	%R/RPD/%D	90/	16	16	6	3.1
Recalculated	%R/RPD/%D	901	16	16	6	2.8
	R (units)	100 (mg/L)	20.0 (mg/kg)	18.7 (mg/kg)	(m3/kg)	(mg/kg)
	True / D / SDR (units)	001	20.06		23.21 (mg/kg)	4.063 (mg/kg
	11	(mg/r)	36 (mg/kg)	00 (mg/kg)	(mg/kg)	(mg//lug)
	Found / S / I (units)	105.60	18.36	(SSR-SR)	As 21. 18	As 4.181
	Element	As	As	ş	As	As
	Type of Analysis	ICP interference check	Laboratory control sample	Matrix spike	Duplicate	ICP serial dilution
	Sample 1D	1631 ICSABA	507 LH06	3136 17	81/L1 bere/9ere	9118 / BID

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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3

LDC #: 24522E4

# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	of
Reviewer:	MG
2nd reviewer:	<b>√</b>

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

A)N A)N A)N	N/A Have results N/A Are results w N/A Are all detect	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?	ts and within the line	ear range of the ICI	⊃γ
Detect equation	ed analyte results for _ on:	# (O , As	were recalcu	lated and verified u	using the following
Concen	tration = (RD)(FV)(Dil) (In. Vol.)	Recalculation:	)(-)		
RD FV In. Vol. Dil	= Raw data conce = Final volume (m = Initial volume (m = Dilution factor	1)	) = 3.	939 Mg/g 6	or mg/kg
#	Sample ID	Analyte	Reported Concentration ( <sup>mg</sup> /kg)	Calculated Concentration (Mg/kg)	Acceptable (Y/N)
	10	As	3.9	3.9	Y
	· · · · · · · · · · · · · · · · · · ·				
			,		
					·
Note:_					
	<del></del>				

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

Collection Date:

November 2, 2010

LDC Report Date:

December 19, 2010

Matrix:

Soil

Parameters:

Perchlorate

Validation Level:

Stage 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-9309-2

Sample Identification

SSAN5-05-0.00\_02\_BPC SSAN5-05-0.00 02 BPCMS

SSAN5-05-0.00\_02\_BPCMSD

SSAN5-05-0.00\_02\_BPCDUP

# Introduction

This data review covers 4 soil samples 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.

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.

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.

# 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 280-9309-2	All analytes reported below the PQL.	J (all detects)	А

# VIII. Overall Assessment

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

# IX. Field Duplicates

No field duplicates were identified in this SDG.

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Perchlorate - Data Qualification Summary - SDG 280-9309-2

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-9309-2	SSAN5-05-0.00_02_BPC	All analytes reported below the PQL.	J (all detects)	А	Sample result verification (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Perchlorate - Laboratory Blank Data Qualification Summary - SDG 280-9309-2

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Perchlorate - Field Blank Data Qualification Summary - SDG 280-9309-2

No Sample Data Qualified in this SDG

# **Tronox Northgate Henderson** VALIDATION COMPLETENESS WORKSHEET

LDC #: 24522F6 SDG #: 280-9309-2 Stage 4 Laboratory: Test America

Date: 12-15-10 Page:\_\_of\_\_ 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
l.	Technical holding times	A	Sampling dates: 11-2-10
lla.	Initial calibration	Α	
llb.	Calibration verification	Α	
ill.	Blanks	A	·
iV	Matrix Spike/Matrix Spike Duplicates	A	MS/MSD
V	Duplicates	A	DUP
VI.	Laboratory control samples	A	LCS/LCSD
VII.	Sample result verification	A	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N	
x	Field blanks	N	

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate FB = Field blank D = Duplicate ·

TB = Trip blank

EB = Equipment blank

# Validated Samples:

9011

1	SSAN5-05-0.00_02_BPC	11	21		31
2	SSAN5-05-0.00_02_BPCMS	12	22	2	32
3	SSAN5-05-0.00_02_BPCMSD	13	23	3	33
4	SSAN5-05-0.00_02_BPCDUP	14	24	,	34
5	PBS	15	25	5	35
6		16	26	3	36
7		17	27	,	37
8		18	28	3	38
9		19	29	)	39
10		20	30	)	40

Notes:		
	_	

LDC #: 24522F6

# **VALIDATION FINDINGS CHECKLIST**

Page:_l	_of_ <u>2</u> _
Reviewer:	
2nd Reviewer:	1~

Method:Inorganics (EPA Method 314.0

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	V			
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?	<b>V</b>			
Were titrant checks performed as required? (Level IV only)			/	
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.		<b>/</b>		
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.	/			
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 $\leq$ 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?	/			
Was an LCS analyzed per extraction batch?	<b>V</b>			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% (85-115% for Method 300.0) QC limits?	<b>√</b>			
VI. Regional Quality Assurance and Quality Control	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,			
Were performance evaluation (PE) samples performed?		/		
Were the performance evaluation (PE) samples within the acceptance limits?			1	

# **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.	<b>V</b>			
IX. Field duplicates				
Field duplicate pairs were identified in this SDG.		<b>V</b>		
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.			1	

LDC # 34533F6

# Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

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

> 314.0 METHOD: Inorganics, Method

\_was recalculated. Calibration date:\_ C104 The correlation coefficient (r) for the calibration of

10-35-10

An initial or 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 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	
Type of Analysis	Analyte	Standard ID	COMC Found (units)	Area True (units)	r or %R	r or %R	Acceptable (Y/N)
Initial calibration		Blank	)	-			
		Standard 1	1.0 (49/1)	0.00303			
		Standard 2	3.5 (	0.00827			
		Standard 3	5.0	0.01638		(=0.49987)	`
	C104	Standard 4	10.0	0.03973	V=0.999973	h9611	<del></del>
		Standard 5	30.00	0.06619		1-11-	-
		Standard 6	40.0 ( 1	0.13068			
		Standard 7	}	i			
Calibration verification		<i>298</i>					
	Cloy	CCVI	9.301 (Mg/L)	9.301 (mg/L) 10.0 (mg/L)	93	93	->
Calibration verification	l			l			ı
		_			l	1	
Calibration verification	Į	1	١		١	1	ı

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# 34532 FG

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

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

> 314.0 METHOD: Inorganics, Method

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

%R = Found x 100

Where,

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

True = concentration of each analyte in the source.

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

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

Original sample concentration Duplicate sample concentration

			•				-
	Acceptable (Y/N)		>	_			>
Reported	%R / RPD		44		93		0.0
Recalculated	%RIRPD		46		25		0.0
	True / D (units)		0.0990 (mg/kg)		1,06 (mg/y)	ð	(mg/kg) 3.71 (mg/kg)
	Found / S (units)		0.0933 (mg/kg) 0.0990 (mg/kg	(SSR-SR)	0.98 (mg/kg)	0	3.71 (mg/kg)
	Element		2010		C104		C104
	Type of Analysis	Laboratory control sample		Matrix spike sample		Duplicate sample	
	Sample ID	5882	700	6) 10	7	0031/00cs	7

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

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LDC#: <u>24522</u>F6

# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	of
Reviewer:	MG
2nd reviewer:	

METH	IOD: Inorganics, Method	d314.0			
Please YN YN YN	N/A Have results N/A Are results with	ow for all questions answered "N". Not ap been reported and calculated correctly? ithin the calibrated range of the instrume ion limits below the CRQL?		e identified as "N/	A".
Comp recalc	ound (analyte) results fould and verified using	or <u># 1, С10 и</u> g the following equation:	repo	orted with a positi	ve detect were
Concen	y = mx + 5	Recalculation: $0.1/578 = 0.0033 \left(\frac{x}{70}\right) -$	0.0002		
ν-	where $m = 0.0033$	361 45 Mg/ = V		0 710 Ma	/ ma/
<b>1</b>	d:(=10 x	2 then (351.45 Mg/	(0.938)	= 3.110	/g or 1/kg
#	Sample ID	V /	Reported Concentration ( <sup>M3</sup> /k <sub>1</sub> )	Calculated Concentration (Mg/kq)	
	(	ClOy	3.7	3.70	Y
		, , , , , , , , , , , , , , , , , , ,			
				<u> </u>	
			1		
Note:_					