

LABORATORY DATA CONSULTANTS, INC.

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

Northgate Environmental Management, Inc.

July 8, 2010

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

SUBJECT:

Tronox LLC Facility, PCS, Henderson, Nevada,

**Data Validation** 

Dear Ms. Arnold,

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

### **LDC Project # 23436:**

### SDG#

### **Fraction**

280-2131-7, 280-2400-10, 280-2500-9 280-2541-2, 280-3059-9, 280-3197-6 280-3584-1, 280-3624-1, 280-3679-1 280-3679-3 Semivolatiles, 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 Superfund Organic Methods Data Review, June 2008
- USEPA, Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, October 2004

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

Sincerely,

Erlinda T. Rauto

**Operations Manager/Senior Chemist** 

23436ST.wpd

		S																							46
		≷																							٥
		တ																							0
		≷																							٥
		S																							٥
		≥																							٥
		S																							0
		≩																							٥
		S																							0
		3																							0
		S																							0
(S:		≥																							0
PC		S	$\dashv$		$\neg$																				0
LLC-Northgate, Henderson NV / Tronox PCS	İ	3	1																						0
5		S	1		$\exists$																				0
/ T		≥	7																						0
≥		S	1																						0
l no		≥																							0
) LE		S																							0
nde		3																							0
유	<sup>7</sup> , €	S	'	,	-	-	5	-		,		,	2	F	9	2	0								17
te,	CLO <sub>4</sub> (314.0)	≥			-	,	0	-	١			-	0	0	0	0	1	-							-
ıga	<sub>5</sub> (2	S	-1	'	-	-	0			-	-				1	•	ı								0
ort	Mg (6020)	≥	,	'	•	'		-			-	-	ı			•	-								-
Ž	20)	S		'	,	-	0		•	,	,	-		ı	٠	•									0
-	<b>M</b> n (6020)	3	-	٠,	1	-	1	,	,	•		ı	•	ı		1	ı								-
	ç ()	S	-	,	ı	·	0	ı			•	,	•	-	1	-	٠								0
LDC #23436 (Tronox	Pb (6020)	≯	·	٠,		'	-	,		ı	,	ı	,	,		1	,								-
(Tr	° (02	S	-	٠	1	·	0	ı	-	ı	•	-	•	ı	•	•	1								0
36	Co (6020)	≷		'	•	'	-	-	•	-	•				-	-	-								-
234	s 20)	S	٠,	-	-	Σ	0	-	-		١	1	2	0	2	- 2	0								6
#	As (6020)	≯	-	0	0	0	~	ı	1		•	-	0	0	0	0	l								2
ă	SVOA (8270C)	S	Ψ.	,	1	'	0	1	1	3	1	4		1	1	1	0								=
	SV (827	≥	0	-	1	٠,	-	0	0	0	0	0		1	1	1	-								2
	<b>∵</b> ₽∃		5/10	5/10	5/10	5/10	5/10	5/10	5/10	5/10	5/10	5/10	5/10	5/10	5/10	5/10	5/10								
	(3) DATE DUE		06/23/10 07/15/10	07/15/10	07/15/10	07/15/10	07/15/10	07/15/10	07/15/10	06/23/10 07/15/10	06/23/10 07/15/10	06/23/10 07/15/10	06/23/10 07/15/10	06/23/10 07/15/10	06/23/10 07/15/10	06/23/10 07/15/10	06/23/10 07/15/10						ŀ		
	μĢ		7	/10					/10	//10	/10	/10	/10	110	110	110	110								
	DATE REC'D		6/23	06/23/10	06/23/10	06/23/10	06/23/10	06/23/10	06/23/10	)6/23	)6/23	)6/23	)6/23	)6/23	)6/23	)6/23	36/23								
				$\exists$						)	Ŭ	Ŭ		_	)	Ŭ		T		$\Box$			T		
4				-19	6-	6-6	1-2	6-6	9-6	9-2	9-2	1-1	1-1	1-1	9-1	9-1	9-3								
e 2B/	SDG#	r/Soi	280-2131-7	280-2400-10	280-2500-9	280-2500-9	280-2541-2	280-3059-9	280-3059-9	280-3197-6	280-3197-6	280-3584-1	280-3624-1	280-3624-1	280-3679-1	280-3679-1	280-3679-3								T/LR
Stage 2B/4	S	Water/Soil	280	280-	280	280	280	280	280	280	280	280	280	280	280	280	280								
		- 27																		 					Ш
EDD	СРС	Matrix:	∢	В	ပ	ပ	۵	ш	ш	ட	ш	ပ	I	I	_	_	_			<u> </u>					Fotal

Attachment 1

DL 04/23/10

LDC #: 23436

SDG #:<u>280-2131-7</u>, <u>280-2400-10</u>, <u>280-2500-9</u>, <u>280-2541-2</u> <u>280-3059-9</u>, <u>280-3197-6</u>, <u>280-3584-1</u>, <u>280-3624-1</u>

<u>280-3679-1, 280-3679-3</u>

Page: 1\_of 1 Reviewer: <u>JE</u> 2nd Reviewer: BC

### Tronox Northgate Henderson Worksheet

EDD Area	Yes	No	NA	Findings/Comments
1. Completeness			<del>,</del>	
Is there an EDD for the associated Tronox validation report?	X			
II. EDD Qualifier Population				
Were all qualifiers from the validation report populated into the EDD?	X			
III. EDD Lab Anomalies				
Were EDD anomalies identified?		X		
If yes, were they corrected or documented for the client?			х	See EDD_discrepancy_ form_LDC23436_070710.doc
IV. EDD Delivery				
Was the final EDD sent to the client?	X			

### Tronox LLC Facility, PCS, Henderson, Nevada Data Validation Reports LDC #23436

Semivolatiles



# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

April 6, 2010

**LDC Report Date:** 

July 6, 2010

**Matrix:** 

Soil

Parameters:

Semivolatiles

Validation Level:

Stage 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-2131-7

Sample Identification

SSAJ8-01-10BPC

### Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination.

  This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

### III. Initial Calibration

Initial calibration was performed using required standard concentrations.

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

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

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

### IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

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

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

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

### V. Blanks

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

Sample FB-04072010-RZD (from SDG 280-2216-2) was identified as a field blank. No semivolatile contaminants were found in this blank with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB-04072010-RZD	4/7/10	Bis(2-ethylhexyl)phthalate	2.2 ug/L	All samples in SDG 280-2131-7

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

### VI. Surrogate Spikes

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

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

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

### XI. Target Compound Identifications

All target compound identifications were within validation criteria.

### XII. Project Quantitation Limit

All compound quantitation and CRQLs were within validation criteria.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-2131-7	All compounds reported below the PQL.	J (all detects)	А

### XIII. Tentatively Identified Compounds (TICs)

Tentatively identified compounds were not reported by the laboratory.

### XIV. System Performance

The system performance was acceptable.

### 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, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-2131-7

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-2131-7	SSAJ8-01-10BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

Tronox LLC Facility, PCS, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-2131-7

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Semivolatiles - Field Blank Data Qualification Summary - SDG 280-2131-7

No Sample Data Qualified in this SDG

## Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

SDG #:	280-2131-7
ahorato	V. Test America

LDC #: 23436A2a

Stage 4

Date:_	7/02/1
Page:_	of /
Reviewer:	JM
2nd Reviewer	

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 4/06 /10
11.	GC/MS Instrument performance check	A	
III.	Initial calibration	A	7 KSD M
IV.	Continuing calibration/ICV	A	2 KSD 12 COV/1W = 25 B
V.	Blanks	4	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	N	client spec
VIII.	Laboratory control samples	A	client spec
IX.	Regional Quality Assurance and Quality Control	N	
Χ.	Internal standards	A	
XI.	Target compound identification	À	
XII.	Compound quantitation/CRQLs	A	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	A	
XV.	Overall assessment of data	A	
XVI.	Field duplicates	N	
XVII.	Field blanks	WZ	FB = FB-04072010- RZD (280-2216-2)

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

TB = Trip blank

EB = Equipment blank

Validated Samples:

(1)

	Soil				
1	SSAJ8-01-10BPC	11	21	31	
2	NB 280- 11504 1-A	12	22	32	
3		13	23	33	
4		14	24	34	
5		15	25	35	
6		16	26	36	
7		17	27	37	
8		18	28	38	
9		19	29	39	
10		20	30	40	

LDC #: 39 476 A29 SDG #: See Cover

### **VALIDATION FINDINGS CHECKLIST**

Page: \\_\ of \\_2 Reviewer: \\_\ \forall V\_i 2nd Reviewer: \\_\

Method: Semivolatiles (EPA SW 846 Method 8270C)

	Ĭ	T*****		
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
		F		
All technical holding times were met.	_			
Cooler temperature criteria was met.				Digitire .
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?				
(II. Initial calibration				
		F		
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?				Control of the Contro
V. Blanks				
Was a method blank associated with every sample in this SDG?				
Was a method blank analyzed for each matrix and concentration?				
Was there contamination in the method blanks? If yes, please see the Blanks			/	
validation completeness worksheet.				
VI, Surrogate spikes			4	
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	ر			Commission of the Commission o
Was an LCS analyzed for this SDG?				

LDC #: 23 436 Aza SDG #: See Cover

### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 2
Reviewer: 2nd Reviewer:

		<del>,</del>	<del></del>	T T T T T T T T T T T T T T T T T T T
Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?	<u></u>		<u> </u>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
X. Internal standards				Control of the second s
Were internal standard area counts within -50% or +100% of the associated calibration standard?				
Were retention times within + 30 seconds from the associated calibration standard?				
XI. Target compound identification		191		
Were relative retention times (RRT's) within $\pm$ 0.06 RRT units of the standard?				
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	1			
Were chromatogram peaks verified and accounted for?				
XII. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XIII. Tentatively identified compounds (TICs)				
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?				
Were relative intensities of the major ions within $\pm$ 20% between the sample and the reference spectra?				
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?				
XIV. System performance				
System performance was found to be acceptable.				
XV. Overall assessment of data				
Overall assessment of data was found to be acceptable.	$\overline{A}$			
KVI. Field duplicates				
Field duplicate pairs were identified in this SDG.		1		Variables in the second
Farget compounds were detected in the field duplicates.			7	
KVII. Field blanks			L	1944 • 10
Field blanks were identified in this SDG.	1			(Contract of the Contract of t
arget compounds were detected in the field blanks.	X			

# VALIDATION FINDINGS WORKSHEET

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

A. Phenol**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenol**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene⁴*	W. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitropheno!*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenoi**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	000. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzyiphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chiorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyi alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	111.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	nnn
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	WW.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	WWW.

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

LDC#: 03 436 A74 SDG #:

# VALIDATION FINDINGS WORKSHEET Field Blanks

Page:_	Reviewer:_	2nd Reviewer:

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

Were field blanks identified in this SDG?

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

Blank units: 4/67/12

Sampling date: 4/67/12

Field blank type: (circle one)(Field Blank DRinsate / Other.

( an Sample Identification Associated Samples: F8-04672010-RZD Blank ID 是任任 Compound CRaL

Associated sample units: Blank units:

Sampling date: Field Blank / Rinsate / Other:

Associated Samples:

	ıtion				
Associated Salliples.	Sample Identification				
Associated					
				-	
71151.					
יין אווואון א					
וכ) ו וכות טומו	Blank ID				
וח בוחוח ישל	Compound				
icia Dialin type, (circle Orie) i icia Dialin / initisate / Other.	Сош				CRQL

5x Phthalates 2x All others

LDC # 23456 # 29 SDG #:

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page:

2nd Reviewer: Reviewer:

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

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

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

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

 $C_x$  = Concentration of compound,

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

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

X = Mean of the RRFs S= Standard deviation of the RRFs,

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
		Calibration		RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
#	Standard ID	Date	Compound (Internal Standard)	( 50 std)	( 50 std)	(Initial)	(Initial)		
_	ICAL	4/20/2010	4/20/2010 1,4-Dioxane (IS1)	0.5633	0.5633	0.5623	0.5623	3.9	3.88
	MSSK		Naphthalene (IS2)	1.0165	1.0165	0.9768	0.9768	12.1	12.09
			Fluorene (IS3)	1.3180	1.3180	1.2472	1.2472	11.8	11.84
			Hexachlorobenzene (IS4)	0.2487	0.2487	0.2372	0.2372	7.3	7.30
			Chrysene (IS5)	1.0840	1.0840	1.0536	1.0536	12.2	12.24
			Benzo(a)pyrene (IS6)	1.0986	1.0986	1.0351	1.0352	4.3	4.30

	Area IS	278805	1080033	647354	1132325	1313329	1354411
٠	Area cpd	196298	1372301	1066485	352069	1779496	1859994
	nc IS/Cpd	40/50	40/20	40/20	40/20	40/20	40/20

Conc	1,4-Dioxane	Naphthalene	Fluorene	Hexachlorob	Chrysene	Benzo(a)py
4.00	0.6098	1.1305	1.4155		1.2500	0.9983
10.00	0.5619	1.0760	1.3980	0.2566	1.1847	1.0322
20.00	0.5728	1.0779	1.3632	0.2532	1.1196	1.0866
50.00	0.5633	1.0165	1.3180	0.2487	1.0840	1.0986
80.00	0.5436	0.9602	1.2264	0.2394	1.0191	1.0452
120.00	0.5571	0.9039	1.1370	0.2323	0.9691	1.0322
160.00	0.5446	0.8412	1.0734	0.2198	0.9201	1.0281
200.00	0.5456	0.8082	1.0461	0.2107	0.8825	0.9600
×	0.5623	0.9768	1.2472	0.2372	1.0536	1.0352
S	0.0218	0.1181	0.1477	0.0173	0.1290	0.0445

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 # 27 436 A20\_ SDG # See Cover

Continuing Calibration Results Verification VALIDATION FINDINGS WORSHEET

Page\_ Reviewer: 2nd Reviewer.

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

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

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF = (Ax)(Cis)/(Ais)(Cx)

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

Ax = Area of compound

Ais = Area of associated internal standard Cis = Concentration of internal standard Cx = Concentration of compound Recalculated ۵ % 6.5 0.2 7.1 2.7 2.5 2.7 Reported 2.7 6.5 7.1 2.7 Recalculated (CC RRF) 1.2138 0.5223 0.9507 0.2377 0.9852 1.0607 Reported (CC RRF) 0.5223 1.2138 0.9852 0.2377 1.0607 Average RRF (Initial RRF) 0.5623 0.9768 1.2472 0.2372 1.0536 1.0351 (IS1) (IS2) (IS3) (184) (185) (186) Compound (Reference IS) Hexachlorobenzene Benzo(a)pyrene Naphthalene 1,4-Dioxane Chrysene Fluorene Calibration 04/20/10 Date Standard ID K2922 # 0

Area IS							
Area Cpd							
Area IS		314591	1232258	759171	1333067	1526995	1433844
Area Cpd		328617	2342930	1842898	633836	3008940	3041867
Concentration	(IS/Cpd)	40/80	40/80	40/80	40/80	40/80	40/80
(S)		(IS1)	(182)	(183)	(IS4)	(185)	(981)
Compound (Reference		1,4-Dioxane	Naphthalene	Fluorene	Hexachlorobenzene	Chrysene	Benzo(a)pyrene
	Area Cpd Area IS Area Cpd	Concentration Area Cpd Area IS Area Cpd (IS/Cpd)	Concentration   Area Cpd   Area IS   Area Cpd   (IS/Cpd)   328617   314591	Concentration (IS1)         Area Cpd (IS/Cpd)         Area IS (IS/Cpd)         Area Cpd (IS/Cpd)	Concentration (IS1)         Area Cpd (IS2)         Area IS (IS2)         Area Cpd (IS2)         Area IS (IS2)         Area Cpd (IS2)         Area IS (IS2)         Area Cpd (IS2)         Area	Concentration (ISJ)         Area Cpd (ISJ)         Area IS (ISJ)         Area Cpd (ISJ)         Area Cpd (ISJ)         Area IS (ISJ)         Area Cpd (ISJ)         Area	Concentration         Area Cpd         Area IS         Area Cpd           (IS/Cpd)         328617         314591         6           (IS2)         40/80         2342930         1232258           (IS3)         40/80         1842898         759171           (IS4)         40/80         633836         1333067           (IS5)         40/80         3008940         1526995

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

Page:	<u>lof_1</u>
Reviewer:_	JU
2nd reviewer:	0/

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found

Sample ID:

SS = Surrogate Spiked

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	100	87. ~	87	87	
2-Fluorobiphenyl		84.1	84	2 7	1 - 7 -
Terphenyl-d14		91 9	92	a v	
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					Difference
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					

23 \$ 96 A 2 a SDG #: See Cover LDC #:

# VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

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

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

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

% Recovery = 100 \* (SC/SA

SSC = Spike concentration SA = Spike added Where:

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

LCS/LCSD samples:

6-A 40511-082 S S

	ัด	pike	š	ike	3	CS	<u> </u>	CSD	GSC FSC -	G
Compound		Added ( りん)	Conce ( 4/5	Concentration ( $\mu_{\zeta}$ / $\mu_{\zeta}$ )	Percent F	Percent Recovery	Percent Recovery	Perovery		
	SUL	C 1CSD	l Cs	l CSD	Ponorted	- Page	C			
						Net all	керопед	Kecalc	Reported	Recalculated
Phenol										
N-Nitroso-di-n-propylamine										
4-Chloro-3-methylphenol										
Acenaphthene	099	\$	22,80	S. S	86	98				
Pentachlorophenol										
Pyrene	2660		N. 24. 4		9,4	4 4				
						<b></b>				

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#: 73436 AZA SDG#: Sre Cover

## VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	lof1
Reviewer:	
2nd reviewer:	

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

Percent solids, applicable to soil and solid matrices only.

<u>Y</u> )	N	N/A
X	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?

Conce	entration	(A. )(RRF)(V.)(V.)(%S)	Example:
$A_{x}$	=	Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D. 🕌 📗 ,
$A_{is}$	=	Area of the characteristic ion (EICP) for the specific internal standard	and a second
l <sub>s</sub>	=	Amount of internal standard added in nanograms (ng)	Conc. = $\frac{(2/3 88\%)(40)(1n/)(1n)}{(1192/45)(0,237)(31,29)(0.926)(0)}$
V <sub>o</sub>	=	Volume or weight of sample extract in milliliters (ml) or grams (g).	
$V_{i}$	=	Volume of extract injected in microliters (ul)	=10 47. V
$V_{t}$	=	Volume of the concentrated extract in microliters (ul)	,
Df	=	Dilution Factor.	2 lovo us/for

2.0	= Factor of 2 to accoun	nt for GPC cleanup				
#	Sample ID	Compound		Reported Concentration ( )	Calculated Concentration (	Qualification
	**************************************					
			,			

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

April 16, 2010

LDC Report Date:

July 7, 2010

Matrix:

Water

Parameters:

Semivolatiles

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-2541-2

Sample Identification

EB-04152010-2RZD

### Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination.

  This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

### III. Initial Calibration

Initial calibration was performed using required standard concentrations.

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

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

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

### IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

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

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

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

### V. Blanks

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

Sample EB-04152010-2RZD 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

The laboratory has indicated that there was insufficient sample volume for analysis of the matrix spike and matrix spike duplicate.

### 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

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 280-2541-2	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, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-2541-2

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-2541-2	EB-04152010-2RZD	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (sp)

Tronox LLC Facility, PCS, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-2541-2

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Semivolatiles - Equipment Blank Data Qualification Summary - SDG 280-2541-2

No Sample Data Qualified in this SDG

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

SDG #: 280-2541-2 Laboratory: Test America

LDC #: 23436D2a

Stage 2B

Page:\_lof Reviewer: 2nd Reviewer:

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

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

	Validation Area		Comments
l.	Technical holding times	A	Sampling dates: 4 16 ho
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	A	3 RSD 12
IV.	Continuing calibration/ICV	A	3 KSD 17 COV/101 = 25 }
V.	Blanks	Α	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	N	Ctient spec Insufficient sample US 10
VIII.	Laboratory control samples		vcs 15
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	MD	EB = 1

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

TB = Trip blank

EB = Equipment blank

Validated Samples:

Water

	VV W/ CO					
1	EB-04152010-2RZD	11	21	3	31	
2	MB 280-11838/1-A	12	22	3	32	
3	/	13	23	3	33	
4		14	24	3	34	
5		15	25	3	35	
6		16	26	3	36	
7		17	27	3	37	
8		18	28	3	38	
9		19	29	3	39	
10		20	30	4	10	

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

April 29, 2010

LDC Report Date:

July 6, 2010

Matrix:

Soil

Parameters:

Semivolatiles

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-3059-9

Sample Identification

SSAO5-05-3BPC

SSAO5-05-4BPC\*\*

SSAO5-05-3BPCMS

SSAO5-05-3BPCMSD

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

### Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination.

  This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

### III. Initial Calibration

Initial calibration was performed using required standard concentrations.

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

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

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

### IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

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

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

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

### V. Blanks

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

Sample FB-04072010-RZC (from SDG 280-2280-2) was identified as a field 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. Surrogate recoveries (%R) were not within QC limits for sample SSAO5-05-4BPC\*\*. Since the samples were diluted out, no data were qualified.

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

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

### XI. Target Compound Identifications

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

### XII. Project Quantitation Limit

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

Sample	Compound	Finding	Flag	A or P
SSAO5-05-3BPC	Benzo(b)fluoranthene	Due to lack of resolution between these compounds in the samples, the laboratory	J (all detects) UJ (all non-detects) J (all detects)	Р
	Benzo(k)fluoranthene	performed the quantitation using the total peak area.	UJ (all non-detects)	

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-3059-9	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, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-3059-9

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-3059-9	SSAO5-05-3BPC	Benzo(b)fluoranthene Benzo(k)fluoranthene	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Project Quantitation Limit (q)
280-3059-9	SSAO5-05-3BPC SSAO5-05-4BPC**	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (sp)

Tronox LLC Facility, PCS, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-3059-9

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Semivolatiles - Field Blank Data Qualification Summary - SDG 280-3059-9

No Sample Data Qualified in this SDG

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

LDC #: 23436E2a Stage 2B/4 SDG #: 280-3059-9 Laboratory: Test America

Date:	162/10
Page:_	of
Reviewer:_	<b>3V</b> C
2nd Reviewer:	Q

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 4/29/ro
11.	GC/MS Instrument performance check	A	
111.	Initial calibration	A	2 RSD 17
IV.	Continuing calibration/ICV	A	ca/101 = 25 }
V.	Blanks	Á	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	A	us
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	À	Not reviewed for Stage 2B validation.
XII.	Compound quantitation/CRQLs	(NZ	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	ND	FB-FB04072010-RZC (SDL 280-2280-)

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate FB = Field blank D = Duplicate TB = Trip blank

EB = Equipment blank

Validated Samples:

tall Indicates sample underwent Stage 4 validation

	361				
1	SSAO5-05-3BPC	11	MB 280-15262/1-A	21	31
2	SSAO5-05-4BPC**	12		22	32
3	SSAO5-05-3BPCMS	13		23	33
4	SSAO5-05-3BPCMSD	14		24	34
5		15		25	35
6		16		26	36
7		17		27	37
8		18		28	38
9		19		29	39
10		20		30	40

### **VALIDATION FINDINGS CHECKLIST**

Method: Semivolatiles (EPA SW 846 Method 8270C)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				resp.
All technical holding times were met.				
Cooler temperature criteria was met.				
II. GC/MS Instrument performance check	1		ı	
Were the DFTPP performance results reviewed and found to be within the specified criteria?				
Were all samples analyzed within the 12 hour clock criteria?				
III. Initial calibration		I .		
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 <u>&gt;</u> 0.990?	_			
Were all percent relative standard deviations (%RSD) $\leq$ 30% and relative response factors (RRF) $\geq$ 0.05?				
IV. Continuing calibration				property and the second
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?		ſ		
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?				
Vi Blanks				
Was a method blank associated with every sample in this SDG?	•			
Was a method blank analyzed for each matrix and concentration?			-	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
VI. Surrogate spikes				
Were all surrogate %R within QC limits?				***
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?				
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?				
VII. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.				
Was a MS/MSD analyzed every 20 samples of each matrix?				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII. Laboratory control samples			- 1	
Was an LCS analyzed for this SDG?	1			

LDC#: 23 476 E39 SDG#: See Cover

### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 2
Reviewer: 12
2nd Reviewer: 2

	T	T	T	
Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?			<del> </del>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		<u> </u>		
IX. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
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				
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		14.0		Property of the state of the st
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				en en president de la companya de l La companya de la co
Overall assessment of data was found to be acceptable.	7	T		
XVI: Field duplicates				
	T	7	4	
Field duplicate pairs were identified in this SDG.	_	-4	_	
Target compounds were detected in the field duplicates.			_}	
(VII. Field blanks				
Field blanks were identified in this SDG.				
arget compounds were detected in the field blanks.				

## VALIDATION FINDINGS WORKSHEET

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

A. Phenoi**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenol**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	VV. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	000. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthaiate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	111.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octy/phthalate**	nnn
N. 2-Nitropheno!**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	WW.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	WWW.

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

23436 EJA #DC#

VALIDATION FINDINGS WORKSHEET

Surrogate Recovery

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

Page:

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

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

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

If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R? If any %R was less than 10 percent, was a reanalysis performed to confirm %R?

#	Date	Sample ID	Surrogate	%R (Limits)	nits)	Qualifi	Qualifications
		(س) ک	NBZ	677	( 00) - 05)	No grant	(my 1 out)
					( )	۵	
		a (4x)	NB2	45	( 1		_
					( )		
					( )		
					( )		
					( )		
					( )		-
					( )		
					( )		
					)		majorna, sprincial de la companya d
					( )		
					( )		
					( )		
					( )		
					( )		
					( )		
					( )		
					( )		
					( )		
					( )		
					( )		
					( )		
					( )		
* QC limi S1 (NBZ) S2 (FBP) S3 (TPH) S4 (PHL)	* QC limits are advisory S1 (NBZ) = Nitrobenzene-d5 S2 (FBP) = 2-Fluorobipheny S3 (TPH) = Terpheny-d14 S4 (PHL) = Phenol-d5	OC Limits (Soil) OC Limits (Water) 35-114 yl 30-115 43-116 18-137 33-141 24-113 10-94		S5 (2FP)= 2-Fluorophenol S6 (TBP) = 2,4,6-Tribromophenol S7 (2CP) = 2-Chlorophenol-d4 S8 (DCB) = 1,2-Dichlorobenzene-d4	QC Limits (Soil) 25-121 19-122 20-130*	QC Limits (Water) 21-100 10-123 33-110* 16-110*	

LDC#: 23 436 E29 SDG #: \_ S\_\_

VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

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

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

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

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

AN NA

MS/MSD. Soil / Water.

V N NA

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?

-	Ç.	di damian	7	MS MS	MSD (-a); i, a.y	777	0 T T T T T T T T T T T T T T T T T T T	31.00
		7/2	Sumodius V	(SIIIIII)	121 (CL-1)	Nro (chillis)	Associated Samples	No mice Car in
		J	3					
						( )		
					( )	·		
				( )	(	( )		
				( )		( )		
				( )				
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )		(		
				(	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )		)		

	Compound	QC Limits (Soil)	RPD (Soil)	QC Limits (Water)	RPD (Water)		Compound	QC Limits (Soil)	RPD (Soil)	QC Limits (Water)	RPD (Water)
Ą	Phenol	26-90%	< 35%	12-110%	< 42%	99	Acenaphthene	31-137%	< 19%	46-118%	< 31%
ن	C. 2-Chlorophenol	25-102%	< 50%	27-123%	< 40%	-	4-Nitrophenol	11-114%	< 50%	10-80%	< 50%
ш	1,4-Dichlorobenzene	28-104%	< 27%	36-97%	< 28%	X X	2,4-Dinitrotoluene	28-89%	< 47%	24-96%	< 38%
-	N-Nitroso-di-n-propylamine	41-126%	< 38%	41-116%	< 38%	T.	Pentachlorophenol	17-109%	< 47%	9-103%	< 50%
œ	1,2,4-Trichlorobenzene	38-107%	< 23%	39-98%	< 28%	77.	Pyrene	35-142%	< 36%	26-127%	< 31%
>	4-Chloro-3-methylphenol	26-103%	< 33%	23-97%	< 42%						

7	\
Ľυ	۲
26	رگر
Y	- 기
3	अ
	[ ِ
#	#
$\mathbf{O}$	Q
ŏ	ă
	S

# VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

Reviewer:

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

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

N/A

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

Qualifications	JAT P (2)								*27	
Associated Samples	7									
Finding	666 HHH UNITSOlved									
Sample ID										
# Date										

Comments: See sample calculation verification worksheet for recalculations

LDC#: 27 FN EV. SDG#: Sec Con

# VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: of / 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:

 $\label{eq:RRF} RRF = (A_x)(C_{is})/(A_{is})(C_x)$  average RRF = sum of the RRFs/number of standards

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

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

%RSD = 100 \* (S/X)

S= Standard deviation of the RRFs,

X = Mean of the RRFs

					Donottod	Pacalculated	Reported	Recalculated	Reported	Recalculated
					Pepulied	Ivecalculated	portodori			
		Calibration			RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
#	Standard ID Date	Date	Compound (Internal Standard)	andard)	(50 std)	(50 std)	(Initial)	(Initial)		
•	140	070000		(154)	0.5630	0.5630	0.5686	0.5686	2.7	2.68
	I AL	0102/21/6	3/12/2010 1,4-Dioxalle					,;;;;	ر	00 0
	MSSK		Naphthalene	(IS2)	1.0281	1.0281	1.0211	1.0211	0.0	0.00
			Fliorene	(183)	1.3033	1.3033	1.2978	1.2978	7.0	96.9
				(10.4)	00000	0.222	0.2313	0.2312	2.4	2.45
			Hexachioropenzene	(134)	0.6360	0.5025				
			Chrysene	(185)	1.0597	1.0597	1.0588	1.0588	7.7	/./4
			0	(108)	1 0050	1 0950	1.0629	1.0629	4.0	4.02
			Derizo(a)pyrerie	(100)	2000:1					

۱	-											
	1,4-Dioxane	0.5835	0.5929	0.5646	0.5630	0.5874	0.5574	0.5566	0.5559	0.5559	0.5686	01,00
	Conc	4.00	10.00	20.00	50.00	80.00	120.00	160.00	200.00		×	
	Area IS	256060	993578	583548	978167	1052500	1028084					
	a cpd	180207	276874	950651	283964	394199	407224					

40/50

40/50

40/50 40/50 40/50

nc IS/Cpd

	4 A-Diovana	Nanhthalene	Fluorene	Hexachlorob	Chrysene	Benzo(a)py
5	- 11	a de la				0,000
4.00	0.5835	1.1259	1.4452		1.1823	0.9940
10.00	0.5929	1.0644	1.3682	0.2384	1.1293	1.0380
20.00		1.0578	1.3466	0.2340	1.1090	1.0835
50,00	0.5630	1.0281	1.3033	0.2320	1.0597	1.0950
80.00	0.5874	1.0313	1.2950	0.2362	1.0775	1.1324
120.00	0.5574	1.0018	1.2565	0.2294	0.9858	1.0769
160.00	0.5566	0.9425	1.1967	0.2264	0.9750	1.0450
200.00		0.9170	1.1712	0.2222	0.9515	1.0380
	0.5559					
×	0.5686	1.0211	1.2978	0.2312	1.0588	1.0629
S	0.0152	0.0673	0.0903	0.0057	0.0819	0.0427

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

LDC # マラ 中ル E マム SDG # See Cover

### Continuing Calibration Results Verification VALIDATION FINDINGS WORSHEET

Page 1 of 1 Reviewer: 2nd Reviewer: 4

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

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

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

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

Ax = Area of compound

Ais = Area of associated internal standard

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

Cis = Concentration of internal standard Cx = Concentration of compound

		Calibration		Average RRF	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Date	Compound (Reference IS)	(Initial RRF)	(CC RRF)	(CC RRF)	Ω%	۷D
-	K3872	05/18/10	1,4-Dioxane (IS1)	0.5700	0.5806	0.5806	1.9	1.9
			(1S2)	1.0211	1.0402	1.0402	1.9	1.9
				1.2978	1.3277	1.3277	2.3	2.3
			Hexachlorobenzene (IS4)	0.2313	0.2302	0.2302	0.5	0.5
			Chrysene (1S5)	1.0588	1.0669	1.0669	0.8	0.8
			Benzo(a)pyrene (IS6)	1.0629	1.1392	1.1392	7.2	7.2
2	K3917	05/18/10						
			Hexachlorobenzene (IS4)	0.2313	0.2302	0.2302	9.0	0.5

		CCV1		CCV2		
Compound (Reference IS)	(S)	Concentration	Area Cpd	Area IS	Area Cpd	Area IS
		(IS/Cpd)				
1,4-Dioxane	(IS1)	40/80	365341	314601		
Naphthalene	(182)	40/80	2563410	1232195		
Fluorene	(183)	40/80	1953157	735542		
Hexachlorobenzene	(184)	40/80	569384	1236654	553547	1202559
Chrysene	(185)	40/80	2723280	1276261		
Benzo(a)pvrene	(186)	40/80	2629449	1154050		
6-16-1						

LDC#:_	73	436 E 29
SDG #:_	Sre	Cover

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

Page:_	lof_1_
Reviewer:	M_/
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

Sample ID: # 7 (IX)

SS = Surrogate Spiked

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	160	49.2	49	49	٥
2-Fluorobiphenyl	1	60.0	66	C 6	
Terphenyl-d14		66.1	66	65	
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#: >3496 E22 SDG #: See Cover

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

Page: lof I 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

SSC = Spiked sample concentration SA = Spike added Where:

SC = Sample concentation

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

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples:

	ŝ	Spike	Sample	Spiked	Spiked Sample	Matrix	Matrix Spike	Matrix Spike Duplicate	Duplicate	US/WSD	SD
Compound	Ad (1/5,	Added $(u_{\zeta}/k_{\zeta})$	Concentration	Concel (	ntration )	Percent Recovery	ecovery	Percent Recovery	ecovery	RPD	
	MS	U MSD	0	MS	MSD	Reported	Recalc	Renorted	Docela	Conception	
Phenol										panoday	Kecalculared
N-Nitroso-di-n-propylamine											
4-Chloro-3-methylphenol											
Acenaphthene	2966	3200	0	2196	2620	74	74	43	20	10	2
Pentachlorophenol										ν,	
Pyrene	2960	3200	of c	2410	2763	pz	75	80	8	14	9.
										<u> </u>	

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

LDC#: 25436 E 22 SDG #: See Coner

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

Page: of Reviewer:\_

2nd Reviewer: 9

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

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

% Recovery = 100 \* (SC/SA

SSC = Spike concentration SA = Spike added Where:

280- 15262/2-A ర్గ LCS/LCSD samples: \_

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

	S	pike	Ś	Spike	J	CS	3	CSD	1 CS/I	CS/I CSD
Compound	A ( US	Added ( 내일/독 )	Conce	Concentration ( ½ /೬ )	Percent	Percent Recovery	Percent Recovery	Recovery	RPD	Q,
	SOI	l CSD	S) I	l CSD	Renorted	Recalc	Reported	Recalc	Renorted	Poralculator
Phenol										
N-Nitroso-di-n-propylamine										
4-Chloro-3-methylphenol										
Acenaphthene	29%	₩	1980	2	74	74				
Pentachlorophenol										
Pyrene	2992	+	2/2		Ç,	80				
			, and the second							

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#: >3436 E2a SDG#: <u>Sre Cor</u>er

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	of1_
Reviewer:_	$\mathcal{M}_{\Lambda}$
2nd reviewer:	

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

P	N	N/A
V	И	N/A

2.0

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?

Concen	tration	$A = \frac{(A_{x})(I_{s})(V_{t})(DF)(2.0)}{(A_{is})(RRF)(V_{o})(V_{i})(\%S)}$
A <sub>x</sub>	=	Area of the characteristic ion (EICP) for the compound to be measured
$A_{is}$	=	Area of the characteristic ion (EICP) for the specific internal standard
l <sub>s</sub>	=	Amount of internal standard added in nanograms (ng)
$V_{o}$	=	Volume or weight of sample extract in milliliters (ml) or grams (g).
$V_{I}$	=	Volume of extract injected in microliters (ul)
$V_t$	=	Volume of the concentrated extract in microliters (ul)
Df	=	Dilution Factor.
%S	=	Percent solids, applicable to soil and solid matrices only.

Factor of 2 to account for GPC cleanup

Example:
Sample I.D
conc. = (342783(40)(1m) )(4)(100) (1036671)(0.2313)(20,23)(0.776)(0.776)
= 9760
2 98 us/y

#	Sample ID	Compound	Reported Concentration	Calculated Concentration	Qualification
	- Vanipio ib	Сомроина		]	Quantication
ļ					
				-	·

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

**Project/Site Name:** 

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

May 4, 2010

LDC Report Date:

July 6, 2010

Matrix:

Soil

Parameters:

Semivolatiles

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-3197-6

Sample Identification

SSAM5-03-4BPC

SSAM5-03-6BPC

SSAM5-03-8BPC

SSAM5-03-10BPC\*\*

SSAM5-03-4BPCMS

SSAM5-03-4BPCMSD

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

### Introduction

This data review covers 6 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination.

  This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

### III. Initial Calibration

Initial calibration was performed using required standard concentrations.

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

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

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

### IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

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

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

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

### V. Blanks

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

Sample FB-04132010-RIG2-RZE (from SDG 280-2400-2) was identified as a field blank. No semivolatile contaminants were found in this blank with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB-04132010-RIG2-RZE	4/13/10	Bis(2-ethylhexyl)phthalate Di-n-octylphthalate	1.1 ug/L 1.6 ug/L	All samples in SDG 280-3197-6

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

### VI. Surrogate Spikes

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

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

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

### XI. Target Compound Identifications

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

### XII. Project Quantitation Limit

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

Sample	Compound	Finding	Flag	A or P
SSAM5-03-6BPC SSAM5-03-8BPC	Benzo(b)fluoranthene	Due to lack of resolution between these compounds in the samples, the laboratory	J (all detects) UJ (all non-detects)	Р
SSAM5-03-10BPC**	Benzo(k)fluoranthene	performed the quantitation using the total peak area.	J (all detects) UJ (all non-detects)	

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P	
All samples in SDG 280-3197-6	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, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-3197-6

SDG	Sample	Compound	Flag	A or P	Reason (Code)	
280-3197-6	SSAM5-03-6BPC SSAM5-03-8BPC SSAM5-03-10BPC**  Benzo(b)fluoranthene  Benzo(k)fluoranthene		J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Project Quantitation Limit (q)	
280-3197-6	SSAM5-03-4BPC SSAM5-03-6BPC SSAM5-03-8BPC SSAM5-03-10BPC**	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (sp)	

Tronox LLC Facility, PCS, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-3197-6

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Semivolatiles - Field Blank Data Qualification Summary - SDG 280-3197-6

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

LDC #: 23436F2a Stage 2B/4 SDG #: 280-3197-6 Laboratory: Test America

Reviewer:\_ 2nd Reviewer:

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 5/04/10
II.	GC/MS Instrument performance check	A	
111.	Initial calibration	A	7. KSV ~
IV.	Continuing calibration/ICV	A	ca/a = 25 %
V.	Blanks	A	
VI.	Surrogate spikes	_A_	
VII.	Matrix spike/Matrix spike duplicates	<u> </u>	
VIII.	Laboratory control samples	A	us
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification		Not reviewed for Stage 2B validation.
XII.	Compound quantitation/CRQLs	SM	Not reviewed for Stage 2B validation.
XIII.	Tentatively identified compounds (TICs)	N	Not reviewed for Stage 2B validation.
XIV.	System performance	A	Not reviewed for Stage 2B validation.
XV.	Overall assessment of data	A	
XVI.	Field duplicates	N	
XVII.	Field blanks	N2	FB = FB-04132010-RIG2-RZE (250-2400-

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

	Soil		,			
1	SSAM5-03-4BPC	11	MB280-15018/1-A	21	31	
2	SSAM5-03-6BPC	12	,	22	32	
3	SSAM5-03-8BPC	13		23	 33	
4	SSAM5-03-10BPC**	14		24	34	
5	SSAM5-03-4BPCMS	15		25	35	
6	SSAM5-03-4BPCMSD	16		26	36	
7		17		27	37	
8		18		28	38	
9		19		29_	39	
10		20		30	40	

LDC #: 23 436 Fra SDG #: See Cover

### **VALIDATION FINDINGS CHECKLIST**

Page: \\_\ of \\_2 Reviewer: \\_\(\forall \tau\) 2nd Reviewer: \_\_\(\forall \)

Method: Semivolatiles (EPA SW 846 Method 8270C)

Method. Sernivolatiles (EPA SVV 846 Method 8270C)	<del>-</del>			
Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.				7
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?		İ		
IV. Continuing calibration				
Was a continuing calibration standard analyzed at least once every 12 hours for		T		The state of the s
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?		İ		
V. Blanks		1.4F 2.7		
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				attack to the many of the control of
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?		Ī		
VII. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.				
Was a MS/MSD analyzed every 20 samples of each matrix?				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII. Laboratory control samples				
Was an LCS analyzed for this SDG?				

LDC #: 93436 F 29 SDG #: See Cover

### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 2
Reviewer: 1/2
2nd Reviewer:

ı		T	7		
	Validation Area	Yes	No	NA	Findings/Comments
	Was an LCS analyzed per extraction batch?		<u> </u>		
	Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
	IX. Regional Quality Assurance and Quality Control				
	Were performance evaluation (PE) samples performed?				
	Were the performance evaluation (PE) samples within the acceptance limits?				
	X. Internal standards				Marie Carlos Car
	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?				
18	XI. Target compound identification				
Ľ	Were relative retention times (RRT's) within ± 0.06 RRT units of the standard?				
	Did compound spectra meet specified EPA "Functional Guidelines" criteria?				
١	Nere chromatogram peaks verified and accounted for?				
ı	GI. Compound quantitation/CRQLs				Art well to
(	Vere the correct internal standard (IS), quantitation ion and relative response factor RRF) used to quantitate the compound?				
V d	Vere compound quantitation and CRQLs adjusted to reflect all sample dilutions and ry weight factors applicable to level IV validation?				
X	III. Tentatively identified compounds (TICs)				The state of the s
v e	/ere the major ions (> 10 percent relative intensity) in the reference spectrum valuated in sample spectrum?				
v re	/ere relative intensities of the major ions within $\pm$ 20% between the sample and the ference spectra?				
e	d the raw data indicate that the laboratory performed a library search for all quired peaks in the chromatograms (samples and blanks)?				
(	V. System performance				species of the control of the contro
3)	stem performance was found to be acceptable.			T	
	Overall assessment of data				
)\	rerall assessment of data was found to be acceptable.				
٧	i. Field duplicates				
ie	eld duplicate pairs were identified in this SDG.			Т	
aı	rget compounds were detected in the field duplicates.	_		╁	
X	II. Field blanks			_1_	
e	d blanks were identified in this SDG.	T		T	
_	get compounds were detected in the field blanks.	_		+	
-	and the work of the state of th			L_	

## 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**	II Benzo(a)nurana##
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroanliine	UU. Phenanthrene	conco(a)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	W. Anthracene	allalidino o'z'. You alla e
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW Carboals	NNK. Uibenz(a,h)anthracene
E. 1,4-Dichlorobenzene**	T. 4-Chloroanilina		Marole Cal Dazole	LLL. Benzo(g,h,i)perylene
		II. 4-Nitrophenol*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ Purene	
H. 2,2'-Oxybis(1-chloropropane)	W 2-Mathylnanhthalom			OOO. N-Nitrosodimethylamine
		LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Puridine
K. Hexachloroethane	Z. 2.4.5-Trichlorophenol			
		oc. 4-Nitroaniine	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	TTT.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octvlphthalate**	
N. 2-Nitrophenol**	CC. Dimethylphthalate	DD 4 D		
		+-bromopnenyi-phenylether	GGG. Benzo(b)fluoranthene	WV.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	WWW.

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

LDC#: 23 436 +22 SDG #: 32 Con-

## VALIDATION FINDINGS WORKSHEET

Field Blanks

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

> METHOD: GC/MS BNA (EPA SW 846 Method 8270C) Y N N/A

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

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

Blank units: 4/A / Associated sample units: 5/A / Associated sample units:

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

7

Associated Samples:

Sample Identification 2 ধ 2. the FB-04182010-RIGZ-RZE Blank ID <u>:</u> EFE Compound

Associated sample units: Blank units:

Sampling date:\_

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

Sample Identification Associated Samples: Blank ID Compound CROL

5x Phthalates 2x All others

1DC# 72 436 F29 SDG# 12 (20)

# VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

Page: of
Reviewer:

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

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

A'N W

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

Qualifications	J/45/4 (9)									
Associated Samples	un resolved peaks	-								
Finding	666, HHH unresolu									
Sample ID	23 4									
# Date										

Comments: See sample calculation verification worksheet for recalculations

LDC#: 23436 Fra SDG#: 10 Cno

# VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: for Reviewer:

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

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

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

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

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

					Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
		Calibration			RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
#	Standard ID	Date	Compound (Internal Standard)	dard)	( 50 std)	( 50 std)	(Initial)	(Initial)		
-	ICAL	5/17/2010	5/17/2010 1,4-Dioxane	(IS1)	0.5858	0.5858	0.6981	0.6981	13.4	13.36
	MSSY		Naphthalene	(182)	1.0095	1.0095	1.0394	1.0395	2.7	2.75
			Fluorene	(183)	1.3686	1.3686	1.4029	1.4029	2.2	2.20
			benzene	(IS4)	0.1973	0.1973	0.2038	0.2038	3.4	3.40
			Chrysene	(185)	1.0983	1.0983	1.1019	1.1019	5.2	5.22
			Benzo(a)pyrene	(186)	1.1920	1.1920	1.1700	1.1700	6.5	09'9

Benz										
Chrysene	1.2054	1.1123	1.0706	1.0983	1.1544	1.0751	1.0837	1.0151	1.1019	0.0575
Hexachlorob		0.1982	0.1951	0.1973	0.2103	0.2101	0.2043	0.2113	0.2038	0.0069
Fluorene	1.4060	1.4052	1.3506	1.3686	1.4468	1.4126	1.4032	1.4302	1.4029	0.0309
Naphthalene	1.0967	1.0414	1.0278	1.0095	1.0612	1.0389	1.0110	1.0291	1.0395	0.0285
1,4-Dioxane		0.8159	0.6727	0.5858	0.7642	0.7371	0.7438	0.5675	0.6981	0.0932
Conc	4.00	10.00	20.00	50.00	80.00	120.00	160.00	200.00	= ×	S

979599

1236178

40/20

465924

797054 202044

40/50 40/50 40/50 40/50

253936

185943

40/20

Area IS

Area cpd

nc IS/Cpd

819260 845794

> 1161122 1089572

731247

20(a)py 1.0396 1.0762 1.1459 1.1920 1.2406

1.2121

1.1700

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.

SDG # See Cover

### Continuing Calibration Results Verification VALIDATION FINDINGS WORSHEET

Page of 2nd Reviewer: Reviewer:\_

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

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

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

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

ave. RRF = initial calibration average RRF

RRF = continuing calibration RRF Ax = Area of compound

Ais = Area of associated internal standard

Cis = Concentration of internal standard Cx = Concentration of compound

		Calibration			Average RRF	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Date	Compound (Reference IS)	Reference IS)	(Initial RRF)	(CC RRF)	(CC RRF)	Ф%	%D
-	Y2435	05/17/10	1,4-Dioxane	(IS1)	0.6981	0.7277	0.7277	4.2	4.2
			Naphthalene	(182)	1.0394	1.0508	1.0508	1.1	1.1
			Fluorene	(ESI)	1.4029	1.4728	1.4728	5.0	5.0
			Hexachlorobenzene	Izene (IS4)	0.2038	0.2109	0.2109	3.5	3.5
			Chrysene	(185)	1.1019	1.1388	1.1388	3.4	3.4
			Benzo(a)pyrene	e (IS6)	1.1700	1.2924	1.2924	10.5	10.5

Compound (Reference 15)		Concentration	Area Cpd	Area IS
		(IS/Cpd)		
1,4-Dioxane	(IS1)	40/80	297217	204223
Naphthalene	(182)	40/80	1685657	802046
Fluorene	(183)	40/80	1374796	466718
Hexachlorobenzene	(184)	40/80	348688	826697
Chrysene	(185)	40/80	1983509	870843
Benzo(a)pyrene	(186)	40/80	1860746	719859

LDC#: 23436 F29 SDG#: Sre Cover

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

Page:	lof_1_
Reviewer:_	JN
2nd reviewer:	W

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found

Sample ID: #

SS = Surrogate Spiked

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	(00)	75.1	75	75	0,
2-Fluorobiphenyl		76.2	76	76	
Terphenyl-d14		86.7	87	87	
Phenol-d5	3	SW 7/	1 9/		, , , , , , , , , , , , , , , , , , ,
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# 73436 F 29 SDG #: See Cover

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

Page: lof 1 Reviewer:\_

2nd Reviewer:\_

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

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

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

SSC = Spiked sample concentration SA = Spike added Where:

SC = Sample concentation

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

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples:

	ds :	ike	Sample	Spiked S	ample	Matrix Spike	Spike	Matrix Spik	Matrix Spike Duplicate	USW/SW	ű
Compound	Adi ( ug ,	Added (4g/koと	Concentration (W5 / kd	Concentration $(45/k)$	rration (k.)	Percent Recovery	ecovery	Percent Recovery	ecovery	RPD	
	MS	MSD	0	MS	O MSD	Reported	Recalc	Reported	Recalc	Reported	Donalculator
Phenol									-		
N-Nitroso-di-n-propylamine											
4-Chloro-3-methylphenol											
Acenaphthene	3160	2950	24	2440	2400	76	76	81	8)		2,1
Pentachlorophenol											
Pyrene	2160	29,50	79	2430	25.40	8)	8	€ 8	48	4	3.5
		_									

Comments: Refer to Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results. 100

LDC#: 23 436 #29 SDG #: See Cover

# VALIDATION FINDINGS WORKSHEET

<u>Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification</u>

Page: of

Reviewer:\_ 2nd Reviewer:

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

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

% Recovery = 100 \* (SC/SA

Where: SSC = Spike concentration SA = Spike added

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

Š LCS/LCSD samples:

V-5/ 8/251 -082

	S	oike	is	oike		y.		000		
Compound	<u>**</u>	Added (Mg/kg)	Conce (MC	Concentration $(\mathcal{U}_{\mathcal{C}}/c,\ )$	Percent	Percent Recovery	Percent	Percent Recovery		
	SOI	O I CSD	SOT	, I CSD	Reported	Pocalc			- 11	
Phenol							oel roden	Kecalc	Keported	Kecalculated
N-Nitroso-di-n-propylamine										
4-Chloro-3-methylphenol										
Acenaphthene	2610	<b>₹</b> 7	2/80	4V	84	73				
Pentachlorophenol		•								
Pyrene	2610	_	2260		87	87				

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results. LDC #: 73 436 7 29 SDG #: Sre Cover

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	<u>l</u> of_1
Reviewer:	JY
nd reviewer:	- W

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

/	$\widehat{Y}$	Ν	N/A
:	X	Ŋ	<b>N/A</b>

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 <sub>x</sub> )(I <sub>x</sub> )(V <sub>x</sub> )(DF)(2.0)
	,s)(RRF)(V,)(Vi)(%S)

Area of the characteristic ion (EICP) for the compound

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

Amount of internal standard added in nanograms (ng)

Volume or weight of sample extract in milliliters (ml) or ٧, grams (g).

V, Volume of extract injected in microliters (ul)

Volume of the concentrated extract in microliters (ul)

Df

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

Sample I.D. # 4 , \_ SS

Conc. =  $\frac{(8/8^{23})(40)(1m/)(100)}{(0,2038)(1/82069)(32.83)(0.843)(0}$ 

= 491, 2 2 496 ug/fy

Factor of 2 to account for GPC cleanup 2.0

		anti or GPC deanup	Reported Concentration	Calculated Concentration	
#	Sample ID	Compound	( )	( )	Qualification
-					
				<u>.</u>	
-					

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

**Project/Site Name:** 

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

May 14, 2010

LDC Report Date:

July 6, 2010

Matrix:

Soil

Parameters:

Semivolatiles

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-3584-1

Sample Identification

SSAK5-04-1BPC

SSAK5-04-2BPC

SSAK5-03-1BPC

SSAK4-02-1BPC

SSAK4-02-1BPCMS

SSAK4-02-1BPCMSD

### Introduction

This data review covers 6 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination.

  This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

### III. Initial Calibration

Initial calibration was performed using required standard concentrations.

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

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

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

### IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

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

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

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

### V. Blanks

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

Sample FB-04072010-RZD (from SDG 280-2216-2) was identified as a field blank. No semivolatile contaminants were found in this blank with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB-04072010-RZD	4/7/10	Bis(2-ethylhexyl)phthalate	2.2 ug/L	All samples in SDG 280-3584-1

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

### VI. Surrogate Spikes

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

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. 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 280-3584-1	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, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-3584-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-3584-1	SSAK5-04-1BPC SSAK5-04-2BPC SSAK5-03-1BPC SSAK4-02-1BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

Tronox LLC Facility, PCS, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-3584-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Semivolatiles - Field Blank Data Qualification Summary - SDG 280-3584-1

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

LDC #: 23436G2a VALIDATION C
SDG #: 280-3584-1
Laboratory: Test America

Stage 2B

Date: 7/61/1
Page: 1 of 1
Reviewer: 2nd Reviewer:

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

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

	Validation Area		Comments
l.	Technical holding times	A	Sampling dates: 5/14 /16
11.	GC/MS Instrument performance check	A	
111.	Initial calibration	A	2 KSD r → COV/10V € 25 €
IV.	Continuing calibration/ICV	A	ca/100 = 252
V.	Blanks	A	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	A	LCS
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	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	SM)	FB = FB-04072010-RZD (from 280-2216-2)

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

TB = Trip blank

EB = Equipment blank

Validated Samples:

Soil

	36.1				
1	SSAK5-04-1BPC	ħ	MB - 280- 16507/1-A	21	31
2	SSAK5-04-2BPC	12	/	22	32
<b>+</b> 3	SSAK5-03-1BPC	13		23	33
4	SSAK4-02-1BPC	14		24	34
5	SSAK4-02-1BPCMS	15		25	35
6	SSAK4-02-1BPCMSD	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. Pentachiorophenoi**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	W. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP, Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	OO. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	111.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	nnn
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	WW.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	WWW.

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

,	
X	>
5	1
و	3
2	٦
7	لا
7 00	
31	·.'
$\ddot{\circ}$	ტ
9	SDG

## VALIDATION FINDINGS WORKSHEET Field Blanks

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

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

Were field blanks identified in this SDG?

Were target compounds detected in the field blanks?

Up /L Associated sample units:  $U_2$  /L  $U_3$  /L  $U_2$  /L  $U_3$  /L Y N N/A

Blank units:

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

ر بي	(VD /						
1/4 8/	1114	uc					
	amples:	Sample Identification					
	Associated Samples:	Sı					
`	ther:						
	Rinsate / O		0-RZD				
اچ	*Field Blank	Blank ID	FB-04072010-RZD	2.2			
oling date: 4 /07/	Field blank type: (circle one) Field Blank) Rinsate / Other	Compound		243			
Samp	-ield						CROL

Associated sample units: Blank units:

Sampling date:

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

Associated Samples:

Compound	Blank ID	Sample Identification	
CRQL			

5x Phthalates 2x All others

23436 925 

VALIDATION FINDINGS WORKSHEET

Page: 1 of

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

Surrogate Recovery

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

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

7 3 Qualifications STACK 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? 12/-120 %R (Limits) Surrogate T 8P Sample ID Date Y N N/A #

QC Limits (Soil) 25-121 19-122 20-130\* S5 (2FP)= 2-Fluorophenol S6 (TBP) = 2,4,6-Tribromophenol S7 (2CP) = 2-Chlorophenol-d4 S8 (DCB) = 1,2-Dichlorobenzene-d4 QC Limits (Water) 35-114 43-116 33-141 10-94 QC Limits (Soil) 23-120 30-115 18-137 24-113 S1 (NBZ) = Nitrobenzene-d5 S2 (FBP) = 2-Fluorobipheny S3 (TPH) = Terphenyl-d14 S4 (PHL) = Phenol-d5 \* QC limits are advisory

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

SUR.2S.wpd

LDC#: 23 436 62 SDG#:

## VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

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

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

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

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 X-KI N/A

MS/MSD. Soil / Water.

		ے	<u> </u>											_	-			
	Qualifications	No great (MED in																
C limits?	Associated Samples	4																
ses (RPD) within the (	RPD (Limits)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
rix? tive percent differenc	MSD %R (Limits)	( )	( )	( )	( )	( )	,	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
samples of each maties (%R) and the rela	MS %R (Limits)	53 (54-120)	( )	(	( )	( )	,		( )	) (	( )	( )	( )	( )	( )	( )	( )	( )
zed every 20 rcent recover	Compound	111																
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?	QI QSW/SW	9/5																
N/A N/A	Date																	
	#			<u></u>											<u> </u>			
_																		

	Compound	QC Limits (Soil)	RPD (Soil)	QC Limits (Water)	RPD (Water)		Compound	QC Limits (Soil)	RPD (Soil)	QC Limits (Water)	RPD (Water)
ď	Phenol	26-90%	< 35%	12-110%	< 42%	GG	GG Acenaphthene	31-137%	< 19%	46-118%	< 31%
ι	C. 2-Chlorophenol	25-102%	× 20%	27-123%	< 40%	Ξ	4-Nitrophenol	11-114%	< 50%	10-80%	< 50%
ш	1,4-Dichlorobenzene	28-104%	< 27%	36-97%	< 28%	KK.	2,4-Dinitrotoluene	28-89%	< 47%	24-96%	< 38%
-;	N-Nitroso-di-n-propylamine	41-126%	< 38%	41-116%	< 38%	TT.	Pentachlorophenol	17-109%	< 47%	9-103%	< 50%
œ		38-107%	< 23%	39-98%	< 28%	22.	Pyrene	35-142%	< 36%	26-127%	< 31%
>	4-Chloro-3-methylphenol	26-103%	< 33%	23-97%	< 42%						

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

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

May 18, 2010

LDC Report Date:

July 6, 2010

Matrix:

Water

Parameters:

Semivolatiles

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-3679-3

Sample Identification

EB-05182010-RZC

### Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

### III. Initial Calibration

Initial calibration was performed using required standard concentrations.

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

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

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

### IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

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

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

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

### V. Blanks

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

Sample EB-05182010-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

The laboratory has indicated that there was insufficient sample volume for analysis of the matrix spike and matrix spike duplicate.

### VIII. Laboratory Control Samples (LCS)

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

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Flag	A or P
LCS/D 280-16560/2-A/3-A (All samples in SDG 280-3679-3)	Pyridine	0 (24-120)	3 (24-120)	-	J- (all detects) R (all non-detects)	Р

### 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 280-3679-3	All compounds reported below the PQL.	J (all detects)	Α

Raw data were not reviewed for this SDG.

### XIII. Tentatively Identified Compounds (TICs)

Raw data were not reviewed for this SDG.

### XIV. System Performance

Raw data were not reviewed for this SDG.

### XV. Overall Assessment

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

### XVI. Field Duplicates

No field duplicates were identified in this SDG.

### Tronox LLC Facility, PCS, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-3679-3

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-3679-3	EB-05182010-RZC	Pyridine	J- (all detects) R (all non-detects)	Р	Laboratory control samples (%R) (I)
280-3679-3	EB-05182010-RZC	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (sp)

Tronox LLC Facility, PCS, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-3679-3

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Semivolatiles - Equipment Blank Data Qualification Summary - SDG 280-3679-3

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson** VALIDATION COMPLETENESS WORKSHEET Stage 2B

LDC #:\_\_\_ 280-3679-3 SDG #: Laboratory: Test America

Reviewer: 2nd Reviewer:

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 51610
11.	GC/MS Instrument performance check	A	
Ш.	Initial calibration	<b>A</b>	2 RD 17
IV.	Continuing calibration/ICV	A	COM = 252
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	N	ctient spec Insufficient sample
VIII.	Laboratory control samples	<u>sw</u>	us b
IX.	Regional Quality Assurance and Quality Control	N	
Χ	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	1/0	EB = 1

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

TB = Trip blank

EB = Equipment blank

Validated Samples:

	Water				
1	EB-05182010-RZC	11	21	31	
2	MB 280-16560/1-A	12	22	32	
3		13	23	33	
4		14	24	34	
5		15	25	35	
6		16	26	36	
7		17	27	37	
8		18	28	38	
9		19	29	39	
10		20	30	40	

# VALIDATION FINDINGS WORKSHEET

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

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

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

LDC#: 23436 Jea SDG #: Se Co.

## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Reviewer: 2nd Reviewer: Page:

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

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

Was a LCS required?

Y N N/A

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

Qualifications	J-/R/P (1)																			:					
Associated Samples	411																			- - - - -					
RPD (Limits)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )		( )	( )	( )		( )	( )	( )	( )	( )	( )	
LCSD %R (Limits)	3 (24-120)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
LCS %R (Limits)	0 (24-120)	( )	( )	(	( )	( )	( )	( )	( )	( )	( )	( )	)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
Compound	5-A RRR																								
TCS/TCSD ID	LCS/B 280-16560/2-A/8-A																								
# Date																									

### Tronox LLC Facility, PCS, Henderson, Nevada Data Validation Reports LDC #23436

Metals



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

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

April 13, 2010

LDC Report Date:

July 6, 2010

Matrix:

Soil

Parameters:

Arsenic

Validation Level:

Stage 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-2400-10

Sample Identification

**SA128-8BPC** 

### Introduction

This data review covers one soil sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Arsenic.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004) as there are no current guidelines for the method stated above.

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

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. ICPMS Tune

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

### III. Calibration

An initial calibration was performed.

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

### IV. Blanks

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

Sample FB-04132010-RIG2-RZE (from SDG 280-2400-2) was identified as a field blank. No arsenic was found in this blank.

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

The frequency of analysis was met.

The criteria for analysis were met.

### VI. Matrix Spike Analysis

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

### VII. Duplicate Sample Analysis

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Internal Standards

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

### X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

### XI. ICP Serial Dilution

ICP serial dilution was not performed for this SDG.

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

All sample result verifications were acceptable.

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-2400-10	All analytes reported below the PQL.	J (all detects)	А

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

No field duplicates were identified in this SDG.

### Tronox LLC Facility, PCS, Henderson, Nevada Arsenic - Data Qualification Summary - SDG 280-2400-10

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-2400-10	SA128-8BPC	All analytes reported below the PQL.	J (all detects)	Α	Sample result verification (PQL) (sp)

Tronox LLC Facility, PCS, Henderson, Nevada Arsenic - Laboratory Blank Data Qualification Summary - SDG 280-2400-10

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Arsenic - Field Blank Data Qualification Summary - SDG 280-2400-10

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson** SHEET

LDC #:	23436B4	_ VALIDATION COMPLETENESS WORKS
SDG #:	280-2400-10	Stage 4
Laborator	y: Test America	<u> </u>

Date: 6-08-16	۷
Page: \_\of	
Reviewer:_CC_	
2nd Reviewer:	_

METHOD: As (EPA SW 846 Method 6020)

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

	Validation Area		Comments
1.	Technical holding times	5	Sampling dates: 4/3/10
11.	ICP/MS Tune	A	
Ш.	Calibration	12	
IV.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	À	
VI.	Matrix Spike Analysis	W	Clientspecisied
VII.	Duplicate Sample Analysis	N	上 ・
VIII.	Laboratory Control Samples (LCS)	A	LCS
IX.	Internal Standard (ICP-MS)	A	
Χ.	Furnace Atomic Absorption QC	N	Moturitzed
XI.	ICP Serial Dilution	N	Not preformed
XII.	Sample Result Verification	A	<u> </u>
XIII.	Overall Assessment of Data	Α	
XIV.	Field Duplicates	$\sim$	
XV	Field Blanks	ND	FB= FB-01/37010-RIG2-RZE (506x 280-2400-Z)

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: 50 ( )

·					T-74	
1	SA128-8BPC	11	PB5	21	31	
2		12		22	32	
3	_	13		23	33	
4		14		24	34	
5		15		25	35	
6		16		26	36	
7		17		27	37	
8		18		28	38	
9		19		29	39	
10		20		30	40	

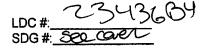
Notes:		
	***************************************	

LDC#: 23436BY SDG#: Secover

### **VALIDATION FINDINGS CHECKLIST**

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

iwethod:Metals (EPA SW 846 Method 6010B/7000/6020)	<del></del>	_		
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?		<u>^</u>		
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				
Was an LCS anaylzed for this SDG?				
Was an LCS analyzed per extraction batch?	4			
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: \_\_of \_\_ Reviewer: \_ 
2nd Reviewer: \_

Validation Area	Yes	No	NA	Findings/Comments
VIII. Furnace Atomic Absorption QC	,			
If MSA was performed, was the correlation coefficients > 0.995?				
Do all applicable analysies have duplicate injections? (Level IV only)				/
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)	:			
Were analytical spike recoveries within the 85-115% QC limits?				
IX. ICP Serial Dilution			1	
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?		_		
Were the performance evaluation (PE) samples within the acceptance limits?			$\perp$	
XII. Sample Result Verification	· · · · · · · · · · · · · · · · · · ·	·		
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?		<u> </u>		
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/		<u>L</u>	
XIV. Field duplicates	<del></del>	·		
Field duplicate pairs were identified in this SDG.				
Target analytes were detected in the field duplicates.		<u> </u>	<u> </u>	
XV. Field blanks	., y	,, <del>,</del>	<del></del>	
Field blanks were identified in this SDG.	/		1	
Target analytes were detected in the field blanks.		1		

LDC#: 2343689 SDG#: SECCOVER

# VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

Page: of Seviewer: G2

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

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

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

Where, Found = concentration (in ug/L) of each analyte 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)						
	GFAA (Initial calibration)						
	CVAA (initial calibration)						
	ICP (Continuing calibration)						
	GFAA (Continuing calibration)					-	
	CVAA (Continuing calibration)						
FCV	ICP/MS (Initial calibration)	AS	9. oh	0,01	(0)	101	<b>)</b> -
CCV(Ora)	ICP/MS (Continuing calibation)	-)	1,02	56.0	[0]	101	

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.

100 #: 2343687 806 #: 588 & COLOR

### VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

Page: of Reviewer: CK

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

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

%R = Found x 100 Where, Found = Con True

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

True = Concentration of each analyte in the source.

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

RPD = <u>IS-DL</u> × 100 (S+D)/2

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

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

%D = ||-SDR| × 100

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

					Recalculated	Reported	
S Ol elumes	Type of Analysis	Element	Found/S/1 (white) (my/kg)	True / D / SDR (units)	%R/RPD/%D	%R/RPD/%D	Acceptable (Y/N)
ICSAR	ICP interference check	\$	104 mg/L	1000ell	h01	601	<b>)</b>
537	Laboratory control sample	-	1.8	202	101	10/	
	Matrix spike		(SSR-SR)		•		,
	Duplicate						
	ICP serial dilution	·					

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

LDC #: 13/3601 SDG #: <u>Secore</u>

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	of
Reviewer:	Ce
2nd reviewer:	

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

Please Y N I Y N I Y N I	N/A	. was reading need tehotted	ated range of the instruments and within the linear range of the ICP?
Detecte followin	ed analyl g equati	te results for	were recalculated and verified using the
Concentr	ration =	(RD)(FV)(Dil) (In. Vol.)(%S)	Recalculation:
RD FV		Raw data concentration Final volume (ml)	100m L(5) (12,68 mg/kg) = 6,8 mg/kg
In. Vol. Dil	=	Initial volume (mi) or weight (G) Dilution factor	(1.049)(0.898)
%S	=	Decimal percent solids	

Sample ID	Analyte	Reported Concentration	Calculated Concentration ( mg/kg )	Acceptable (Y/N)
	<u>As</u>	6,8	68	4
	. •			

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

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

April 15, 2010

LDC Report Date:

July 6, 2010

Matrix:

Soil

Parameters:

Arsenic

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-2500-9

Sample Identification

SA165-2BPC\*\* SA131-7BPC

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

### Introduction

This data review covers 2 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Arsenic.

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

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

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. ICPMS Tune

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

### III. Calibration

An initial calibration was performed.

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

### IV. Blanks

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

Sample EB-04152010-RIG2-RZE (from SDG 280-2500-2) was identified as an equipment blank. No arsenic was found in this blank.

Sample FB-04132010-RIG2-RZE (from SDG 280-2400-2) was identified as a field blank. No arsenic was found in this blank.

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

The frequency of analysis was met.

The criteria for analysis were met.

### VI. Matrix Spike Analysis

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

### VII. Duplicate Sample Analysis

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Internal Standards

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

### X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

### XI. ICP Serial Dilution

ICP serial dilution 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 280-2500-9	All analytes reported below the PQL.	J (all detects)	А

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

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

No field duplicates were identified in this SDG.

### Tronox LLC Facility, PCS, Henderson, Nevada Arsenic - Data Qualification Summary - SDG 280-2500-9

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-2500-9	SA165-2BPC** SA131-7BPC	All analytes reported below the PQL.	J (all detects)	А	Sample result verification (PQL) (sp)

Tronox LLC Facility, PCS, Henderson, Nevada Arsenic - Laboratory Blank Data Qualification Summary - SDG 280-2500-9

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Arsenic - Equipment Blank Data Qualification Summary - SDG 280-2500-9

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Arsenic - Field Blank Data Qualification Summary - SDG 280-2500-9

No Sample Data Qualified in this SDG

LDC #:

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

	•	_	٠,	_	_	_	-	-	_	•	 •	_	•	•	•
е	!	2	3	/4											

Date G-76-10
Page: Lof)
Reviewer: 02
2nd Reviewer:

SDG #: 280-2500-9 Laboratory: Test America

METHOD: As (EPA SW 846 Method 6020)

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

	Validation Area		Comments
l.	Technical holding times	A	Sampling dates: 4/15/10
Π.	ICP/MS Tune	A	
111.	Calibration	A	
IV.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	A	
VI.	Matrix Spike Analysis	$\mathcal{N}$	Clientspecified
VII.	Duplicate Sample Analysis	N	L
VIII.	Laboratory Control Samples (LCS)	A	LCS
IX.	Internal Standard (ICP-MS)	A	
X.	Furnace Atomic Absorption QC	N	Notutilized Not preformed
XI.	ICP Serial Dilution	$\mathcal{N}$	No+ preformed
XII.	Sample Result Verification	A	Not reviewed for Stage 2B validation.
XIII.	Overall Assessment of Data	P	
XIV.	Field Duplicates	$\mathcal{N}$	
XV	Field Blanks	NO	FB= FB-01132010-RIGZ-RZE (506&280-2 EB= EB-04152010-RIGZ-RZEC506% 280-2 detected D= Duplicate

N = Not provided/applicable SW = See worksheet

R = Rinsate FB = Field blank

D = Duplicate TB = Trip blank

EB = Equipment blank

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

			 	·····		
1	SA165-2BPC**	11	21		31	
2	SA131-7BPC	12	22		32	
3		13	23		33	
4		14	24		34	
5		15	25		35	
6		16	26		36	
7		17	27		37	
88		18	28		38	
9		19	29		39	
10		20	30		40	

Notes:				
	<del>.</del>	 	 	

23436 LDC#: <del>24236</del>C4 SDG#: <u>Sec Caler</u>

### **VALIDATION FINDINGS CHECKLIST**

	Page:	ι	_of	2
	Reviewer:		R	
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.			<b>-</b>	
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		<u></u>		
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?			·	

23435 LDC#: 24236C4 SDG#: Sec Caret

### **VALIDATION FINDINGS CHECKLIST**

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

Validation Area	Yes	No	NA	Findings/Comments			
VIII. Furnace Atomic Absorption QC							
If MSA was performed, was the correlation coefficients > 0.995?							
Do all applicable analysies have duplicate injections? (Level IV only)			_				
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)							
Were analytical spike recoveries within the 85-115% QC limits?			/				
IX. ICP Serial Dilution							
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?	<u></u>						
If the %Rs were outside the criteria, was a reanalysis performed?		<u> </u>					
XI. Regional Quality Assurance and Quality Control							
Were performance evaluation (PE) samples performed?		_					
Were the performance evaluation (PE) samples within the acceptance limits?			_				
XII. Sample Result Verification	····		·····				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?							
XIII. Overall assessment of data							
Overall assessment of data was found to be acceptable.							
XIV. Field duplicates							
Field duplicate pairs were identified in this SDG.		_					
Target analytes were detected in the field duplicates.							
XV. Field blanks							
Field blanks were identified in this SDG.	/						
Target analytes were detected in the field blanks.							

1DC# 23436C4 SDG#: SECOVER

# VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

Page: of Reviewer: GZ

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

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

%R = Found x 100 True

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

Acceptable (Y/N) Reported % X 0 Recalculated %R 0 50.0 True (ug/L) 5°97 Found (ug/L) 5 0 0, 7.8 Element AS ICP/MS (Continuing calibation) GFAA (Continuing calibration) CVAA (Continuing calibration) ICP (Continuing calibration) ICP/MS (Initial calibration) Type of Analysis GFAA (Initial calibration) CVAA (Initial calibration) ICP (Initial calibration) 20/61:02) Standard ID FC F

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.

73238CH SDG #SERCELON LDC #:

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

2nd Reviewer: Page: Reviewer:

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

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

%R = Found × 100 True

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

True = Concentration of each analyte in the source.

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

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

RPD = <u>IS-DI</u> x 100 (S+D)/2

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

%D = II-SDR x 100

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

-			(				
					Recalculated	Raported	
Sample ID	Type of Analysis	Element	Found / S / I (units)	True / D / SDR (units)	%R / RPD / %D	%R/RPD/%D	Acceptable (Y/N)
HS B	(CP interference check	B	10426	100 mg/L	ાઉવ	501	)_+
537	Laboratory control sample	7		Sal 800 02	101	(O)	Z
>	Matrix spike		(SSR-SR)	\			
>	Duplicate				,		
>	ICP serial dilution						

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

LDC #: 23(36C9 SDG #: Secore)

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page;_	C of
Reviewer:	æ
2nd reviewer:_	

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

Please Y N I Y N I Y N I	V/A		ge of the instruments and within the linear range of the ICP?
Detecte followin	ed analyl g equati	e results for	were recalculated and verified using the
Concentr	ation =	(RD)(FV)(Dil) (In. Vol.)(%S)	Recalculation:
RD FV	=	Raw data concentration Final volume (ml)	(100mL)(5)(12.10me/L) = 6.4mg/kg
In. Vol. Dil %S	=	Initial volume (ml) or weight (G) Dilution factor Decimal percent solids	(0.904) (1.05g)

Sample ID	Analyte	Reported Concentration ( MR (C )	Calculated Concentration	Acceptable (Y/N)
	As	6,4	64	Y
	·		·	
	· · · · · · · · · · · · · · · · · · ·			
	·			
				· · · · · · · · · · · · · · · · · · ·
			:	

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

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

April 16, 2010

LDC Report Date:

July 6, 2010

Matrix:

Water

Parameters:

Metals

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-2541-2

Sample Identification

EB-04152010-2-RZD

### Introduction

This data review covers one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Metals. The metals analyzed were Arsenic, Cobalt, Lead, Magnesium, and Manganese.

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

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

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. ICPMS Tune

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

### III. Calibration

An initial calibration was performed.

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

### IV. Blanks

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

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
ICB/CCB	Cobalt	0.0237 ug/L	All samples in SDG 280-2541-2

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-04152010-2-RZD	Cobalt	0.13 ug/L	1.0U ug/L

Sample EB-04152010-2-RZD was identified as an equipment blank. No metal contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Analyte	Concentration	Associated Samples
EB-04152010-2-RZD	4/16/10	Lead Cobalt Manganese Magnesium	0.18 ug/L 0.13 ug/L 17 ug/L 66 ug/L	No associated samples in this SDG

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

The frequency of analysis was met.

The criteria for analysis were met.

### VI. Matrix Spike Analysis

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

### VII. Duplicate Sample Analysis

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

### VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Percent recoveries (%R) 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 was not performed for this SDG.

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

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-2541-2	All analytes reported below the PQL.	J (all detects)	Α

Raw data were not reviewed for this SDG.

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

No field duplicates were identified in this SDG.

### Tronox LLC Facility, PCS, Henderson, Nevada Metals - Data Qualification Summary - SDG 280-2541-2

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-2541-2	EB-04152010-2-RZD	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (PQL) (sp)

### Tronox LLC Facility, PCS, Henderson, Nevada Metals - Laboratory Blank Data Qualification Summary - SDG 280-2541-2

SDG	Sample	Analyte	Modified Final Concentration	A or P	Code
280-2541-2	EB-04152010-2-RZD	Cobalt	1.0U ug/L	А	bl

Tronox LLC Facility, PCS, Henderson, Nevada Metals - Equipment Blank Data Qualification Summary - SDG 280-2541-2

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET Stage 2B

LDC #: 23436D4	VALIDATION COMPLETENESS V
SDG #: 280-2541-2	Stage 2B
Laboratory: Test America	<del>-</del>

Date: 6-26-1
Page:of
Reviewer: 02
2nd Reviewer:

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
1.	Technical holding times	A	Sampling dates: 4/16/10
11.	ICP/MS Tune	A	
111.	Calibration	A	
IV.	Blanks	SW	
V.	ICP Interference Check Sample (ICS) Analysis	A	
VI.	Matrix Spike Analysis	$\mathcal{N}$	Client specified
VII.	Duplicate Sample Analysis	N	
VIII.	Laboratory Control Samples (LCS)	A	LCS/D
IX.	Internal Standard (ICP-MS)	A	
Χ.	Furnace Atomic Absorption QC	$\wedge$	Not Desormed
XI.	ICP Serial Dilution	N	Not mesormed
XII.	Sample Result Verification	N	d
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	N	
XV	Field Blanks	SW	EB=1 Cno associated Samples

N.	ote:	
17	ULC.	

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:

water

1	- <b>(20</b> EB-04152010-2 <del>RZD</del>	11	PBU	21	31	
2		12		22	32	
3		13		23	33	
4		14		24	34	
5		15		25	35	
6		16		26	36	
7		17		27	37	
8		18		28	38	
9		19		29	39	
10		20		30	40	

Notes:

LDC #: 2343604 SDG #: 580 CONO?

### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page: of Reviewer: 2nd reviewer:

All circled elements are applicable to each sample.

Sample ID	Matrix	Target Analyte List (TAL)
1		Al, Sb(As) Ba, Be, Cd, Ca, Cr, Co, Cu, Fe(Pb, Mg, Mn) Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <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, Ti, V, Zn, Mo, B, Si, CN,
		Al. Sb. As. Ba. Be, Cd. Ca. Cr. Co. Cu. Fe, Pb. Mg. Mn. Hg. Ni. K. Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al. Sb. As. Ba. Be. Cd. Ca. Cr. Co. Cu. Fe. Pb. Mg. Mn. Hg. Ni. K. Se. Ag. Na. Ti, V. Zn. Mo. B. Si. CN.
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN',
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN',
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN',
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN',
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Analysis Method
ICP		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
ICP Trace		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
ICP-MS		Al, Sb(As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Cb, Mg, Mn) Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN',
GFAA		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,

Comments:	Mercury by C	VAA IT performed		 
			·	

LDC #: 23-(340)-/ SDG #: <u>S-PC CO</u> - ON **METHOD:** Trace Metals (EPA SW 846 Method 6010/7000)

Sample Concentration units, unless otherwise noted:

VALIDATION FINDINGS WORKSHEET PB/ICB/CCB QUALIFIED SAMPLES
Soil preparation factor applied: NAT
Associatec Samples: +

(Reason: b)

Reviewer: | Samples with analyte concentrations within five times the associated ICB, CCB or PB concentration are listed above with the dentifications 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. ਲ ପ୍ର ₹ ¥ g As 쩞 8 င် 8 ď ₽ Ξ £ æ Ą ₽ z ᅩ တ္တ ₹ F > Ñ **6** Sample Identification 0.1/51.0 Blank Action Limit Maximum ICB/CCB\* 0,0237 (ng/L) Maximum PB\* (ng/L) Analyte Maximum PB\* (mg/Kg) Ξ̈ Sp Ва Be පි ပ္ပ ပိ 5 Š Ö Ę. <u>а</u> £ Se β Š Zu Z

SDG #: See Cover LDC #: 23436D4

# VALIDATION FINDINGS WORKSHEET Field Blanks

Reviewer:\_\_ 2nd Reviewer:

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

Were field blanks identified in this SDG? YN 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: 4/16/10 Soil factor applied

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

No Associated Samples

Associated Samples:

Reason Code: be

Sample Identification Action Level 17 99 Blank ID 0.18 0.13 99 17 Analyte ပိ 퇻 βg 요

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".

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

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

May 17, 2010

LDC Report Date:

July 6, 2010

Matrix:

Soil

Parameters:

Arsenic

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-3624-1

Sample Identification

SSAM6-03-1BPC SSAM6-03-5BPC SSAM6-03-5BPCMS SSAM6-03-5BPCMSD

### Introduction

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

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

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

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. ICPMS Tune

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

### III. Calibration

An initial calibration was performed.

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

### IV. Blanks

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

Sample FB-04072010-RZC (from SDG 280-2280-2) was identified as a field blank. No arsenic was found in this blank.

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

The frequency of analysis was met.

The criteria for analysis were met.

### VI. Matrix Spike Analysis

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

### VII. Duplicate Sample Analysis

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

### VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Percent recoveries (%R) 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 analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-3624-1	All analytes reported below the PQL.	J (all detects)	Α

Raw data were not reviewed for this SDG.

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

No field duplicates were identified in this SDG.

### Tronox LLC Facility, PCS, Henderson, Nevada Arsenic - Data Qualification Summary - SDG 280-3624-1

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-3624-1	SSAM6-03-1BPC SSAM6-03-5BPC	All analytes reported below the PQL.	J (all detects)	А	Sample result verification (PQL) (sp)

Tronox LLC Facility, PCS, Henderson, Nevada Arsenic - Laboratory Blank Data Qualification Summary - SDG 280-3624-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Arsenic - Field Blank Data Qualification Summary - SDG 280-3624-1

No Sample Data Qualified in this SDG

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

Stage 2B

	Date:	6-29	<del>1</del> -10
	Page:_	<u>l</u> of	1
	Reviewer:	CP	
2nd	Reviewer:	V	

SDG #: 280-3624-1 Laboratory: Test America

LDC #:

METHOD: As (EPA SW 846 Method 6020)

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 5/7/16
II.	ICP/MS Tune	A	
	Calibration	A	
IV.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	p	
VI.	Matrix Spike Analysis	Ä	m3/D
VII.	Duplicate Sample Analysis	N	
VIII.	Laboratory Control Samples (LCS)	A	LCS
IX.	Internal Standard (ICP-MS)	A	
X.	Furnace Atomic Absorption QC	$\mathcal{N}$	Noturinzed
XI.	ICP Serial Dilution	A	
XII.	Sample Result Verification	N	
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	Ν	
XV	Field Blanks	ND	FB=FB-01072010-RZC

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

TB = Trip blank

EB = Equipment blank

Validated Samples: \( \)

		T.,	୧୫୬5	Ī	T.	
1	SSAM6-03-1BPC	11	(0)	21	31	
2	SSAM6-03-5BPC	12		22	32	
3	SSAM6-03-5BPCMS	13		23	33	
4	SSAM6-03-5BPCMSD	14		24	34	
5		15		25	35	
6		16		26	36	
7		17		27	37	
8		18		28	38	
9		19		29	39	
10		20		30	40	

Notes:			

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

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

May 18, 2010

LDC Report Date:

July 6, 2010

Matrix:

Soil

Parameters:

Arsenic

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-3679-1

Sample Identification

SSAM6-04-1BPC\*\*

SSAM6-04-5BPC

SSAJ2-03-1BPC

SSAJ2-03-5BPC\*\*

SSAM6-04-1BPCMS

SSAM6-04-1BPCMSD

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

### Introduction

This data review covers 6 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Arsenic.

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

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

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination.

  This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. ICPMS Tune

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

### III. Calibration

An initial calibration was performed.

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

### IV. Blanks

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

Sample EB-05182010-RZC (from SDG 280-3679-3) was identified as an equipment blank. No arsenic was found in this blank.

Samples FB-04072010-RZC (from SDG 280-2280-2) and FB-04072010-RZD (from SDG 280-2216-2) were identified as field blanks. No arsenic was found in these blanks.

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

The frequency of analysis was met.

The criteria for analysis were met.

### VI. Matrix Spike Analysis

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

### VII. Duplicate Sample Analysis

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Internal Standards

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

### X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

### XI. ICP Serial Dilution

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

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

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

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-3679-1	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

No field duplicates were identified in this SDG.

### Tronox LLC Facility, PCS, Henderson, Nevada Arsenic - Data Qualification Summary - SDG 280-3679-1

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-3679-1	SSAM6-04-1BPC** SSAM6-04-5BPC SSAJ2-03-1BPC SSAJ2-03-5BPC**	All analytes reported below the PQL.	J (all detects)	А	Sample result verification (PQL) (sp)

Tronox LLC Facility, PCS, Henderson, Nevada Arsenic - Laboratory Blank Data Qualification Summary - SDG 280-3679-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Arsenic - Equipment Blank Data Qualification Summary - SDG 280-3679-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Arsenic - Field Blank Data Qualification Summary - SDG 280-3679-1

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson** VALIDATION COMPLETENESS WORKSHEET Stage 2B/4

		6-19-	-11
	Date:	0 01	IC.
	Page:_	1 of 1	
	Reviewer:	0/2	
2nd	Reviewer:	$\sim$	

SDG #: 280-3679-1 Laboratory: Test America

2343614

LDC #:

METHOD: As (EPA SW 846 Method 6020)

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 5/18/10
11.	ICP/MS Tune	A	
111.	Calibration	A	
IV.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	A	
VI.	Matrix Spike Analysis	A	ms/D
VII.	Duplicate Sample Analysis	$\sim$	
VIII.	Laboratory Control Samples (LCS)	A	LCS
IX.	Internal Standard (ICP-MS)	A	
X.	Furnace Atomic Absorption QC	$\mathcal{N}$	Notutitized
XI.	ICP Serial Dilution	A	
XII.	Sample Result Verification	A	Not reviewed for Stage 2B validation.
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	Ν	
XV	Field Blanks	NO	EB=EB-05182010-RZC, FB=FB-04072010-RZC,

Note:

A = Acceptable

N = Not provided/applicable SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

(280-3676-3) ed D = Duplicate

TB = Trip blank EB = Equipment blank (2802280-2)

FB-04072010-1820 (280-2216-2)

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

<del> </del>	So.		Serwent Stage 4 valida	ation		
1	SSAM6-04-1BPC**	11	GB5	21	31	
2	SSAM6-04-5BPC	12		22	32	
3	SSAJ2-03-1BPC	13		23	33	
4	SSAJ2-03-5BPC**	14		24	34	
5	SSAM6-04-1BPCMS	15		25	35	,
6	SSAM6-04-1BPCMSD	16		26	36	
7		17		27	37	
8		18		28	38	
9		19		29	39	
10		20		30	40	

Notes:	

LDC#: 2343674 SDG#: Sec Exer

### **VALIDATION FINDINGS CHECKLIST**

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times	·	<b></b>	<u></u>	
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 anaylzed for this SDG?				
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?				

LDC #: 23436 [1] SDG #: <u>Seo caet</u>

### **VALIDATION FINDINGS CHECKLIST**

Page:	_of
Reviewer:	82
Reviewer:	

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

LDC#. 2343&2\Y SDG#: 566COV6\?

# Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer:\_ Reviewer:

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

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

%R = Found x 100 True

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

True = concentration (in ug/L) of each analyte in the ICV or CCV source

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	% %	0%	Acceptable
	ICP (Initial calibration)					NW.	(AIN)
	GFAA (Initial calibration)	,					
	CVAA (Initial calibration)						
	ICP (Continuing calibration)						
	GFAA (Continuing calibration)						
	CVAA (Continuing calibration)						
) <u> </u>	ICP/MS (Initial calibration)	Δς		3 3 -			
> 3		<u> </u>	7 - 7	0.04	104	h01	<i>&gt;</i> -
(E1:10)	(CUC2:13)   ICP/MS (Continuing calibation)	$\rightarrow$	51,7	50,03	103	103	7
							_

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

479878Z "FDC" SDG #: SER COLON

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

Reviewer: 2nd Reviewer: Page:

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

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

%R = Found × 100 True

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

True = Concentration of each analyte in the source.

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

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

RPD = <u>IS-DI</u> x 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  $\times$  5)

	Acceptable (Y/N)	2-				7
Reported	%R / RPD / %D	(0)	46	201	6	h 6
Recalculated	%R / RPD / %D	201	97	201	Ь	9,650
	True / D / SDR ( <del>units</del> ) MR-/ KS	100 regli	20	21,2	73,5	25/6
=	Found/8/1 (K)	107 regle	19,3	(SSR-SR)	25,6	d'N
	Element	As				$\rightarrow$
	Type of Analysis	ICP interference check	Laboratory centrol sample	Matrix spike	Duplicate	ICP serial dilution
	Sample ID	ICS AB	527	S	9/5	

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

LDC #: 23436I4 SDG #: <u>Secore</u>

#### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

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

Detected analyte results for	Y N N/A Are re	ons below for all questions answered "N results been reported and calculated of esults within the calibrated range of the Il detection limits below the CRDL?	LUITECHIV /		
(in. Vol.)(%S)  RD = Raw data concentration  FV = Final volume (ml)  In. Vol. = Initial volume (ml) or weight (G)  Dil = Dilution factor  %S = Decimal percent solids  Reported Concentration  Concentration Acceptable  (MS S) (MS S)	Detected analyte resu following equation:	alts for	<u>.</u>	were recalculated and	d verified using the
Sample ID  Analyte  Reported Concentration Concentration (M2/S) (M6/S) (Y/N)	1.10/1	<u>FV)(Dil)</u> Recal		, .	
Sample ID  Analyte  Reported Concentration Concentration (M2/S) (M6/S) (Y/N)	FV = Final vol. = Initial vol. Dil = Dilution	olume (mi) olume (mi) or weight (G) n factor	$\frac{(\omega_{m}L)(5)}{(0.923)(1.00)}$	7,62m/L) 1000 =	4.0mg/k
	Sample ID	Analyte	Reported Concentration	Calculated Concentration	Acceptable
		AS	4.0	4.0	Y
					•
		1			
				·	
				· · · · · · · · · · · · · · · · · · ·	

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

May 18, 2010

LDC Report Date:

July 6, 2010

Matrix:

Water

Parameters:

Arsenic

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-3679-3

Sample Identification

EB-05182010-RZC

#### Introduction

This data review covers one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Arsenic.

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

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

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. ICPMS Tune

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

#### III. Calibration

An initial calibration was performed.

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

#### IV. Blanks

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

Sample EB-05182010-RZC was identified as an equipment blank. No arsenic was found in this blank.

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

The frequency of analysis was met.

The criteria for analysis were met.

#### VI. Matrix Spike Analysis

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

#### VII. Duplicate Sample Analysis

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

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

Laboratory control samples were reviewed for each matrix as applicable. Percent recoveries (%R) 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 was not performed for this SDG.

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

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-3679-3	All analytes reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

#### XIII. Overall Assessment of Data

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

#### XIV. Field Duplicates

No field duplicates were identified in this SDG.

#### Tronox LLC Facility, PCS, Henderson, Nevada Arsenic - Data Qualification Summary - SDG 280-3679-3

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-3679-3	EB-05182010-RZC	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (PQL) (sp)

Tronox LLC Facility, PCS, Henderson, Nevada Arsenic - Laboratory Blank Data Qualification Summary - SDG 280-3679-3

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Arsenic - Field Blank Data Qualification Summary - SDG 280-3679-3

No Sample Data Qualified in this SDG

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

Stage 2B

Date:6-29-10	)
Page: Lof 1	
Reviewer:	
2nd Reviewer:	

METHOD: As (EPA SW 846 Method 6020)

280-3679-3

Laboratory: Test America

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: S/K6/10
[].	ICP/MS Tune	Ð	
III.	Calibration	A	
IV.	Blanks	9	
V.	ICP Interference Check Sample (ICS) Analysis	J.	
VI.	Matrix Spike Analysis	Ŋ	Client specified
VII.	Duplicate Sample Analysis	2	
VIII.	Laboratory Control Samples (LCS)	A	LCS/D
IX.	Internal Standard (ICP-MS)	Ð	
X.	Furnace Atomic Absorption QC	7	Noturirzed
XI.	ICP Serial Dilution	N	Noturirzed
XII.	Sample Result Verification	2	
XIII.	Overall Assessment of Data	8	
XIV	Field Duplicates	$\sim$	
XV	Field Blanks	ND	EB=1 (no associated samples)

Note:

LDC #:

SDG #:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

TB = Trip blank

EB = Equipment blank

Validated Samples:

water\_

			· · · · · · · · · · · · · · · · · · ·		 	
1	EB-05182010-RZC	11	BOW	21	31	
2		12		22	32	
3		13		23	33	
4		14	•	24	34	
5		15		25	35	
6		16		26	36	
7		17		27	37	
8		18		28	38	
9		19		29	39	
10		20		30	40	

Notes:			

#### Tronox LLC Facility, PCS, Henderson, Nevada Data Validation Reports LDC #23436

Perchlorate



## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

April 16, 2010

**LDC Report Date:** 

July 6, 2010

Matrix:

Soil

Parameters:

Perchlorate

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-2541-2

Sample Identification

SSAL5-05-2BPC

SSAL5-05-4BPC

SSAL5-05-6BPC

SSAL5-05-8BPC

SSAL5-05-10BPC

#### Introduction

This data review covers 5 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.

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

Raw data were not reviewed for this SDG.

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

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. Calibration

#### a. Initial Calibration

All criteria for the initial calibration were met.

#### b. Calibration Verification

Calibration verification frequency and analysis criteria were met.

#### III. Blanks

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

Sample FB-04072010-RZD (from SDG 280-2216-2) was identified as a field blank. No perchlorate was found in this blank.

#### IV. Matrix Spike/Matrix Spike Duplicates

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

#### V. Duplicates

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

#### VI. Laboratory Control Samples

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

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

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-2541-2	All analytes reported below the PQL.	J (all detects)	Α

Raw data were not reviewed for this SDG.

#### **VIII. Overall Assessment**

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

#### IX. Field Duplicates

No field duplicates were identified in this SDG.

#### Tronox LLC Facility, PCS, Henderson, Nevada Perchlorate - Data Qualification Summary - SDG 280-2541-2

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-2541-2	SSAL5-05-2BPC SSAL5-05-4BPC SSAL5-05-6BPC SSAL5-05-8BPC SSAL5-05-10BPC	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (sp)

Tronox LLC Facility, PCS, Henderson, Nevada Perchlorate - Laboratory Blank Data Qualification Summary - SDG 280-2541-2

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Perchlorate - Equipment Blank Data Qualification Summary - SDG 280-2541-2

No Sample Data Qualified in this SDG

## Northanta Handaraan

Date: 6-29-10
Page: <u></u> of <u></u> ∫
Reviewer: C2

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
	Technical holding times	A	Sampling dates: 4 / 16/10
lla.	Initial calibration	A	
ilb.	Calibration verification	A	
HI.	Blanks	A	
· IV	Matrix Spike/Matrix Spike Duplicates	$\lambda$	Client specified
V	Duplicates	N	
VI.	Laboratory control samples	A	LCS/D
VII.	Sample result verification	N	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N	
X	Field blanks	ND	FB=FB-0407Z010-RZD

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

(280-2216-2) D = Duplicate

TB = Trip blank
EB = Equipment blank

Validated Samples: 50:1

LDC #: 23436D6 SDG #: 280-2541-2 Laboratory: Test America

	<u></u>				
1	SSAL5-05-2BPC	11 PBS	21	31	
2	SSAL5-05-4BPC	12	22	32	4
3	SSAL5-05-6BPC	13	23	33	
4	SSAL5-05-8BPC	14	24	34	
5	SSAL5-05-10BPC	15	25	35	
6		16	26	36	
7		17	27	37	
8		18	28	38	
9		19	29	39	
10		20	30	40	

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** 

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

May 17, 2010

LDC Report Date:

July 6, 2010

Matrix:

Soil

Parameters:

Perchlorate

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-3624-1

#### Sample Identification

SSAN5-02-1BPC

SSAN5-02-5BPC\*\*

SSAM6-02-1BPC

SSAM6-02-5BPC

SSAM6-03-1BPC

SSAM6-03-5BPC

SSAN5-02-1BPCMS

SSAN5-02-1BPCMSD

SSAN5-02-1BPCDUP

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

#### Introduction

This data review covers 9 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.

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

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

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. Calibration

#### a. Initial Calibration

All criteria for the initial calibration were met.

#### b. Calibration Verification

Calibration verification frequency and analysis criteria were met.

#### III. Blanks

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

Sample FB-04072010-RZC (from SDG 280-2280-2) was identified as a field blank. No perchlorate was found in this blank.

#### IV. Matrix Spike/Matrix Spike Duplicates

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

#### V. Duplicates

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

#### VI. Laboratory Control Samples

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

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

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

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-3624-1	All analytes reported below the PQL.	J (all detects)	A

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

#### VIII. Overall Assessment

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

#### IX. Field Duplicates

No field duplicates were identified in this SDG.

#### Tronox LLC Facility, PCS, Henderson, Nevada Perchlorate - Data Qualification Summary - SDG 280-3624-1

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-3624-1	SSAN5-02-1BPC SSAN5-02-5BPC** SSAM6-02-1BPC SSAM6-02-5BPC SSAM6-03-1BPC SSAM6-03-5BPC	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (sp)

Tronox LLC Facility, PCS, Henderson, Nevada Perchlorate - Laboratory Blank Data Qualification Summary - SDG 280-3624-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Perchlorate - Field Blank Data Qualification Summary - SDG 280-3624-1

No Sample Data Qualified in this SDG

## Tronox Northgate Henderson

	ronox noringate nendereen
LDC #: 23436H6	VALIDATION COMPLETENESS WORKSHEET
SDG #: 280-3624-1	_ Stage 2B/4
Laboratory: Test America	_

Date 6-29-10
Page: <u></u> of ∫
Reviewer: CF
2nd Reviewer: 🖊 🧼

METHOD: (A	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
I.	Technical holding times	A	Sampling dates: 5/17/10
lla.	Initial calibration	A	
IIb.	Calibration verification	A	
III.	Blanks	19	,
IV	Matrix Spike/Matrix Spike Duplicates	A	msp
V	Duplicates	A	D.P.
VI.	Laboratory control samples	A	LCS/D
VII.	Sample result verification	A	Not reviewed for Stage 2B validation.
VIII.	Overall assessment of data	A,	
IX.	Field duplicates	~	
Lx	Field blanks	NO	FB = FB-04072010-RZC

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

C280-2280-2)
D = Duplicate
TB = Trip blank
EB = Equipment blank

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

	5011		-			
1	SSAN5-02-1BPC	11	(B)	21	31	
2	SSAN5-02-5BPC**	12		22	32	
3	SSAM6-02-1BPC	13		23	33	
4	SSAM6-02-5BPC	14		24	34	
5	SSAM6-03-1BPC	15		25	35	
6	SSAM6-03-5BPC	16		26	36	
7	SSAN5-02-1BPCMS	17		27	37	
8	SSAN5-02-1BPCMSD	18		28	38	
9	SSAN5-02-1BPCDUP	19		29	39	
10		20		30	40	

Notes:		

LDC#: 23436+16 SDG#: SEE COVER

#### **VALIDATION FINDINGS CHECKLIST**

Page: of A
Reviewer: 2
2nd Reviewer: V

Method: Inorganics (EPA Method See Cover)

IMETROO: Inorganics (EPA Method See Guer)							
Validation Area	Yes	No	NA	Findings/Comments			
I. Technical holding times							
All technical holding times were met.							
Cooler temperature criteria was met.		<u> </u>					
II. 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?							
Were titrant checks performed as required? (Level IV only)							
Were balance checks performed as required? (Level IV only)			_				
III. Blanks							
Was a method blank associated with every sample in this SDG?							
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.							
IV. Matrix spike/Matrix spike duplicates and Duplicates							
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	\						
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.		ſ					
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq$ 20% for waters and $\leq$ 35% for soil samples? A control limit of $\leq$ CRDL( $\leq$ 2X CRDL for soil) was used for samples that were $\leq$ 5X the CRDL, including when only one of the duplicate sample values were $\leq$ 5X the CRDL.							
V. Laboratory control samples							
Was an LCS anayized for this SDG?							
Was an LCS analyzed per extraction batch?							
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% (85-115% for Method 300.0) QC limits?	/						
VI. Regional Quality Assurance and Quality Control							
Were performance evaluation (PE) samples performed?							
Were the performance evaluation (PE) samples within the acceptance limits?			/				

LDC#: 23436H6 SDG#: See cavest

#### **VALIDATION FINDINGS CHECKLIST**

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

Validation Area	Yes	No	NA	Findings/Comments
VII. Sample Result Verification				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/	_		
Were detection limits < RL?				
VIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.				
IX. Field duplicates				
Field duplicate pairs were identified in this SDG.			_	
Target analytes were detected in the field duplicates.			/	
X. Field blanks				
Field blanks were identified in this SDG.				
Target analytes were detected in the field blanks.		7		

100 #: 23436#6 spg #: <u>58000</u>00

# Initial and Continuing Calibration Calculation Verification Validatin Findings Worksheet

-	Page: of	Reviewer:	2nd Reviewer:
			~

Method: Inorganics, Method \_

314.0

\_ was recalculated.Calibration date: 5/17/10The correlation coefficient (r) for the calibration of ClO

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

%R = Found X 100

Where,

Found = concentration of each analyte measured in the analysis of the ICV or CCV solution

True

True = concentration of each analyte in the ICV or CCV source

					Recalculated	Reported	Acceptable
Type of analysis	Analyte	Standard	Conc. (ug/l)	Area	r or r²	r or r²	(Y/N)
Initial calibration		s1		0.00137			
		\$2	2.5	0.00562	0.999878	0.999700	\ \ \ \ \
		83	5	0.01434	·		)-
	5	\$4	10	0.03176			
		S5	20	0.06198			
		98	40	0.12605			
Calibration verification		ICV	02	Fonduge)	701	1	
Calibration verification	$\rightarrow$	20	$\mathcal{R}$	33,397 111	111		>
Calibration verification	0.00						

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 #: SDG #:

## VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

Page: 2nd Reviewer: Heviewer:

METHOD: Inorganics, Method Seccover

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

Where, %R = Found x 100

Found =

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

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

RPD = <u>is-Di</u> × 100 Where, (S+D)/2

Original sample concentration

ii ii O

Duplicate sample concentration

	•						
		-	Found / s	1	Recalculated	Reported	
Sample IU	Type of Analysis	Element	(unita) KS	(unite)(MG	%R / RPD	%R / RPD	Acceptable
S	Laboratory control sample	C/04	6.103	0,0980	901	501	7-
· /	Matrix spike semple		(88R-8R)	11.2	021	7117	
6	Duplicate sample	<b> </b>	340	337		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

LDC #: 23436Hb SDG #: Seecoler

#### VALIDATION FINDINGS WORKSHEET

Page: of Reviewer: 2nd reviewer:

Spg #: See Cover Sample Calculation Verification

5DG #:_	Sections	<u>Gample Galculation ver</u>	INCAUOTI	neviewe 2nd reviewe	er: er:
METHO	D: Inorganics, Metho	od Secoul			
Please s Y N N Y N N Y N N	/A Have results w	been reported and calculated correctly?  ithin the calibrated range of the instrumention limits below the CRQL?	•	are identified as '	'N/A".
Compou recalcula	nd (analyte) results tated and verified using	for Claying the following equation:	repo	rted with a positi	ve detect were
Concentration - C	tion = sffset) Preps id) (1000)	Recalculation:  O.04	1256+0.001° 0.0032 .891)(1000	(10)(100)	= 16
(% Sol	id)(1000)	. (0	.89 I) (1000	). 	
#	Sample ID	Analyte	Reported Concentration (1/0/16)	Calculated Concentration (YYS   KG)	Acceptable (Y/N)
	2	C104	160	160	7
					·
				·	
<b> </b>	, , , , , , , , , , , , , , , , , , ,				

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

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

May 18, 2010

**LDC Report Date:** 

July 6, 2010

Matrix:

Soil

Parameters:

Perchlorate

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-3679-1

#### Sample Identification

SSAM6-04-1BPC\*\*

SSAM6-04-5BPC

SSAL6-01-1BPC

SSAL6-01-5BPC

SSAL6-02-1BPC

SSAL6-02-5BPC

SSAJ2-03-1BPC

SSAJ2-03-5BPC\*\*

SSAM6-04-1BPCMS

SSAM6-04-1BPCMSD

SSAM6-04-1BPCDUP

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

#### Introduction

This data review covers 11 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.

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

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

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. Calibration

#### a. Initial Calibration

All criteria for the initial calibration were met.

#### b. Calibration Verification

Calibration verification frequency and analysis criteria were met.

#### III. Blanks

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

Sample EB-05182010-RZC (from SDG 280-3679-3) was identified as an equipment blank. No perchlorate was found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Analyte	Concentration	Associated Samples
EB-05182010-RZC	5/18/10	Perchlorate	3.3 ug/L	SSAM6-04-1BPC** SSAM6-04-5BPC

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

Samples FB-04072010-RZD (from SDG 280-2216-2) and FB-04072010-RZC (from SDG 280-2280-2) were identified as field blanks. No perchlorate was found in these blanks.

#### IV. Matrix Spike/Matrix Spike Duplicates

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

#### V. Duplicates

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

#### VI. Laboratory Control Samples

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

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

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

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-3679-1	All analytes reported below the PQL.	J (all detects)	А

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

#### VIII. Overall Assessment

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

#### IX. Field Duplicates

No field duplicates were identified in this SDG.

#### Tronox LLC Facility, PCS, Henderson, Nevada Perchlorate - Data Qualification Summary - SDG 280-3679-1

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-3679-1	SSAM6-04-1BPC** SSAM6-04-5BPC SSAL6-01-1BPC SSAL6-01-5BPC SSAL6-02-1BPC SSAL6-02-5BPC SSAJ2-03-1BPC SSAJ2-03-5BPC**	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (sp)

Tronox LLC Facility, PCS, Henderson, Nevada Perchlorate - Laboratory Blank Data Qualification Summary - SDG 280-3679-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Perchlorate - Equipment Blank Data Qualification Summary - SDG 280-3679-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Perchlorate - Field Blank Data Qualification Summary - SDG 280-3679-1

No Sample Data Qualified in this SDG

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

Stage 2B/4

	Date:	6-29-10	)
	Page:_	_of	
	Reviewer:	02	
2nd	Reviewer:	~	

LDC #:\_2343616 SDG #: 280-3679-1 Laboratory: Test America

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
Technical holding times	A	Sampling dates: 5/18/( O
Initial calibration	A	
Calibration verification	A	
Blanks	A	
Matrix Spike/Matrix Spike Duplicates	A	ms/p
Duplicates	A	ap.
Laboratory control samples	A	LCS/D
Sample result verification	A	Not reviewed for Stage 2B validation.
Overall assessment of data	A	
Field duplicates	$\wedge$	
Field blanks	Sw	FB=FB-04072010-RZD FB-04072010-RZC (280-226-2)

Note:

A = Acceptable

N = Not provided/applicable

ND = No compounds detected

D = Duplicate

EB = EB-05182010-RZC (250-3679-3)

SW = See worksheet

R = Rinsate FB = Field blank TB = Trip blank

EB = Equipment blank

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

1_	SSAM6-04-1BPC**	11	SSAM6-04-1BPCDUP	21	PB5	31	
2	SSAM6-04-5BPC	12		22		32	
3	SSAL6-01-1BPC	13		23		33	
4	SSAL6-01-5BPC	14		24		34	
5	SSAL6-02-1BPC	15		25		35	
6	SSAL6-02-5BPC	16		26		36	
7	SSAJ2-03-1BPC	17		27		37	
8	SSAJ2-03-5BPC**	18		28		38	
9	SSAM6-04-1BPCMS	19		29		39	
10	SSAM6-04-1BPCMSD	20		30		40	

Notes:		

LDC#: 23436TY SDG#: SECOVER

#### **VALIDATION FINDINGS CHECKLIST**

	Page:	_of
Re	viewer:_	œ
2nd Re	viewer:	

Method: Inorganics (EPA Method See Cover)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.				
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?		•		
Were titrant checks performed as required? (Level IV only)				
Were balance checks performed as required? (Level IV only)			_	
III. Blanks				
Was a method blank associated with every sample in this SDG?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		-		
IV. Matrix spike/Matrix spike duplicates and Duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	1	1		
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.	\	-		
Were the MS/MSD or duplicate relative percent differences (RPD) ≤ 20% for waters and ≤ 35% for soil samples? A control limit of ≤ CRDL(≤ 2X CRDL for soil) was used for samples that were ≤ 5X the CRDL, including when only one of the duplicate sample values were ≤ 5X the CRDL.				
V. 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% (85-115% for Method 300.0) QC limits?				
VI. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				

LDC#: 23436J4 SDG#: <u>See caver</u>

#### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 2 Reviewer: 2

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?	1			
Were detection limits < RL?	/			·
VIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			
IX. Field duplicates				
Field duplicate pairs were identified in this SDG.			-	
Target analytes were detected in the field duplicates.			/	
X. Field blanks				
Field blanks were identified in this SDG.				
Target analytes were detected in the field blanks.		_		

LDC #: 2343616

SDG #: See Cover

**VALIDATION FINDINGS WORKSHEET** 

Page: Reviewer:\_ 2nd Reviewer:

Field Blanks

METHOD: Inorganics, EPA Method See Cover

Were field blanks identified in this SDG?

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

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

EB Sampling date: 5/18/10 Soil factor applied 10x Field blank type: (circle one) Field Blank / Rinsate / Other:

Reason Code: be

	tification						
nples: 1, 2	Sample Identification					•	
Associated Samples: 1, 2							
As							
ier: (EB)		No Qualifiers					
Rinsate / Oth	Action Limit		0.33				
ield blank type: (circle one) Field Blank / Rinsate / Other:	Blank ID	EB-05182010-RZC (SDG#: 280-3679-3)	3.3				
ield blank type: (circle one	Analyte		CIO4				

# Initial and Continuing Calibration Calculation Verification Validatin Findings Worksheet

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

Method: Inorganics, Method 214.0

The correlation coefficient (r) for the calibration of  $\overline{\Box}\cup$  was recalculated.Calibration date:  $\overline{5/17/10}$ 

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

%R = Found X 100

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

	The state of the s				Recalculated	Reported	Acceptable
Type of analysis	Analyte	Standard	Conc. (ug/I)	Area	r or r²	r or r²	(Y/N)
Initial calibration		s1		0.00138			
		s2	2.5	0.00562	0.999878	0.999699	
		s3	5	0.01434			2
		84	10	0.03176			
		\$5	20	0.06198			
		9S	40	0.12605			
Calibration verification		ICV	92	Fand (Light) 20.471	701	1	
Calibration verification		CCV	30	25028	107		
Calibration verification	$\rightarrow$		10	719.01	901		<b>&gt;</b>

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.\_ 7343675 LDC #: SDG #:

# VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

Page: Reviewer: 2nd Reviewer:

METHOD: Inorganics, Method Secolar

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

Where, %R = Found x 100

Found =

True ==

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.

 $RPD = 1S-D_1 \times 100$  Where, (S+D)/2

ii ii O Q

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

Original sample concentration Duplicate sample concentration

			( a / busing		Reculculated	Reported	
Sample ID	Typs of Analysis	Element	(weethes) (mg/fs)	(enter) (X/K)	%R / RPD	%R / RPD	Acceptable (Y/N)
Labor	Laboratory control sample						
5		00-	103	84.3	J 0	/>0/	J_
Matrib	Matrix spike sample		0000012 0000622	0000012	 	<i>h</i> 11	
Dupk	Duplicate semple	$\rightarrow$	32,0000	301000	0	5	

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

TOTCLC.6

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	of
Reviewer:	de
2nd reviewer:	

METHOD: inorganics, Method	ecall.	
Please see qualifications below for all q  Y N N/A Have results been repo Y N N/A Are results within the ca Y N N/A Are all detection limits to the call detectio	rted and calculated co alibrated range of the i	. Not applicable questions are identified as "N/A".  orrectly?  nstruments? reported with a positive detect were
recalculated and verified using the follo  Concentration =  ARA - OFFSCH (DF)(ProFactor)	wing equation:  Recalculation:	(20000)(100)(0,047+0,019) 0.0032 -3.2x10-6
Slope / Solid	• •	(0.923) (10.38)

#	Sample ID	Analyte	Reported Concentration (MALS)	Calculated Concentration (MRICS)	Acceptable (Y/N)
	\	. ClOy	320000	3200000	7
			<u> </u>		
					·
		·			
					·

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** 

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

May 18, 2010

**LDC Report Date:** 

July 6, 2010

Matrix:

Water

Parameters:

Perchlorate

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-3679-3

Sample Identification

EB-05182010-RZC

EB-05182010-RZCMS

EB-05182010-RZCMSD

EB-05182010-RZCDUP

#### Introduction

This data review covers 4 water 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.

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

Raw data were not reviewed for this SDG.

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

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. Calibration

#### a. Initial Calibration

All criteria for the initial calibration were met.

#### b. Calibration Verification

Calibration verification frequency and analysis criteria were met.

#### III. Blanks

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

Sample EB-05182010-RZC was identified as an equipment blank. No perchlorate was found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Analyte	Concentration	Associated Samples
EB-05182010-RZC	5/18/10	Perchlorate	3.3 ug/L	No associated samples 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 analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-3679-3	All analytes reported below the PQL.	J (all detects)	Α

Raw data were not reviewed for this SDG.

#### **VIII. Overall Assessment**

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

#### IX. Field Duplicates

No field duplicates were identified in this SDG.

#### Tronox LLC Facility, PCS, Henderson, Nevada Perchlorate - Data Qualification Summary - SDG 280-3679-3

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-3679-3	EB-05182010-RZC	All analytes reported below the PQL.	J (all detects)	А	Sample result verification (sp)

Tronox LLC Facility, PCS, Henderson, Nevada Perchlorate - Laboratory Blank Data Qualification Summary - SDG 280-3679-3

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Perchlorate - Equipment Blank Data Qualification Summary - SDG 280-3679-3

No Sample Data Qualified in this SDG

#### **Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET** Stage 2B

Date: 6 C4 IC
Page: 1_of 1_
Reviewer: _ C
2nd Reviewer

LDC #: 23436J6 SDG #: 280-3679-3 Laboratory: Test America

METHOD: (Analyte)	Perchlorate (EPA Method 314.0	)
, , , , , , , , , , , , , , , , , , , ,		

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 5/16/10
lla.	Initial calibration	A	
IIb.	Calibration verification	A	
111.	Blanks	A	
IV	Matrix Spike/Matrix Spike Duplicates	A	m3/D
V	Duplicates	A	DY
VI.	Laboratory control samples	A	LCS/D
VII.	Sample result verification	N	·
VIII.	Overall assessment of data	A_	
łX.	Field duplicates	N	
x	Field blanks	SW	EB= (no associated samples)

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:

Valid	ated Samples: WQ	ter				
1	EB-05182010-RZC	11	PBW	21	31	
2	EB-05182010-RZCMS	12		22	32	
3	EB-05182010-RZCMSD	13		23	33	
4	EB-05182010-RZCDUP	14		24	34	
5		15		25	35	
6		16		26	36	
7		17		27	37	
8		18		28	38	
9		19		29	39	
10		20		30	40	

Notes:			
•			

23436 LDC #: <u>24236</u>J6

SDG #: See Cover

**VALIDATION FINDINGS WORKSHEET** 

Field Blanks

METHOD: Inorganics, EPA Method See Cover

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

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

Blank units: ug/L Associated sample units: mg/Kg Sampling date: 5/18/10 Soil factor applied 10x Field blank type: (circle one) Field Blank / Rinsate / Other: (EB)

Reason Code: be

		, ,			<del></del>	1	
	Sample Identification						
səldı							
ssociated sam							
nples: No a							
Associated Samples: No associated samples							
ler: (EB)							
Field blank type: (circle one) Field Blank / Rinsate / Other.	Action Limit		0.33				
	Blank ID	<b>-</b>	3.3				
	Analyte						
Field blank	An		CIO4				