

# LABORATORY DATA CONSULTANTS, INC.

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Northgate Environmental Management, Inc.

November 3, 2010

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

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

**Data Validation** 

Dear Ms. Arnold,

Enclosed are the final validation reports for the fraction listed below. These SDGs were received on October 12, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

# **LDC Project # 24177:**

## SDG#

# **Fraction**

280-7233-1, 280-7545-1 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** 

Shaded cells indicate Stage 4 validation (all other cells are Stage 2B validation). These sample counts do not include MS/MSD, and DUPs

EDD	Stage 2B/4	100 mg	LDC #24177 (Tronox LLC-Northgate, Henderson NV / Tronox PCS Additional Sampling)	12		ខ្ម	ĕ	3	강	orthg	ıga	te, t	len	ders	30n	⋛	Ę	<u>ق</u> ا	ŏ	ద్ద	3 A	dit	000	3   S	a a	틻	<u>(6</u>		. 1020.4		4 5 E		
TDC	SDG#	DATE REC'D	(3) DATE DUE	SV (827	SVOA (8270C)	As (6020)		Pb (6020)		<b>M</b> n (6020)		CLO <sub>4</sub> (314.0)																					
Matrix:	ix: Water/Soil			3	S	Χ	S	3	S	N S	≥ (2	S	≥	S	N S	N S	S	≯	S	≥	S	3	S	8	≶ ′S	S /	≥	S	3	S	ν ×	≥	S
∢	280-7233-1	10/12/10	11/02/10	-	9	$\overline{}$	9	,	<u> </u>	0		'				_		_	_	_			$\dashv$	$\dashv$	$\dashv$		_			$\dashv$	$\dashv$	$\dashv$	
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В	280-7545-1	10/12/10	11/02/10	-	ı	0	F	0	1 (	0 1	1 0	-																					
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Attachment 1

DL 10/12/10

LDC #: <u>24177</u> SDG #: <u>280-7233-1</u>, <u>280-7545-1</u>

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

# Tronox Northgate Henderson Worksheet

EDD Area	Yes	No	NA	Findings/Comments
I. Completeness				**************************************
Is there an EDD for the associated Tronox validation report?	X			
II. EDD Qualifier Population			i	
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_LDC24177_110210.doc
IV. EDD Delivery				
Was the final EDD sent to the client?	X			

# Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

September 8, 2010

LDC Report Date:

October 28, 2010

Matrix:

Soil/Water

Parameters:

Semivolatiles

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-7233-1

# Sample Identification

SSAQ3-02-1BPC\*\*

SSAQ3-02-1BPC FD

SSAP5-03-10BPC\*\*

SSAP5-03-1BPC

SSAP5-03-2BPC

SSAP5-03-3BPC

SSAP5-03-4BPC

SSAP5-03-5BPC

EB-09082010 1

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

## Introduction

This data review covers 8 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

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

# I. Technical Holding Times

All technical holding time requirements were met.

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

# II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

## III. Initial Calibration

Initial calibration was performed using required standard concentrations.

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

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

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

# IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

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

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

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

# V. Blanks

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

Method Blank ID	Extraction Date	Compound TIC (RT in minutes)	Concentration	Associated Samples
MB 280-32100/1-A	9/19/10	Bis(2-ethylhexyl)phthalate	97.3 ug/Kg	SSAP5-03-10BPC** SSAP5-03-1BPC SSAP5-03-5BPC
MB 280-32471/1-A	9/22/10	Bis(2-ethylhexyl)phthalate	86.3 ug/Kg	SSAP5-03-2BPC SSAP5-03-3BPC SSAP5-03-4BPC

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

Sample	Compound TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
SSAP5-03-10BPC**	Bis(2-ethylhexyl)phthalate	140 ug/Kg	140U ug/Kg
SSAP5-03-5BPC	Bis(2-ethylhexyl)phthalate	100 ug/Kg	100U ug/Kg
SSAP5-03-3BPC	Bis(2-ethylhexyl)phthalate	100 ug/Kg	100U ug/Kg
SSAP5-03-4BPC	Bis(2-ethylhexyl)phthalate	100 ug/Kg	100U ug/Kg

Sample EB-09082010\_1 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 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) and relative percent differences (RPD) were within QC limits.

# IX. Regional Quality Assurance and Quality Control

Not applicable.

## X. Internal Standards

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

# XI. Target Compound Identifications

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

# XII. Project Quantitation Limit

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

Sample	Compound	Finding	Flag	A or P
SSAQ3-02-1BPC** SSAP5-03-1BPC SSAP5-03-2BPC	Benzo(b)fluoranthene Benzo(k)fluoranthene	quantitation using the total peak area.	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-7233-1	All compounds reported below the PQL.	J (all detects)	Α

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

# XIII. Tentatively Identified Compounds (TICs)

Tentatively identified compounds were not reported by the laboratory.

# XIV. System Performance

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

## XV. Overall Assessment

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

# XVI. Field Duplicates

Samples SSAQ3-02-1BPC\*\* and SSAQ3-02-1BPC\_FD were identified as field duplicates. No semivolatiles were detected in any of the samples with the following exceptions:

	Concentra	tion (ug/Kg)				
Compound	SSAQ3-02-1BPC**	SSAQ3-02-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Benzo(b)fluoranthene	31	350U	-	319 (≤350)	-	-
Benzo(g,h,i)perylene	19	350U	-	331 (≤350)	-	-

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG 280-7233-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-7233-1	SSAQ3-02-1BPC** SSAP5-03-1BPC SSAP5-03-2BPC	Benzo(b)fluoranthene Benzo(k)fluoranthene	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Compound quantitation and CRQLs (q)
280-7233-1	SSAQ3-02-1BPC** SSAQ3-02-1BPC_FD SSAP5-03-10BPC** SSAP5-03-1BPC SSAP5-03-2BPC SSAP5-03-3BPC SSAP5-03-4BPC SSAP5-03-5BPC EB-09082010_1	All compounds reported below the PQL.	J (all detects)	Α	Project Quantitation Limit (sp)

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG 280-7233-1

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
280-7233-1	SSAP5-03-10BPC**	Bis(2-ethylhexyl)phthalate	140U ug/Kg	А	bl
280-7233-1	SSAP5-03-5BPC	Bis(2-ethylhexyl)phthalate	100U ug/Kg	А	þļ
280-7233-1	SSAP5-03-3BPC	Bis(2-ethylhexyl)phthalate	100U ug/Kg	А	bl
280-7233-1	SSAP5-03-4BPC	Bis(2-ethylhexyl)phthalate	100U ug/Kg	А	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Semivolatiles - Equipment Blank Data Qualification Summary - SDG 280-7233-1

No Sample Data Qualified in this SDG

# **Tronox Northqate Henderson**

_DC #:	24177A2a	VALIDATION COMPLETENESS WORKSHEET
SDG #:	280-7233-1	Stage 2B/4
_aborato	ry: Test America	<del>-</del>

Page: | of / Reviewer: N4 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: 9 /08 /10
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	A	% KSD r~
IV.	Continuing calibration/ICV	A	7. KSD r7 CW/W 6252
V.	Blanks	N2	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	N	client Spec
VIII.	Laboratory control samples	A	us b
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	A	Not reviewed for Stage 2B validation.
XII.	Compound quantitation/CRQLs	SAY	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	SM	D = 1,~
XVII.	Field blanks	M	EB = 9

Note:

SW = See worksheet

A = Acceptable N = Not provided/applicable

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate TB = Trip blank

EB = Equipment blank

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

	Soi   + v	vate	<u> </u>			
+1	SSAQ3-02-1BPC** D S	111	MB 280-31791 1-A	21	31	
2 1	SSAQ3-02-1BPC_FD	12 >	MB 280 - 32/00/1-A	22	32	
<b>⅓</b> 7	SSAP5-03-10BPC**	13	MB280- 20979 /-A	-23	33	
4 >	SSAP5-03-1BPC	14 4	MB280- 32471	24-	34	
5 4	SSAP5-03-2BPC	15		25	35	
6 4	SSAP5-03-3BPC	16		26	36	
7 4	SSAP5-03-4BPC	17		27	37	
8 7	SSAP5-03-5BPC	18		28	38	
- 3 9	EB-09082010_1	19		29	39	
10		20	,	30	40	

# VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
Reviewer: JV

2nd Reviewer: 4

Method: Semivolatiles (EPA SW 846 Method 8270C)

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Validation Area	Yes	No	NA	Findings/Comments
L'Tachnical holding times 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2				(Arrived Lose)
All technical holding times were met.	K			
Cooler temperature criteria was met.		1		
ii. GCMS insurined performance check				<b>Fig. 1.2</b> (C. 1.2) (C. 1.2)
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. Intitial Califoration Region Communication Communicat				
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 enforation 359 35 35 35 35 35 35 35 35 35 35 35 35 35				* All as paint of the second
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?				
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?				
Were all percent differences (%D) $\leq$ 25% and relative response factors (RRF) $\geq$ 0.05?				
/ Bankers				
Was a method blank associated with every sample in this SDG?				
Was a method blank analyzed for each matrix and concentration?		-		
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
Were all surrogate %R within QC limits?				A STATE OF THE STA
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?				
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.			-	
Was a MS/MSD analyzed every 20 samples of each matrix?				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
Was an LCS analyzed for this SDG?	1			

LDC #: 24177 A 24

# VALIDATION FINDINGS CHECKLIST

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

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		/		
D. Regional Guality Assurance and Cataliny, Stigra				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?	MT CONST. S	2 t 10 1 10 1 10 1 10 10 10 10 10 10 10 10		
kelijienekeli (njesa alika ja				BOOK OF THE CO.
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 zapa oznostru denincatar				Appropriate and an experience
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?				
All Congon (Quantum CROL)				A CONTROL OF THE STATE OF THE S
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?		,		
XIII. a enadikely kiertified compounds (1138) (1846)	7			
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?				
Were relative intensities of the major ions within $\pm$ 20% between the sample and the reference spectra?				/
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?				
System performance was found to be acceptable.		"Section Colors Selection	e personal de la companya de la comp	·
	$\overline{A}$	(120.92 		
Overall assessment of data was found to be acceptable.				
Field duplicate pairs were identified in this SDG.	4			
Target compounds were detected in the field duplicates.	Δ			
XVII FOR EMER				
Field blanks were identified in this SDG.			[	
Target compounds were detected in the field blanks.				

# VALIDATION FINDINGS WORKSHEET

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

A. Phenol™	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenol™	III. Benzo(a)pyrene™
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol™	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene™	VV. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-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. Diethyiphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1) <sup>⊷</sup>	FFF. Di-n-octylphthalate**	ກກກ
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.

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77	(
X	, ,
#	#: 50
20	DG

# VALIDATION FINDINGS WORKSHEET Blanks

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rage:	Reviewer:	2nd Reviewer:

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

Was a method blank analyzed for each matrix?

Was a method blank analyzed for each concentration preparation level?

Was a method blank associated with every sample? 

X N N/A Was the blank contaminated? If yes, please see qualification below. Blank extraction date: 9 1/4 1/6 Blank analysis date: 1/20 1/20

W Associated Samples:

Ø

\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \							
8	, Sample Identification						
2 4	Samp						
amples:							
Associated Samples:		8	h/				
<b>▼</b>			100				
		1-4 3	140 /u	,			
:	Blank ID	18 280 - 32100 1-A 3	973				
2		MB	EFE				
Conc. units: 49 /kg	Compound						
Conc. uni			7				
		XS	486.5				

Associated Samples: Blank extraction date: 9/22/6 Blank analysis date: 10/01/0 46 /kc/ Conc. units:

r S

	-						
7	100/4	,					
•} -∀	n/ ao1						
80-32 471	86.3						
MB	ESEE						
<u></u>	\$						
	4 4-1/14 50-32 471/A-4 6	1 4 V 1 4 6 50 - 32 471 / -4 6 100 / 41	η φ 32 φ.	## 20-3247/-4 6 EEF 86.3 100/4	mb 20-3247/-4 6  EEF 86.3 100/4	mb 20-3247/-4 6  (FEF 86.3 100/4	mb 20-3247/-4 6  (FEF 86.3 100/4

5x Phthalates 2x all others

# LDC# 24177 # 20

# Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

l of) Page: Reviewer: 2nd Reviewer:

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

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

Were 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? A N N/A

Qualifications	J/45/P										
Associated Samples	owed	ara,									
Finding	6GG, HHH pealls unrespowed	lab need total peak	for grantitution								
Sample ID	4 5	,									
Date											
#											

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24177A2a

# **VALIDATION FINDINGS WORKSHEET**

Field Duplicates

Page:_	<u> </u>
Reviewer:	24
2nd Reviewer:	9_

JVA	ĻΙ	HUU
		NA
abla	M	ΝΔ

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

Compound Name	Conc (	ug/Kg)	RPD	Diff	Diff Limits	Quals
Compound Name	1	2	(≤50%)	DIIT	DIII LIMIKS	(Parent Only)
Benzo(b)fluoranthene	31	350U		319	≤350	
Benzo(g,h,i)perylene	19	350U		331	≤350	

V:\FIELD DUPLICATES\24177A2a.wpd

LDC#: 24177422

# VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: of A

Reviewer: 2nd Reviewer:

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

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

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

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

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

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

S= Standard deviation of the RRFs,

X = Mean of the RRFs

					Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
		Calibration			RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
#	Standard ID Date	Date	Compound (Internal Standard)	ndard)	( 50 std)	( 50 std)	(Initial)	(Initial)		
-	ICAL	8/27/2010	8/27/2010 1,4-Dioxane	(181)	0.5926	0.5926	0.5795	0.5795	3.7	3.74
	MSS K		Naphthalene	(IS2)	1.0571	1.0571	1.0015	1.0015	8.9	8.92
			Fluorene	(IS3)	1.3180	1.3180	1.2421	1.2421	7.9	7.87
			Hexachlorobenzene	(IS4)	0.2424	0.2424	0.2313	0.2313	6.1	6.04
			Bis(2-ethylhexyl)phthalate	(185)	0.6574	0.6574	0.6075	0.6075	7.1	7.14
			Benzo(g,h,i)perylene	(186)	1.1231	1.1231	1.0199	1.0199	7.5	7.53

onc IS/Cpd         Area cpd           40/50         127636           40/50         884641           40/50         648342           40/50         200827           40/50         624253	
	Area IS
	172314
	11 669515
	12 393544
	27 662745
The second secon	53 759660
40/50 1096793	3 781265

Conc	1,4-Dioxane	Naphthalene	Fluorene	Hexachlorob	Bis(2-eh)phtha	Benzo(g,h,i)per
4.00	0.5778	1.1018	1.3240		0.5199	0.9595
10.00	0.6003	1.0722	1.3327	0.2454	0.5982	1.0450
20.00	0.6103	1.0714	1.3075	0.2448	0.6428	1.0900
50.00	0.5926	1.0571	1.3180	0.2424	0.6574	1.1231
80.00	0.5842	1.0008	1.2564	0.2335	0.6408	1.0769
120.00	0.5678	0.9489	1.1901	0.2252	0.6113	1.0108
160.00	0.5547	0.8964	1.1248	0.2168	0.6051	0.9476
200.00	0.5485	0.8636	1.0833	0.2109	0.5841	9906:0
×	0.5795	1.0015	1.2421	0.2313	0.6075	1.0199
S =	0.0217	0.0893	0.0977	0.0140	0.0434	0.0768

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

LDC# 24 177 Ax

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

<u>م</u> م Page: Reviewer:

2nd Reviewer:

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

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

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

 $A_x = Area of Compound$ 

 $C_x$  = Concentration of compound,

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

S= Standard deviation of the RRFs,

X = Mean of the RRFs

/erage RRF = sum of the RRFs/number of standards RSD = 100 * (S/X)	Compound (Internal	0/45/2040 4 4 0:2022
f the RRFs/ı	Calibration Date	0/45/2040
/erage RRF = sum o .RSD = 100 * (S/X)	Standard ID	الاحا
/erage F RSD = .	#	_

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
	Calibration			RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
Standard ID Date	Date	Compound (Internal Standard)	dard)	( 50 std)	( 50 std)	(Initial)	(Initial)		
ICAL	9/15/2010	9/15/2010 1,4-Dioxane	(181)	see r2 calculations	SU				
MSS D		Naphthalene	(182)	1.0439	1.0439	1.0514	1.0514	4.8	4.84
		Fluorene	(183)	1.2658	1.2658	1.2948	1.2948	6.7	7.89
		Hexachlorobenzene	(1S4)	0.2207	0.2207	0.2343	0.2343	10.0	9.97
		Bis(2-ethylhexyl)phthalate (IS5)	(185)	see r2 calculations	SU				
		Benzo(g,h,i)perylene (	(186)	1.0014	1.0014	1.0046	1.0046	11.2	11.18

Area IS	244285	934927	603765	1043323	1161848	1028955
Area cpd	185677	1220003	022330	287832	856940	1287934
nc IS/Cpd	40/50	40/20	40/20	40/20	40/20	40/20

Conc	1,4-Dioxane	Naphthalene	Fluorene	Hexachlorob	Bis(2-eh)phtha	Benzo(g,h,i)per
4.00	12	0.9661	1.1493		72	0.8279
10.00		1.0094	1.1995	0.2011		0.8797
20.00		1.0162	1.2174	0.2187		0.9418
50.00		1.0439	1.2658	0.2207		1.0014
80.00		1.0792	1.3399	0.2351		1.0538
120.00		1.0903	1.3533	0.2393		1.0832
160.00		1.0932	1.3954	0.2547		1.1164
200.00		1.1130	1.4378	0.2704	-	1.1329
×		1.0514	1.2948	0.2343		1.0046
S		0.0509	0.1022	0.0234		0.1123

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# 24177 Ava

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: 3 of 4 Reviewer: 36 2nd Reviewer: 2

METHOD:

GC EPA SW 846 Method 8270C

1,4-Dioxane Parameter:

		·	>	×	X^2
Date	Column	Compound	area ratio	conc ratio	
09/15/2010	Not specified	1,4-Dioxane	0.0984	0.100	
			0.1765	0.250	
			0.3444	0.500	
			0.7601	1.250	
			1.2284	2.000	
		,	1.8454	3.000	
			2.3578	4.000	
			3.0143	5.000	

	- 17/104 Feb 2017	100,000,000,000,000	
Regression Output:		Reported	
Constant	0.03708	11 0	-0.065000
Std Err of Y Est	0.02621	- Control of the Cont	
R Squared	0.99950	1.2 =	0.998700
No. of Observations	8.00000	The state of the s	A TOTAL CONTRACTOR OF THE PARTY
Degrees of Freedom	000009	The state of the s	
			- mm.s.vii.
X Coefficient(s) 0.56	0.591839	= E	0.590000
Std Err of Coef. 0.00	0.005410		

0.9837 0.7058 0.6081 0.6142 0.6151 0.5895	0.6760
--	--------

LDC # 2417 A 29

# VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: 4 of
Reviewer: 1

METHOD: GC EPA SW 846 Method 8270C

Parameter: Bis(2-ethylhexyl)phthalate

Y Y
Compound
Bis(2-ethylhexyl)phthalate
(matrix)
Trans.

0.2544 0.4063 0.5112 0.5901 0.6254 0.6431 0.6543 0.6543

Regression Output:				
		Reported		
Constant	-0.06985	11 0	0.065400	Г
Std Err of Y Est	0.01727	The state of the s		
R Squared	0.99983	12 =	0.995900	
No. of Observations	8.00000			
Degrees of Freedom	000000	and the second s		
X Coefficient(s)	0.666275	" E	0.637700	
Std Err of Coef.	0.003564			Π

LDC # 24177422

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

\ of -Page\_\_\_ Reviewer. 2nd Reviewer.

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

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

ave. RRF = initial calibration average RRF

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

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

RRF = continuing calibration RRF

Ax = Area of compound

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)	IS)	(Initial RRF)	(CC RRF)	(CC RRF)	, %D	Ω%
7	K6629	09/22/10	1,4-Dioxane	(IS1)	0.5795	0.6104	0.6104	5.3	5.3
			Naphthalene	(182)	1.0015	1.0651	1.0651	6.3	6.3
			Fluorene	(183)	1.2421	1.3197	1.3197	6.2	6.2
			Hexachlorobenzene	(184)	0.2313	0.2436	0.2436	5.3	5.3
			Bis(2-ethylhexyl)phthalate	e (IS5)	0.6075	0.7180	0.7180	18.2	18.2
			Benzo(g,h,i)perylene	(9SI)	1.0199	1.1145	1.1145	9.3	9.3
2	D8768	9/20/2010	1,4-Dioxane	(181)	80000	68600	68625	14.3	14.3
			Naphthalene	(IS2)	1.051	1.105	1.105	5.1	5.1
	MSS D		Fluorene	(183)	1.295	1.363	1.363	5.3	5.3
			Hexachlorobenzene	(184)	0.234	0.247	0.247	5.5	5.6
			Bis(2-ethylhexyl)phthalate	e (IS5)	80000	89100	89054	11.3	11.4
			Benzo(a.h.i)pervlene	(981)	1 005	1 100	1 100	9.4	5.0

Compound (Reference IS)	(	Concentration	Area Cpd	Area IS	Area Cpd	Area IS
		(IS/Cpd)				
1,4-Dioxane	(IS1)	40/80	325560	266683	236496	225113
Naphthalene	(182)	40/80	2174804	1020961	1906904	862593
Fluorene	(183)	40/80	1584055	600149	1446320	530492
Hexachlorobenzene	(184)	40/80	478292	981724	447244	904839
Bis(2-ethylhexyl)phthalate	(185)	40/80	1493039	1039694	1464779	1062945
Benzo(g,h,i)perylene	(186)	40/80	2399208	1076340	1920793	873138
		-				

LDC #: 74 177 A 29

# VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page:	_lof_1_
Reviewer:_	N
d raviowar.	Á

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found

SS = Surrogate Spiked

Sample ID:

1	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	100	62.0	62	62	0
2-Fluorobiphenyl		16,4	64	<b>6</b> ¢	
Terphenyl-d14	1	73.1	73	73	
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					

LUC#: 24/77 AM

# VALIDALION FINDINGS WORKSHEEL

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

Page: Lof 1 2nd Reviewer: Reviewer:

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

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

Where: % Recovery = 100 \* (SC/SA

SSC = Spike concentration SA = Spike added

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

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

7801 ž LCS/LCSD samples:

379V2-A

	S	pike	Š	pike		SU	Ĉ	GSD	   SJ	CS/I CSD
Compound	<b>A</b> (%)	Added ( Mg /k_ )	Conce	Concentration ( 45 /E)	Percent Recovery	Secovery	Percent Recovery	Secovery	7.	RPD
	1.05	l CSD	SUI	1 CSD	Reported	Recalc	Reported	Recalc	Reported	Receiptinged
Phenoi										
N-Nitroso-di-n-propylamine										
4-Chloro-3-methylphenol								-		
Acenaphthene	0972	NA	0% ox	KA.	76	16				
Pentachlorophenol										
Pyrene	2660	7	2 / 00		79	62				
			_							

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#: 4177 Am

# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	1	_of_	1
Reviewer:		N	1
2nd reviewer:			
		9	
		,	

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

 $(A_x)(I_s)(DF)$ 

Y N N/A Y N N/A

Concentration =

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?

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

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

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

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

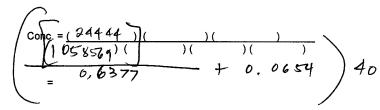
RRF = Relative response factor of the calibration standard.

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

Df = Dilution factor.

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

Example:



= 4.00

		Reported	Calculated	
Sample ID	Compound	Concentration ( )	Concentration (	Qualification
	final cu	$\kappa = (4.064)$	) (IM) (10	( m
		4		
		= 144,	5	
		2 140	ug /tg	
			U	
	11-11-11-11-11-11-11-11-11-11-11-11-11-			
	Sample ID		final conc. = (4.069) = 149.	Sample ID Compound ( ) ( ) $firal curc. = (4.064) (IM) (IC)$ $30.79 (0.9)$ $= 144, ($

# Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

September 8, 2010

LDC Report Date:

October 27, 2010

Matrix:

Soil/Water

Parameters:

Arsenic and Manganese

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-7233-1

# Sample Identification

SSAQ3-02-1BPC\*\*

SSAQ3-02-1BPC FD

SSAL8-02-2BPC

SSAL8-02-3BPC

SSAL8-02-4BPC\*\*

SSAL8-02-2BPC FD

SSAJ2-06-6BPC

SSAL2-06-7BPC

SSAJ2-06-8BPC\*\*

EB-09082010 1

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

### Introduction

This data review covers 9 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Arsenic and Manganese.

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

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

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

The following are definitions of the data qualifiers:

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

# I. Technical Holding Times

All technical holding time requirements were met.

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

## II. ICPMS Tune

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

### III. Calibration

An initial calibration was performed.

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

### IV. Blanks

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

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
PB (prep blank)	Manganese	0.0642 mg/Kg	SSAL8-02-2BPC SSAL8-02-3BPC SSAL8-02-4BPC** SSAL8-02-2BPC_FD

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

Sample EB-09082010\_1 was identified as an equipment blank. No arsenic or manganese was found in this blank.

# V. ICP Interference Check Sample (ICS) Analysis

The frequency of analysis was met.

The criteria for analysis were met.

# VI. Matrix Spike Analysis

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

# VII. 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 analysis was not performed by the laboratory.

# 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 280-7233-1	All analytes reported below the PQL.	J (all detects)	A

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

## XIII. Overall Assessment of Data

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

# XIV. Field Duplicates

Samples SSAQ3-02-1BPC\*\* and SSAQ3-02-1BPC\_FD and samples SSAL8-02-2BPC and SSAL8-02-2BPC\_FD were identified as field duplicates. No arsenic or manganese was detected in any of the samples with the following exceptions:

	Concentral	tion (mg/Kg)	DDD	Difference		
Analyte	SSAQ3-02-1BPC**	SSAQ3-02-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	3.1	3.4	9 (≤50)	-	-	-

	Concentrati	ion (mg/Kg)	DDD	Difference		
Analyte	SSAL8-02-2BPC	SSAL8-02-2BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	12	16	29 (≤50)	. <b>-</b>	-	-
Manganese	2000	1900	5 (≤50)	-	-	-

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic and Manganese - Data Qualification Summary - SDG 280-7233-1

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-7233-1	SSAQ3-02-1BPC** SSAQ3-02-1BPC_FD SSAL8-02-2BPC SSAL8-02-3BPC SSAL8-02-4BPC** SSAL8-02-2BPC_FD SSAJ2-06-6BPC SSAL2-06-7BPC SSAJ2-06-8BPC** EB-09082010_1	All analytes reported below the PQL.	J (all detects)	А	Sample result verification (PQL) (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic and Manganese - Laboratory Blank Data Qualification Summary - SDG 280-7233-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Arsenic and Manganese - Equipment Blank Data Qualification Summary - SDG 280-7233-1

No Sample Data Qualified in this SDG

# Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

LDC #: 24177A4 VALIDATION COMPLETENESS
SDG #: 280-7233-1 Stage 2B/4
Laboratory: Test America

Date: 10-27-10
Page: <u>1</u> of <u>1</u>
Reviewer: CZ_
2nd Reviewer:

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

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

	Validation Area		Comments
l.	Technical holding times	A	Sampling dates: 9/8/10
11.	ICP/MS Tune	A	
III.	Calibration	A	
IV.	Blanks	SW	
V.	ICP Interference Check Sample (ICS) Analysis	A	
VI.	Matrix Spike Analysis	$\sim$	Client specified
VII.	Duplicate Sample Analysis	$\sim$	L.
VIII.	Laboratory Control Samples (LCS)	A	LCS/D
IX.	Internal Standard (ICP-MS)	A	
Χ.	Furnace Atomic Absorption QC	$\mathcal{N}$	NOTUE FRED
XI.	ICP Serial Dilution	$\mathcal{N}$	Notpensoned
XII.	Sample Result Verification	A	Not reviewed for Stage 2B validation.
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	SW	(1,2),(3,6)
ΧV	Field Blanks	NO	E0=10

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate FB = Field blank

R = Rinsate

D = Duplicate TB = Trip blank

EB = Equipment blank

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

	2011 1 2000.			
1	SSAQ3-02-1BPC** 5	11	21 885	31
2	SSAQ3-02-1BPC_FD	12	22 834	32
3	SSAL8-02-2BPC	13	23	33
4	SSAL8-02-3BPC	14	24	34
5	SSAL8-02-4BPC**	15	25	35
6	SSAL8-02-2BPC_FD	16	26	36
7	SSAJ2-06-6BPC	17	27	37
8	SSAL2-06-7BPC	18	28	38
9	SSAJ2-06-8BPC**	19	29	39
10	EB-09082010_1 W	20	30	40

Notes:	

LDC# WINAY

# **VALIDATION FINDINGS CHECKLIST**

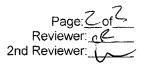
Page: of Reviewer: 2nd Reviewer:

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

Validation Area	Yes	No	NA	Findings/Comments			
I. Technical holding times							
All technical holding times were met.	/						
Cooler temperature criteria was met.							
II. ICP/MS Tune	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.			$\rangle$				
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq$ 20% for waters and $\leq$ 35% for soil samples? A control limit of +/- RL(+/-2X RL for soil) was used for samples that were $\leq$ 5X the RL, including when only one of the duplicate sample values were $\leq$ 5X the RL.			/				
VII. Laboratory control samples							
Was an LCS anaylzed for this SDG?							
Was an LCS analyzed per extraction batch?	_/						
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?							

LDC# 24177A4

# **VALIDATION FINDINGS CHECKLIST**



Validation Area	Yes	No	NA	Findings/Comments		
VIII. Furnace Atomic Absorption QC						
If MSA was performed, was the correlation coefficients > 0.995?						
Do all applicable analysies have duplicate injections? (Level IV only)						
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)			/			
Were analytical spike recoveries within the 85-115% QC limits?			/			
IX. ICP Serial Dilution	,					
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the MDL (ICP)/>100X the MDL(ICP/MS)?		\ 				
Were all percent differences (%Ds) < 10%?			/			
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.			/			
X. Internal Standards (EPA SW 846 Method 6020/EPA 200.8)						
Were all the percent recoveries (%R) within the 30-120% (6020)/60-125% (200.8) of the intensity of the internal standard in the associated initial calibration?	/					
If the %Rs were outside the criteria, was a reanalysis performed?						
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.		7				

LDC#\_VIM

## VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

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

All circled elements are applicable to each sample.

Sample ID Matrix	Target Analyte List (TAL)
1,7-,7-10	Al, Sb, Ās, 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> ,
3-6	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN
·	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
`	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, 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,
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	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,
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	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,
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·	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
	Analysis Method
ICP	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
ICP-MS	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
GEAA	Al, Sh, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN

Comments: Mercury by CVAA if performed

## LDC #: 24177A4

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

mg/Kg Sample Concentration units, unless otherwise noted:

Soil preparation factor applied: 100x x 5xdil Associated Samples: 3-6

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	Maximum Maximum Action PB* ICB/CCB* Limit Qu* (mg/Kg) (ug/L)	$\dashv$	
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a - The listed analyte concentration is the highest ICB, CCB, or PB detected in the analysis of each element. Note:

LDC#: 24177A4

## **VALIDATION FINDINGS WORKSHEET** Field Duplicates

_ /	,
Page: \(\frac{\cappa}{2}\)	_of
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METHOD: Metals (EPA Method 6010B/6020/7000)

$\Gamma$	Ŷ	M	NA	
	Y	N	NA	

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

	Concentration	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	1	2	RPD	Difference	Limits	(Parent Only)
Arsenic	3.1	3.4	9			

V:\FIELD DUPLICATES\FD\_inorganic\24177A4.wpd

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	3	6	RPD	Difference	Limits	(Parent Only)
Arsenic	12	16	29			
Manganese	2000	1900	5			

241778 # DOI

# Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

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

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

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

%R = Found × 100 True

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

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
	ICP (Initial calibration)						
TC?	ICP/MS (Initial calibration)	A.	717	0/5	10.5	(05	)~
	CVAA (Initial calibration)						-
	ICP (Continuing calibration)						
CCV (cours)	ICP/MS (Continuing calibration)	M	<b>50,</b> 3	8	101	(0)	<u>}                                    </u>
	CVAA (Continuing calibration)						
	GFAA (Initial calibration)				-		
	GFAA (Continuing calibation)						

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

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

Page: Lof 2nd Reviewer.

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

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

%R = Found x 100 True

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

True = Concentration of each analyte in the source.

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

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

Where,

S = Original sample concentration D = Duplicate sample concentration

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

%D = ||-SDR| × 100

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

	ē.				Recalculated	Reported	
Sample ID	Type of Analysis	Element	Found 18/1 KG (units) MR KG	True / D / SDR (umits)	%R/RPD/%D	%R/RPD/%D	Acceptable (Y/N)
ACS ES	ICP interference check	P5	AS 103 Ngh	100 mg/L	(2.0)	20	)-
105	Laboratory control sample	Mn	h'&1	20	25	76	7
>	Matrix spike		(SSR-SR)			·	
ک	Duplicate						
N	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#. 24MAH

## VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page: <u>↓</u>	of \
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METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

Y N N/A Are results within the ca Y N N/A Are all detection limits b Detected analyte results for equation:	ted and calculated correctly librated range of the instrumelow the CRDL?	/? nents and within the line were recalcu	ear range of the IC	
Concentration = \(\frac{(RD)(FV)(Dil)}{(In. Vol.)}\)  RD = Raw data concentration  FV = Final volume (ml)  In. Vol. = Initial volume (ml) or weight (0)  Dil = Dilution factor	Recalculation  I (2)	0m L (5) (509) 03g) (092)	3ugli) =	26,87mg/
# Sample ID	Analyte	Reported Concentration (max)	Calculated Concentration (W2-(155)	Acceptable (Y/N)
5	AS Mr	670	670	
Note:				

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

September 16, 2010

LDC Report Date:

October 27, 2010

Matrix:

Soil

Parameters:

Metals

**Validation Level:** 

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-7545-1

## Sample Identification

SSAO7-09-1\_01\_BPC

SSAO7-09-5 01 BPC

SSAO7-09-5 01 BPC FD

SSAO7-09-10 01 BPC\*\*

SSAO5-07-1 01 BPC

SSAO5-07-5 01 BPC

SSA07-09-5 01 BPCMS

SSA07-09-5 01 BPCMSD

SSAO5-07-5 01 BPCMS

SSAO5-07-5 01 BPCMSD

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

## Introduction

This data review covers 10 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Arsenic, Lead, and Manganese.

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

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

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

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

### II. ICPMS Tune

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

## III. Calibration

An initial calibration was performed.

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

## IV. Blanks

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

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
PB (prep blank)	Lead Manganese	0.0216 mg/Kg 0.0438 mg/Kg	SSAO7-09-1_01_BPC SSAO7-09-10_01_BPC**
ICB/CCB	Manganese	0.435 ug/L	SSAO7-09-1_01_BPC SSAO7-09-10_01_BPC**
PB (prep blank)	Lead Manganese	0.0206 mg/Kg 0.105 mg/Kg	SSAO7-09-5_01_BPC SSAO7-09-5_01_BPC_FD
ICB/CCB	Manganese	0.507 ug/L	SSAO7-09-5_01_BPC SSAO7-09-5_01_BPC_FD

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

No field blanks were identified in this SDG.

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

The frequency of analysis was met.

The criteria for analysis were met.

## VI. Matrix Spike Analysis

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

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
SSAO7-09-5_01_BPCMS/MSD (SSAO7-09-1_01_BPC SSAO7-09-5_01_BPC SSAO7-09-5_01_BPC_FD SSAO7-09-10_01_BPC** SSAO5-07-1_01_BPC)	Arsenic	-	186 (75-125)	•	J+ (all detects)	

## VII. Duplicate Sample Analysis

Duplicate 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 280-7545-1	All analytes reported below the PQL.	J (all detects)	A

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

## XIII. Overall Assessment of Data

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

## XIV. Field Duplicates

Samples SSAO7-09-5\_01\_BPC and SSAO7-09-5\_01\_BPC\_FD were identified as field duplicates. No metal contaminants were detected in any of the samples with the following exceptions:

	Concentra	tion (mg/Kg)	222	5.0		
Analyte	SSAO7-09-5_01_BPC	SSAO7-09-5_01_BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Arsenic	20	15	29 (≤50)	-	-	-
Lead	84	56	40 (≤50)	-	. <del>-</del>	-
Manganese	3600	3300	9 (≤50)	-	-	•

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Metals - Data Qualification Summary - SDG 280-7545-1

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-7545-1	SSAO7-09-1_01_BPC SSAO7-09-5_01_BPC SSAO7-09-5_01_BPC_FD SSAO7-09-10_01_BPC** SSAO5-07-1_01_BPC	Arsenic	J+ (all detects)	А	Matrix spike/Matrix spike duplicates (%R) (m)
280-7545-1	SSAO7-09-1_01_BPC SSAO7-09-5_01_BPC SSAO7-09-5_01_BPC_FD SSAO7-09-10_01_BPC** SSAO5-07-1_01_BPC SSAO5-07-5_01_BPC	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (PQL) (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Metals - Laboratory Blank Data Qualification Summary - SDG 280-7545-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Metals - Equipment Blank Data Qualification Summary - SDG 280-7545-1

No Sample Data Qualified in this SDG

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

LDC #:\_ 24177B4 SDG #: 280-7545-1 Stage 2B/4 Laboratory: Test America

Date:	10-27-10
Page:	
Reviewer:	02
2nd Reviewer:	~

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 9/16/10
11.	ICP/MS Tune	A	
111.	Calibration	A	
IV.	Blanks	3W	
V.	ICP Interference Check Sample (ICS) Analysis	A	,
VI.	Matrix Spike Analysis	SW	mS/1)
VII.	Duplicate Sample Analysis	N	
VIII.	Laboratory Control Samples (LCS)	A	LCS
IX.	Internal Standard (ICP-MS)	A	
X.	Furnace Atomic Absorption QC	N	Not utilized
XI.	ICP Serial Dilution	A	
XII.	Sample Result Verification	<b>A</b>	Not reviewed for Stage 2B validation.
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	54	(2,3)
ΧV	Field Blanks	N	

Note:

A = Acceptable

N = Not provided/applicable SW = See worksheet

ND = No compounds detected

R = Rinsate FB = Field blank D = Duplicate

TB = Trip blank

EB = Equipment blank

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

,		5011		
1	SSAO7-09-1_01_BPC	11	21 835 (1,4-6)	31
2	SSAO7-09-5_01_BPC	12	22 (2)	32
3	SSAO7-09-5_01_BPC_FD	13	23	33
4	SSAO7-09-10_01_BPC**	14	24	34
5	SSAO5-07-1_01_BPC	15	25	35
6	SSAO5-07-5_01_BPC	16	26	36
7	SSAO7-09-5_01_BPCMS	17	27	37
8	SSAO7-09-5_01_BPCMSD	18	28	38
9	SSAO5-07-5_01_BPCMS	19	29	39
10	SSAO5-07-5_01_BPCMSD	20	30	40

Notes:			

## **VALIDATION FINDINGS CHECKLIST**

Page: 1 of 7 Reviewer: 02 2nd Reviewer: V

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.		<u> </u>		
II. ICP/MS Tune				
Were all isotopes in the tuning solution mass resolution within 0.1 amu?	/			
Were %RSD of isotopes in the tuning solution ≤5%?				
III. Calibration		_		
Were all instruments calibrated daily, each set-up time?		,		
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?				A A CONTRACTOR OF THE CONTRACT
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 #: 24177BY

## **VALIDATION FINDINGS CHECKLIST**

Page: Z of Z Reviewer: c C 2nd Reviewer: \_\_\_\_\_

Validation Area	Yes	No	NA	Findings/Comments		
VIII. Furnace Atomic Absorption QC						
If MSA was performed, was the correlation coefficients > 0.995?						
Do all applicable analysies have duplicate injections? (Level IV only)						
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)			_			
Were analytical spike recoveries within the 85-115% QC limits?						
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?		ſ				
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.		_				
Target analytes were detected in the field duplicates.				)		
XV. Field blanks						
Field blanks were identified in this SDG.			_			
Target analytes were detected in the field blanks.			_	·		

LDC#: WIMBY

## VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page:	_of/
Reviewer:_	CR
2nd reviewer:_	<u> </u>

All circled elements are applicable to each sample.

Sample ID	_Matrix	Target Analyte List (TAL)
1-4		Al, Sb, As) Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Ph, Mg, Mn Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
56		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,
50-7.8		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,
1 9 10		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,
\(\cup 1_1 \cup 1_2 \cup		
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Ał, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		Analysis Method.
ICP		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
ICP-MS		Al, Sb(A), Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, PD Mg, Mn Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
GEAA		Al Sh As Ba Be Cd Ca Cr Co Cu Fe Ph Mg Mn Hg Ni K Se Ag Na Tl V Zn Mo R Si CN

Comments:_	Mercury by CVAA if performed	
***************************************	*	

## LDC #: 24177B4

Soil preparation factor applied: 100x x 5xdil VALIDATION FINDINGS WORKSHEET PB/ICB/CCB QUALIFIED SAMPLES METHOD: Trace metals (EPA SW 864 Method 6010B/6020/7000)

Associated Samples: mg/Kg

Sample Concentration units, unless otherwise noted:

Page:

e Maximum Maximum N	Maximum ICB/CCB <sup>a</sup>	n Action	No Qualifiers					
(ng/L)	2.7							
0.435								

Sample Co	ncentration u	Sample Concentration units, unless otherwise noted: mg/Kg	otherwise not	ted: mg/k		Associated Samples: 2, 3	2,3				
	Company of State of S										
Analyte	Maximum PB <sup>a</sup> (mg/Kg)	Analyte Maximum Maximum Maximum PB <sup>a</sup> PB <sup>a</sup> ICB/CCB <sup>a</sup> (mg/Kg) (ug/L) (ug/L)	Maximum ICB/CCB <sup>a</sup> (ug/L)	Action Limit	No Qualifiers			1001000			
Pb	0.0206						and the state of t				1177 A. L.
Mn	0.105		0.507								

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

LOCALO # POPI

# VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Reviewer: 2nd Reviewer.

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

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

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

Were matrix spike percent recoveries (%R) within the control limits of 75-125?) If the sample concentration exceeded the spike concentration by a factor

of 4 or more, no action was taken.

Were all duplicate sample relative percent differences (RPD ≤ 20% for water samples and ≤35% for soil samples?

Y(N) N/A Wer

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

		_		_	_	 _	_	_	 		 		 	_	_	_	==
Qualifications	7+db+/A (m)	1000al (LCS in)															
Associated Samples	20 4 5 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4	1-5															
		Ĺ															
MSD %Recovery	1 86																
MS %Recovery																	
Analyte	ΑS	AS															
Matrix	\$														9000000		
OI OSW/SW	2/8									A STATE OF THE STA		and provided and design and desig	***************************************	THE PART AND ADMINISTRATION OF THE PART AND ADMINISTRATION OF			
#	Ш																

Comments:

LDC#: 24177B4

## **VALIDATION FINDINGS WORKSHEET** Field Duplicates

Page:	of
Reviewer:	
nd Reviewer:	1

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

W	N	NA_
$\backslash Y$	N	NA
· \	$\overline{}$	

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

V:\FIELD DUPLICATES\FD\_inorganic\24177B4.wpd

	Concentrati	on (mg/kg)	(≤50)	(mg/Kg)	(mg/Kg)	Qualifications
Analyte	2	3	RPD	Difference	Limits	(Parent Only)
Arsenic	20	15	29			
Lead	84	56	40			
Manganese	3600	3300	9			

LDC# 2477/89

# Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Reviewer. Page: 2nd Reviewer:

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

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

%R = Found x 100 True

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

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
	ICP (Initial calibration)						
ICU	ICP/MS (Initial calibration)	qd	6/1h	Q15	(03)	103	7
	CVAA (Initial calibration)						
	ICP (Continuing calibration)						
CEUCIER	CEU/18:30 ICP/MS (Continuing calibration)	MN	21/5	B	701	20)	5
	CVAA (Continuing calibration)						
	GFAA (Initial calibration)	,				The second secon	
	GFAA (Continuing calibation)						

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

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

2nd Reviewer: Reviewer

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

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

%R = Found × 100

Where, Found = Concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation,

Found = SSR (spiked sample result) - SR (sample result).

True = Concentration of each analyte in the source.

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

RPD =  $[S-D] \times 100$ (S+D)/2

Where,

S = Original sample concentration D = Duplicate sample concentration

%D = [I-SDR] × 100

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

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

			4		Recalculated	Reported	
Sample ID	Type of Analysis	Element	Found / S / I (units) (XX)	True / D / SDR (amptis)	%R/RPD/%D	%R/RPD/%D	Acceptable (Y/N)
ICS AR	ICP interference check	(A)	93,6 mg/L	100 28/	hb	hb	2
<657	Laboratory control sample	Mn	18,4	70	26	76	
10	Matrix spike	B	(SSR-SR)	20,6	38	88	
01/6	Duplicate		19,01	5/12	8	7	
<u>O</u>	ICP serial dilution	Mn	300	355	1,67	28.0	

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 #: 24MB4

## VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page: <u>↓</u>	of
Reviewer:_	ac_
2nd reviewer:_	

METHOD: Trace Metals (EPA	A SW 846 Method 6010/6020/7000)			
Y N N/A Have results W	ow for all questions answered "N". Not apple been reported and calculated correctly? within the calibrated range of the instrumer tion limits below the CRDL?	nts and within the line	ear range of the IC	
Concentration = \(\frac{(RD)(FV)(Dil)}{(ln. Vol.)}\)  RD = Raw data concervation = Final volume (min. Vol. = Initial volume (min. Vol. = Dilution factor	Recalculation:    OCN	LX3 (4.12, 1000)	= 7,	1378/1cg
# Sample ID	Analyte	Reported Concentration (YYR (A)	Calculated Concentration	Acceptable (Y/N)
	Ph MA	3, L 7, 1 350	3,2 7.1 350	
Note:				

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

September 16, 2010

**LDC Report Date:** 

October 27, 2010

Matrix:

Soil

Parameters:

Perchlorate

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-7545-1

## Sample Identification

SSAM6-06-1 01 BPC

SSAM6-06-5 01 BPC

SSAM6-06-10 01 BPC\*\*

SSAM6-05-1 01 BPC

SSAM6-05-5 01 BPC

SSAM6-05-10 01 BPC

SSAM5-05-1 01 BPC

SSAM5-05-1 01 BPC FD

SSAM5-05-5 01 BPC

SSAM5-05-10 01 BPC

SSAM6-06-1 01 BPCMS

SSAM6-06-1 01 BPCMSD

SSAM6-06-1 01 BPCDUP

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

## Introduction

This data review covers 13 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.

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

## The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. Calibration

## a. Initial Calibration

All criteria for the initial calibration were met.

## b. Calibration Verification

Calibration verification frequency and analysis criteria were met.

## III. Blanks

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

No field blanks were identified in this SDG.

## IV. Matrix Spike/Matrix Spike Duplicates

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

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
SSAM6-06-1_01_BPCMS/MSD (All samples in SDG 280-7545-1)	Perchlorate	128 (75-125)	127 (75-125)	-	J+ (all detects)	А

## V. Duplicates

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

## VI. Laboratory Control Samples

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

## VII. Sample Result Verification and Project Quantitation Limit

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

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-7545-1	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

Samples SSAM5-05-1\_01\_BPC and SSAM5-05-1\_01\_BPC\_FD were identified as field duplicates. No contaminant concentrations was detected in any of the samples with the following exceptions:

	Concentra	tion (mg/Kg)				
Analyte	SSAM5-05-1_01_BPC	SSAM5-05-1_01_BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
Perchlorate	180	180	0 (≤50)	-	· <u>-</u>	-

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Perchlorate - Data Qualification Summary - SDG 280-7545-1

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
280-7545-1	SSAM6-06-1_01_BPC SSAM6-06-5_01_BPC SSAM6-06-10_01_BPC** SSAM6-05-1_01_BPC SSAM6-05-5_01_BPC SSAM6-05-10_01_BPC SSAM5-05-1_01_BPC SSAM5-05-1_01_BPC_FD SSAM5-05-5_01_BPC SSAM5-05-10_01_BPC	Perchlorate	J+ (all detects)	Α	Matrix spike/Matrix spike duplicates (%R) (m)
280-7545-1	SSAM6-06-1_01_BPC SSAM6-06-5_01_BPC SSAM6-06-10_01_BPC** SSAM6-05-1_01_BPC SSAM6-05-5_01_BPC SSAM6-05-10_01_BPC SSAM5-05-1_01_BPC SSAM5-05-1_01_BPC_FD SSAM5-05-5_01_BPC SSAM5-05-10_01_BPC	All analytes reported below the PQL.	J (all detects)	А	Sample result verification (sp)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Perchlorate - Laboratory Blank Data Qualification Summary - SDG 280-7545-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Perchlorate - Equipment Blank Data Qualification Summary - SDG 280-7545-1

No Sample Data Qualified in this SDG

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

Stage 2B/4

Date:	0-22-10

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Reviewer: 2nd Reviewer:

SDG #: 280-7545-1 Laboratory: Test America

LDC #: 24177B6

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
I,	Technical holding times	A	Sampling dates: 9-16-16
IIa.	Initial calibration	A	
IIb.	Calibration verification	A	
III.	Blanks	A	
IV	Matrix Spike/Matrix Spike Duplicates	SW	m31)
V	Duplicates	A	OR
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	5W	(7,8)
х	Field blanks	$\wedge$	

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

TB = Trip blank EB = Equipment blank

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

	<u>soil</u>	_				.,,	
1	SSAM6-06-1_01_BPC	11	SSAM6-06-1_01_BPCMS	21	PB5	31	
2	SSAM6-06-5_01_BPC	12	SSAM6-06-1_01_BPCMSD	22		32	
3	SSAM6-06-10_01_BPC**	13	SSAM6-06-1_01_BPCDUP	23		33	
4	SSAM6-05-1_01_BPC	14		24		34	
5	SSAM6-05-5_01_BPC	15		25		35	
6	SSAM6-05-10_01_BPC	16		26		36	
7	SSAM5-05-1_01_BPC	17		27		37	
8	SX/SAM5-05-1_01_BPC_FD	18		28		38	
9	SSAM5-05-5_01_BPC	19		29		39	
10	SSAM5-05-10_01_BPC	20		30		40	

Notes:			

## **VALIDATION FINDINGS CHECKLIST**

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Method: Inorganics (EPA Method See Cover-

Method: 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.				
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)				<u> </u>
III. Blanks				
Was a method blank associated with every sample in this SDG?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/		
IV. Matrix spike/Matrix spike duplicates and Duplicates				7
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.				
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq$ 20% for waters and $\leq$ 35% for soil samples? A control limit of $\leq$ CRDL( $\leq$ 2X CRDL for soil) was used for samples that were $\leq$ 5X the CRDL, including when only one of the duplicate sample values were $\leq$ 5X the CRDL.				
V. Laboratory control samples		,		
Was an LCS anaylzed for this SDG?	/			
Was an LCS analyzed per extraction batch?	/			
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?		/		
Nere the performance evaluation (PE) samples within the acceptance limits?			7	. <u>-</u>

LDC #: 7417136

## **VALIDATION FINDINGS CHECKLIST**

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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.	1			
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	

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# VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

**METHOD**: Inorganics, EPA Method 3/4.0

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

N/A

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

Were matrix spike percent recoveries (%R) within the control limits of 75-125) If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken. Y N N/A

Were all duplicate sample relative percent differences (RPD) < 20% for water samples and <35% for soil samples?

N N/A Were all duplic tever in the contract of

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

Qualifications	Jtdet/A (m)								
RPD (Limits) Associated Samples	110								
RPD (Limits)			Addition						
MSD %Recovery	127								
MS %Recovery	128								
Analyte	6104							A CONTRACTOR OF THE CONTRACTOR	
Matrix (A) eQ	1 1								
ardsw/sw	71/12								

Comments:

L	D	C#:	24177B6	

## **VALIDATION FINDINGS WORKSHEET** Field Duplicates

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METHOD: Inorganics (Method: See Cover )

Æ	Y	h	NA	
$\mathcal{I}$	Y	N	NA	

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

V:\FIELD DUPLICATES\FD\_inorganic\24177B6.wpd

	Concentration (mg/kg)		(≤50)	(mg/Kg)	(mg/Kg)	Qualifications	
Analyte	7	8	RPD	Difference	Limits	(Parent Only)	
Perchlorate	180	180	0				

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

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METHOD: Inorganics, Method SEC COVER

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

 $%R = Found \times 100$ 

Where,

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

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

Where, RPD =  $|S-D|_{X} \times 100$ (S+D)/2

" " O

Original sample concentration Duplicate sample concentration

	***********		•		Recalculated	Renorted	
Sample ID	Type of Analysis	Element	Found / S / K	True / D (units) pre / K	%R/RPD	%R / RPD	Acceptable (Y/N)
{	Laboratory control sample	(	) )	5			
3		5- C[0-	0,105	(A)	501	50)	)
	Matrix spike sample		(SSR-SR)				
_			9592	0502	123	821	
5	Duplicate sample		3868	912h	0	0	1
		7	)				

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

DC# 2417736

# Validation Findings Worksheet Initial and Continuing Calibration Calculation Verification

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Method: Inorganics, Method  $\overline{\mathcal{SULO}}$ 

The correlation coefficient (r) for the calibration of  $\overline{ClO_{\mathcal{H}}}$  was recalculated.Calibration date:  $\overline{R/27llO}$ 

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, Fo

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

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

					Recalculated	Reported	Acceptable
Type of analysis	Analyte	Standard	Conc. (ug/l)	Area	r or r²	r or r²	(Y/N)
Initial calibration		s1	1.000	0.00355			
		s2	2.5	0.00738	0.999887	0.999773	,
	-	s3	22	0.02			7
	Cor	84	10	0.03			
		SS	20	0.06			_
		9s	40	0.12			
1100 minutes		1001	102	Pandlinge)	5		
Calibration verification			3	10.01	)		
Calibration verification		CCV	30	35,048 110	110	1	<u>.</u>
:	$\rightarrow$		5	10,002	hol		
Calibration verification		)	$\supset$	ر ا ا	-		

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 #. 2417 1836

## **VALIDATION FINDINGS WORKSHEET**

Sample Calculation Verification

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Reviewer:	CZ.
nd reviewer:	

Please Y N Y N Y N Comprecato	e see qualifications below N/A Have results volume N/A Are all detections below N/A Are all detections below N/A Are all detections below N/A Are all detections below N/A Are all detections below N/A Are all detections below N/A Are all detections below N/A Are all detections below N/A Are all detections below N/A Are all detections below N/A Are all detections below N/A Are all detections below N/A Are results below N/A Are all detections below N/A Are all detections below N/A Are results below N/A Are all detections below N/A Are	ng the following equation:	nts?	orted with a positi	ve detect were
#	Sample ID	Analys	Reported Concentration	Calculated Concentration	Acceptable
	Sample ID	Analyte	(mg/kg)	1 (Mells)	(Y/N)
		COH	1000		
-					
-					
	,				
	· · · · · · · · · · · · · · · · · · ·				
		· · · · · · · · · · · · · · · · · · ·			
			<u> </u>		
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Note:_					