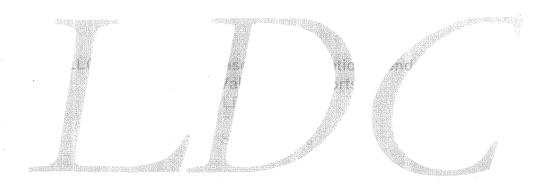
## Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Data Validation Reports LDC# 21257

Semivolatiles



## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

June 16 through June 23, 2008

LDC Report Date:

August 21, 2009

Matrix:

Water

Parameters:

Semivolatiles

Validation Level:

Stage 2B

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2844538

### Sample Identification

PB061608B

PC-40B

H-48B

MC-66BD

MC-66BDRE

MC-65B

MC-66B

MC-66BRE

PC-37B

PC-72B

M-94BX

M-94BXRE

MC-62B

#### Introduction

This data review covers 13 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

#### III. Initial Calibration

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 15.0% for each individual compound and less than or equal to 30.0% for calibration check compounds (CCCs).

In the case where %RSD was greater than 15.0%, the laboratory used a calibration curve to evaluate the compound. All coefficients of determination (r²) were greater than or equal to 0.990.

For the purposes of technical evaluation, all compounds were evaluated against the 30.0% (%RSD) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

Average relative response factors (RRF) for all semivolatile target compounds and system performance check compounds (SPCCs) were greater than or equal to 0.05 as required.

### 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).

For the purposes of technical evaluation, all compounds were evaluated against the 25.0% (%D) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

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 RRF values were greater than or equal to 0.05.

#### 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
SBLK1	6/23/08	Butylbenzylphthalate Di-n-butylphthalate Diethylphthalate Naphthalene	0.31 ug/L 1.8 ug/L 0.22 ug/L 0.060 ug/L	PB061608B PC-40B H-48B

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	Reported	Modified Final
	TIC (RT in minutes)	Concentration	Concentration
PB061608B	Naphthalene	0.085 ug/L	0.085U ug/L
PC-40B	Naphthalene	0.048 ug/L	0.048U ug/L
H-48B	Diethylphthalate	0.20 ug/L	0.20U ug/L
	Naphthalene	0.066 ug/L	0.066U ug/L

Sample FB062408GWAREA1 (from SDG R2844650) was identified as a field blank. No semivolatile contaminants were found in this blank with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB062408GWAREA1	6/24/08	Diethylphthalate Naphthalene	0.18 ug/L 0.075 ug/L	PC-40B H-48B MC-66BD MC-66BDRE MC-65B MC-66B MC-66BRE PC-37B PC-72B M-94BX M-94BX M-94BXRE MC-62B

Sample PB061608B was identified as a pump blank. No semivolatile contaminants were found in this blank with the following exceptions:

Pump Blank ID	Sampling Date	Compound	Concentration	Associated Samples
PB061608B	6/16/08	Bis (2-ethylhexyl) phthalate Naphthalene	0.21 ug/L 0.085 ug/L	PC-40B H-48B MC-66BD MC-66BDRE MC-65B MC-66B MC-66BRE PC-37B PC-72B M-94BX M-94BXRE MC-62B

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

Sample	Compound	Reported Concentration	Modified Final Concentration
PC-40B	Naphthalene	0.048 ug/L	0.048U ug/L
H-48B	Bis(2-ethylhexyl)phthalate Naphthalene Diethylphthalate	0.22 ug/L 0.066 ug/L 0.20 ug/L	0.22U ug/L 0.066U ug/L 0.20U ug/L
MC-66BD	Naphthalene	0.038 ug/L	0.038U ug/L
MC-66BDRE	Naphthalene	0.048 ug/L	0.048U ug/L
MC-66B	Naphthalene	0.056 ug/L	0.056U ug/L
MC-66BRE	Naphthalene	0.056 ug/L	0.056U ug/L
PC-72B	Naphthalene	0.066 ug/L	0.066U ug/L
M-94BX	Naphthalene Diethylphthalate	0.047 ug/L 0.14 ug/L	0.047U ug/L 0.14U ug/L
M-94BXRE	Naphthalene Diethylphthalate	0.047 ug/L 0.19 ug/L	0.047U ug/L 0.19U ug/L
MC-62B	Naphthalene	0.075 ug/L	0.075U ug/L
PC-37B	Diethylphthalate	0.13 ug/L	0.13U ug/L

### 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 with the following exceptions:

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Flag	A or P
LCS/D1 (PB061608B PC-40B H-48B SBLK1)	Di-n-butylphthalate Di-n-octylphthalate	200 (50-120) 138 (50-120)	168 (50-120) 144 (50-120)	-	J+ (all detects) J+ (all detects)	Р
LCS/D1 (PB061608B PC-40B H-48B SBLK1)	1,4-Dioxane Pyridine	42 (50-120) 32 (50-120)	44 (50-120) 33 (50-120)	-	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	Р
LCS/D2 (MC-66BD MC-66BDRE MC-65B MC-66B MC-66BRE PC-37B PC-72B M-94BX M-94BXRE MC-62B SBLK2)	Di-n-butylphthalate Di-n-octylphthalate Pyridine	134 (50-120) 140 (50-120) 180 (50-120)	128 (50-120) 134 (50-120)	- - -	J+ (all detects) J+ (all detects) J+ (all detects)	Р

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Flag	A or P
LCS/D2 (MC-66BD MC-66BDRE MC-65B MC-66B MC-66BRE PC-37B PC-72B M-94BX M-94BXRE MC-62B SBLK2)	1,4-Dioxane	40 (50-120)	40 (50-120)	-	J- (all detects) UJ (all non-detects)	Р

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

All internal standard areas and retention times were within QC limits with the following exceptions:

Sample	Internal Standards	Area (Limits)	Compound	Flag	A or P
MC-66BD	Perylene-d12	0 (153834-615336)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) R (all non-detects)	А
MC-66B	Perylene-d12	15000 (153834-615336)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) UJ (all non-detects)	А
MC-66BDRE	Perylene-d12	0 (141763-567050)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) R (all non-detects)	A

Sample	Internal Standards	Area (Limits)	Compound	Flag	A or P
MC-66BRE	Perylene-d12	0 (141763-567050)	Di-n-octylphthalate Benzo (b)fluoranthene Benzo (k)fluoranthene Benzo (a) pyrene Indeno (1,2,3-cd) pyrene Dibenz (a, h) anthracene Benzo (g, h, i) perylene	J (all detects) R (all non-detects)	A
M-94BX	Perylene-d12	0 (141763-567050)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) R (all non-detects)	А
M-94BXRE	Perylene-d12	0 (141763-567050)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) R (all non-detects)	А

### XI. Target Compound Identifications

Raw data were not reviewed for this SDG.

### XII. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R2844538	All compounds reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

### XIII. Tentatively Identified Compounds (TICs)

Raw data were not reviewed for this SDG.

### XIV. System Performance

Raw data were not reviewed for this SDG.

### XV. Overall Assessment

The overall assessment of data was acceptable. In the case where more than one result was reported for an individual sample, the least technically acceptable results were rejected as follows:

Sample	Compound	Flag	A or P
MC-66BDRE MC-66BRE M-94BXRE	All TCL compounds	x	А

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

### XVI. Field Duplicates

Samples MC-66BD and MC-66B and samples MC-66BDRE and MC-66BRE were identified as field duplicates. No semivolatiles were detected in any of the samples with the following exceptions:

	Concentral	tion (ug/L)				
Compound	MC-66BD	MC-66B	RPD (Limits)	Difference (Limits)	Flags	AorP
1,4-Dioxane	0.36	0.38	-	0.02 (≤2.0)	-	-
Naphthalene	0.038	0.056	-	0.018 (≤0.20)	-	-

	Concentrat	tion (ug/L)				
Compound	MC-66BDRE	MC-66BRE	RPD (Limits)	Difference (Limits)	Flags	A or P
1,4-Dioxane	0.36	0.32	-	0.04 (≤2.0)	-	-
Naphthalene	0.048	0.056	-	0.008 (≤0.20)	-	-

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG R2844538

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R2844538	PB061608B PC-40B H-48B MC-66BD MC-66BDRE MC-65B MC-66B MC-66BRE PC-37B PC-72B M-94BX M-94BXRE MC-62B	Di-n-butylphthalate Di-n-octylphthalate	J+ (all detects) J+ (all detects)	Р	Laboratory control samples (%R) (I)
R2844538	PB061608B PC-40B H-48B	1,4-Dioxane Pyridine	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	Р	Laboratory control samples (%R) (I)
R2844538	MC-66BD MC-66BDRE MC-66B MC-66BRE PC-37B PC-72B M-94BX M-94BXRE MC-62B	1,4-Dioxane	J- (all detects) UJ (all non-detects)	Р	Laboratory control samples (%R) (I)
R2844538	MC-66BD MC-66BDRE MC-65B MC-66B MC-66BRE PC-37B PC-72B M-94BX M-94BXRE MC-62B	Pyridine	J+ (all detects)	Р	Laboratory control samples (%R) (l)
R2844538	MC-66BD MC-66BDRE MC-66BRE M-94BX M-94BXRE	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) R (all non-detects)	А	Internal standards (area) (i)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R2844538	MC-66B	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) UJ (all non-detects)	А	Internal standards (area) (i)
R2844538 PB061608B PC-40B H-48B MC-66BD MC-66BDRE MC-66B MC-66B MC-66BRE PC-37B PC-72B M-94BX M-94BXRE MC-62B		All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
R2844538	MC-66BDRE MC-66BRE M-94BXRE	All TCL compounds	х	Α	Overall assessment of data (o)

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG R2844538

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
R2844538	PB061608B	Naphthalene	0.085U ug/L	А	bl
R2844538	PC-40B	Naphthalene	0.048U ug/L	А	bl
R2844538	H-48B	Diethylphthalate Naphthalene	0.20U ug/L 0.066U ug/L	А	bl

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles- Field Blank Data Qualification Summary - SDG R2844538

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
R2844538	PC-40B	Naphthalene	0.048U ug/L	Α	bf,bp
R2844538	H-48B	Naphthalene	0.066U ug/L	А	bf,bp

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
R2844538	H-48B	Bis(2-ethylhexyl)phthalate	0.22U ug/L	А	bp
R2844538	H-48B	Diethylphthalate	0.20U ug/L	А	bf
R2844538	MC-66BD	Naphthalene	0.038U ug/L	А	bf,bp
R2844538	MC-66BDRE	Naphthalene	0.048U ug/L	А	bf,bp
R2844538	MC-66B	Naphthalene	0.056U ug/L	А	bf,bp
R2844538	MC-66BRE	Naphthalene	0.056U ug/L	А	bf,bp
R2844538	PC-72B	Naphthalene	0.066U ug/L	А	bf,bp
R2844538	M-94BX	Naphthalene	0.047U ug/L	А	bf,bp
R2844538	M-94BX	Diethylphthalate	0.14U ug/L	А	bf
R2844538	M-94BXRE	Naphthalene	0.047U ug/L	А	bf,bp
R2844538	M-94BXRE	Diethylphthalate	0.19U ug/L	А	bf
R2844538	MC-62B	Naphthalene	0.075U ug/L	А	bf,bp
R2844538	PC-37B	Diethylphthalate	0.13U ug/L	А	bf

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

LDC #:_	21257A2a
SDG #:	R2844538

Stage 2B

Laboratory: Columbia Analytical Services

Date: 8/1 /69 Reviewer: 3V 2nd Reviewer:

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/16-23/08
11.	GC/MS Instrument performance check	A	
111.	Initial calibration	A	2 RSD r
IV.	Continuing calibration/ICV	JVGWA	COV/101 < 25 %
V.	Blanks	SW	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	N	Client spec
VIII.	Laboratory control samples	SW	Client spec US 10
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	SW	
XI.	Target compound identification	N	
XII.	Compound quantitation/CRQLs	N	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	N	
XV.	Overall assessment of data	SW	
XVI.	Field duplicates	SW	$D_1 = 4.7$ $D_2 = 5.8$
XVII.	Field blanks	SW	PB=1 +B0624084NARZAI(R2844600)

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:

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	VULTEY						· · · · · · · · · · · · · · · · · · ·
1 1	PB061608B	11	M-94B <b>X</b>	<del>1</del> 21 /	SBLKI	31	
2 1	PC-40¢ B	12	M-94BRE	22 Y	SBYEZ	32	
3 1	H-48B	13	MC-62B	23	,	33	
4	мс-666ВD В	14		24		34	
5	MC-066BDRE DY	15		25		35	
6	MC-65B	16		26		36	
7	мс-66B <b>Д</b>	17		27		37	,
8	MC-66BRE 🗓 ✓	18		28		38	
9	PC-37B	19		29		39	
10	PC-72B	20		30		40	

# **VALIDATION FINDINGS WORKSHEET**

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

A. Phenol**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenol**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene™	VV. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol™	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	171. octachlorostyrene
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	uuu 1,4. Dioxane
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	WV.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	www.

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

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# VALIDATION FINDINGS WORKSHEET **Blanks**

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

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

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

Was a method blank analyzed for each matrix? Was a method blank analyzed for each concentration preparation level?

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

 $\sqrt{N N/A}$  Was the blank contaminated? If yes, please see qualification below. Blank extraction date:  $\frac{b}{a^2/a^2}$  Blank analysis date:  $\frac{b}{a^2/a^2}$ 

Associated Samples: Conc. units: ଏବ

Blank ID

Compound

SBUKI

0.3 ∞,

AAA

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2

(79)

Sample Identification 0,066/4 U. 20 /W  $\Lambda$ 

0.048/4

D, 085

0.060 0.22

5

0.5

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

_		 	 	

LDC # 2127428 SDG#:

# VALIDATION FINDINGS WORKSHEET Field Blanks

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

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

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

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

Associated sample units: الم Blank units:

Sampling date:

PB Field blank type. (circle

AM except

	Field Diamk type: (circle one) Field Diamk / Kimsale / Other.	rield Dialik	/ Ririsale / Oli	IIEI.	Associat	Associated Samples.			\		
	Compound	Blank ID				š	Sample Identification	tion			
			٦	~	4	5	7	8	Ф	11	γ
20.1	333	0.21		0. 22/y							
9. 7	5	0,085	0,085 0,048/11 0,066		b, 038 /4	0,048/4	n/250 0	N/2500	n/ 990'0	14 6,078/4 0,048/4 0.050/4 0.050/4 0,066/4 0.047 /4 0.047 /4	0.047 /4
	CRQL										

Associated sample units: Blank units:\_

s Love

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

d Blank / Rinsate / Other: Associated Samples:	ink ID Sample Identification	13	, 2)	0,085 0,075/4			
/ Rinsate / Other:		13	,	W/2/0,0	, /		<b>***</b>
Field Blank	Blank ID		12,0	5800			
Field blank type: (circle one) Field Blank / Rinsate / Other:	Compound		133				CROI

5x- phthalatio 2x - all others

See Carry LDC #: 21257 SDG#:

# VALIDATION FINDINGS WORKSHEET

Field Blanks

Page: Zof Reviewer: TVC 2nd Reviewer:\_

(+q)

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

Blank units: "9 / Associated sample units: "49 / Associated sample units: "40 / Associated sam

Were field blanks identified in this SDG?

Y N/A

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

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

Associated Samples:

All except

Compound	F <i>B 06240</i> 2 Blank ID	FB06240 & GWAREA /		,	Sa	Sample Identification	tion			
	801 be/ 9	2 8	3	4	8	7	×	6 41	9	1
11	0.18		0.20/4					0.13/4		0.14/4
S	5/0.0	0.048/4	0.066/4	18600	6,075 0.048/4 0.066/4 0.038/4 0.048/4 0.055 1/4 0.056/4 0	0.65% /U	17950.0	•	0.066/4 6.047/4	6.047 M
			,	, ,	•	,	• (			
CROL										

Associated sample units:\_ Sampling date: Blank units:

Ş Same

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

Associated Samples:

Compound	Blank ID			Sample k	Sample Identification		
	≠B	۲)	3				
71	0.18	16 0.18 0.19/4					
S	26.0	1/2500 h/540.0 250.0 S	h/560.0				
			•				
CRQL							

5x Phthalates 2x all others

LDC#27424 SDG #: La (2000)

# VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Page: of / Reviewer: 2nd Reviewer:

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

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

| N/A | N/A | Was a LCS required?

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

	Ś	\$					3	<u>`</u>			_														
A I I I I I I I I I I I I I I I I I I I	1 + A.A. (D	J-1/4T 13	7 16th /b	7-115			7	1	T+ 4.4.10																
Accordated Servoles	1-2 52171	l					4-13 SBUX																		
RPD (Limits)		)	(	(	( )	(	(	)		( )	( )	(	`	`	)	( )	· )	(	( )	`	•	( )	( )	(	
LCSD %R (Limits)	168 (20-130)	( ) 74	144 ( )	33 ( 1)	( )	•	(00/-05) 80/	_	34 ( ) ,	( )	(	(	( )	( )	(	( )	(	( )	( )	( )	( )	( )	( )	( )	]( )
LCS %R (Limits)	1061-65) 005		( ) 861	32 ( 1 )	( )	( )	134 (50-120)	( 1 ) 04	140	`	(	( )	( )	( )	( )	( )	( )		( )	( )	( )	( )	( )	( )	
Compound	XX	ทุกท	キキト	204	120		XX	nnn	FFF	KKK															
CSACSD ID	1 45/20 1						16s/b 2																		
# Date																									

LDC# 2/257 ANA SDG#:

# VALIDATION FINDINGS WORKSHEET Internal Standards

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

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

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

Were all internal standard area counts within -50 to +100 of the associated calibration standard? Y N/A

Were the retention times of the internal standards within +/- 30 seconds of the retention times of the associated calibration standard?

		_							7#4	KK	<u> </u>					
Qualifications	J/R/A (i		J/MJ/A	J/R/A				>	(qual FFF 669, 444)	111						
RT (Limits)				) )												
Area (Limits)	( 153834- (153836)		1 5 000 21	0 (141763-567050	0	0	/) 0	>								
Internal Standard	PRW		PRY				1									
Sample ID	4		7	 5	Ø		اع	-								
Date																
#																_

\* QC limits are advisory IS1 (DCB) = 1,4-Dichlorobenzene-d4 IS2 (NPT) = Naphthalene-d8 IS3 (ANT) = Acenaphthene-d10

IS4 (PHN) = Phenanthrene-d10 IS5 (CRY) = Chrysene-d12 IS6 (PRY) = Perylene-d12

he recoveries - no R.T.s.)

LDC#: 21257 479 SDG#:

# **VALIDATION FINDINGS WORKSHEET** Overall Assessment of Data

Page: \\_of\_

Reviewer: 2nd Reviewer:

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

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

All available information pertaining to the data were reviewed using professional judgement to compliment the determination of the overall quality of the data.

Y N N/A

Was the overall quality and usability of the data acceptable?

#	Date	Sample ID	Finding	Associated Samples	Qualifications	
		587	Remus for IS work	Is waterde contenia	(0) ¥/X	6)
			-			
]	Comments					

LDC #:_	212574 29
SDG #:	Su Corey

### VALIDATION FINDINGS WORKSHEET Field Duplicates

Page:	of
Reviewer:	30%/
2nd reviewer:	47

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

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

Were field duplicate pairs identified in this SDG? Were target compounds identified in the field duplicate pairs?

	Concentrati	one us 1	Diff
. Compound	4	7	- <del>RPD-</del>
нии	0.36	<del>4</del> , 6,38	0,02 (= 2,0)
S	0.038	0,056	0.018 (60.20)
		<u> </u>	<u>.</u>

	Concentration	us/L,	
Compound	5	8	RPD
unn	0, 36	0.32	0,04 (= 2,0)
	0.048	0,056	0.008 (40,20)
·			

	Concentration ( )		
Compound			RPD
			·
	·		
·	·		·

·	Concentration (		
Compound			RPD .
·			

### LDC Report# 21257B2a

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

**Project/Site Name:** 

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

June 24 through June 27, 2008

LDC Report Date:

September 17, 2009

Matrix:

Water

Parameters:

Semivolatiles

Validation Level:

Stage 4

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2844650

### Sample Identification

M-44B

M-95B

H-49AB

M-68B

FB062408GWAREA1

M-7BBMS

MC-45B

M-7BBMSD

MC-53B

M-23B

M-23BRE

MC-97B

VIO 01 D

MC-94B

MC-94BRE

MW-16B

M-5AB

EB062608GW3

M-61B

M-88BB

M-7BB

M-67B

M-6AB

M-57AB

M-57ABRE

### Introduction

This data review covers 24 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

#### III. Initial Calibration

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 15.0% for each individual compound and less than or equal to 30.0% for calibration check compounds (CCCs).

In the case where %RSD was greater than 15.0%, the laboratory used a calibration curve to evaluate the compound. All coefficients of determination (r²) were greater than or equal to 0.990.

For the purposes of technical evaluation, all compounds were evaluated against the 30.0% (%RSD) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

Average relative response factors (RRF) for all semivolatile target compounds and system performance check compounds (SPCCs) were greater than or equal to 0.05 as required.

### 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).

For the purposes of technical evaluation, all compounds were evaluated against the 25.0% (%D) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

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

Date	Compound	%D	Associated Samples	Flag	A or P
6/26/08	Pyridine	44.4	All samples in SDG R2844650	J+ (all detects)	А

All of the continuing calibration RRF values were greater than or equal to 0.05.

### \*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
SBLK2	6/30/08	Butylbenzylphthalate Di-n-butylphthalate Diethylphthalate Bis(2-ethylhexyl)phthalate Naphthalene	0.39 ug/L 1.7 ug/L 0.13 ug/L 0.25 ug/L 0.040 ug/L	MC-45B MC-53B M-23B M-23BRE MC-97B MC-94B MC-94BRE MW-16B M-5AB EB062608GW3 M-61B M-88BB M-7BB M-67B M-67B M-67B M-57AB M-57AB M-57ABRE M-95B M-95B M-68B

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	Reported	Modified Final
	TIC (RT in minutes)	Concentration	Concentration
MC-53B	Butylbenzylphthalate	0.71 ug/L	0.71U ug/L
M-23B	Diethylphthalate	0.17 ug/L	0.17U ug/L
	Naphthalene	0.056 ug/L	0.056U ug/L
M-23BRE	Diethylphthalate	0.20 ug/L	0.20U ug/L
	Naphthalene	0.066 ug/L	0.066U ug/L

Sample	Compound	Reported	Modified Final
	TIC (RT in minutes)	Concentration	Concentration
MC-97B	Di-n-butylphthalate	1.5 ug/L	1.5U ug/L
	Diethylphthalate	0.20 ug/L	0.20U ug/L
	Naphthalene	0.067 ug/L	0.067U ug/L
MC-94B	Bis(2-ethylhexyl)phthalate	0.75 ug/L	0.75U ug/L
	Naphthalene	0.070 ug/L	0.070U ug/L
MC-94BRE	Bis(2-ethylhexyl)phthalate	0.75 ug/L	0.75U ug/L
	Naphthalene	0.070 ug/L	0.070U ug/L
MW-16B	Bis(2-ethylhexyl)phthalate	0.54 ug/L	0.54U ug/L
M-5AB	Naphthalene	0.048 ug/L	0.048U ug/L
EB062608GW3	Diethylphthalate	0.38 ug/L	0.38U ug/L
	Naphthalene	0.050 ug/L	0.050U ug/L
M-67B	Naphthalene	0.049 ug/L	0.049U ug/L
M-6AB	Bis(2-ethylhexyl)phthalate	0.26 ug/L	0.26U ug/L
	Naphthalene	0.059 ug/L	0.059U ug/L
M-57AB	Naphthalene	0.040 ug/L	0.040U ug/L
M-57ABRE	Naphthalene	0.040 ug/L	0.040U ug/L
M-95B	Bis(2-ethylhexyl)phthalate	0.022 ug/L	0.022U ug/L
	Naphthalene	0.038 ug/L	0.038U ug/L
M-68B	Naphthalene	0.041 ug/L	0.041U ug/L

Sample EB062608GW3 was identified as an equipment blank. No semivolatile contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB062608GW3	6/26/08	Diethylphthalate Naphthalene	0.38 ug/L 0.050 ug/L	MW-16B M-5AB

Sample FB062408GWAREA1 was identified as a field blank. No semivolatile contaminants were found in this blank with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB062408GWAREA1	6/16/08	Naphthalene Diethylphthalate	0.075 ug/L 0.18 ug/L	M-44B H-49AB MC-45B MC-53B M-23BRE M-23BRE MC-97B MC-94B MC-94BRE M-61B M-88BB M-7BB M-67B M-67B M-6AB M-57AB M-57ABRE M-95B M-68B

<sup>\*</sup>Sample PB061608B (from SDG R2844538) was identified as a pump blank. No semivolatile contaminants were found in this blank with the following exceptions:

Pump Blank ID	Sampling Date	Compound	Concentration	Associated Samples
PB061608B	6/16/08	Bis(2-ethylhexyl)phthalate Naphthalene	0.21 ug/L 0.085 ug/L	M-44B H-49AB MC-45B MC-53B M-23BRE M-23BRE MC-97B MC-94B MC-94BRE M-61B M-88BB M-7BB M-67B M-67B M-6AB M-57AB M-57ABRE M-95B M-68B

<sup>\*</sup>Removed samples MW-16B and M-5AB from above table.

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

Sample	Compound	Reported Concentration	Modified Final Concentration
H-49AB	Naphthalene	0.080 ug/L	0.080U ug/L
	Diethylphthalate	0.15 ug/L	0.15U ug/L

Sample	Compound	Reported Concentration	Modified Final Concentration
M-23B	Naphthalene	0.056 ug/L	0.056U ug/L
	Diethylphthalate	0.17 ug/L	0.17U ug/L
M-23BRE	Naphthalene	0.066 ug/L	0.066U ug/L
	Diethylphthalate	0.20 ug/L	0.20U ug/L
MC-97B	Naphthalene	0.067 ug/L	0.067U ug/L
	Diethylphthalate	0.20 ug/L	0.20U ug/L
MC-94B	Bis(2-ethylhexyl)phthalate	0.75 ug/L	0.75U ug/L
	Naphthalene	0.070 ug/L	0.070U ug/L
MC-94BRE	Bis(2-ethylhexyl)phthalate	0.75 ug/L	0.75U ug/L
	Naphthalene	0.070 ug/L	0.070U ug/L
M-5AB	Naphthalene	0.048 ug/L	0.048U ug/L
M-67B	Naphthalene	0.049 ug/L	0.049U ug/L
M-6AB	Bis(2-ethylhexyl)phthalate	0.26 ug/L	0.26U ug/L
	Naphthalene	0.059 ug/L	0.059U ug/L
M-57AB	Naphthalene	0.040 ug/L	0.040U ug/L
M-57ABRE	Naphthalene	0.040 ug/L	0.040U ug/L
M-95B	Bis(2-ethylhexyl)phthalate	0.22 ug/L	0.22U ug/L
	Naphthalene	0.038 ug/L	0.038U ug/L
M-68B	Naphthalene	0.041 ug/L	0.041U ug/L

### VI. Surrogate Spikes

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

### VII. Matrix Spike/Matrix Spike Duplicates

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

Spike ID (Associated Samples)	Compound	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
M-7BBMS/MSD (M-7BB)	Di-n-butylphthalate	320 (50-150)	200 (50-150)	46 (≤30)	J+ (all detects)	A
M-7BBMS/MSD (M-7BB)	Pyridine	38 (50-150)	34 (50-150)	-	J- (all detects) UJ (all non-detects)	Α

Although the MSD percent recovery (%R) was not within QC limits for one compound, the MS percent recovery (%R) was within QC limits and no data were qualified.

### VIII. Laboratory Control Samples (LCS)

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

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Flag	A or P
LCS/D1 (MW-16B M-5AB EB062608GW3 SBLK1)	Di-n-butylphthalate Di-n-octylphthalate	134 (50-120) 140 (50-120)	128 (50-120) 134 (50-120)	-	J+ (all detects) J+ (all detects)	P
LCS/D1 (MW-16B M-5AB EB062608GW3 SBLK1)	1,4-Dioxane	40 (50-120)	40 (50-120)	* <u>-</u> -	J- (all detects) UJ (all non-detects)	Р

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Flag	A or P
LCS/D2 (MC-45B MC-53B M-23B M-23BRE MC-97B MC-94B MC-94BRE MW-16B M-5AB EB062608GW3 M-61B M-88BB M-7BB M-67B M-67B M-67B M-67B M-67B M-67B M-57AB M-57AB M-57ABRE M-95B M-68B SBLK2)	Di-n-butylphthalate	460 (50-120)	480 (50-120)	-	J+ (all detects)	Р
LCS/D2 (MC-45B MC-53B M-23BRE M-23BRE MC-97B MC-94B MC-94BRE MW-16B M-5AB EB062608GW3 M-61B M-88BB M-7BB M-67B M-67B M-67B M-57AB M-57AB M-57ABRE M-95B M-68B SBLK2)	Pyridine	14 (50-120)	0 (50-120)	200 (≤30)	J- (all detects) R (all non-detects)	P

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

All internal standard areas and retention times were within QC limits with the following exceptions:

Sample	Internal Standards	Area (Limits)	Compound	Flag	A or P
M-23B	Perylene-d12	0 (141763-567050)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) R (all non-detects)	A
M-23BRE	Perylene-d12	0 (127170-508680)	Di-n-octylphthalate Benzo (b) fluoranthene Benzo (k) fluoranthene Benzo (a) pyrene Indeno (1,2,3-cd) pyrene Dibenz (a,h) anthracene Benzo (g,h,i) perylene	J (all detects) R (all non-detects)	A
MC-94B	Perylene-d12	46528 (127170-508680)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) R (all non-detects)	A
MC-94BRE	Perylene-d12	27916 (127170-508680)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) R (all non-detects)	A
M-57AB	Perylene-d12	24566 (127170-508680)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) R (all non-detects)	A
M-57ABRE	Perylene-d12	1051 (127170-508680)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) R (all non-detects)	A

### XI. Target Compound Identifications

All target compound identifications were within validation criteria.

### XII. Project Quantitation Limit

All project quantitation limits were within validation criteria.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R2844650	All compounds reported below the PQL.	J (all detects)	А

### XIII. Tentatively Identified Compounds (TICs)

Tentatively identified compounds were not reported by the laboratory.

### XIV. System Performance

The system performance was acceptable.

#### XV. Overall Assessment

The overall assessment of data was acceptable. In the case where more than one result was reported for an individual sample, the least technically acceptable results were rejected as follows:

Sample	Compound	Flag	A or P
M-23BRE MC-94BRE M-57ABRE	All TCL compounds	×	A

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

### XVI. Field Duplicates

No field duplicates were identified in this SDG.

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG R2844650

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R2844650	M-44B H-49AB FB062408GWAREA1 MC-45B MC-53B M-23B M-23BRE MC-97B MC-94B MC-94BRE MW-16B M-5AB EB062608GW3 M-61B M-88BB M-7BB M-67B M-6AB M-57AB M-57ABRE M-95B M-95B M-95B M-68B	Pyridine	J+ (all detects)	A	Continuing calibration (ICV %D) (c)
R2844650	M-7BB	Di-n-butylphthalate	J+ (all detects)	А	Matrix spike/Matrix spike duplicates (%R)(RPD) (m,ld)
R2844650	M-7BB	Pyridine	J- (all detects) UJ (all non-detects)	А	Matrix spike/Matrix spike duplicates (%R)(m)
R2844650	MW-16B M-5AB EB062608GW3	Di-n-butylphthalate Di-n-octylphthalate	J+ (all detects) J+ (all detects)	Р	Laboratory control samples (%R) (I)
R2844650	MW-16B M-5AB EB062608GW3	1,4-Dioxane	J- (all detects) UJ (all non-detects)	Р	Laboratory control samples (%R) (I)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R2844650	MC-45B MC-53B M-23B	Di-n-butylphthalate	J+ (all detects)	Р	Laboratory control samples (%R) (I)
	M-23BRE MC-97B MC-94B				
	MC-94BRE MW-16B M-5AB				
	EB062608GW3 M-61B M-88BB				
	M-7BB M-67B M-6AB M-57AB		·		
	M-57ABRE M-95B M-68B				
R2844650	MC-45B MC-53B	Pyridine	J- (all detects) R (all non-detects)	Р	Laboratory control samples (%R) (I)
	M-23B M-23BRE MC-97B		(		Campios (vary ty
	MC-94B MC-94BRE MW-16B M-5AB	·			
	EB062608GW3 M-61B M-88BB			-	
	M-7BB M-67B M-6AB M-57AB				
	M-57ABRE M-95B M-68B				
R2844650	M-23B M-23BRE MC-94B	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene	J (all detects) R (all non-detects)	Α	Internal standards (area) (i)
·	MC-94BRE M-57AB M-57ABRE	Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene			

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R2844650	M-44B H-49AB FB062408GWAREA1 MC-45B MC-53B M-23B M-23BRE MC-97B MC-94B MC-94BRE MW-16B M-5AB EB062608GW3 M-61 B M-88BB M-7BB M-67B M-6AB M-57AB M-57ABRE M-95B M-95B M-68B	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
R2844650	M-23BRE MC-94BRE M-57ABRE	All TCL compounds	Х	А	Overall assessment of data (o)

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG R2844650

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
R2844650	MC-53B	Butylbenzylphthalate	0.71U ug/L	А	bl
R2844650	M-23B	Diethylphthalate Naphthalene	0.17U ug/L 0.056U ug/L	А	bl
R2844650	M-23BRE	Diethylphthalate Naphthalene	0.20U ug/L 0.066U ug/L	А	bl
R2844650	MC-97B	Di-n-butylphthalate Diethylphthalate Naphthalene	1.5U ug/L 0.20U ug/L 0.067U ug/L	А	bl
R2844650	MC-94B	Bis(2-ethylhexyl)phthalate Naphthalene	0.75U ug/L 0.070U ug/L	А	bl
R2844650	MC-94BRE	Bis(2-ethylhexyl)phthalate Naphthalene	0.75U ug/L 0.070U ug/L	А	bl

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
R2844650	MW-16B	Bis(2-ethylhexyl)phthalate	0.54U ug/L	А	bl
R2844650	M-5AB	Naphthalene	0.048U ug/L	А	bl
R2844650	EB062608GW3	Diethylphthalate Naphthalene	0.38U ug/L 0.050U ug/L	А	bl
R2844650	M-67B	Naphthalene	0.049U ug/L	А	bl
R2844650	M-6AB	Bis(2-ethylhexyl)phthalate Naphthalene	0.26U ug/L 0.059U ug/L	A	bl
R2844650	M-57AB	Naphthalene	0.040U ug/L	А	bl
R2844650	M-57ABRE	Naphthalene	0.040U ug/L	А	bl
R2844650	M-95B	Bis(2-ethylhexyl)phthalate Naphthalene	0.22U ug/L 0.038U ug/L	А	bl
R2844650	M-68B	Naphthalene	0.041U ug/L	А	bl

### \*Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles- Field Blank Data Qualification Summary - SDG R2844650

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
R2844650	H-49AB	Diethylphthalate	0.15U ug/L	А	bf
R2844650	H-49AB	Naphthalene	0.080U ug/L	Α	bf,bp
R2844650	M-23B	Diethylphthalate	0.17U ug/L	А	bf
R2844650	M-23B	Naphthalene	0.056U ug/L	А	bf,bp
R2844650	M-23BRE	Naphthalene	0.066U ug/L	А	bf,bp
R2844650	M-23BRE	Diethylphthalate	0.20U ug/L	Α	bf
R2844650	MC-97B	Naphthalene	0.067U ug/L	Α	bf,bp

SDG	Sample	Compound	Modified Final Concentration	Å or P	Code
R2844650	MC-97B	Diethylphthalate	0.20U ug/L	А	bf
R2844650	MC-94B	Naphthalene	0.070U ug/L	А	bf,bp
R2844650	MC-94B	Bis(2-ethylhexyl)phthalate	0.75U ug/L	А	bp
R2844650	MC-94BRE	Naphthalene	0.070U ug/L	А	bf,bp
R2844650	MC-94BRE	Bis(2-ethylhexyl)phthalate	0.75U ug/L	А	bp
R2844650	M-5AB	Naphthalene	0.048U ug/L	А	be,bf
R2844650	M-67B	Naphthalene	0.049U ug/L	А	bf,bp
R2844650	M-6AB	Naphthalene	0.059U ug/L	Α	bf,bp
R2844650	M-6AB	Bis(2-ethylhexyl)phthalate	0.26U ug/L	· A	bp
R2844650	M-57AB	Naphthalene	0.040U ug/L	А	bf,bp
R2844650	M-57ABRE	Naphthalene	0.040U ug/L	А	bf,bp
R2844650	M-95B	Naphthalene	0.038U ug/L	Α	bf,bp
R2844650	M-95B	Bis(2-ethylhexyl)phthalate	0.22U ug/L	Α	bp
R2844650	M-68B	Naphthalene	0.041U ug/L	Α	bf,bp

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

LDC #:	2125/B2a
SDG #:	R2844650

Stage 4 R2844650

Page: | of Reviewer:

Laboratory: Columbia Analytical Services

2nd Reviewer:

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 4/24 - 27/08
11.	GC/MS Instrument performance check	A	
· III.	Initial calibration	A	2 RSD P
IV.	Continuing calibration/ICV	SW	COV/OV EZEZ
V.	Blanks	SW	
VI.	Surrogate spikes	A	A A A A A A A A A A A A A A A A A A A
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	SN)	LCS/D
IX.	Regional Quality Assurance and Quality Control	N	
Х.	Internal standards	SW	
XI.	Target compound identification	<u> </u>	
XII.	Compound quantitation/CRQLs	A	
XIII.	Tentatively identified compounds (TICs)	N	
(IV.	System performance	A	
XV.	Overall assessment of data	SW)	
(VI.	Field duplicates	N	
VII.	Field blanks	SW)	+B=3 EB=13 PB=PB061608B +rem R2844538

Note:

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate FB = Field blank D = Duplicate

TB = Trip blank

EB = Equipment blank

Validated Samples:

	wate	Y					
1 1	M-44B	11	MW-16B	21	M-95B	31	SBUEL
2	H-49AB	12	M-5AB	22	M-68B	32	SBUCY
7 3 1	FB062408GWAREA1	13	EB062608GW3	23	M-7BBMS	33	
4	MC-45B	14	M-61B	24	M-7BBMSD	34	
5	MC-53B	15	M-88BB	25		35	
6	M-23B	16	M-7BB	26		36	
7	M-23BRE	17	M-67B	27		37	
8	MC-97B	18	М-6АВ	28		38	
9	MC-94B	19	M-57AB	29		39	
10	MC-94BRE	20	M-57ABRE	30		40	

### **VALIDATION FINDINGS CHECKLIST**

Method: Semivolatiles (EPA SW 846 Method 8270C)

		T	<del></del>	T	
Validation Area	Yes	No	NA	Findings/Comr	nents
All technical holding times were met.	1-	1			
Cooler temperature criteria was met.		1			
Decembration and Comments of the Comments of t					
Were the DFTPP performance results reviewed and found to be within the specified criteria?	1/				
Were all samples analyzed within the 12 hour clock criteria?					1,1
al Maliarocate Alicas					
Did the laboratory perform a 5 point calibration prior to sample analysis?	/				
Were all percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?	/	1		er og kommune Kommune blever	
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?					
iya sooofii ya su su daa a					
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?	/			s sais said Sainteir Mark	
Were all percent differences (%D) ≤ 25% and relative response factors (RRF) ≥ 0.05?		/			
Was a method blank associated with every sample in this SDG?					
Was a method blank analyzed for each matrix and concentration?					- Age 24
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		San Francisco	ilo:		Talles Statistics and States
		20 .70 20			
Were all surrogate %R within QC limits?		-			
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?			7		1,43.4
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?			7		- A48 1
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.					in Marky
Was a MS/MSD analyzed every 20 samples of each matrix?	$\exists$		$\dashv$		
Were the MS/MSD percent recoveries (%R) and the relative percent differences			$\neg$	<u>an ann an Aire ann an Aire ann an Aire</u> An Aige ann an Aire	
RPD) within the QC limits?	****				
				and the second second	
Was an LCS analyzed for this SDG?					

LDC#: 21257 Bra SDG#: Sce Cover

### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 2
Reviewer: 500
2nd Reviewer: 0

-Validation Area	Y	'es	No	NA		Finding	s/Comments	s'
Was an LCS analyzed per extraction batch?	_/<				reign -			å i ja
Were the LCS percent recoveries (%R) and relative percent difference (RPD) we the QC limits?	vithin	-						
Zerostore Silver Silver voltare de la company de la compan								
Were performance evaluation (PE) samples performed?			1					
Were the performance evaluation (PE) samples within the acceptance limits?		g-8- o	8773 SOA			10 m ( 10 m ( 10 m 10 m 10 m 10 m 10 m 1		
						and the second		
Were internal standard area counts within -50% or +100% of the associated calibration standard?			/					
Were retention times within ± 30 seconds from the associated calibration stand	ard?		OVERNOR I	359 11 187	- (2.50 - 8.10 - 10.00 - 10.00 - 10.00 - 10.00 - 10.00 - 10.00 - 10.00 - 10.00 - 10.00 - 10.00 - 10.00 - 10.00			Vilita-kassa legistikki
XI Toop so the second s								
Were relative retention times (RRTs) within ± 0.06 RRT units of the standard?	_ /							
Did compound spectra meet specified EPA "Functional Guidelines" criteria?		$\dashv$						
Were chromatogram peaks verified and accounted for?								
Were the correct internal standard (IS), quantitation ion and relative response fa (RRF) used to quantitate the compound?	actor	4						
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions dry weight factors applicable to level IV validation?	s and	1	/					-
XIII Seriesius Program (Program of Control o								
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 reference spectra?	d the						in Parking.	
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.		1		eri rezi pr				
Overall assessment of data was found to be acceptable.	/	1	14	1	ina spec			
Field duplicate pairs were identified in this SDG.								A PAPER IN
Target compounds were detected in the field duplicates.		T	$\dashv$	7		<u> </u>	<u>and the second </u>	
Field blanks were identified in this SDG.		1			er Ali			
		$\dagger$	+	$\dashv$				80. 1 · · · · · · · · · · · · · · · · · ·
Target compounds were detected in the field blanks.						<u> </u>		

# VALIDATION FINDINGS WORKSHEET

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

A. Phenol**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenoi**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C, 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	W. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH, 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g.h.i)perviene
E. 1,4-Dichlorobenzene™	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-buty/phthalate	MMM. Bis(2-Chlorolsopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene™	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Apiline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	000. N-Nitrosodimethylamine
H. 2,2'-0xybis(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-propylamine⁴	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR, Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	OO. 4-Nitroaniline	DDD, Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	TTT. Octo chlory ct.
M. Isophorone	BB. 2-Nitroaniline	QQ, N-Nitrosodiphenylamine (1)**		
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	92	
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	WWW.

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

LDC#: 21 257 Bzg SDG#:

SDG #: 34 (227) METHOD: GC/MS BNA (EPA SW 848 Method 8270C)

VALIDATION FINDINGS WORKSHEET

Continuing Calibration

2nd Reviewer:

Page: Reviewer:

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

N N/A

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

	71		7		_	-		, , , , , , , , , , , , , , , , , , ,		_	_		_			-	_				,	-		<u>.</u>								
	Qualifications	J+ 145/A (c)														"Rin grad																
Associated Samples	AH 1 1 11	11. 4.9/KS																					7									
(Limit: >0.05)																																
(Limit: <25.0%).	44.4																									**	-					
₽	4																															
ARass	100	(2)																														
16/26/08							1							-																	+	
	A R Q 2 2 Associated Samples		PR928         RRR (4)         (Limit: >0.05)         Associated Samples         Qualifications           (10v)         A4 +         A1 + B1ks         This Acts A	AR938 RRR (F) 44.4 All + Blks Samples Qualifications (100)	ARA28 RRR (F) 44.4 ATT ASSOCIATED Associated Samples Qualifications (100)  (100)  ATT + B/RS  TH ASTRACE  THE ASTRACE  (100)	AR928 RRR (F) 44.4 AND Associated Samples Qualifications (100)	AR928 RRR (F) 44.4 A AH ARSocieted Samples Qualifications (1cm) AH + BRS J+ Act / AC	AR928 RRR (F) 44.4 All + BRS Jet Actions (12mit; 25.0%) Associated Samples Qualifications (16V)	ARA28 RRR (F) 44.4 A AII + BIRS THISTONS  (100)  AP 1 + BIRS THIST A A A A A A A A A A A A A A A A A A A	みR928         R.R.R. (エ)         (Limit: >0.05)         Associated Samples         Qualifications           (1cv)         44.4         Att + Blks         J + Act	ARA28 RRR (F) 44.4 AIT AIRS Associated Samples Qualifications (Limit: 20.05) Associated Samples Qualifications (Limit: 20.05) Associated Samples Qualifications (Limit: 20.05) Ari + Bilks It Acts / A	A R 9.2 8         R R R (Ω)         (Limit: 50.05)         Associated Samples         Qualifications           (1ω)         (1ω)         44.4         All + B/k S         Σ + Λετ / A	AR938 RRR (F) 44 t (Limit: >0.05) Associated Samples Qualifications (Limit: >0.05) Associated Samples Qualifications (Limit: >0.05) Aft + 19/kS \(\text{T} \text{T} \text{Acts.} \text{A}	AR928 RRR (F) 44.4 Authority (Limit 20.05) Associated Samples Qualifications (Limit 20.05) Associated Samples (Limit 20.05) Associated (Limit 20.05) As	AR 928R.R.R. (4)(Limit >0.05)Associated SamplesQualifications $(10u)$ $A4$ $L$ $A4$ $L$ $A4$ $L$ $A4$ $L$ $A4$ $L$	ARGRE RR (F) 44 t (Linit 26.05) Associated Samples Qualifications (Linit 26.05) Associated Samples Qualifications (100)	A R 938         R R R R R R         (Limit 20.05)         Associated Samples         Qualifications           (10v)         AP R R R R R         AP R R R R R R R R R R R R R R R R R R R	A R 928         R R R (4)         (LImit >0.05)         Associated Samples         Qualifications           (100)         44.4         All 4.8/R5         T + Acts. A	ARAZZ R.R. (±) 44 + A. (Linit 50.05) Associated Samples Qualifications  (160) At A. (Linit 50.05) Associated Samples Qualifications  At A. (Linit 50.05) Associated Samples Qualifications  At A. (Linit 50.05) Associated Samples Qualifications  (160) At A. (Linit 50.05) Associat	A R 9.2 B         R R R         (μ)         (Limit > 0.05)         Associated Samples         Qualifications           (1cv)         (1cv)         4.4 £         (Limit > 0.05)         Atf + B/R S         Σ + Λε'Σ / A	ARA28 R.R.R. (Limit: 50.55) Associated Samples Qualifications  ALL + BLR T ALL + ALL + ALL + BLR T ACLT A  ALL + BLR T ACLT A  ALL + BLR T ACLT A  Audifications  ALL + BLR T ACLT A  Audifications	ARA28 RRR (1 Innit 25.0%) (Limit 26.0%) Associated Samples Qualifications (1cv) A44 A A44 A44 A44 A44 A44 A44 A44 A44	A R 9.2 8 R R R G) 44 F Conditions Samples Qualifications  (101) A 1 + 10 R S	A R 9.2 R         R R R         (4)         4.4 ±         (Limit 20.65)         Associated Samples         Qualifications           (1cv)         (1cv)         4.4 ±         H1 + B/R S         Σ + Λεts / Λ	A E θ 2 B         R R R         (1)         4 4 ±         (Limit 20.83)         Associated Samples         Qualifications           (10x)         (10x)         At I + B R S         Σ + Λ ε L Λ Λ Λ         Δ + Λ ε L Λ Λ Λ         Δ + Λ ε L Λ Λ Λ         Δ + Λ ε L Λ Λ Λ Λ         Λ + Λ ε L Λ Λ Λ         Λ + Λ ε L Λ Λ Λ         Λ + Λ ε L Λ Λ Λ         Λ + Λ ε L Λ Λ Λ Λ Λ Λ Λ Λ Λ Λ Λ Λ Λ Λ Λ Λ Λ	4 / 2 / 2 / 2 / 2 / 2 / 2 / 2 / 2 / 2 /	4/24/68	4 (24.68) A R A 2 (Limit 25.0%) (Limit 26.0%) Associated Sumples Qualifications (10.1) (10.1	2. \( \frac{2.6.68}{\text{Low}} \) \( \frac{2.6.68}{\text{Low}} \) \( \frac{2.6.68}{\text{Low}} \) \( \frac{2.6.68}{\text{Low}} \) \( \frac{2.6.6}{\text{Low}} \) \( \frac{2.6.6}{\text{Low}} \) \( \frac{4.4}{\text{Low}}	1. (2 1.6 8	1 (γ 1 6 8 γ R 4 8 R R R R) 4 4 4 2 (Innit 25 6 9) Associated Samples Occupies to the state of	( \( \frac{1}{2} \lambda \

21 257 B2A

## **VALIDATION FINDINGS WORKSHEET** Blanks

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

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

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

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

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

Y/N N/A

N N/A Was the blank contaminated? If yes, please see qualification below. Blank extraction date: 7/61 /08

Associated Samples:

Conc. units:

4

1/0500 28/2 3 ď 0.048/4 (79) 0.54/4 3 0,067 /y 0.070/y 0.070/y 22.0 Sample Identification 2 0.75 0 + 4 1.5/4 0, 20 8 0.066 R ٥ 2 0,050 0. 1 0.71 70 SBLEY 0.040 0:25 Blank ID 0.0 0,34 **424** 4 4 A ム X Compound

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

21257 B 2a See Cores SDG#: LDC#\_

## VALIDATION FINDINGS WORKSHEET Field Blanks

Page: of Reviewer: 2nd Reviewer:\_

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

Were target compounds detected in the field blanks? Were field blanks identified in this SDG?

Y N/A X/N N/A

Associated sample units: 45 Blank units: Ng /L

<del>2</del>B Field blank type: (circle one) Field Blank / Rinsate / Other. Sampling date: 6 /6 /6 8

1,2,4-10,14-22 P.B. 1.

(bb; bt)

0.049 0,648/4 4112 0,070/4 0,75/4 0.070/4 0.75/4 Sample Identification +B= 0.067/4 0,20 Associated Samples: 4/330.0 n/250.0 10,17/4 0.080/4 <u>ه</u> 0.075 0, (8 PB 06 16 08B 0.085 Blank ID 0,2/ EFE S Compound

Associated sample units: Sampling date: Blank units:

CROL

2x-14 ATTE SX- Phthalato

CROL

21257Bra LDC #: SDG #:

## VALIDATION FINDINGS WORKSHEET Field Blanks

METHOD: GC/MS BNA (EPA SW 846 Method 8270)
Y N N/A Were field blanks identified in this SDG?

Were target compounds detected in the field blanks?

Sampling date: Blank units:

Field blank type: (c

7	The control of the co	י יכום חומו י	/ Nilisale / Oli	ASSOCIA	C D Associated Samples:						1
	Compound	Blank ID			U	Sample Identification	Hon.				Г
		3	7			olinian oldun					T
	77	0,38									$\top$
	5	0.050	0.050 0 048/11								T
			7 2 2 2								_
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										,	Ī
			- 1								
											Τ
_											Ì
							*: :				
	CRQL							. V .m			T
											-

Associated sample units: Blank units:

Sampling date:

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

Associated Samples:

Compound Blank ID								
	Compound	ı		v,	amnia Identifica	tion		
	HHISTANDAS I PRICERA (SERVICE AND SERVICE	5						
		,						
							-	
					:			
	CROL			- 1.				

LDC# 21257826 SDG#: L

## VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Page: Reviewer: 2nd Reviewer.

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 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 8270)

MS/MSD. Soil / Water.

N N/A

X N N N

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

_								
*	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (1 lowles)	i da		
		25/24	××	320 (58-18m)	300 37.50	-1	Associated Samples	Qualifications
					1	78 (20)	91	Starty (m. ld)
			KKK	-	(911-24) 221	_		1 100 / 000 0
			RRR	38 ( ) )	34 (50-150)	,		A 244
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	Compound	(Soil)	RPD (Soll)	QC Limits (Water)	RPD (Water)		Compound	QC Umits	RPD	QC Limits	RPD	
∢ .	Pheno	26-90%	~32%	12-110%	< 42%	99	Acenaphthene	31-137%	(30il) < 18%	(Water) 46-118%	(Water)	
ر											71	<u>.</u>
اد	Z-Chiorophenol	25-102%	× 20%	27-123%	× 40%	=	4. Nitrombonel	27.77				T
ui	1.4-Dichlorobenzene	30 4049/					1111001101	11-114%	< 50%	10-80%	× 20%	
		%+01-07	<27%	36-97%	v 28%	¥.	2.4-Dinitrotolume	28.80%	/470/	0,000		Γ
-	N-Nitroso-di-n-propylamine	41-126%	< 38%	41-116%	/300/	ļ		2000	2/ 1/2	24-90%	< 38%	٦
Ω	1 2 4 Trichlorahamman			8/2	% %		Pentachlorophenoi	17-109%	× 47%	9-103%	× 50%	
	ייליין	38-107%	< 23%	39-98%	< 28%	2	Dyrana	35 (108)	,000			T
>	4-Chloro-3-methylphenol	76 4001					2001	92-145%	× 30%	26-127%	×31%	
		40-103%	× 33%	23-97%	< 42%							Γ

LDC #: 21 257 \$24 SDG #: 54 Gray

## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Page: 1 of 1 2nd Reviewer: Reviewer:

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

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	Qualifications	J+ det /p Ct	一下をナイカー	a) +ar + -	4	Ne spect ( USD ,			Nome of Close	- 1	J+ dets/P (1 1)	No peal (VCS.	7	J-/R/P (11 1)															A Market Control of the Control of t
	Associated Semples	(2nds 6-1							4-12 CRIV-	1			,																
The Page			(		77 (1.20.1			`		,			-	( 02 ) 207	(	,	( )		•					-	)	( )	,	~	( )
LCSD %R (Limits)	124 (2) 96		- 2	134 ( 1/2)	(	,		-	188 (50-120)	( ) 08%	4.7	11/4 /	, , ,						,	(	( )	,				Service of Company	,		
LCS %R (Limits)	134 (50-12)	-	1 21.	2	180 ( 1	•	,	1	(18 (50-120)	460 ( )	46	) 24	4						`		(	(	)				( )		
Compound	. XX	141111	27.7	44	KKK			A A A	#37	××	מממ	トドロ	RAR																
OI OSOTOSO ID	105/201							VC/P >	1																				
Date								2												1									
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LDC # 21.257 B 24 SDG #:

# VALIDATION FINDINGS WORKSHEET

of 1

Reviewer: Page:

2nd Reviewer:

Internal Standards

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) X/N/N/A

YN N/A

Were all internal standard area counts within -50 to +100 of the associated calibration standard?

KK K G66 Qualifications R/A FFF 171 Were the retention times of the internal standards within +/- 30 seconds of the retention times of the associated calibration standard? Thak RT (Limits) 508 680 050 4 95 127170-141 763 -Area (Limits) 46 528 24 566 27 916 1501 ه 0 Internal Standard PRY PRY Sample ID 20 2 2 4 Date #

IS1 (DCB) = 1,4-Dichlorobenzene-d4 IS2 (NPT) = Naphthalene-d8 IS3 (ANT) = Acenaphthene-d10 QC Ilmits are advisory

IS4 (PHN) = Phenanthrene-d10 IS5 (CRY) = Chrysene-d12 IS6 (PRY) = Perylene-d12

LDC #: 21257 by

## VALIDATION FINDINGS WORKSHEET Overall Assessment of Data

Page:

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

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

All available information pertaining to the data were reviewed using professional judgement to compliment the determination of the overall quality of the data.

Was the overall quality and usability of the data acceptable? Y)M N/A

	T	Τ	Τ	T	T	T	T	T	1	T		Τ	7		T	T	7	
Qualifications	(n) A/X																	
Associated Samples	15 excedano.																	
Finding	Confirmation rune for																	
Sample ID	7 10, 20																	
Date																	Comments:	
#											L			1			Com	

SDG #: Sec Gree

# VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: of Reviewer: 0/c

METHOD: GC/MS BNA (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

 $RRF = (A_{\nu})(C_{\nu})/(A_{\nu})(C_{\nu})$  average RRF = sum of the RRFs/humber of standards %RSD = 100 \* (S/X)

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

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

Recalculated %RSD 286 635 20 2 もんな オーナ Reported 14.00 3.97 5.57 %RSD 5.04 14.14 9.9 Receimined Average RRF pr. ray : 203 1,902 (inItial) 6 0 . 640 076 Average RRF : 23. Reported 0.0 6.734 1.903 1.6+6 1.028 RRF (-0 std) Recalculated 1221 1.008 659'0 1.00 1 . 887 RRF ( ), o std) Reported 800. 0,659 1.882 1.23/ 100: Compound (Reference Internal Standard) Bis(2-ethylhexyl)phthalate (5th internal standard) Bis(2-ethylhexyl)phthalate (5th internal standard) Bis(2-ethylhexyl)phthalate (5th internal standard) Pentachiorophenol (4th internal standard) Pentachlorophenol (4th internal standard) Pentachlorophenol (4th internal standard) Benzo(a)pyrene (6th internal standard) Benzo(a)pyrene (6th internal standard) Naphthalene (2nd internal standard) Naphthalene (2nd internal standard) Naphthalene (2nd internal standard) Elucrade (3rd internal standard) Fluorene (3rd internal standard) Fluorene (3rd internal standard) Phenot (1st internal standard) Phenol (1st internal standerd) Phenol (1st internal standard) 80/20/9 Calibration Date Standard ID 747 #

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

Benzo(a)pyrene (6th internal standard)

SDG#: See Core

# VALIDATION FINDINGS WORKSHEET Continuing Calibration Results Verification

Reviewer: 0/2 2nd Reviewer:

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

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

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF = (A<sub>x</sub>)(C<sub>a</sub>)/(A<sub>a</sub>)(C<sub>a</sub>)

Where: ave. RRF = initial calibration average RRF RF = continuing calibration RRF

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

KKF = continuing calibration RRF

A<sub>x</sub> = Area of compound,

C<sub>x</sub> = Concentration of compound,

<u>_</u>									
					Reported	Recalculated	Reported	Receiptand	-
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF	RRF	σ,	Ω%	10
-	ARARY	21/20/9	6 /2x /1 / Present 1st internal attended to 1 / 15 / 15 / 2		22				
		0		1.402	2.057	2.057	<u>্</u>	8	
				1.028	869.	1.64X	P 7	0 7	_
			Fluorene (3rd internal standard)	1.640	1.514	777	1	7.7	
			Pontachiorophenal (4th internal standard) UV	1.619	1.107	72.	, 0	- 0	
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.724	6,819	0,814	12	13.1	
	. <		Renzo(a)puzene (6th internal standard)	1. 26 3	1.283	1.762	7 9	6.6	
7	11051	7/01/68	7/61 /6 & - Phenol-(1st internal standard) RRR		2054	7 057	\(\times\)	9 0	
$\int$			Naphthalene (2nd internal standard)		1.17.0	25	0 4	0,0	
			Ellucreng (3rd internal standard)		200	X7	7	4.7	
			Rentschlorenbend (4th internal standard) 1/1		12.7.7	5000	7.7	7.7	
			Rie(2) athirthe atheres of the control of (7)		2000	1,097	T, V, 7,	7.7	
			o (Signary) principles (Sin Internal standard)		0,306	908.0	1 N	11, 3	
٠	Ac xxx	1/-1/-	tandard)		1.327	1.327	10, 3	16. 2	
<u>.</u>	. 1	1100118	Pheno+(1st internal standard) RRR		2,061	2,061	4.0	9	
L			Naphthalene (2nd internal standard)		1.62	7 6 7 -	7.0	6, 2	
			Fluorene (3rd internal standard)		1 8 -	1 2 7	200	6, 5	
			Pentachforophenol (4th internal standard) 1717		#Q+ ::	189	9.5	9.5	
		-	Bi-/2 - th- th- th- th- th- th- th- th- th- t		1.077	1, 677	5.7	, L	_
			Dis(z-ethylhexyl)phthalate (5th internal standard)		0,809	0,809	11.7	1,7	
			Benzo(a)pyrene (6th internal standard)	<u> </u>	1.32X	1. 3.2			
						:			

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

LDC# 2727 Bya See Gret SDG #:

## Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Reviewer: Page:

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

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

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF =  $(A_x)(C_u)/(A_u)(C_x)$ 

ave. RRF = initial calibration average RRF RRF = continuing calibration RRF A<sub>x</sub> = Area of compound, C<sub>x</sub> = Concentration of compound, ( Where:

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

L								
					Reported	Receipting	Renorted	Roceliniano
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	RRF	RRF	σ%.	Ο%.
7	45061	2/03/08	Phenol (1st internal standard) RRR	1.962	7.054	2005	7 9	7 41
$\perp$			Naphthalene (2nd internal standard)	1.623	690.	٠ 90 .	2.4	2 /2
			Fluorent (3rd internal standard)	1.640	1.477	4		2 8
1			Pentachlorophenel (4th internal standard) M V/	1.019	7601	1,001		77
1			Bis(2-ethylhexyl)phthelate (5th internal standard)	20.0	0.786	0.786	9,0	9,19
L			Renzo(a)nirena (6th Internal standard)	1, 263	ンガ!	1.295	2,7	20.0
7			Phenot (1st internal standard)				0 ,,	
			Naphthalene (2nd internal standard)					
		À	Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)			-	·	
			Bis(2-ethylhexyl)phthalate (5th Internal standard)					
			Benzo(a)pyrene (6th internal standard)					
m			Phenol (1st internal standard)					
$\perp$			Naphthalene (2nd internal standard)					
$\perp$			Fluorene (3rd internal standard)					
1			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					-
			Benzo(a)pyrene (6th Internal standard)					

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

SDG #: STE Cover

### LIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page:	<u>lof_1</u>
Reviewer:	300
nd reviewer:_	V

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

Sample ID: #

Where: SF = Surrogate Found SS = Surrogate Spiked

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	2,06	1-41	71	71	01
2-Fluorobiphenyl		1.21	61	61	
Terphenyl-d14		1.70	85	85	}
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			t-		
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	V 10	15					
2-Fluorobiphenyl							
Terphenyl-d14		- 200 W		1 911			
Phenol-d5							
2-Fluorophenoi			5				
2,4,6-Tribromophenol		*4.					
2-Chlorophenol-d4		1					
1,2-Dichlorobenzene-d4				i		ar e	

LDC#: PAZET Bra SDG #: See Cover

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

Page: lof Reviewer: 2nd Reviewer:

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

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

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

Where: SSC = Spiked sample concentration SA = Spike added

SC = Sample concentation

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

MSC = Matrix spike concentration

MSDC = Matrix splke duplicate concentration

MS/MSD

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											大海 计记录 化氯
		Spike	Sample	Spiked Sample	Sample	Matrix Spike	Spike	Matrix Spike Duplicate	a Duplicate	MS/MSD	dsi
Compound	Addigo ( NS /	ided (1)	Concentration ( 1/2/U	Concentrati	itration カメ	Percent Recovery	ecovery	Percent Recovery	acoverv	Cax	
	MS	MSD		MS	MSD	Renorted	Recei	Panartad	olego O	11	
Phenoi										ogrooday	Recalcillated
N-Nitroso-di-n-propylamine									-		
4-Chloro-3-methylphenol											
Acenaphthene	0.500	0, 500	Ф	0.470	6.480 56	\$	8	46	26	1,	-
Pentachlorophenol											
Pyrene	6.500	(ms.0	· .\	0,410	0.41	\ \ \	82	9	4	-	
					8				1		
								,.			·
			-		·						
ď.											

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

LDC# 2127 BY

# VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

Page: lof L

Reviewer: 2nd Reviewer:

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

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

% Recovery = 100 \* (SC/SA

Where: SSC = Spike concentration SA = Spike added

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

LCS/LCSD samples:

	8	sike	Ī	oike	31	CS	<b>51</b>	l CSD	l CS/I	GS/I GSD
Compound	<b>₩</b>	Added (Ms /し)	Concer (μ <sub>α</sub>	Concentration $(u_{\alpha}/l_{\alpha})$	Percent Recovery	Recovery	Percent F	Percent Recovery	RPD	Q
	108	LCSD	1.05	1 CSD	Reported	Recalc	Reported	Rocalc	Panortad	Doceland
Phenol										
N-Nitroso-di-n-propylamine									-	
4-Chloro-3-methylphenol										
Acenaphthene	0,50	0.50	0,480	6. Pro	96	90	9 9	64	7	
Pentachlorophenol										
Pyrene	O. 50	6.5	0.46.0	0.970	90	26	24	20	p	1.
									· · · · · · · · · · · · · · · · · · ·	<u></u>

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. SDG #: Sre Cover

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

Page:_	Lot 1
Reviewer:	TVZ
nd reviewer:	<u>a</u>

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

1	Y	N	N/A
	<b>Y</b> /	N	N/A

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

Conc	entration	$n = \frac{(A_{a})(I_{a})(V_{a})(DF)(2.0)}{(A_{a})(RRF)(V_{a})(V_{a})(WS)}$	Example:	
A <sub>x</sub> .	=	Area of the characteristic ion (EIQP) for the compound to be measured	Sample I.D. 6 1, 4- 5, 000	me
A <sub>is</sub>	=	Area of the characteristic ion (EICP) for the specific internal standard		
1,	=	Amount of internal standard added in nanograms (ng)	Conc. = (9417)(1)(1m1	)( (CO))( )
V.	=	Volume or weight of sample extract in milliliters (ml) or grams (g).	(90837)(0.574)(160m))(	)( )
V,	· =	Volume of extract injected in microliters (ul)	= 0,17 ug 1	
V <sub>t</sub>	=	Volume of the concentrated extract in microliters (ul)		
Df	=	Dilution Factor.		
%S	=	Percent solids, applicable to soil and solid mátrices only.		* * *

2.0	= Factor of 2 to account	for GPC cleanup			4
#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration	Qualification
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### Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

June 26, 2008

LDC Report Date:

August 17, 2009

Matrix:

Soil

Parameters:

Semivolatiles

Validation Level:

Stage 2B

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2844666

### Sample Identification

SA180-0.5B

SA180-0.5BD

SA180-10B

SA180-10BDL

SA180-20B

SA180-30B

SA57-0.5B

SA57-10B

SA57-20B

SA57-20BDL

SA57-30B

SA57-10BD

SA180-10BMS

**SA180-10BMSD** 

### Introduction

This data review covers 14 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

### III. Initial Calibration

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 15.0% for each individual compound and less than or equal to 30.0% for calibration check compounds (CCCs).

In the case where %RSD was greater than 15.0%, the laboratory used a calibration curve to evaluate the compound. All coefficients of determination (r<sup>2</sup>) were greater than or equal to 0.990.

For the purposes of technical evaluation, all compounds were evaluated against the 30.0% (%RSD) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

Average relative response factors (RRF) for all semivolatile target compounds and system performance check compounds (SPCCs) were greater than or equal to 0.05 as required.

### 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).

For the purposes of technical evaluation, all compounds were evaluated against the 25.0% (%D) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

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 RRF values were greater than or equal to 0.05.

### 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
SBLK1	7/10/08	Butylbenzylphthalate Phenanthrene	9.0 ug/L 1.8 ug/L	All samples in SDG R2844666

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
SA180-0.5B (5x)	Butylbenzylphthalate	65 ug/Kg	65U ug/Kg
SA180-10B	Butylbenzylphthalate	15 ug/Kg	15U ug/Kg
SA180-10BDL (3x)	Butylbenzylphthalate Phenanthrene	33 ug/Kg 9.8 ug/Kg	33U ug/Kg 9.8U ug/Kg
SA180-20B (3x)	Butylbenzylphthalate	41 ug/Kg	41U ug/Kg
SA57-10B (3x)	Phenanthrene	9.0 ug/Kg	9.0U ug/Kg
SA57-20B	Butylbenzylphthalate	43 ug/Kg	43U ug/Kg
SA57-30B (5x)	Butylbenzylphthalate	73 ug/Kg	73U ug/Kg
SA57-10BD (5x)	Phenanthrene	5.8 ug/Kg	5.8U ug/Kg

No field blanks were identified in this SDG.

### VI. Surrogate Spikes

Surrogates were added to all samples and blanks as required by the method. Surrogate recoveries (%R) were not within QC limits for sample SA57-20B. Since the sample was diluted out, no data were qualified.

### VII. Matrix Spike/Matrix Spike Duplicates

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

Spike ID (Associated Samples)	Compound	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
SA180-10BMS/MSD (SA180-10B SA180-10BDL)	Di-n-butylphthalate	157 (50-150)	46 (50-150)	46 (≤30)	J (all detects) UJ (all non-detects)	А

### VIII. Laboratory Control Samples (LCS)

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

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Flag	A or P
LCS/D1 (All samples in SDG R2844666)	Di-n-butylphthalate	143 (50-120)	150 (50-120)	-	J+ (all detects)	Р

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

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

### XI. Target Compound Identifications

Raw data were not reviewed for this SDG.

### XII. Project Quantitation Limit

All project quantitation limits were within validation criteria with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P
SA180-10B SA57-20B	Di-n-butylphthalate	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	А

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R2844666	All compounds reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

### XIII. Tentatively Identified Compounds (TICs)

Raw data were not reviewed for this SDG.

### XIV. System Performance

Raw data were not reviewed for this SDG.

### XV. Overall Assessment

The overall assessment of data was acceptable. In the case where more than one result was reported for an individual sample, the least technically acceptable results were rejected as follows:

Sample	Compound	Flag	A or P
SA180-10B SA57-20B	Di-n-butylphthalate	×	А
SA180-10BDL SA57-20BDL	All TCL compounds except Di-n-butylphthalate	X	А

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

### XVI. Field Duplicates

Samples SA180-0.5B and SA180-0.5BD and samples SA57-10B and SA57-10BD were identified as field duplicates. No semivolatiles were detected in any of the samples with the following exceptions:

	Concentrat	ion (ug/Kg)	RPD	Difference		
Compound	SA180-0.5B	SA180-0.5BD	(Limits)	(Limits)	Flags	A or P
Anthracene	24	40U	-	16 (≤40)	-	-
Benzo(a)anthracene	zo(a)anthracene 160 29 -		-	131 (≤40)	J (all detects)	А
Benzo(a)pyrene	110	32	-	78 (≤40)	J (all detects)	А
Benzo(b)fluoranthene	110	32	-	78 (≤40)	J (all detects)	А
Benzo(g,h,i)perylene	74	27	-	47 (≤40)	J (all detects)	А
Benzo(k)fluoranthene	110	33	-	77 (≤40)	J (all detects)	А
Butylbenzylphthalate	65	1000U	-	935 (≤1000)	-	-
Di-n-Butylphthalate	1700	1300	-	400 (≤1000)	-	-
Indeno(1,2,3-cd)pyrene	69	23	<del>.</del>	46 (≤40)	J (all detects)	А
Chrysene	190	46	-	144 (≤40)	J (all detects)	А
Dibenzo(a,h)anthracene	23	40U	-	17 (≤40)	-	-
Fluoranthene	350	71	-	279 (≤40)	J (all detects)	А
Hexachlorobenzene	20	19	-	1 (≤40)	-	-
Phenanthrene	190	41	-	149 (≤40)	J (all detects)	А
Pyrene	270	51	-	219 (≤40)	J (all detects)	А

	Concentrat	ion (ug/Kg)	200	D'''		
Compound	SA57-10B	SA57-10BD	RPD (Limits)	Difference (Limits)	Flags	A or P
Di-n-Butylphthalate	850	1500	-	650 (≤920)	-	-
Diethylphthalate	62	920U	-	858 (≤920)	-	-
Fluoranthene	4.4	36U	-	31.6 (≤36)	-	-

	Concentrati	on (ug/Kg)		D:#*		
Compound	SA57-10B	SA57-10BD	RPD (Limits)	Difference (Limits)	Flags	A or P
Phenanthrene	9.0	5.8	-	3.2 (≤36)	-	-

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG R2844666

SDG	Sample	Compound	Flag	AorP	Reason (Code)
R2844666	SA180-10B SA180-10BDL	Di-n-butylphthalate	J (all detects) UJ (all non-detects)	А	Matrix spike/Matrix spike duplicates (%R)(RPD) (m, ld)
R2844666	SA180-0.5B SA180-0.5BD SA180-10B SA180-10BDL SA180-20B SA180-30B SA57-0.5B SA57-10B SA57-20B SA57-20BDL SA57-30B SA57-10BD	Di-n-butylphthalate	J+ (all detects)	Р	Laboratory control samples (%R) (I)
R2844666	SA180-10B SA57-20B	Di-n-butylphthalate	J (all detects)	А	Project Quantitation Limit (e)
R2844666	SA180-0.5B SA180-0.5BD SA180-10B SA180-10BDL SA180-20B SA180-30B SA57-0.5B SA57-10B SA57-20B SA57-20BDL SA57-30B SA57-10BD	All compounds reported below the PQL.	J (all detects)	А	Project quantitation Limit (sp)
R2844666	SA180-10B SA57-20B	Di-n-butylphthalate	Х	А	Overall assessment of data (o)
R2844666	SA180-10BDL SA57-20BDL	All TCL compounds except Di-n-butylphthalate	Х	А	Overall assessment of data (o)
R2844666	SA180-0.5B SA180-0.5BD	Benzo(a) anthracene Benzo(a) pyrene Benzo(b) fluoranthene Benzo(g,h,i) perylene Benzo(k) fluoranthene Indeno(1,2,3-cd) pyrene Chrysene Fluoranthene Phenanthrene Pyrene	J (all detects)	А	Field duplicates (Difference) (fd)

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG R2844666

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
R2844666	SA180-0.5B (5x)	Butylbenzylphthalate	65U ug/Kg	А	bl
R2844666	SA180-10B	Butylbenzylphthalate	15U ug/Kg	А	bl
R2844666	SA180-10BDL (3x)	Butylbenzylphthalate Phenanthrene	33U ug/Kg 9.8U ug/Kg	А	bl
R2844666	SA180-20B (3x)	Butylbenzylphthalate	41U ug/Kg	А	bl
R2844666	SA57-10B (3x)	Phenanthrene	9.0U ug/Kg	А	bl
R2844666	SA57-20B	Butylbenzylphthalate	43U ug/Kg	А	bl
R2844666	SA57-30B (5x)	Butylbenzylphthalate	73U ug/Kg	А	bl
R2844666	SA57-10BD (5x)	Phenanthrene	5.8U ug/Kg	А	bl

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles- Field Blank Data Qualification Summary - SDG R2844666

No Sample Data Qualified in this SDG

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

LI	D	С	#:	21	2	25	7	(	"	2	<u>a</u>	

R2844666

SDG #:

Laboratory: Columbia Analytical Services

Stage 2B

Reviewer: 3V7 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: 6 /26 /08
11.	GC/MS Instrument performance check	À	
111.	Initial calibration	A	7 RSD F~
IV.	Continuing calibration/ICV	A	ca/10 = 252
V.	Blanks	SW)	
VI.	Surrogate spikes	Wک	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	SW	vcs/b
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	N	
XII.	Compound quantitation/CRQLs	SW	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	N	
XV.	Overall assessment of data	SN)	
XVI.	Field duplicates	sw	$0, = 1, 2$ $0_2 = 8, 17$
XVII.	Field blanks	2	,

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

D = Duplicate TB = Trip blank

FB = Field blank

EB = Equipment blank

Validated Samples:

5611

	>0	1/					
1	SA180-0.5B <b>1</b> 1	11	SA57-30B	<del>  +</del>   21	SBLK1	31	
2	SA180-0.5BD <b>p</b>	12	SA57-10BD $p_{\checkmark}$	22		32	
3	SA180-10B	13	SA180-10BMS	23		33	
4	SA180-10BDL	14	SA180-10BMSD	24		34	
5	SA180-20B	15		25		35	
6	SA180-30B	16		26		36	
7	SA57-0.5B	17		27		37	
8	SA57-10B D	18		28		38	
9	SA57-20B	19		29	100,000,000,000,000,000	39	
10	SA57-20BDL	20		30		40	

# **VALIDATION FINDINGS WORKSHEET**

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

A. Phenol**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenol**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chiorophenol	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-Methyinaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyi alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	III. Octachions styrene
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	uuu 1,4. Diexare
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	WV.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	www.

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

LDC# 2/257 (24 SDG #:

# VALIDATION FINDINGS WORKSHEET **Blanks**

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

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

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

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

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

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

 $\frac{\sqrt{N N/A}}{8 \text{lank extraction date:}}$  Was the blank contaminated? If yes, please see qualification below.

Associated Samples: Mg/Rg Conc. units:

(97)

(xx)8 (xx) M. Xe Xe Sample Identification (3x) 4 /2 4 (xx) 33/4 8.6 e ie M 5 2(5x) 4 (XS) 190 65 Blank ID 8 6.0 SBUKI 2 AAA Compound

か

Sample Identification	11 (Sx) 12 (Sx)	73/4	5.8/4			
Blank ID	sbiki	9.0	1.8			
Compound		AAA	nn			

(19)

as above

Associated Samples:

Same

Blank analysis date:\_

Blank extraction date:\_

Conc. units:

2/27 CM LDC#

VALIDATION FINDINGS WORKSHEET

Surrogate Recovery

Reviewer:\_\_ Page:

2nd Reviewer.\_

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

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

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

Y)N N/A KANA NA

Date	Sample ID	Surrogate	%R (Lim	ts)	Qualifications	
	(xre) 6	TPH	20	(50-54)	No quel	
		# BP		( )		
				( )		
			-	(		
				( )		
				( )		
				( )		
				( )		
				( )		
				( )		
				( )		
				( )		
				( )		
				( )		
				(		
				( )		
				( )		
				( )		
				(		
				( )		
				( )		
its are advisory  = Nitrobenzene-d5  = 2-Fluorobipheny  = Terpheny-d14  = Phenol-d5	QC Limits (Soll) 23-120 30-115 18-137 24-113	<i>თ თ თ თ</i>	Fluorophenol .,4,6-Tribromophenol :-Chlorophenol-d4 1,2-Dichlorobenzene-d4	QC Limits (Soil) 25-121 19-122 20-130*	QC Limits (Water) 21-100 10-123 33-110*	
	bate  Date  Line advisory  = 2-Fluorobiphenyl = Terphenyl-d14 = Phenol-d5	ory QC Limits (Soil) 2-120 sighenly 30-115 h-d14 18-137 5-5 2-4-13	Sample ID   Surroga	9 (32 X) T PH E D  N B Z  # B P  # B P  OC Limits (Soil) OC Limits (Water) S5 (2F)= 2-Fluorophenol 30-115 (10-94 S8 (DCB) = 12-Dichlordenize (DCB)	Sample   D	Sample   D   Surrogate   WR (Linits)

7127C29 SDG#: LDC #:

# VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Page: 1 of 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 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 Y N N/A

MS/MSD. Soil / Water.

ă #	Date MS/MSD ID	MSMSD ID Compound %R (Limits) %R (Limits) Associ	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
	13/14	66	( )	(	( %) 29	3 4	SM/SM) John NA
		DD	( )	( )	~		
		$\gamma V$	( )		353		>
		××	(osi-us) Ls1	(251-05) H	46 ( )		J/NJ /4 (m. 1)
		77	( ) 0,6	~	( ) /9		
		22	,	, ,	72 , 72		100000
		nnn	( )	( )	SY ( )		
		NU	(24-124)		69		(SW)
		SS	(051-05) Lt	( )	( ) 6,9		
		Μ	( ) 55	( )	( ) 29		-
		5	(. )	( )	So ( )		(MS Ma
		7	( ) 4b	( )	56 ( )		: GSW)
		1777	( )	( )	43 ( )		(MS/M)
		24	( )	( )	1 ) 25		
		RRR	1 1 2	46 (50-150)	23	->	V (Ves/pin
			( )	( )	( )		
			,	`			

_	Compound	QC Limits	RPD	QC Limits	RPD		,	QC Limits	RPD	QC Limits	RPD
Α̈́	Phend	26-90%	.≤35%	12-110%	< 42%	၅၅	Acenaphthene	31-137%	(30II) < 19%	( <b>Vate</b> ) 46-118%	(water) ≤ 31%
ن	2-Chlorophenol	25-102%	< 50%	27-123%	< 40%	. =	4-Nitrophenol	11-114%	< 50%	10-80%	× 50%
шi	1,4-Dichlorobenzene	28-104%	< 27%	36-97%	< 28%	ξ		28-89%	< 47%	24-96%	< 38%
-	N-Nitroso-di-n-propylamine	41-126%	×38%	41-116%	< 38%	Ë	Pentachiorophenol	17-109%	< 47%	9-103%	× 50%
ď	1,2,4-Trichlorobenzene	38-107%	< 23%	39-98%	< 28%	72	Pyrene	35-142%	× 36%	26-127%	< 31%
>	4-Chloro-3-methylphenol	26-103%	< 33%	23-97%	< 42%						

LDC# 2/257 C22 SDG#: 24 Care

# VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Page: 1 of 1

Reviewer: 01/2 2nd Reviewer:

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

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

Y N N/A Was a LCS required?

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

	9	7	?	_		-																			
Qualifications	J+404/2 (8	No and (M) Men																							
Associated Samples	A11 + B/K																								
RPD (Limits)	( )	( )	(	(	•	•	)	)	( )	( )	)	( )	(	(	(	(	(	( )	( )	( )	( )	( )	( )	( )	( )
LCSD %R (Limits)	150 (50-120)	135 ( 1)	( )	( )	( )	( )	( )	( )	,	( )	( )	( )	( )	( )		( )	( )		( )	( )	(		( )	· ·	)
LCS %R (Llmtts)	143 (50-120)	180 (1)	( )	( )	( )	( )	( )	( )		( )	,	,			7	( )	( )	)	( )	`	( )	( )	( )	( )	( )
Compound	××	444																							
CSVCSD ID	LCS/p 1	,																							-
# Date																									

LDC # 2/ 25-7 C22 SDG #:

VALIDATION FINDINGS WORKSHEET

Compound Quantitation and Reported CRQLs

Page:

2nd Reviewer: Reviewer:

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

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

Y N N/A

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

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

Qualifications	Jacks / (e)									
Associated Samples										
Finding	XX > and rongo									
Sample ID	3 9									
Date										
#										

Comments: See sample calculation verification worksheet for recalculations

LDC# 21257624 SDG #:

# **VALIDATION FINDINGS WORKSHEET** Overall Assessment of Data

Page: Reviewer:

2nd Reviewer: \_

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

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

All available information pertaining to the data were reviewed using professional judgement to compliment the determination of the overall quality of the data.

Was the overall quality and usability of the data acceptable? YN NA

#	Date	Sample ID	Finding	Associated Samples	Qualifications	
		3, 4	XX > cy range		X/A Co	0)
		4, 10	All exapt XX di)		•	+
Comn	Comments:					

LDC#: 21257C2 SDG#:See cover

### **VALIDATION FINDINGS WORKSHEET**

**Field Duplicates** 

Page:	1 of /
Reviewer:	<i>5</i> V4
2nd Reviewer:	

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

Y N NA

Were field duplicate pairs identified in this SDG?

Y/N NA

Were target analytes detected in the field duplicate pairs? Y/N NA

Compound Name	Conc (	ug/Kg)	RPD	Diff	Quals	
Compound Name	1	2	(≤50%)	Dill	(Parent Only)	
Anthracene	24	40U		16 (40)		
Benzo(a)anthracene	160	29		131 ( 40 )	3 dots/A (	fd)
Benzo(a)pyrene	110	32		78		
Benzo(b)fluoranthene	110	32		78		
Benzo(g,h,i)perylene	74	27		47		
Benzo(k)fluoranthene	110	33		77	J	
Butylbenzylphthalate	65	1000U		935 (41000)	_	
Di-n-Butylphthalate	1700	1300		400	-	
Indeno(1,2,3-cd)pyrene	69	23		46 (440)	Jaco/A	
Chrysene	190	46		144		
Dibenzo(a,h)anthracene	23	40U		17	-	
Fluoranthene	350	71		279	J dets/A	
Hexachlorobenzene	20	19		1	_	
Phenanthrene	190	41		149	Jack/A	
Pyrene	270	51		219		\ \mathcal{V}

Compayed Name	Conc (	ug/Kg)	RPD	Diff	Quals
Compound Name	8	12	(≤50%)	Dill	(Parent Only)
Di-n-Butylphthalate	850	1500		650 (=920)	
Diethylphthalate	62	920U		858	j
Fluoranthene	4.4	36U		31.6 (= 36)	•
Phenanthrene	9.0	5.8		3.2	<del></del>

### LDC Report# 21257D2a

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

Collection Date:

June 29 through June 30, 2008

LDC Report Date:

September 15, 2009

Matrix:

Water

Parameters:

Semivolatiles

Validation Level:

Stage 2B

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2844768

Sample Identification

M-79B

M-126B

M-84B

M-14ADBF

M-14ABF

#### Introduction

This data review covers 5 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

#### III. Initial Calibration

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 15.0% for each individual compound and less than or equal to 30.0% for calibration check compounds (CCCs).

In the case where %RSD was greater than 15.0%, the laboratory used a calibration curve to evaluate the compound. All coefficients of determination (r<sup>2</sup>) were greater than or equal to 0.990.

For the purposes of technical evaluation, all compounds were evaluated against the 30.0% (%RSD) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

Average relative response factors (RRF) for all semivolatile target compounds and system performance check compounds (SPCCs) were greater than or equal to 0.05 as required.

### 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).

For the purposes of technical evaluation, all compounds were evaluated against the 25.0% (%D) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

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 RRF values were greater than or equal to 0.05.

### 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
SBLK1	7/2/08	Naphthalene	0.060 ug/L	All samples in SDG R2844768

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
M-126B	Naphthalene	0.075 ug/L	0.075U ug/L
M-14ADBF	Naphthalene	0.047 ug/L	0.047U ug/L
M-14ABF	Naphthalene	0.056 ug/L	0.056U ug/L

Sample FB062408GWAREA1 (from SDG R2844650) was identified as a field blank. No semivolatile contaminants were found in this blank with the following exceptions:

Sampling Field Blank ID Date		Compound	Concentration	Associated Samples
FB062408GWAREA1	6/24/08	Diethylphthalate Naphthalene	0.18 ug/L 0.075 ug/L	All samples in SDG R2844768

Sample PB061608B (from SDG R2844538) was identified as a pump blank. No semivolatile contaminants were found in this blank with the following exceptions:

Pump Blank ID	Sampling Date	Compound	Concentration	Associated Samples
PB061608B	6/16/08	Bis(2-ethylhexyl)phthalate Naphthalene	0.21 ug/L 0.085 ug/L	All samples in SDG R2844768

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

Sample	Compound	Reported Concentration	Modified Final Concentration
М-79В	Diethylphthalate	0.21 ug/L	0.21U ug/L
	Bis(2-ethylhexyl)phthalate	0.24 ug/L	0.24U ug/L
M-126B	Naphthalene	0.075 ug/L	0.075U ug/L
M-14ADBF	Diethylphthalate	0.22 ug/L	0.22U ug/L
	Naphthalene	0.047 ug/L	0.047U ug/L
M-14ABF	Diethylphthalate	0.13 ug/L	0.13U ug/L
	Naphthalene	0.056 ug/L	0.056U ug/L
M-84B	Diethylphthalate	0.13 ug/L	0.13U ug/L

### 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 with the following exceptions:

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Flag	A or P
LCS/D1 (All samples in SDG R2844768)	Di-n-butylphthalate Diethylphthalate	280 (50-120) 128 (50-120)	240 (50-120) 126 (50-120)	-	J+ (all detects) J+ (all detects)	Р
LCS/D1 (All samples in SDG R2844768)	1,4-Dioxane	46 (50-120)	46 (50-120)	-	J- (all detects) UJ (all non-detects)	Р
LCS/D1 (All samples in SDG R2844768)	Pyridine	5 (50-120)	10 (50-120)	67 (≤30)	J- (all detects) R (all non-detects)	Р

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

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

### XI. Target Compound Identifications

Raw data were not reviewed for this SDG.

### XII. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R2844768	All compounds reported below the PQL.	J (all detects)	A

Raw data were not reviewed for this SDG.

### XIII. Tentatively Identified Compounds (TICs)

Raw data were not reviewed for this SDG.

### XIV. System Performance

Raw data were not reviewed for this SDG.

#### XV. Overall Assessment

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

### XVI. Field Duplicates

Samples M-14ADBF and M-14ABF were identified as field duplicates. No semivolatiles were detected in any of the samples with the following exceptions:

	Concentration (ug/Kg)		DDD	Difference		
Compound	M-14ADBF	M-14ABF	RPD (Limits)	Difference (Limits)	Flags	A or P
Diethylphthalate	0.22	0.13	- ·	0.09 (≤4.7)	-	-

### Revision 1

	Concentration (ug/Kg)			B.//		
Compound	M-14ADBF	M-14ABF	RPD (Limits)	Difference (Limits)	Flags	A or P
Naphthalene	0.047	0.056	-	0.009 (≤0.19)	-	-

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG R2844768

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R2844768	M-79B M-126B M-84B M-14ADBF M-14ABF	Di-n-butylphthalate Diethylphthalate	J+ (all detects) J+ (all detects)	Р	Laboratory control samples (%R) (I)
R2844768	M-79B M-126B M-84B M-14ADBF M-14ABF	1,4-Dioxane	J- (all detects) UJ (all non-detects)	P	Laboratory control samples (%R) (l)
R2844768	M-79B M-126B M-84B M-14ADBF M-14ABF	Pyridine	J- (all detects) R (all non-detects)	Р	Laboratory control samples (%R) (l)
R2844768	M-79B M-126B M-84B M-14ADBF M-14ABF	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (sp)

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG R2844768

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
R2844768	M-126B	Naphthalene	0.075U ug/L	А	bl
R2844768	M-14ADBF	Naphthalene	0.047U ug/L	А	bl
R2844768	M-14ABF	Naphthalene	0.056U ug/L	А	bl

### \*Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles- Field Blank Data Qualification Summary - SDG R2844768

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
R2844768	M-79B	Diethylphthalate	0.21U ug/L	А	bf

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
R2844768	M-79B	Bis(2-ethylhexyl)phthalate	0.24U ug/L	Α	bp
R2844768	M-126B	Naphthalene	0.075U ug/L	А	bp, bf
R2844768	M-14ADBF	Diethylphthalate	0.22U ug/L	А	bf
R2844768	M-14ADBF	Naphthalene	0.047U ug/L	А	bp, bf
R2844768	M-14ABF	Diethylphthalate	0.13U ug/L	Α	bf
R2844768	M-14ABF	Naphthalene	0.056U ug/L	А	bp, bf
R2844768	M-84B	Diethylphthalate	0.13U ug/L	А	bf

<sup>\*</sup>Corrected codes in above table.

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		Honox Northgate Henderson	
LDC #:_	21257D2a	VALIDATION COMPLETENESS WORKSHEET	Date: <u>୬ /ፋ</u> / ዕ
SDG #:	R2844768	Stage 2B	Page: <u>1</u> of <u>)</u>
Laborato	ry: Columbia Ana	ytical Services	Reviewer: 🐠
	•		2nd Reviewer:

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/29-30/08
II.	GC/MS Instrument performance check	Á	
III.	Initial calibration	A	20 RSD rz
IV.	Continuing calibration/ICV	A	2 RSD r2 COV/101 = 252
V.	Blanks	SW)	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	2	Client Spec
VIII.	Laboratory control samples	SW	Client Spec US AP
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	N	
XII.	Compound quantitation/CRQLs	N	<b>\</b>
XIII.	Tentatively identified compounds (TICs)	N	,
XIV.	System performance	N	
XV.	Overall assessment of data	A	
XVI.	Field duplicates	SW	D = 4,5
XVII.	Field blanks	SM	PB = PB061608b (from R2844538)
Note:	A = Acceptable ND = No	compounds	PB = PB0616085 (from R 2844538)  FB = FB0624086W AREA! (from R 2844650)  G detected D = Duplicate  D = Duplicate  D = Duplicate  D = Duplicate

N = Not provided/applicable SW = See worksheet

R = Rinsate FB = Field blank TB = Trip blank

EB = Equipment blank

Validated Samples:

Water

1	M-79B	11	21	31	
2	M-126B	12	22	32	
3	M84B	13	23	33	
4	M4ADBF P	14	24	34	
5	M-14ABF D	15	25	35	
6	SBKI	16	26	36	
7		17	27	37	
8		18	28	38	
9		19	29	39	
10		20	30	40	

# VALIDATION FINDINGS WORKSHEET

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

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

2/257 Dra SDG#: LDC #:\_

# VALIDATION FINDINGS WORKSHEET

Reviewer: 3 Page:

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

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

Was a method blank analyzed for each matrix?

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

Was the blank contaminated? If yes, please, see qualification below. Blank extraction date: \$702/881ank analysis date: \$702/68

Sample Identification -\* Associated Samples: h/ 250 'A 0.075/4 0.047 090'0 Spikl Blank ID Compound Conc. units: 49

Blank analysis date:\_ Blank extraction date:\_

Associated Samples:

Sample Identification

Blank ID Compound Conc. units:

LDC# 21257 D2a SDG #: See Goney

# VALIDATION FINDINGS WORKSHEET Field Blanks

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

METHOD: GC/MS BNA (EPA SW 846 Method 8270)
Y N N/A Were field blanks identified in this SDG?

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

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

Blank units: 4/2 Associated sample units: 4/2 C

Sampling date: 6/6/6/8

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

Ž Associated Samples:

(67)

Compound	Blank ID				San	Sample Identification	ıtion		
P. Commission of the Commissio	8001 20 df		2	4	5				
243	0,21 0,24/4	0,24/4							
S	580.0		0.075/y	h 250.0 W 40.0 W 250	h 250.0				
				,					
CROL									

Blank units: 45 /L Associated sample units: 45 /L Sampling date: 44 / 48 Field Blank Rinsate / Other:

Associated Samples:

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tion	ک	n/41.0	0.056/4	,		
Sample Identification	4	0.13/4 0.22/4 0.13/4	0.047/4 0.056/4			
S	3	h/<1.0				
	2		0.075 /u			
		0.21/4				
	GWAKEA!					
Blank ID	114	6.18	5.075			
Compound Blank ID	The second of th	71	5			CRQL

2x- ay atress SX- PHTHALLES

\$3 32 33

LDC#: 21257 224 SDG #: 672 67

# VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Page: Reviewer: 2nd Reviewer:

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

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

YN N/A

Was a LCS required?

YN N/A

Were the LCS/LCSD percent recoveries (%R) and the relative percent differences (RPD) within

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

LDC #: 2/257 D24 SDG #: See Core/

### VALIDATION FINDINGS WORKSHEET <u>Field Duplicates</u>

Page:_	
Reviewer:_	1376
2nd reviewer:	-J

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

Were field duplicate Were target compou	pairs identi unds identif	tified in this SDG? fied in the field duplicate	e pairs?	
		1	- 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1	1
Compound	i	<u>Concentration</u>	5	D)ff
	LL	200	0, 13	0.09 (4.7)
	S	0. 22		
		0, 047	0,056	0.009 (= 0.19)
		<u> </u>	<u>L</u>	<u> </u>
		Concentration		T
Compound	, F		1	PPD
Compound	<del></del>			RPD
***************************************		<del></del>		
		1		
	<del></del>	<u> </u>	1	·
			1	
		Concentration	1	
Compound				RPD
			,	
		Concentration		4
Compound				RPD
	-			
:			,	

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

June 30 through July 2, 2008

LDC Report Date:

August 20, 2009

Matrix:

Soil

Parameters:

Semivolatiles

Validation Level:

Stage 4

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2844797

### Sample Identification

SA207-0.5B

SA207-0.5BDL

SA207-10B

**SA207-10BRE** 

SA207-20B

SA207-30B

SA207-40B

SA181-0.5B

SA181-10B

SA181-10B

SA181-30B

SA181-35B

SA207-30BMS

SA207-30BMSD

#### Introduction

This data review covers 14 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008) as there are no current guidelines for the method stated above.

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Field duplicates are summarized in Section XVI.

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- 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.
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- 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).
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- 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 15.0% for each individual compound and less than or equal to 30.0% for calibration check compounds (CCCs).

In the case where %RSD was greater than 15.0%, the laboratory used a calibration curve to evaluate the compound. All coefficients of determination (r<sup>2</sup>) were greater than or equal to 0.990.

For the purposes of technical evaluation, all compounds were evaluated against the 30.0% (%RSD) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

Average relative response factors (RRF) for all semivolatile target compounds and system performance check compounds (SPCCs) were greater than or equal to 0.05 as required.

### 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).

For the purposes of technical evaluation, all compounds were evaluated against the 25.0% (%D) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

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

Date	Compound	%D	Associated Samples	Flag	A or P
7/10/08	Nitrobenzene	66.9	SA207-0.5BDL SA207-10BRE SA181-0.5B SA181-20B SA181-30B SA181-35B SBLK2	J+ (all detects)	А

All of the continuing calibration RRF values were greater than or equal to 0.05.

### 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
SBLK1	7/7/08	Bis(2-ethylhexyl)phthalate Di-n-octylphthalate	460 ug/Kg 26 ug/Kg	SA207-0.5B SA207-0.5BDL SA207-10B SA207-20B SA207-30B SA207-40B SA181-0.5B SA181-10B SA181-20B SA181-35B
SBLK2	7/10/08	Butylbenzylphthalate Phenanthrene	9.0 ug/Kg 1.8 ug/Kg	SA207-10BRE

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
SA207-20B	Bis(2-ethylhexyl)phthalate	66 ug/Kg	66U ug/Kg
SA207-30B	Bis(2-ethylhexyl)phthalate	430 ug/Kg	430U ug/Kg
SA207-40B	Bis(2-ethylhexyl)phthalate	340 ug/Kg	340U ug/Kg
SA181-0.5B	Bis(2-ethylhexyl)phthalate	140 ug/Kg	140U ug/Kg

Sample	Compound TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
SA207-40B	Bis(2-ethylhexyl)phthalate	340 ug/Kg	340U ug/Kg
SA181-0.5B	Bis(2-ethylhexyl)phthalate	140 ug/Kg	140U ug/Kg
SA181-10B	Bis(2-ethylhexyl)phthalate	170 ug/Kg	170U ug/Kg
SA181-20B	Bis(2-ethylhexyl)phthalate	360 ug/Kg	360U ug/Kg
SA181-30B	Bis(2-ethylhexyl)phthalate	130 ug/Kg	130U ug/Kg
SA181-35B	Bis(2-ethylhexyl)phthalate	63 ug/Kg	63U ug/Kg
SA207-10BRE (8x)	Butylbenzylphthalate	110 ug/Kg	110U ug/Kg

No field blanks were identified in this SDG.

### VI. Surrogate Spikes

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

Sample	Surrogate	%R (Limits)	Compound	Flag	A or P
SA207-10BRE (8x)	Nitrobenzene-d5 2-Fluorobiphenyl	25 (45-135) 27 (45-135)	All TCL compounds	J- (all detects) UJ (all non-detects)	А

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Flag	A or P
LCS/D2 (SA207-10BRE) SBLK2)	Di-n-butylphthalate Bis(2-ethylhexyl)phthalate	143 (50-120) 180 (50-120)	150 (50-120) 135 (50-120)	<u>-</u> -	J+ (all detects) J+ (all detects)	P

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

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

### XI. Target Compound Identifications

All target compound identifications were within validation criteria.

### XII. Project Quantitation Limit

All project quantitation limits were within validation criteria with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P	
SA207-0.5B	Hexachlorobenzene Octachlorostyrene	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	А	

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R2844538	All compounds reported below the PQL.	J (all detects)	А

### XIII. Tentatively Identified Compounds (TICs)

Tentatively identified compounds were not reported by the laboratory.

### XIV. System Performance

The system performance was acceptable.

### XV. Overall Assessment

The overall assessment of data was acceptable. In the case where more than one result was reported for an individual sample, the least technically acceptable results were rejected as follows:

Sample	Compound	Flag	A or P
SA207-0.5B	Hexachlorobenzene Octachlorostyrene	×	А
SA207-0.5BDL	All TCL compounds except Hexachlorobenzene Octachlorostyrene	х	А
SA207-10BRE	All TCL compounds	Х	А

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

### XVI. Field Duplicates

No field duplicates were identified in this SDG.

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG R2844797

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R2844797	SA207-0.5BDL SA207-10BRE SA181-0.5B SA181-20B SA181-30B SA181-35B	Nitrobenzene	J+ (all detects)	А	Continuing calibration (ICV %D) (c)
R2844797	SA207-10BRE (8x)	All TCL compounds	J- (all detects) UJ (all non-detects)	А	Surrogate recovery (%R) (s)
R2844797	SA207-10BRE	Di-n-butylphthalate Bis(2-ethylhexyl)phthalate	J+ (all detects) J+ (all detects)	Р	Laboratory control samples (%R) (l)
R2844797	SA207-0.5B	Hexachlorobenzene Octachlorostyrene	J (all detects) J (all detects)	А	Project Quantitation Limit (e)
R2844797	SA207-0.5B SA207-0.5BDL SA207-10B SA207-10BRE SA207-20B SA207-30B SA207-40B SA181-0.5B SA181-10B SA181-20B SA181-30B SA181-35B	All compounds reported below the PQL.	J (all detects)	Α	Project Quantitation Limit (sp)
R2844797	SA207-0.5B	Hexachlorobenzene Octachlorostyrene	X X	А	Overall assessment of data (o)
R2844797	SA207-0.5BDL	All TCL compounds except Hexachlorobenzene Octachlorostyrene	х	Α	Overall assessment of data (o)
R2844797	SA207-10BRE	All TCL compounds	Х	Α	Overall assessment of data (o)

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG R2844797

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code	
R2844797	SA207-20B	Bis(2-ethylhexyl)phthalate	66U ug/Kg	А	Ы	

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
R2844797	SA207-30B	Bis(2-ethylhexyl)phthalate	430U ug/Kg	А	bl
R2844797	SA207-40B	Bis(2-ethylhexyl)phthalate	340U ug/Kg	А	bl
R2844797	SA181-0.5B	Bis(2-ethylhexyl)phthalate	140U ug/Kg	А	bl
R2844797	SA181-10B	Bis(2-ethylhexyl)phthalate	170U ug/Kg	А	bl
R2844797	SA181-20B	Bis(2-ethylhexyl)phthalate	360U ug/Kg	А	bl
R2844797	SA181-30B	Bis(2-ethylhexyl)phthalate	130U ug/Kg	А	bl
R2844797	SA181-35B	Bis(2-ethylhexyl)phthalate	63U ug/Kg	А	bl
R2844797	SA207-10BRE (8x)	Butylbenzylphthalate	110U ug/Kg	A	bl

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles- Field Blank Data Qualification Summary - SDG R2844797

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

SDG #: R2844797 Stag

Stage 4

Date: 8/11/69
Page: 1 of )
Reviewer: 3V6
2nd Reviewer: Q

Laboratory: Columbia Analytical Services

LDC #: 21257E2a

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
<u>l.</u>	Technical holding times	A	Sampling dates: 6/20- 7/02/08
II.	GC/MS Instrument performance check	A	, and the second
111.	Initial calibration	À	₹ RSD r
IV.	Continuing calibration/ICV	ŚN)	2 RSD r 2 COV /10V € 25 Z
V.	Blanks	SW	
VI.	Surrogate spikes	SW)	
VII.	Matrix spike/Matrix spike duplicates	SW)	
VIII.	Laboratory control samples	SW	Ves /D
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	À	
XII.	Compound quantitation/CRQLs	SW	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	A	
XV.	Overall assessment of data	SW	
XVI.	Field duplicates	N	
XVII.	Field blanks	N	

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

TB = Trip blank

EB = Equipment blank

Validated Samples:

Soil

	1	SA207-0.5B	11	SA181-30B	21	SBLKI	31	
	2	SA207-0.5BDL	12	SA181-35B	22	SBLK2	32	
×	3	, SA207-10B	13	SA207-30BMS	23		33	
X	<sub>4</sub> >	SA207-10BB RE	14	SA207-30BMSD	24		34	
	5	SA207-20B	15		25		35	
ł	6	SA207-30B	16		26		36	
	7	SA207-40B	17		27		37	
	8	SA181-0.5B	18		28		38	
	9	SA181-10B	19		29		39	
	10	SA181-20B	20		30		40	

LDC #: 2/ 257 E 2A SDG #: See Cover

### **VALIDATION FINDINGS CHECKLIST**

Page: 1 of 2
Reviewer: 506
2nd Reviewer: 0

Method: Semivolatiles (EPA SW 846 Method 8270C)

Metriod: Sernivolatiles (EPA SW 646 Metriod 6270C)	Ī.,			
Validation Area	Yes	No	NA	Findings/Comments
1. Technical holding times (1998) 1. Tec				A Part Control of the State of
All technical holding times were met.				
Cooler temperature criteria was met.			Y-07000	
II. GC/MS instrument performance check				
Were the DFTPP performance results reviewed and found to be within the specified criteria?	_			
Were all samples analyzed within the 12 hour clock criteria?				
III. Initial calibration				Talan in the second sec
Did the laboratory perform a 5 point calibration prior to sample analysis?				
Were all percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?				
Was a curve fit used for evaluation?				
Did the initial calibration meet the curve fit acceptance criteria of ≥ 0.990?	/			
Were all percent relative standard deviations (%RSD) ≤ 30% and relative response factors (RRF) ≥ 0.05?		-		
IV. Continuing calibration				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?				
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?				
Were all percent differences (%D) ≤ 25% and relative response factors (RRF) ≥ 0.05?		/		
V Blanks 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2				
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?	yes one ex			
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?				AAAAAA
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?		/		
VIII) Espando y como estrolar de la como esta				
Was an LCS analyzed for this SDG?	با		<u> </u>	

LDC #: 2/257 E 24 SDG #: See Cover

### **VALIDATION FINDINGS CHECKLIST**

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

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?			\	
X. Regional Quality Assurance and Challify Council				And the second s
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?			_	
X. Internal standards		-2		
Were internal standard area counts within -50% or +100% of the associated calibration standard?				
Were retention times within ± 30 seconds from the associated calibration standard?				
XI. Target compound identification				A Paragraphic Control of the Control
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?	A 2000000		oi: 300 c 200 a 20	
XII Compound quantilation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?		•		
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XIII. Tentatively scentified compounds (TiCs)	0.00			
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)?		/		
Occupation of the second of th				
System performance was found to be acceptable.			271 275558	
Overella Spesiment (spesiment)				appropriately to the second se
Overall assessment of data was found to be acceptable.		/		
Field duplicate pairs were identified in this SDG.				
		-		
Target compounds were detected in the field duplicates.				
XVII. Field blanks: Add and a finite of the state of the		,		
Field blanks were identified in this SDG.		$\triangle$		
Target compounds were detected in the field blanks.				

# **VALIDATION FINDINGS WORKSHEET**

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

A. Phenoi**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenol**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	VV. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	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	III. Octachlorostyrene
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	uuu 1,4- Dioxane
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	VVV.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	WWW.

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

21257E LDC#

VALIDATION FINDINGS WORKSHEET

Continuing Calibration

SDG#

of )

Page: Reviewer 2nd Reviewer:

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

S DANA

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

Was a continuing calibration standard analyzed at least once every 12 hours of sample analysis for each instrument?

Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's ? Were all %D and RRFs within the validation criteria of <25 %D and >0.05 RRF ?

	Qualifications	0+ 443/A (c)	1													-	
Associated Semples		2 4 8 10-12 SALKZ															
Finding RRF (Limit: >0.05)																	
Finding %D (Limit: <25.0%)	0 77	66.		-											-		
Compound	1																
Standard ID	AS 134	(111)	 														
	7/10/69	,						-									

from on (Passing 101 for 10AL acquire) on 6/26

×13674

LDC#: 2/257 1 2a SDG #: See Commy

## VALIDATION FINDINGS WORKSHEET Blanks

Page: 2nd Reviewer:\_ Reviewer:

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

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

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

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

Y/N N/A Was the blank contaminated? If yes, please see qualification below. ટ

(19)

S O 120/ 260 9 Sample Identification All exapt 2 404 Associated Samples: 7 340 430/4 و 7 S e Blank ID 97 40 SBKI FFF 五年 Compound Sonc. units:

(79)Associated Samples: Blank extraction date: 7/10 /68 Blank analysis date: 7/11 /68 Conc. units:

	11	T	T	ī	ī		 
						-	
tion							
Sample Identification							
Ss							
	(kx)	n/011	(04)	)			
Blank ID	Sb42	9.0	1.8				
0		AAA	hП				
Compound							

3.6 2

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

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

2/257 826

LDC#:

Surrogate Recovery

Reviewer: Page:

2nd Reviewer.

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

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

X N N/A

Qualifications ⋖ mal N 20 45-135) %R (Limits) 7 25 90 ⊗ 9 NBZ **473** Surrogate FBP AII 40.0x X<sub>0</sub> (8x) Sample ID d 3 Date #

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

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

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

S5 (2FP)= 2-Fluorophenol

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

QC Limits (Soil) 23-120 30-115 18-137 24-113

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

LDC#: 21257 £24 SDG#: C. Cas/

# VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Page: 1 of 1 Reviewer: 3VC 2nd Reviewer: 0

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

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

Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water. YN N/A

Was a MS/MSD analyzed every 20 samples of each matrix?

Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

No level (BR MCC), Qualifications	Jacks 16)			1 R (M. (M. 0)	1 R (M, 14,0)	्रान्य						
Associated Samples Qua	9 JA		_	74								
	30) 6	(		<u> </u>	7	7 7						
RPD (Limits)	( ) 45 (	) 88 (										
MSD %R (Limits)	( )	( )	C ( C) ITE	_	~					1-1-11-1-1-1-1-1		
MS %R (Limits)	( )	( )	32 (50-150)									
Compound	AAA	××										
DI OSWISM	13/14											
# Date												

	Compound	QC Limits (Soil)	RPD (Soil)	QC Limits (Water)	RPD (Water)		Compound	QC Limits	RPD (Soll)	QC Limits (Water)	RPD (Water)
Ą	Phenol	26-90%	~35% ≥	12-110%	<u>≤ 42%</u>	99	Acenaphthene	31-137%	≥ 19%	46-118%	<u> </u>
Ö	C. 2-Chlorophenol	25-102%	> 50%	27-123%	< 40%	=	4-Nitrophenol	11-114%	< 50%	10-80%	< 50%
wi	1,4-Dichlorobenzene	28-104%	< 27%	36-97%	< 28%	X X	2,4-Dinitrotoluene	28-89%	< 47%	24-96%	%8E >
٦	N-Nitroso-di-n-propylamine	41-126%	< 38%	41-116%	< 38%	Ë	Pentachlorophenol	17-109%	< 47%	9-103%	%0 <u>5</u> >
œ	1,2,4-Trichlorobenzene	38-107%	< 23%	39-98%	< 28%	.72	Pyrene	35-142%	%9E >	26-127%	<31%
>	4-Chloro-3-methylphenol	26-103%	< 33%	23-97%	< 42%						

LDC# 21257 E2 SDG #: 164

## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Page: 10f /

Reviewer:

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

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

Was a LCS required?

Y(N,N/A)

Were the LCS/LCSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits? Y IN ANA

163   Dec   LGSALGSD   Compound   WR Links   WR Links   RPD Links   Modelle Sandas   Wolfields   WR Links		71 1/	<b>5</b> 3	رد (	7	<u>د</u> ور		7																			
Date   LCS/D   Compound   Set Liceta   Se	No Guax	C7 *** **	7.40	1017	1507 OSM	18	4																				
Deta   LCSLCSD   Compound   SAR (Limits)   SAR (L	Associated Semples	All excent 4 SBIL																									
Data   LCS/LOD   Compound   WR (Limits)	RPD (Limits)		)	,			( )	( )	,	(	( )	(	(	)			( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	
Date LCS/LCSD ID Compound LCS/D   EEE RKK LCS/D Z XX LCS/D Z XX  L	LCSD %R (Limits)	~	1 ~				1	~	( )	(	( )	( )	( )	(	(	( )	( )	( )	( )	( )	( )	( )	( )		( )	( )	
Date LCS/LCSD ID  LCS/D    LCS/D    LCS/D 2	LCS %R (Limits)	(80 (50-120)	47 ( ) )	,			180 ( )	( )	( )	)	( )	( )		( )	( )	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	)	
27 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3	Compound	3.33	RKK			XX	<i>9.</i> 33																				
<del></del>	TCS/TCSD ID	1 9/877				LG/D 2																					
	Date																										
	*										1	+	+	†	+	+	1	+	┪	1	1	$\dashv$	1		1	$\dashv$	

LDC # 21 257 £ 22 SDG#: Se Cor

## Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

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

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

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

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? X N N/A

Qualifications	J dus 1 (e)		(								
Associated Samples	rang	j									
Finding	ho < TTT SS	,									
Sample ID											
Date											
#											

Comments: See sample calculation verification worksheet for recalculations

LDC #: 21257 E2A SDG #: Su (mr/

# VALIDATION FINDINGS WORKSHEET Overall Assessment of Data

Page: 1 of 1
Reviewer: 7/C
2nd Reviewer:

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

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

All available information pertaining to the data were reviewed using professional judgement to compliment the determination of the overall quality of the data.

Y) N N/A Was the overall quality and usability of the data acceptable?

k						
#	Date	Sample ID	Finding	Associated Samples	Qualifications	
			SS TTT > call	rang	XX	(@)
		ત	All except SS TIT	T dil		(6)
		7	\$			(0)
Juo	Comments					
5						

LDC# 2/257 E29 SDG #: Sec Coper

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Reviewer: 2nd Reviewer:

Page: of

METHOD: GC/MS BNA (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,)(C,,)(A,,)(C,) average RRF = sum of the RRFs/number of standards %RSD = 100 \* (S/X)

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

 $A_{\rm b}$  = Area of associated internal standard  $G_{\rm b}$  = Concentration of internal standard X = Mean of the RRFs

L									
				Reported	Recalculated	Reported	Receiculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	RRF (1.60 std)	RRF (1.60 std)	Average RRF (initial)	Average RRF (initial)	%RSD	%RSD
	1541	89/2/9	Phonol (1st internal standard) 1, 4- Di Mane	0,534	45.0	425.0	b. 5774	7.14	7.17
		3	Naphthalene (2nd internal standard)	1,001	1.007	1.028	1.028	3.95	156%
			Elluoren $e$ (3rd internal standard) $^{1}\mathcal{L}$	1.251	1.237	1.640	1.640	74.14	14.15
			<u>Pentachlarophen</u> ol (4th internal standard) $\mathcal{U}\mathcal{U}$	1.008	3 as-1	1.019	1.019	5.57	82'5
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0,639	6-653	pc/ '0	pr/2	15.6	9.88
			Benzo(a)pyrene (6th internal standard)	1-114	1.114	1.203	جمر '(	14.65	14.64
2	1921	7/10/5	- Ohemol (1st internal standard) 1, 4、り)のバル・	(10) 0.585	(10) 0.585	0.616	0.616	11.94	26-11
		60,	Naphthalene (2nd internal standard)	6.963	6,963	1. 624	tro'	444	443
			Fluoreme (3rd internal standard)	1.345	1.945	1.214	1.24	6.0'5	40.9
			Rentachlorophenol (4th internal standard) $\mathcal{U}_{\mathcal{U}}$	456.0	7-560	1.025	1. 027	417	44
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0,868	8 08 - 2	0.727	1960	\$6.8	268
			Benzo(a)pyrene (8th internal standard)	1. 354	1.359	1,117	1. 177	75.01	10.53
3			Phenol (1st internal standard)						
			Naphthalene (2nd internal standard)						
			Fluorene (3rd internal standard)						
			Pentachlorophenol (4th internal standard)						
			Bis(2-ethylhexyl)phthalate (5th internal standard)						
			Benzo(a)pyrene (6th internal standard)						

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

SDG#: See Gover

# VALIDATION FINDINGS WORKSHEET Continuing Calibration Results Verification

Page: lof 1.
Reviewer: Ne.

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

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

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF =  $(A_x)(C_k)/(A_k)(C_x)$ 

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

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

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

					Reported	Recalculated	Reported	Recalculated	П
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Φ%	<b>0%</b>	
_	45089	7/07/08	Phemot (1st internal standard) · UUV	0,574	285'0	L85'0	2,3	٧,٧	
	,		Naphthalene (2nd internal standard)	1.028	1.056	1.056	۲.۶	2.7	
			Fluorene (3rd internal standard)	1.640	1, 523	1.323	19,3	1.9.3	
			Pentachiorophenol (4th internal standard) UV	1.019	1.082	<b>~89~</b>	ف	e'-	Γ
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.724	877.0	844,0	5.7	7.5	Π
			Benzo(a)pyrene (6th internal standard)	ر 20°، ۲	1,286	1.283	6.4	p · 9	
2	AS 127	59/01/6	Phenof (1st internal standard) $MMM$	0.616	0,65/	0.651	4.5	7:5	ſ
			Naphthalene (2nd internal standard)	1.024	1, 060	990'1	3,5	3.8	Π
			Fluorene (3rd internal standard)	1, 214	1.269	692.1	4. S	4.5	Γ
			Pentachlorophenol (4th internal standard) V (/	1,027	1.063	890.1	3,5	3.5	Π
			Bis(2-ethythexyl)phthalate (5th internal standard)	0.762	0.780	082.0	7'8	+'~	
			Renzo(a)pyrene (6th internal standard)	1, 177	1.217	1.217	3,4	3·6	П
ო	AS147	24.12	Phenot (1st internal standard) UNV	0.616	0,678	8620	10,1	101	
			Naphthalene (2nd internal standard)	1.024	1.082	1.082	4.8	5.7	
			Eluorene (3rd internal standard)	1.214	1.328	826·1	4.6	7-6	
			Pentaehlorophenol (4th internal standard) $MU$	1.027	1.113	1. 113	<i>7</i> × 8	8.4	
			Bis(2-ethylhexyl)phthalate (5th internal standard)	6.762	D. 846	0.846	9.11	0 11	
			Benzo(a)pyrene (6th internal standard)	1,177	1,270	1270	7-4	2, 9	

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

LDC#:	21	25	7	E	Za
SDG #:	ς	u	Cu	γ	<b>~</b>

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

Page:_	l_of/
Reviewer:_	JVE
2nd reviewer:	

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found

SS = Surrogate Spiked

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	46.7	(7.02 x5)	53	53	6
2-Fluorobiphenyl	1	(12,53×5)	94	94	1
Terphenyl-d14		(9.31xs)	70	70	
Phenoi-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

(2 ppm) (1M) (1M) = 66.7

Sample ID:

300

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

Sample ID:

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

LDC #: 21257 E29 SDG #: See Cover

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

Page: lof 1 Reviewer: 2nd Reviewer:

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

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

SC = Sample concentation

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

Where: SSC = Spiked sample concentration SA = Spike added

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples: \_\_

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

12/14

Concentration (Lyke) MSD Reported MS MSD Reported MSD Reported MSD Reported MSD Reported MSD MSD Reported MSD MSD Reported MSD MSD Reported MSD	E	Spil	ę,	Sample	Spiked (	Sample	Matrix Spike	Spike	Matrix Spike Duplicate	a Duplicate	MS/MSD	ISD
149   149   145   169   972   168   972   168   972   972   972   972   972   972   972   972   972   972   972   972   972   972   973	Compound	PA (2)	(2g)	Concentration ( Mc/b)	Concen (V)	tration	Percent R	ecovery	Percent Recovery	acovery	RPD	Q
o-din-propylamine (49 149 0 97.3 78.3 65 lorophenol 149 149 4 145 169 97		MS	MSD		WS	MSD	Reported	Recalc	Renorted	Recalc	Reported	Recalculated
o-din-propylamine												
149 149 0 97.3 78.3 65 lorophenol   149 149   145   109   97   97   97   97   97   97   97	so-di-n-propylamine											
Identification of the contraction of the contractio	ro-3-methylphenol											
149   145   109   97   199   97   145   169   97   97   97   97   97   97   97		149	149	0-	97.3	78.5	وک	65	53	.73	んく	27
149 149 \$ 145 109 97	hlorophenol									,		
		149	149	<b>→</b>	571	60	97	47	73	73	28	×

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

LDC# 2127 E29 SDG #: See Corer

# VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

Page: of 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:

% Recovery = 100 \* (SC/SA

Where: SSC = Spike concentration SA = Spike added

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

LCS/LCSD samples:

	g.	ike	ďS	ike	1	CS	101	CSD		CS/I CSD
Compound	Added (12 /C1)	ded	Concer (セン)	Concentration (いく/に)	Percent Recovery	Зесочегу	Percent F	Percent Recovery	RPD	Qc
	1.03	USDI /	l CS	/ I CSD	Reported	Recalc	Reported	Recalc	Renorted	Recalculated
Phenol										
N-Nitroso-di-n-propylamine										
4-Chloro-3-methylphenol										
Acenaphthene	133,3	133.7	88	16	29	29	89	89	~	۴
Pentachlorophenoi										\
Pyrene	130.3	6.661	126.69	132,47	86	7 6	98	66	B	

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

LDC#:_	21	257E	26
SDG #	Cce	Carel	

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

Page:_	lof_/1_
Reviewer:	SVE
2nd reviewer:	

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

	Y	N	N/A
,	Y	Ŋ	N/A

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

Conc	entratio	$n = (A_{b})(1)(V_{b})(DF)(2.0)$ $(A_{b})(RRF)(V_{b})(V_{b})(%S)$	Example:
			l <del>4</del> .1
A <sub>x</sub>	=	Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D:
$A_{is}$	=	Area of the characteristic ion (EICP) for the specific internal standard	
l <sub>s</sub>	=	Amount of internal standard added in nanograms (ng)	$\frac{28776 \times 1.6 \times 5}{381370 \times 1.028} \times 1.6 \times 5 \times 10^{11} \times 10^{11}$
$V_{o}$	=	Volume or weight of sample extract in milliliters (ml) or grams (g).	
$V_i$	=	Volume of extract injected in microliters (ul)	= 48.14 mg/Ee
$V_{t}$	=	Volume of the concentrated extract in microliters (ul)	γ
Df	=	Dilution Factor.	
%S	=	Percent solids, applicable to soil and solid matrices only.	

2.0	= Factor of 2 to accour	nt for GPC cleanup				
#	Sample ID	Compound	C	Reported concentration ( )	Calculated Concentration ( )	Qualification
	<u> </u>					
						***************************************
		·				
				,,,,		
		<u></u>				
				-		

### LDC Report# 21257F2a

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

Project/Site Name:

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 1 through July 2, 2008

LDC Report Date:

September 18, 2009

Matrix:

Water

Parameters:

Semivolatiles

Validation Level:

Stage 2B

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2844803

### Sample Identification

M-55B

M-55DB

M-78B

M-65B

EB070208GW1

M-78BMS

M-78BMSD

### Introduction

This data review covers 7 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

### III. Initial Calibration

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 15.0% for each individual compound and less than or equal to 30.0% for calibration check compounds (CCCs).

In the case where %RSD was greater than 15.0%, the laboratory used a calibration curve to evaluate the compound. All coefficients of determination (r²) were greater than or equal to 0.990.

For the purposes of technical evaluation, all compounds were evaluated against the 30.0% (%RSD) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

Average relative response factors (RRF) for all semivolatile target compounds and system performance check compounds (SPCCs) were greater than or equal to 0.05 as required.

### 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).

For the purposes of technical evaluation, all compounds were evaluated against the 25.0% (%D) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

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 RRF values were greater than or equal to 0.05.

### \*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
SBLK1	7/7/08	Acenaphthene Diethylphthalate Naphthalene Phenanthrene	0.020 ug/L 0.22 ug/L 0.080 ug/L 0.040 ug/L	All samples in SDG R2844803

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	Reported	Modified Final
	TIC (RT in minutes)	Concentration	Concentration
M-55B	Diethylphthalate	0.20 ug/L	0.20U ug/L
	Naphthalene	0.088 ug/L	0.088U ug/L
M-55DB	Diethylphthalate	0.23 ug/L	0.23U ug/L
	Naphthalene	0.10 ug/L	0.10U ug/L
M-78B	Diethylphthalate	0.18 ug/L	0.18U ug/L
	Naphthalene	0.066 ug/L	0.066U ug/L
M-65B	Diethylphthalate	0.26 ug/L	0.26U ug/L
	Naphthalene	0.13 ug/L	0.13U ug/L
EB070208GW1	Naphthalene	0.088 ug/L	0.088U ug/L
	Phenanthrene	0.059 ug/L	0.059U ug/L

Sample EB070208GW1 was identified as an equipment blank. No semivolatile contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB070208GW1	7/2/08	Butylbenzylphthalate Di-n-butylphthalate Diethylphthalate Dimethylphthalate Bis(2-ethylhexyl)phthalate 2-Methylnaphthalene Naphthalene Phenanthrene	0.33 ug/L 3.0 ug/L 5.9 ug/L 3.0 ug/L 0.62 ug/L 0.039 ug/L 0.088 ug/L 0.059 ug/L	M-65B

Sample FB062408GWAREA1 (from SDG R2844650) was identified as a field blank. No semivolatile contaminants were found in this blank with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB062408GWAREA1	6/24/08	Diethylphthalate Naphthalene	0.18 ug/L 0.075 ug/L	M-55B M-55DB M-78B

Sample PB061608B (from SDG R2844538) was identified as a pump blank. No semivolatile contaminants were found in this blank with the following exceptions:

Pump Blank ID	Sampling Date	Compound	Concentration	Associated Samples
PB061608B	6/16/08	Bis(2-ethylhexyl)phthalate Naphthalene	0.21 ug/L 0.085 ug/L	M-55B M-55DB M-78B

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

Sample	Compound	Reported Concentration	Modified Final Concentration
M-65B	Di-n-butylphthalate	1.4 ug/L	1.4U ug/L
	Diethylphthalate	0.26 ug/L	0.26U ug/L
	2-Methylnaphthalene	0.066 ug/L	0.066U ug/L
	Naphthalene	0.13 ug/L	0.13U ug/L
M-55B	Diethylphthalate	0.20 ug/L	0.20U ug/L
	Naphthalene	0.088 ug/L	0.088U ug/L
M-55DB	Diethylphthalate	0.23 ug/L	0.23U ug/L
	Naphthalene	0.10 ug/L	0.10U ug/L

Sample	Compound	Reported Concentration	Modified Final Concentration
M-78B	Diethylphthalate	0.18 ug/L	0.18U ug/L
	Naphthalene	0.066 ug/L	0.066U ug/L

<sup>\*</sup>Corrected compound concentrations in table above.

### VI. Surrogate Spikes

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

### VII. Matrix Spike/Matrix Spike Duplicates

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

Spike ID (Associated Samples)	Compound	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
M-78BMS/MSD (M-78B)	Pyridine	33 (50-150)	45 (50-150)	32 (≤30)	J- (all detects) UJ (all non-detects)	А

### VIII. Laboratory Control Samples (LCS)

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

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Flag	A or P
LCS/D1 (All samples in SDG R2844803)	Pyridine	30 (50-120)	28 (50-120)	-	J- (all detects) UJ (all non-detects)	Р

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

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

### XI. Target Compound Identifications

Raw data were not reviewed for this SDG.

### XII. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R2844803	All compounds reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

### XIII. Tentatively Identified Compounds (TICs)

Raw data were not reviewed for this SDG.

### XIV. System Performance

Raw data were not reviewed for this SDG.

### XV. Overall Assessment

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

### XVI. Field Duplicates

Samples M-55B and M-55DB were identified as field duplicates. No semivolatiles were detected in any of the samples with the following exceptions:

	Concentration (ug/Kg)					A or P
Compound	M-55B	M-55DB	RPD Difference (Limits) (Limits		Flags	
Diethylphthalate	0.20	0.23	-	0.03 (≤5.1)	-	-
1,4-Dioxane	1.0	0.96	-	0.04 (≤2.0)	-	-
Naphthalene	0.088	0.10	-	0.012 (≤0.20)	-	-

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG R2844803

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R2844803	M-78B	Pyridine	J- (all detects) UJ (all non-detects)	Α	Matrix spike/Matrix spike duplicates (%R)(RPD) (m, ld)
R2844803	M-55B M-55DB M-78B M-65B EB070208GW1	Pyridine	J- (all detects) UJ (all non-detects)	Р	Laboratory control samples (%R) (I)
R2844803	M-55B M-55DB M-78B M-65B EB070208GW1	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (sp)

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG R2844803

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
R2844803	M-55B	Diethylphthalate Naphthalene	0.20U ug/L 0.088U ug/L	А	bl
R2844803	M-55DB	Diethylphthalate Naphthalene	0.23U ug/L 0.10U ug/L	А	bl
R2844803	M-78B	Diethylphthalate Naphthalene	0.18U ug/L 0.066U ug/L	А	bl
R2844803	M-65B	Diethylphthalate Naphthalene	0.26U ug/L 0.13U ug/L	А	bl
R2844803	EB070208GW1	Naphthalene Phenanthrene	0.088U ug/L 0.059U ug/L	А	bl

### \*Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles- Field Blank Data Qualification Summary - SDG R2844803

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
R2844803	M-65B	Di-n-butylphthalate Diethylphthalate 2-Methylnaphthalene Naphthalene	1.4U ug/L 0.26U ug/L 0.066U ug/L 0.13U ug/L	А	be
R2844803	M-55B	Diethylphthalate	0.20U ug/L	А	bf
R2844803	M-55B	Naphthalene	0.088U ug/L	А	bf,bp
R2844803	M-55DB	Diethylphthalate	0.23U ug/L	А	bf
R2844803	M-55DB	Naphthalene	0.10U ug/L	Α	bf,bp
R2844803	M-78B	Diethylphthalate	0.18U ug/L	Α	bf
R2844803	M-78B	Naphthalene	0.066U ug/L	Α	bf,bp

<sup>\*</sup>Corrected codes in above table.

### Tronox Northgate Henderson

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LDC#:_	21257F2a	VALIDATION COMPLETENESS WORKSHEET
SDG #:_	R2844803	Stage 2B
		• • • • • • • • • • • • • • • • • • • •

Page: 1 of Reviewer: 2nd Reviewer:

Laboratory: Columbia Analytical Services

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

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

	Validation Area		Comments
l.	Technical holding times	A	Sampling dates: 7/61-02/08
11.	GC/MS Instrument performance check	A	
111.	Initial calibration	A	% RSD € 307 r~ W/CCV € 25 %
IV.	Continuing calibration/ICV	A	W/COV & 25 }
V.	Blanks	SW	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	WZ	
VIII.	Laboratory control samples	Sw	LCS/D
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	N	
XII.	Compound quantitation/CRQLs	N	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	N	
XV.	Overall assessment of data	A	
XVI.	Field duplicates	≤W	b = 1,2
XVII.	Field blanks	SW)	EB = 5 PB = PB061608B from R28445\$8

Note:

A = Acceptable N = Not provided/applicable SW = See worksheet

ND = No compounds detected

FB = Field blank

R = Rinsate

D = Duplicate

TB = Trip blank

EB = Equipment blank

Validated Samples:

101 / Bun .

	WATE	<u> </u>			
1	м-55B <b>у</b>	11	SBUKI	21	31
2	M-55DB <i>D</i>	12		22	32
3	M-78B	13		23	33
4	M65B	14		24	34
5	EB070208GW1	15		25	35
6	M-78BMS	16		26	36
7	M-78BMSD	17		27	37
8	,	18		28	38
9		19		29	39
10		20		30	40

## **VALIDATION FINDINGS WORKSHEET**

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

A Phenoi™	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachloropheno!	II. 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-Dinkrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)peryiene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	ii, 4-Nitrophenoi*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachiorobutadiene⁴	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-Methyinaphthalene	LL. Diethyiphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
l. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chiorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyi alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	TTT. Octachlorostyrene
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	uuu 1,4-Dioxane
N. 2-Nitrophenol™	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	w.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	WWW.

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

21257 FW	La Corr
LDC#:	SDG #:_

## VALIDATION FINDINGS WORKSHEET

Blanks

2nd Reviewer: Q

Page: Reviewer:

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

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

Was a method blank analyzed for each matrix?

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

VN N/A Was the blank contaminated? If yes, please see qualification below.

Associated Samples: Conc. units:

茅

Compound	Blank ID				S	Sample Identification	
	SBLKI		7	3	4	2	
99	0.020						
77	0,22	0.20 /y	0.23/4	0.18/4	5.23/4 0.18/4 0.20 /4 (5.4)	(N. 4)	
S	080.0	0,080 0.088/4	0,10/4	0.066/4	0.10/4 0.066/4 0.13/4 0.08/11	11/200	
NN	0,040				(6,20)	6.20 0.059/4	
						,	

Blank analysis date: Blank extraction date:

	ation				
	Sample Identification				
::					
Associated Samples:					
Associ					
	Blank ID				
conc. units:	Compound				
5					

21257 Fx LDC#: SDG#:

VALIDATION FINDINGS WORKSHEET

Page: 1 of 19

Reviewer: 31/6

2nd Reviewer:\_\_\_

Field Blanks

Were target compounds detected in the field blanks? Y N N/A Were target compounds detected in the Were target and the Were target compounds detected in the Were target and the Were targe METHOD: GC/MS BNA (EPA SW 846 Method 8270)
Y N N/A Were field blanks identified in this SDG?

Associated Samples: 땁 Field blank type: (circle one) Field Blank / Rinsate / Other:\_

4

	7,			7	1			
tion								
Sample Identification								
	4		1.4/4	0.26 /y			0.066/y	a 13/u
Blank iD	Ŋ	0.33	3.0	5.9	3.0	0.62	0.039	0.088
pun		AAA	XX	77	ઝ	去在在	Я	5
Compound								CROL

Associated sample units: Blank units:

Sampling date:\_

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

Associated Samples:

Sample Identification 6.20 0.059 Blank ID Compound

sx- phthatale

2x- 40 opens

FBLKASC2tronox.wpd

SDG #: See Govery LDC# 2/257

**VALIDATION FINDINGS WORKSHEET** Field Blanks

Page: Reviewer: 2nd Reviewer:

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

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

Were target compounds detected in the field blanks?

49 /L Associated sample units: 45 Blank units:

PB Sampling date: 6 /16 /6 /3 Field blank type: (circle one) Field Blank / Rinsate / Other:

1 3

Field blank type: (circle one) Field Blank / Rinsate / Other: PB Associated Samples:	Blank ID Sample Identification	PB 06 16 08 P   2   3	(2)	5 0.085 0.088/4 15 10/4 12.066/4 5 0.085 0	1		
Field Blank / R	Blank ID	PB 06 16 08B	0,2/	0.085 0.			
Field blank type: (circle one)	Compound	<b>美工作</b>	E TH	S			Zac

Associated sample units: 46 Blank units: 46 /L Sampling date: 6

3

( t q)

Compound	Blank ID				Sai	Sample Identification	ition		
THE THE TANK OF THE PROPERTY O	## FB 0634 08 GW AREA!	SW AREA!		7	٤ .				
717	81.0 77		0,20/4	6.20/4 0.23/4	0,18/4				
5	270.0		8.088/11	3.088/4 0,10/4 0,066/4	0,066/U				
								•	
CROL									

ax- ny etress SX- Phthalates

125 12 LDC#: SDG #:

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

Page: Of 9 Reviewer:\_\_\_ 2nd Reviewer.

METHOD: GC/MS BNA (EPA SW 848 Method 8270)

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

Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated Y)N N/A

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?

( US/LADIL) 3 3 Qualifications N & gral Associated Samples 6 3 RPD (Limits) 9 n y (47-1)9) ( asi-as ) なニシ (611-36) (211-65) (42-130) 139-122 (411-45) (511753) (511 - 15)(54-819) (31-22) (47-116 55-13 MSD %R (Limits) 7 V N B σ ч Ø ß (47-119) (512-)13) (21-12) (411-45) をニタ (ST - PR) ( 611-98 ) (47-116) (42 - 190)(光三万) (50-150) (5(1-15) (54-51A) MS %R (Limits) 56 ry ev E 33 ñ 35 ^ M 0 'n オナエ Compound 111 K K K 900  $\frac{3}{2}$ ИИ RRR 99 アア ξ MS/MSD ID Date Y N NA

A. Phenot		(Soil)	RPD (Soll)	QC Limits (Water)	RPD (Water)		Compound	QC Limits (Soil)	RPD (Soll)	QC Limits (Water)	· RPD (Water)
		26-90%		12-110%	<u>≤ 42%</u>	99 .	Acenaphthene	31-137%	<u>≤</u> 19%	46-118%	≥31%
C. 2-Chlorophenol	rophenol	25-102%	× 50%	27-123%	< 40%	=	4-Nitrophenol	11-114%	< 50%	10-80%	< 50%
E. 1,4-Dic	,4-Dichlorobenzene	28-104%	< 27%	%26-9E	< 28%	χ Έ	2,4-Dinitrotoluene	28-89%	< 47%	24-96%	< 38%
J. N-NITO	N-Nitroso-di-n-propylamine	41-126%	< 38%	41-116%	< 38%	Ë	Pentachiorophenol	17-109%	< 47%	9-103%	< 50%
R. 1,2,4-T	1,2,4-Trichlorobenzene	38-107%	< 23%	39-98%	< 28%	Z	Pyrene	35-142%	< 36%	26-127%	<31%
V. 4-Chlor	4-Chloro-3-methylphenol	26-103%	< 33%	23-97%	< 42%						

SDG #: 2/257 FX

## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Page: 1 of / Reviewer: 3772 2nd Reviewer: 4

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

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

N/A. Was a LCS required?

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

ſ		1		T	T	Т	T	Т	T	T	Т	T	Τ	Τ	T	T	Τ	T	T	Τ	T	Т	Т	T	T
	N A CALL OF THE PARTY OF THE PA	The guest MS/	₹																						
Accordated Remotes	4 トトン								,																
RPD (Limits)					`	-	`	`		`	(		,	,	,	~	•	( )	(	(	-	( )	`	~	,
LCSD %R (Linits)	+8 (50-120)	78 (	-		^		`	^	( )	1	_		( )	( )		( )	(	( )	`	( )	( )	( )	7		~
LCS %R (Limits)	45 (30-120)	- 1 · %	-	( )	( )	^ )	^ )	( )		( )	( )	) (	(	1 (	( )	( )	,	( )	( )	( )	( )	( )	( )	( )	( )
	חחת	•																							
O GCST/CS)	18/21	•												į											
Ø Date																									

LDC# 21257 F24

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

Reviewer:\_\_ 2nd reviewer:

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

Y	N	N/A
$(\mathbf{X}$	N	N/A

	Concentration ( U 5 /L)		p://
Compound		\ \gamma \	Diff FRED.
1-1	0.20	0,23	0.03 (45.1)
чиц	1.0	0.96	0.04 ( 2.0)
<u>.</u>	0,088	0.10	0.012 (40.20)
		3	1
		<u>.</u>	
	Concentrati	on ( )	
Compound	Concentrati	00 ( )	RPD
Compound	Concentrati		RPD
Compound	Concentrati	on ( )	RPD
Compound	Concentrati		RPD
Compound	Concentrati	on ( )	RPD

	Concentratio	p.4	·
Compound			RPD
	1		
	•		
		•	

	Concentration ()		
, Compound			RPD .
·			

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

**Project/Site Name:** 

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 7 through July 8, 2008

LDC Report Date:

August 20, 2009

Matrix:

Soil

Parameters:

Semivolatiles

Validation Level:

Stage 2B

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2844862

### Sample Identification

SA47-0.5B

SA47-10B

SA47-20B

SA47-30B

SA47-35B

SA183-0.5B

SA67-0.5B

SA67-10B

SA67-20B

SA67-30B

SA67-35B

RSAN2-0.5B

RSAN2-10B

RSAN2-20B

RSAN2-10BMS

RSAN2-10BMSD

### Introduction

This data review covers 16 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

### III. Initial Calibration

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 15.0% for each individual compound and less than or equal to 30.0% for calibration check compounds (CCCs).

In the case where %RSD was greater than 15.0%, the laboratory used a calibration curve to evaluate the compound. All coefficients of determination (r<sup>2</sup>) were greater than or equal to 0.990.

For the purposes of technical evaluation, all compounds were evaluated against the 30.0% (%RSD) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

Average relative response factors (RRF) for all semivolatile target compounds and system performance check compounds (SPCCs) were greater than or equal to 0.05 as required.

### 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).

For the purposes of technical evaluation, all compounds were evaluated against the 25.0% (%D) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

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 RRF values were greater than or equal to 0.05.

#### 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
SBLK1	7/16/08	Bis(2-ethylhexyl)phthalate Phenanthrene	360 ug/Kg 1.2 ug/Kg	All samples in SDG R2844862

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	Reported	Modified Final
	TIC (RT in minutes)	Concentration	Concentration
SA47-10B	Bis(2-ethylhexyl)phthalate	140 ug/Kg	140U ug/Kg
SA47-20B	Bis(2-ethylhexyl)phthalate	340 ug/Kg	340U ug/Kg
	Phenanthrene	2.4 ug/Kg	2.4U ug/Kg
SA47-30B	Bis(2-ethylhexyl)phthalate	430 ug/Kg	430U ug/Kg
SA47-35B	Bis(2-ethylhexyl)phthalate	140 ug/Kg	140U ug/Kg
	Phenanthrene	2.3 ug/Kg	2.3U ug/Kg
SA183-0.5B	Bis(2-ethylhexyl)phthalate	240 ug/Kg	240U ug/Kg
	Phenanthrene	1.5 ug/Kg	1.5U ug/Kg
SA67-0.5B	Bis(2-ethylhexyl)phthalate	110 ug/Kg	110U ug/Kg
SA67-10B	Bis(2-ethylhexyl)phthalate	240 ug/Kg	240U ug/Kg
	Phenanthrene	1.3 ug/Kg	1.3U ug/Kg
SA67-20B	Phenanthrene	2.0 ug/Kg	2.0U ug/Kg
SA67-30B	Bis(2-ethylhexyl)phthalate	380 ug/Kg	380U ug/Kg
	Phenanthrene	2.3 ug/Kg	2.3U ug/Kg
SA67-35B	Bis(2-ethylhexyl)phthalate	210 ug/Kg	210U ug/Kg
RSAN2-0.5B	Bis(2-ethylhexyl)phthalate	110 ug/Kg	110U ug/Kg
	Phenanthrene	2.2 ug/Kg	2.2U ug/Kg
RSAN2-10B	Bis(2-ethylhexyl)phthalate	110 ug/Kg	110U ug/Kg
	Phenanthrene	2.2 ug/Kg	2.2U ug/Kg

Sample	Compound	Reported	Modified Final
	TIC (RT in minutes)	Concentration	Concentration
RSAN2-20B	Bis(2-ethylhexyl)phthalate	100 ug/Kg	100U ug/Kg
	Phenanthrene	2.2 ug/Kg	2.2U ug/Kg

No field blanks were identified in this SDG.

#### VI. Surrogate Spikes

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

#### VII. Matrix Spike/Matrix Spike Duplicates

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

Spike ID (Associated Samples) Compound		MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P	
RSAN2-10BMS/MSD Bis(2-ethylhexyl)phthalate (RSAN2-10B)		159 (50-150)	250 (50-150)	32 (≤30)	J (all detects)	Р	

Although the MS/MSD percent recoveries (%R) were not within QC limits for two compounds, the MS and LCS/D percent recoveries (%R) were within QC limits and no data were qualified.

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

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

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Flag	A or P
LCS/D1 (All samples in SDG R2844862)	Bis(2-ethylhexyl)phthalate	270 (50-120)	323 (50-120)	-	J+ (all detects)	Р

#### IX. Regional Quality Assurance and Quality Control

Not applicable.

#### X. Internal Standards

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

#### XI. Target Compound Identifications

Raw data were not reviewed for this SDG.

#### XII. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R2844862	All compounds reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

#### XIII. Tentatively Identified Compounds (TICs)

Raw data were not reviewed for this SDG.

#### XIV. System Performance

Raw data were not reviewed for this SDG.

#### XV. Overall Assessment

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

#### XVI. Field Duplicates

No field duplicates were identified in this SDG.

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG R2844862

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R2844862	RSAN2-10B	Bis(2-ethylhexyl)phthalate	J (all detects)	Р	Matrix spike/Matrix spike duplicates (%R)(RPD) (m, ld)
R2844862	SA47-0.5B SA47-10B SA47-20B SA47-30B SA47-35B SA183-0.5B SA67-0.5B SA67-10B SA67-20B SA67-30B SA67-35B RSAN2-0.5B RSAN2-0.5B RSAN2-10B	Bis(2-ethylhexyl)phthalate	J+ (all detects)	Р	Laboratory control samples (%R) (I)
R2844862	SA47-0.5B SA47-10B SA47-20B SA47-35B SA47-35B SA67-0.5B SA67-10B SA67-20B SA67-20B SA67-30B SA67-35B RSAN2-0.5B RSAN2-0.5B	All compounds reported below the PQL.	J (all detects)	Α	Project Quantitation Limit (sp)

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG R2844862

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
R2844862	SA47-10B	Bis(2-ethylhexyl)phthalate	140U ug/Kg	А	bl
R2844862	SA47-20B	Bis(2-ethylhexyl)phthalate Phenanthrene	340U ug/Kg 2.4U ug/Kg	A	bl
R2844862	SA47-30B	Bis(2-ethylhexyl)phthalate	430U ug/Kg	А	bl
R2844862	SA47-35B	Bis(2-ethylhexyl)phthalate Phenanthrene	140U ug/Kg 2.3U ug/Kg	А	bl

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
R2844862	SA183-0.5B	Bis(2-ethylhexyl)phthalate Phenanthrene	240U ug/Kg 1.5U ug/Kg	А	ld
R2844862	SA67-0.5B	Bis(2-ethylhexyl)phthalate	110U ug/Kg	А	bl
R2844862	SA67-10B	Bis(2-ethylhexyl)phthalate Phenanthrene	240U ug/Kg 1.3U ug/Kg	А	ld
R2844862	SA67-20B	Phenanthrene	2.0U ug/Kg	А	bl
R2844862	SA67-30B	Bis(2-ethylhexyl)phthalate Phenanthrene	380U ug/Kg 2.3U ug/Kg	А	ld
R2844862	SA67-35B	Bis(2-ethylhexyl)phthalate	210U ug/Kg	А	bl
R2844862	RSAN2-0.5B	Bis(2-ethylhexyl)phthalate Phenanthrene	110U ug/Kg 2.2U ug/Kg	А	bl
R2844862	RSAN2-10B	Bis(2-ethylhexyl)phthalate Phenanthrene	110U ug/Kg 2.2U ug/Kg	А	bl
R2844862	RSAN2-20B	Bis(2-ethylhexyl)phthalate Phenanthrene	100U ug/Kg 2.2U ug/Kg	А	ld

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles- Field Blank Data Qualification Summary - SDG R2844862

No Sample Data Qualified in this SDG

#### **Tronox Northgate Henderson**

LDC #:	21257G2a		VALIDA <sup>-</sup>		ĔTENE		SHEET
SDG #:	R2844862		_	Sta	age 2B		
Laborator	y: Columbia	Analytical	Services				

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

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 7 /07- 08/08
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	A	20 RSD r2 car/ca & 25 2
IV.	Continuing calibration/ICV	A	ca/a 6252
V.	Blanks	SW	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	SM	
VIII.	Laboratory control samples	2W	165/b
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	N	
XII.	Compound quantitation/CRQLs	N	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	Ν	
XV.	Overall assessment of data	A	
XVI.	Field duplicates	N	
XVII.	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:

Cail

	2011				<del></del>		
1	SA47-0.5B	11	SA67-35B	21	SBIEL	31	
2	SA47-10B	12	RSAN2-0.5B	22		32	
3	SA47-20B	13	RSAN2-10B	23		33	
4	SA47-30B	14	RSAN2-20B	24		34	
5	SA47-35B	15	RSAN2-10BMS	25		35	
6	SA183-0.5B	16	RSAN2-10BMSD	26		36	
7	SA67-0.5B	17		27		37	
8	SA67-10B	18		28		38	
9	SA67-20B	19		29		39	
10_	SA67-30B	20		30		40	

# **VALIDATION FINDINGS WORKSHEET**

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

A Phenol**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenol**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	VV. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene⁴	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chioro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chiorophenyl-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-Chioronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	ПТ.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	ກກກ
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	WV.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	WWW.

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

LDC#: 21 257 624 SDG #:

## **VALIDATION FINDINGS WORKSHEET** Blanks

Reviewer: TV6 Page: of 2nd Reviewer:

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

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

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

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

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

 $\sqrt{N/A}$  Was the blank contaminated? If yes, please see qualification below. Blank extraction date:  $\sqrt{\frac{16}{68}}$  Blank analysis date:  $\sqrt{\frac{236}{6}}$ 

2.0/4 240 N -ر در Ø 240/4 Sample Identification 140/2 8.33 430 'n 7 Associated Samples: \_ 340 <u>4</u> 12 Blank ID SBIKI 360 7. **七七年** 2 Compound Conc. units:

1860 4 Blank analysis date:\_ Blank extraction date:\_ Conc. units:

above Ş same

Associated Samples:

Compound	Blank ID				S	Sample Identification	ion	
	SB1K)	10	11	12	13	14		
<b>年</b>	360	h/086	m/ o10	h/o11 h/	110/11	h/001		
nn		1/8.2	(5'0)	12.2/4	2.2/4	2.2/4		
		,	)	,	,	,		

LDC#: 21257 624 SDG#: Ca Groy

# VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Page: Reviewer: 2nd Reviewer:

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

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

Note 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

N N A

MS/MSD. Soil / Water.

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

MSi ٤ Qualifications No rack JA dets/P No mus Associated Samples 20 RPD (Limits) 32 (52/25) MSD %R (Limits) 232 40 173 (20-120)MS %R (Limits) 29 4 Compound 市内市 RRR MS/MSD ID Date Y N M/A

	Compound	QC Limits (Soil)	RPD (Soll)	QC Limits (Water)	RPD (Water)		Compound	QC Limits (Soli)	RPD (Soil)	QC Limits (Water)	RPD (Water)
Ą.	Phenol	26-90%	~32% ~	12-110%	<u>&lt; 42%</u>	9g .	Acenaphthene	31-137%	<u>≤ 19%</u>	46-118%	≥ 31%
Ö	2-Chlorophenol	25-102%	× 20%	27-123%	< 40%	=	4-Nitrophenoi	11-114%	< 50%	10-80%	× 20%
ш	1,4-Dichlorobenzene	28-104%	< 27%	36-97%	< 28%	χ Ξ	2,4-Dinitrotoluene	28-89%	< 47%	24-96%	< 38%
٦	N-Nitroso-di-n-propylamine	41-126%	< 38%	41-116%	< 38%	Ë	Pentachlorophenot	17-109%	< 47%	9-103%	< 50%
œ	1,2,4-Trichlorobenzene	38-107%	< 23%	39-98%	< 28%	12	Pyrene	35-142%	< 36%	26-127%	< 31%
>	4-Chloro-3-methylphenol	26-103%	< 33%	23-97%	< 42%						

LDC# 2/257 624 SDG #: 524 Corry

## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Page: Reviewer:

2nd Reviewer:

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

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

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

# Date	CSACSD ID		LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Serroles	Ottailfoodlane
	14/571	EFF	270 (50-126)	323 (50-120)		411 + 8K	J+ dets/p 11 1
			( )	( )	( )		
			( )	( )			
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### Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 8 through July 11, 2008

**LDC Report Date:** 

August 31, 2009

Matrix:

Water

Parameters:

Semivolatiles

Validation Level:

Stage 2B

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2844866

#### Sample Identification

M-39B

TR-2B

M-69B

M-69BRE

I-BB

I-BBRE

M-96BF

M-48B

TR-4B

CLD3-RB

CLD1-RB

M-124B

M-123B

M-96BFMS

M-96BFMSD

#### Introduction

This data review covers 15 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P
M-124B M-123B	All TCL compounds	Cooler temperature was reported at 8°C upon receipt by the laboratory.	Cooler temperature must be 4±2°C .	J- (all detects) UJ (all non-detects)	А

#### II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

#### III. Initial Calibration

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 15.0% for each individual compound and less than or equal to 30.0% for calibration check compounds (CCCs).

In the case where %RSD was greater than 15.0%, the laboratory used a calibration curve to evaluate the compound. All coefficients of determination (r²) were greater than or equal to 0.990.

For the purposes of technical evaluation, all compounds were evaluated against the 30.0% (%RSD) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

Average relative response factors (RRF) for all semivolatile target compounds and system performance check compounds (SPCCs) were greater than or equal to 0.05 as required.

#### 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).

For the purposes of technical evaluation, all compounds were evaluated against the 25.0% (%D) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

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 RRF values were greater than or equal to 0.05.

#### V. Blanks

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

Sample FB062408GWAREA1 (from SDG R2844650) was identified as a field blank. No semivolatile contaminants were found in this blank with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB062408GWAREA1	6/24/08	Diethylphthalate Naphthalene	0.18 ug/L 0.075 ug/L	All samples in SDG R2844866

Sample PB061608B (from SDG R2844538) was identified as a pump blank. No semivolatile contaminants were found in this blank with the following exceptions:

Pump Blank ID	Sampling Date	Compound	Concentration	Associated Samples
PB061608B	6/16/08	Bis(2-ethylhexyl)phthalate Naphthalene	0.21 ug/L 0.085 ug/L	M-39B TR-2B M-69B M-69BRE I-BB I-BBRE M-96BF M-48B CLD3-RB CLD1-RB M-124B M-123B

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

Sample	Compound	Reported Concentration	Modified Final Concentration
M-39B	Naphthalene	0.075 ug/L	0.075U ug/L

Sample	Compound	Reported Concentration	Modified Final Concentration
TR-2B	Bis(2-ethylhexyl)phthalate	0.22 ug/L	0.22U ug/L
M-96BF	Naphthalene Diethylphthalate	0.066 ug/L 0.14 ug/L	0.066U ug/L 0.14U ug/L
M-48B	Bis(2-ethylhexyl)phthalate Naphthalene Diethylphthalate	0.25 ug/L 0.12 ug/L 0.26 ug/L	0.25U ug/L 0.12U ug/L 0.26U ug/L
CLD3-RB	Naphthalene	0.094 ug/L	0.094U ug/L
M-124B	Naphthalene	0.056 ug/L	0.056U ug/L
M-39B	Naphthalene	0.075 ug/L	0.075U ug/L
CLD1-RB	Diethylphthalate Naphthalene	0.13 ug/L 0.075 ug/L	0.13U ug/L 0.075U ug/L

#### VI. Surrogate Spikes

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

#### VII. Matrix Spike/Matrix Spike Duplicates

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

Spike ID (Associated Samples)	Compound	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
M-96BFMS/MSD (M-96BF)	Pyridine	33 (50-150)	40 (50-150)	-	J- (all detects) UJ (all non-detects)	А

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

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

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Flag	A or P
LCS/D1 (M-39B TR-2B M-69B M-69BRE I-BB I-BBRE M-96BF M-48B TR-4B)	Pyridine	16 (50-120)	0 (50-120)	200 (≤30)	J (all detects) R (all non-detects)	Р
LCS/D2 (CLD3-RB CLD1-RB M-124B M-123B)	1,4-Dioxane Pyridine	35 (50-120) 30 (50-120)	43 (50-120) 25 (50-120)	-	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	Р

#### IX. Regional Quality Assurance and Quality Control

Not applicable.

#### X. Internal Standards

All internal standard areas and retention times were within QC limits with the following exceptions:

Sample	Internal Standards	Area (Limits)	Compound	Flag	A or P
I-BB	Perylene-d12	80747 (115242-460968)	Di-n-octylphthalate Benzo (b)fluoranthene Benzo (k)fluoranthene Benzo (a) pyrene Indeno (1,2,3-cd) pyrene Dibenz (a,h) anthracene Benzo (g,h,i) perylene	J (all detects) UJ (all non-detects)	А
I-BBRE	Perylene-d12	19439 (115242-460968)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) R (all non-detects)	А
M-69B	Perylene-d12	41859 (115242-460968)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) R (all non-detects)	А

Sample	Internal Standards	Area (Limits)	Compound	Flag	A or P
M-69BRE	Perylene-d12	29800 (115242-460968)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) R (all non-detects)	A

#### XI. Target Compound Identifications

Raw data were not reviewed for this SDG.

#### XII. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R2844866	All compounds reported below the PQL.	J (all detects)	Α

Raw data were not reviewed for this SDG.

#### XIII. Tentatively Identified Compounds (TICs)

Raw data were not reviewed for this SDG.

#### XIV. System Performance

Raw data were not reviewed for this SDG.

#### XV. Overall Assessment

The overall assessment of data was acceptable. In the case where more than one result was reported for an individual sample, the least technically acceptable results were rejected as follows:

Sample	Compound	Flag	A or P
M-69BRE I-BBRE	All TCL compounds	x	А

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

#### XVI. Field Duplicates

No field duplicates were identified in this SDG.

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG R2844866

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R2844866	M-124B M-123B	All TCL compounds	J- (all detects) UJ (all non-detects)	Α	Cooler temperature (st)
R2844866	M-96BF	Pyridine	J- (all detects) UJ (all non-detects)	А	Matrix spike/Matrix spike duplicates (%R) (m)
R2844866	M-39B TR-2B M-69B M-69BRE I-BB I-BBRE M-96BF M-48B TR-4B	Pyridine	J (all detects) R (all non-detects)	Р	Laboratory control samples (%R)(RPD) (I)
R2844866	CLD3-RB CLD1-RB M-124B M-123B	1,4-Dioxane Pyridine	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	Р	Laboratory control samples (%R) (I)
R2844866	I-BB	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) UJ (all non-detects)	A	Internal standards (area) (i)
R2844866	I-BBRE M-69B M-69BRE	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) R (all non-detects)	A	Internal standards (area) (i)
R2844866	M-39B TR-2B M-69B M-69BRE I-BB I-BBRE M-96BF M-48B TR-4B CLD3-RB CLD1-RB M-124B M-123B	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R2844866	M-69BRE I-BBRE	All TCL compounds	Х	А	Overall assessment of data (o)

#### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG R2844866

#### No Sample Data Qualified in this SDG

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles- Field Blank Data Qualification Summary - SDG R2844866

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
R2844866	M-39B	Naphthalene	0.075U ug/L	А	bp,bf
R2844866	TR-2B	Bis(2-ethylhexyl)phthalate	0.22U ug/L	А	bp
R2844866	M-96BF	Naphthalene	0.066U ug/L	А	bp,bf
R2844866	M-96BF	Diethylphthalate	0.14U ug/L	А	bf
R2844866	M-48B	Bis(2-ethylhexyl)phthalate	0.25U ug/L	Α	bp
R2844866	M-48B	Naphthalene	0.12U ug/L	А	bp,bf
R2844866	M-48B	Diethylphthalate	0.26U ug/L	А	bf
R2844866	CLD3-RB	Naphthalene	0.094U ug/L	Α	bp,bf
R2844866	CLD1-RB	Naphthalene	0.075U ug/L	А	bp,bf
R2844866	CLD1-RB	Diethylphthalate	0.13U ug/L	А	bf
R2844866	M-124B	Naphthalene	0.056U ug/L	А	bp,bf

#### **Tronox Northgate Henderson**

LDC #:	21257H2a	VALIDATION COMPLETENESS WORKSHEET
SDG #:_	R2844866	Stage 2B
Laborato	ry: Columbia A	alytical Services

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	SW	Sampling dates: 7/68 – 11 /08
11.	GC/MS Instrument performance check	¥	
111.	Initial calibration	A	2 KSD r
IV.	Continuing calibration/ICV	JVENT A	COV/100 = 25 %
V.	Blanks	A	
VI.	Surrogate spikes	Ā	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	WZ	KS/D
IX.	Regional Quality Assurance and Quality Control	N	·
Χ.	Internal standards	SW	
XI.	Target compound identification	N	
XII.	Compound quantitation/CRQLs	N	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	N	
XV.	Overall assessment of data	SN)	
XVI.	Field duplicates	N	
XVII.	Field blanks	700	PB = PB061608B from R2844538

FB = FB0624086W AREA; from R2844650

Note: A = Acceptable

N = Not provided/applicable SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate TB = Trip blank

EB = Equipment blank

Validated Samples:

	Water				***************************************		
1 )	M-39B	11	CLD1-RB	27	SBLKI	31	
2 )	TR-2B	12 2	M-124B	<del>2</del> 2	SBIKZ	32	
3	M-69B	137	M-123B	23		33	***************************************
4	M-69BRE	14	M-96BFMS	24		34	
5	I-BB	15 1	M-96BFMSD	25		35	
6 1	I-BBRE	16		26		36	MANAGEMENT AND
7 1	M-96BF	17		27		37	
8 1	M-48B	18		28	<u> </u>	38	
9 1	TR-4B	19		29		39	
102	CLD3-RB	20		30		40	

# **VALIDATION FINDINGS WORKSHEET**

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

A. Phenol™	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenol**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	W. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	000. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	OO. 4-Nitroaniline	DDD, Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	тт. Оста chloro styrone
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate™	uuu 174 - Dioxane
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	ww.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	www.

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

LDC #:		7 H 2a
SDG #:	Sa	Cores

#### VALIDATION FINDINGS WORKSHEET Technical Holding Times

Page:	of	1
Reviewer:	$ar{ar{\iota}}$	VC
2nd Reviewer:	7	

All circled dates have exceeded the technical holding times.

YN N/A Were all cooler temperatures within validation criteria?

Sample ID	Matrix	Preserved	Sampling Date	Extraction date	Analysis date	Total # of Days	Qualifie
12, 13	Cor	ter t	emp = 8°C				J-/UJ /A
							,
	-						

#### **TECHNICAL HOLDING TIME CRITERIA**

Water:

Extracted within 7 days, analyzed within 40 days.

Soil:

Extracted within 14 days, analyzed within 40 days.

LDC# 21257#72 See Cores SDG #:

# **VALIDATION FINDINGS WORKSHEET** Field Blanks

Page: of 2 Reviewer: N6

2nd Reviewer:\_

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

Were target compounds detected in the field blanks?

Slank units: 45/L Associated sample units: 45/L

Sampling date: 6/6/8 X N N/A

Were field blanks identified in this SDG?

3:

0,17

0,056/4 h 6 0.094/4 0.075/4 All except Sample Identification Associated Samples: ~ 0.7 0,25 1/220 0.22 N Field blank type: (circle one) Field Blank / Rinsate / Other: 0.075 PB 06 16 08B 0.085 Blank ID 0, 2/ EFE S Compound

Associated sample units: Blank units:

Sampling date:

CROL

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

Associated Samples:

Compound	Blank ID		Sa	Sample Identification	fion		
	L.						
							-
CROL							

2x- ny others SX- Phthalates

LDC# 7 27# 20 SDG #:

# **VALIDATION FINDINGS WORKSHEET**

Field Blanks

Page: 76f 7 Reviewer:\_ 2nd Reviewer:

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

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

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

Blank units: 45 / Associated sample units: 45 / Associated sample

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

=

0.050/11 Sample Identification 0,075/4 0.13/4 Associated Samples: 0.094/4 0,26, 6.12 1/2900 0.14 /y FBOG 2408 GW XREAD Blank ID W/2100 6/24/08 20,0 0.78 Compound

Associated sample units:\_ Blank units:

Sampling date:

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

Associated Samples:

Compound	Blank ID	Sample Identification	
CRQL			

LDC #: 21257 #24 SDG #: Su Car

# VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Page: 1 of 1 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 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 K)N N/A

MS/MSD. Soil / Water.

Y (N) N/A	<b>₹</b>	MS/MSD. Soil / Water. Was a MS/MSD analyzed every 20 samples of each Were the MS/MSD percent recoveries (%R) and the	r. rzed every 2t rcent recove	o samples iries (%R)	of each me and the rela	matrix? relative perc	ent differen	matrix? relative percent differences (RPD) within the QC limits?	QC limits?		
*	Date	GI OSW/SW	Compound	- X	MS %R (Limite)	W %	MSD %R (1 im/ts)	( a) Coo	A Company of Company o		
		14/15	//	40	( <u>at 154</u> )	4	加气场		4	) No FLA!	(76/5)7)
			171	28	( 36-119)		25 (36-119)	( )			
			RRR	33	(651-05)	40	(50-150)	( )		J-/45/A	( in the second
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					( )		( )	( )			
-					( )		( )	( )			
					( )		( )	( )			
					( )		(	(			

	Compound	QC Limits (Soil)	RPD (Soll)	QC Limits (Water)	RPD (Water)		Compound	QC Limits (Soll)	RPD (Soll)	QC Limits (Water)	RPD (Water)
₹	Phenol	26-90%	%3€>	12-110%	<u>≤ 42%</u>	99	Acenaphthene	31-137%	< 19%	46-118%	<u>&lt;</u> 31%
ن	2-Chlorophenol	25-102%	< 50%	27-123%	< 40%	11.	4-Nitrophenol	11-114%	< 50%	10-80%	× 60%
ш	1,4-Dichlorobenzene	28-104%	< 27%	36-97%	< 28%	χ Έ	2,4-Dinitrotoluene	%8-87	< 47%	24-96%	< 38%
٦	N-Nitroso-di-n-propylamine	41-126%	< 38%	41-116%	< 38%	Ë	Pentachlorophenol	17-109%	< 47%	9-103%	< 50%
œ	1,2,4-Trichlorobenzene	38-107%	< 23%	39-98%	< 28%	22.	Pyrene	35-142%	× 36%	26-127%	< 31%
>	4-Chloro-3-methylphenol	26-103%	< 33%	23-97%	< 42%						

LDC#: 21257H2x SDG #: See (was)

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

Page: \_of\_ Reviewer:

2nd Reviewer. \_

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

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

| N/A | Was a LCS required? Y N N/A

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

Date   LCSALCED ID   Compound   SR (Limba)   SR (Limba)   SR (Limba)   SR (Limba)   SR (Limba)   Compound   SR (Limba)   Compound   SR (Limba)   Compound   SR (Limba)   Compound   Compo	L		The state of the s								
VUU   35 (50-120)   42 (50-120)   10 (10)	*	Date	LCSACSD ID	Compound	, , , , , , , , , , , , , , , , , , ,	.cs Limits)	LCSC	<b>1</b>	( a)		
RRK       16 (1) 0 (1) 30 (30)         (1) (1) (1) (1) (1)       (1) (1) (1)         LUMY 35 (50-120) 43 (50-120)       (1) (1) (1)         RPK       30 (1) (1) (1) (1)         (1) (1) (1) (1) (1)       (1) (1) (1)         (1) (1) (1) (1) (1)       (1) (1) (1)         (1) (1) (1) (1) (1)       (1) (1) (1)         (2) (1) (1) (1) (1) (1)       (1) (1) (1)         (2) (1) (1) (1) (1) (1)       (1) (1) (1)         (2) (1) (1) (1) (1) (1)       (1) (1) (1)         (2) (1) (1) (1) (1) (1)       (1) (1) (1)         (2) (1) (1) (1) (1) (1)       (1) (1) (1)         (2) (1) (1) (1) (1) (1)       (1) (1) (1)         (2) (1) (1) (1) (1) (1)       (1) (1) (1)			1 cs/p 1	מממ	35	( 52/- 05 )		30-12n )		Associated Samples	Qualifications
NUM 25 (50-120) (1) (1) (1) (1) (1) (1) (1) (1) (1) (1			,	RRR	5	^ 		_	1~	L	10 /0 /1 115/1950
UMUY       255 (50-120)       43 (50-120)       ( )       (								Ŷ	~		(A) (A)
uuu       35 (50-120)       ( )	L					1	_	^	(		
UMU       35       (30-120)       (1)       (1)         PPR       30       (1)       (1)       (1)         (1)       (1)       (1)       (1)         (1)       (1)       (1)       (1)         (1)       (1)       (1)       (1)         (1)       (1)       (1)       (1)         (1)       (1)       (1)       (1)         (1)       (1)       (1)       (1)         (1)       (1)       (1)       (1)         (1)       (1)       (1)       (1)         (1)       (1)       (1)       (1)         (2)       (1)       (1)       (1)         (2)       (1)       (1)       (1)         (2)       (1)       (1)       (1)         (2)       (1)       (1)       (1)         (2)       (1)       (1)       (1)         (2)       (2)       (2)       (2)       (2)         (2)       (2)       (2)       (2)       (2)         (2)       (2)       (2)       (2)       (2)         (2)       (2)       (2)       (2)       (2) <t< th=""><th></th><th></th><th></th><th></th><th></th><th>,</th><th>_</th><th>(</th><th>( )</th><th></th><th></th></t<>						,	_	(	( )		
			762/2 2	מממ	35	(50-120)		1901-02	`	10-13 CRIFZ	J-1/1 1
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DC #: 2	SDG #:

## VALIDATION FINDINGS WORKSHEET Internal Standards

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

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

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

Were all internal standard area counts within -50 to +100 of the associated calibration standard? Y) N N/A

Were the retention times of the internal standards within +/- 30 seconds of the retention times of the associated calibration standard?

	:-	-				- -	7	HH	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	Ш							
Qualifications	JMI/A /		J/R/A					(grue FFF 6GB HH	1 1 1 555 KKK								
Area (Limits) RT (Limits) Qu	460 968)																
	460																
	1 77	-					3										
	80747 ( 115 242 -		19 439	4: 800	11 059	29 800											
Internal Standard	PRY																
Sample ID	5 #		9	r		4											
Date																	
#			-7-	*													
			भे	7													

IS1 (DCB) = 1,4-Dichlorobenzene-d4 IS2 (NPT) = Naphthalene-d8 IS3 (ANT) = Acenaphthene-d10 \* QC limits are advisory

(x < 251 K)

IS4 (PHN) = Phenanthrene-d10 IS5 (CRY) = Chrysene-d12 IS6 (PRY) = Perylene-d12

LDC# 21257 HM SDG# Su Cover

# **VALIDATION FINDINGS WORKSHEET** Overall Assessment of Data

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

2nd Reviewer: \_\_

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

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

All available information pertaining to the data were reviewed using professional judgement to compliment the determination of the overall quality of the data.

Was the overall quality and usability of the data acceptable? Y N/A

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Qualifications	*/*										
Associated Samples	IS ontside limits										
Finding	runs for										
Sample ID	97										
Date											
#											

Comments:

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

**Project/Site Name:** 

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 8 through September 8, 2008

LDC Report Date:

August 18, 2009

Matrix:

Soil

Parameters:

Semivolatiles

Validation Level:

Stage 2B

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2844885

#### Sample Identification

RSAN2-30B

RSAN2-30BD

RSAN2-35B

RSAO2-0.5B

RSAO2-10B

RSAO2-20B

RSAO2-20BD

RSAO2-30B

RSAO2-33B

10/102 000

SA183-10B

SA183-10BD

SA183-20B

SA183-30B

SA183-33B

RSA04-0.5B

RSA04-10B

RSA04-20B

RSA04-30B RSA04-36B

RSA04-30BMS

RSA04-30BMSD

#### Introduction

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

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

#### III. Initial Calibration

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 15.0% for each individual compound and less than or equal to 30.0% for calibration check compounds (CCCs).

In the case where %RSD was greater than 15.0%, the laboratory used a calibration curve to evaluate the compound. All coefficients of determination (r<sup>2</sup>) were greater than or equal to 0.990.

For the purposes of technical evaluation, all compounds were evaluated against the 30.0% (%RSD) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

Average relative response factors (RRF) for all semivolatile target compounds and system performance check compounds (SPCCs) were greater than or equal to 0.05 as required.

#### 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).

For the purposes of technical evaluation, all compounds were evaluated against the 25.0% (%D) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

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 RRF values were greater than or equal to 0.05.

#### 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
SBLK1	7/15/08	Diethylphthalate Phenanthrene	3.8 ug/Kg 1.5 ug/Kg	All samples in SDG R2844885

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	Reported	Modified Final
	TIC (RT in minutes)	Concentration	Concentration
RSAN2-30B	Diethylphthalate	9.6 ug/Kg	9.6U ug/Kg
	Phenanthrene	3.0 ug/Kg	3.0U ug/Kg
RSAN2-30BD	Diethylphthalate	11 ug/Kg	11U ug/Kg
RSAN2-35B	Phenanthrene	2.4 ug/Kg	2.4U ug/Kg
RSAO2-0.5B	Diethylphthalate	9.3 ug/Kg	9.3U ug/Kg
	Phenanthrene	2.9 ug/Kg	2.9U ug/Kg
RSAO2-10B	Diethylphthalate	8.8 ug/Kg	8.8U ug/Kg
	Phenanthrene	2.9 ug/Kg	2.9U ug/Kg
RSAO2-20B	Phenanthrene	2.5 ug/Kg	2.5U ug/Kg
RSAO2-20BD	Diethylphthalate	4.5 ug/Kg	4.5U ug/Kg
	Phenanthrene	2.3 ug/Kg	2.3U ug/Kg
RSAO2-30B	Diethylphthalate	15 ug/Kg	15U ug/Kg
RSAO2-33B	Diethylphthalate	8.7 ug/Kg	8.7U ug/Kg
	Phenanthrene	2.9 ug/Kg	2.9U ug/Kg
SA183-10B	Diethylphthalate	8.8 ug/Kg	8.8U ug/Kg
SA183-10BD	Diethylphthalate	6.2 ug/Kg	6.2U ug/Kg
	Phenanthrene	2.5 ug/Kg	2.5U ug/Kg
SA183-20B	Diethylphthalate	6.5 ug/Kg	6.5U ug/Kg
	Phenanthrene	2.6 ug/Kg	2.6U ug/Kg

Sample	Compound TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
SA183-30B	Diethylphthalate	8.4 ug/Kg	8.4U ug/Kg
SA183-33B	Diethylphthalate	14 ug/Kg	14U ug/Kg
RSA04-10B	Diethylphthalate Phenanthrene	7.2 ug/Kg 2.1 ug/Kg	7.2U ug/Kg 2.1U ug/Kg
RSA04-20B	Diethylphthalate Phenanthrene	7.6 ug/Kg 2.5 ug/Kg	7.6U ug/Kg 2.5U ug/Kg
RSA04-30B	Diethylphthalate	11 ug/Kg	11U ug/Kg
RSA04-36B	Diethylphthalate	9.6 ug/Kg	9.6U ug/Kg

No field blanks were identified in this SDG.

#### VI. Surrogate Spikes

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

#### VII. Matrix Spike/Matrix Spike Duplicates

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

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

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

#### IX. Regional Quality Assurance and Quality Control

Not applicable.

#### X. Internal Standards

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

### XI. Target Compound Identifications

Raw data were not reviewed for this SDG.

### XII. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R2844885	All compounds reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

### XIII. Tentatively Identified Compounds (TICs)

Raw data were not reviewed for this SDG.

### XIV. System Performance

Raw data were not reviewed for this SDG.

### XV. Overall Assessment

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

### XVI. Field Duplicates

Samples RSAN2-30B and RSAN2-30BD, samples RSAO2-20B and RSAO2-20BD, and samples SA183-10B and SA183-10BD were identified as field duplicates. No semivolatiles were detected in any of the samples with the following exceptions:

	Concentrati	ion (ug/Kg)	555	D:#*		
Compound	RSAN2-30B	RSAN2-30BD	RPD (Limits)	Difference (Limits)	Flags	A or P
Butylbenzylphthalate	260U	5.3	-	254.7 (≤260)	-	
Diethylphthalate	9.6	11	-	1.4 (≤280)	-	-
Phenanthrene	3.0	6.2	-	3.2 (≤11)	-	-

	Concentrati	ion (ug/Kg)	DDD	D:#		
Compound	RSAO2-20B	RSAO2-20BD	RPD (Limits)	Difference (Limits)	Flags	A or P
Butylbenzylphthalate	6.4	230U	-	224 (≤230)	-	-
Diethylphthalate	250U	4.5	-	245.5 (≤250)	-	-
Phenanthrene	2.5	2,3	-	0.2 (≤9.7)	-	-

	Concentrat	ion (ug/Kg)		D:#*		
Compound	SA183-10B	SA183-10BD	RPD (Limits)	Difference (Limits)	Flags	AorP
Butylbenzylphthalate	3.2	190U	-	186.8 (≤190)	-	-
Diethylphthalate	8.8	6.2	-	2.6 (≤190)	-	-
Phenanthrene	3.1	2.5	-	0.6 (≤7.5)	-	_

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG R2844885

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R2844885	RSAN2-30B RSAN2-30BD RSAN2-35B RSAO2-0.5B RSAO2-10B RSAO2-20BD RSAO2-20BD RSAO2-33B SA183-10B SA183-10BD SA183-20B SA183-30B SA183-33B RSA04-0.5B RSA04-10B RSAO4-20B RSAO4-30B RSAO4-36B	All compounds reported below the PQL.	J (all detects)	Α	Project Quantitation Limit (sp)

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG R2844885

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
R2844885	RSAN2-30B	Diethylphthalate Phenanthrene	9.6U ug/Kg 3.0U ug/Kg	А	bl
R2844885	RSAN2-30BD	Diethylphthalate	11U ug/Kg	А	bl
R2844885	RSAN2-35B	Phenanthrene	2.4U ug/Kg	Α	bl
R2844885	RSAO2-0.5B	Diethylphthalate Phenanthrene	9.3U ug/Kg 2.9U ug/Kg	. А	bl
R2844885	RSAO2-10B	Diethylphthalate Phenanthrene	8.8U ug/Kg 2.9U ug/Kg	А	bl
R2844885	RSAO2-20B	Phenanthrene	2.5U ug/Kg	А	bl
R2844885	RSAO2-20BD	Diethylphthalate Phenanthrene	4.5U ug/Kg 2.3U ug/Kg	А	bl
R2844885	RSAO2-30B	Diethylphthalate	15U ug/Kg	А	bl

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
R2844885	RSAO2-33B	Diethylphthalate Phenanthrene	8.7U ug/Kg 2.9U ug/Kg	А	ld
R2844885	SA183-10B	Diethylphthalate	8.8U ug/Kg	А	bl
R2844885	SA183-10BD	Diethylphthalate Phenanthrene	6.2U ug/Kg 2.5U ug/Kg	А	bl
R2844885	SA183-20B	Diethylphthalate Phenanthrene	6.5U ug/Kg 2.6U ug/Kg	А	Ы
R2844885	SA183-30B	Diethylphthalate	8.4U ug/Kg	А	Ы
R2844885	SA183-33B	Diethylphthalate	14U ug/Kg	А	bl
R2844885	RSA04-10B	Diethylphthalate Phenanthrene	7.2U ug/Kg 2.1U ug/Kg	А	bl
R2844885	RSA04-20B	Diethylphthalate Phenanthrene	7.6U ug/Kg 2.5U ug/Kg	А	bl
R2844885	RSA04-30B	Diethylphthalate	11U ug/Kg	А	bl
R2844885	RSA04-36B	Diethylphthalate	9.6U ug/Kg	А	bl

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles- Field Blank Data Qualification Summary - SDG R2844885

No Sample Data Qualified in this SDG

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

SDG #:_	R2844885
Laborat	y: Columbia Analytical Services

LDC #: 21257I2a

Stage 2B

Reviewer: 4V6 2nd Reviewer:

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

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

	Validation Area		Comments
l.	Technical holding times	A	Sampling dates: 7/08 - 69 /08
11.	GC/MS Instrument performance check	Á	. •
111.	Initial calibration	A	2 RSD r~
IV.	Continuing calibration/ICV	JUSWA	Ca/12 £ 25 }
V.	Blanks	WZ	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	SW)	
VIII.	Laboratory control samples	SN)	LCS
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	Á	
XI.	Target compound identification	N	
XII.	Compound quantitation/CRQLs	N	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	N	
XV.	Overall assessment of data	A	
XVI.	Field duplicates	SW)	$D_1 = 1.2$ $D_2 = 6.7$ $D_3 = 6.11$
XVII.	Field blanks	7	

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

TB = Trip blank

EB = Equipment blank

Validated Samples:

Soil

		2011						
1	RSAN2-30B	p <sub> </sub>	11	SA183-10BD <b>/</b>	, 21	RSA04-30BMSD	31	SBULI
2	RSAN2-30BD	b,	12	SA183-20B	22		32	
3	RSAN2-35B		13	SA183-30B	23		33	
4	RSAO2-0.5B		14	SA183-33B	24		34	
5	RSAO2-10B		15	RSA04-0.5B	25		35	
6	RSAO2-20B	$\rho_{\gamma}$	16	RSA04-10B	26		36	
7	RSAO2-20BD	$\mathcal{V}_{\checkmark}$	17	RSA04-20B	27		37	
8	RSAO2-30B		18	RSA04-30B	28		38	
9	RSAO2-33B		19	RSA04-36B	29		39	
10	SA183-10B	$\mathcal{D}_{\lambda}$	20	RSA04-30BMS	30		40	

# **VALIDATION FINDINGS WORKSHEET**

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

A. Phenol**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenol**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	VV. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine⁴	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	T1T.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	nnn
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	ww.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	WWW.

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

LDC# 21257 IZA Su Cores SDG #:

## VALIDATION FINDINGS WORKSHEET Blanks

2nd Reviewer: Q Reviewer:\_ Page:

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

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

Was a method blank analyzed for each matrix?

Was a method blank analyzed for each concentration preparation level?

Was a method blank associated with every sample? 

 $\overline{X}$ , N N/A Was the blank contaminated? If yes, please see qualification below. Blank extraction date: 7/15/68 Blank analysis date: 7/18/68

Ē

(99)

7 83 y 2.3/4 4.5/4 2 7 Sample Identification 7 8.8 /u 8,9 2.9 200 Associated Samples: 4,4 3 9.6 /W 3.0/ Blank ID SBLKI ÿ ⊗ 77 Compound Conc. units:\_

(5x)= M.o.

\*( )\*

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

rbre Ţ Associated Samples:

Compound	Blank ID				San	mple Identifica	tion			
	581K1	9	1	7	13	14	91	17	18	
		7	,	, ,		•	,	,	,	ŀ

		_		_	T	<del>r</del>	<del>r</del>	Г
	61	9.6 14	(3.2)					
	18	11/1	3.7					
	17	7.6/4	2,5/4					
tion	91	h/c.7	1/1.5					
Sample Identification	14	14/4	(4.6)					
Ş	13	11/78	(3.4)					
	7	16.5/W 8.4/W	2.5/4 2.6/4 (	,				
	ll l	6.2/4	2.5/4	•				
	Q	8.8/4	(3.1)*	)				
Blank ID	581K1	3,8	1.5					
Compound		77	nn					

SDG#: 21257 126 SDG#: 54 Grey

# VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Page: of 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".

Y N N/A

Was a MS/MSD analyzed every 20 samples of each matrix?

Y N N/A

Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

	Date	CI OSM/SW	Compound	MS %R (Limits)	MSD (Limits)	RPD (Limits)	Associated Samples	Guations	
41		(5/02	XX	( )	(05/-05) 88/		18	No greet (MS	<u>}                                    </u>
		,		( )		_			<u>\</u>
				( )	( )	( )			1
1				( )	( )	( )			
				( )	( )	( )			<u></u>
				( )	( )	, ,			
				( )	( )	( )			ı
1				( )	( )	( )			
				( )	( )	(			_
				( )	( )	( )			
				(. )	(	( )			_
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				( )	( )	( )			
				( )	( )	( )			
				( )	( )	( )			
				( )	( )	( )			
				( )	( )	(			

	Compound	QC Limits (Soil)	RPD (Soll)	QC Limits (Water)	RPD (Water)		Compound	QC Limits (Soli)	RPD (Soll)	QC Limits (Water)	RPD (Water)
ď	Phenol	76-90%	~32% ▼	12-110%	<u>≤ 42%</u>	99	Acenaphthene	31-137%	≥ 19%	46-118%	<u>&lt;</u> 31%
υj	2-Chlorophenol	25-102%	< 50%	27-123%	< 40%	=i	4-Nitrophenol	11-114%	< 50%	10-80%	× 20%
шi	1,4-Dichlorobenzene	28-104%	< 27%	36-97%	< 28%	<del>χ</del> Ξ.	2,4-Dinitrotoluene	28-89%	< 47%	24-96%	< 38%
٦	N-Nitroso-di-n-propylamine	41-126%	< 38%	41-116%	< 38%	Ë	Pentachlorophenol	17-109%	< 47%	9-103%	< 50%
œ	1,2,4-Trichlorobenzene	38-107%	< 23%	39-98%	< 28%	Z.	Pyrene	35-142%	× 36%	26-127%	< 31%
>	4-Chloro-3-methylphenol	26-103%	< 33%	23-97%	< 42%						

LDC#: 21257 I 29 SDG #: Sec

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

Page: 2nd Reviewer: Reviewer

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

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".  $\frac{V_{NN/N/A}}{V_{NN/A}}$  Was a LCS required?

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

- المستعدي	_	7.4	<u>:                                    </u>		·				_			,								<del></del>					<del>,</del>
Qualifications	No ruel (MCm	CH CON SW)																							
Associated Samples	41 + BK																								
RPD (Limits)		( )	(		( )	( )	( )	( )	, ,	( )		(	)		(	· ·		( )	)	( )	•	( )	)	`	( )
LCSD %R (Limits)	(		)		^	( )	( )	( )	, ,	( )	( )	( )	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
LCS %R (Limits)	(28 50-120)	(1) 82/	(	( )	( )	( )	( )	( )	,		( )	( )	( )	( )	( )	( )	( )		( )	( )	( )	( )	( )	( )	
Compound	XX	433 4																							
CSACSD ID	( 531																								~
Date																									
*																							·		

LDC #: 2/257 [ 22 SDG #: Ly Correr

### VALIDATION FINDINGS WORKSHEET Field Duplicates

Page:_	
Reviewer:	JVL
2nd reviewer:	

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

Y N N/A Y N N/A Were field duplicate pairs identified in this SDG?

Were target compounds identified in the field duplicate pairs?

	Concentratio	nug/kg,		Dilt	Parent
Compound	(	2		-RPD	Parent
AAA	260 U	5, 3	254.7	(£ 260)	~
LL	9.6	11	1.4	( £ 280 )	_
ИИ	3,0	6,2	3,2	(£11)	٠

	Concentration	n, 45/kg)		Di4	Parent
Compound	6	7		RPD_	nly
AAA	6.4	230 y	224	(£ 230)	`
LL	2504	4,5	245.5	(£ 250)	, ,
ии	2,5	2,3	0, 2	(=9,7)	٠

	Concentration	in ing legy			Paren
Compound	10	1/		RPD	mh
AAA	3.2	190 U	186.8	(£190)	-1
L L	8. 8	6,2	2.6	1	_
· uu	3.]	2,5	0,6	(=7.5)	_
·					

	Concentration ( )	
Compound		RPD
·	·	

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

Project/Site Name:

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 9 through July 10, 2008

LDC Report Date:

August 20, 2009

Matrix:

Soil

Parameters:

Semivolatiles

Validation Level:

Stage 2B

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2844902

### Sample Identification

SA46-0.5B

**SA48-35BMS** 

SA46-10B

SA48-35BMSD

SA46-20B

SA46-30B

SA46-30BD

SA48-0.5B

SA48-10B

SA48-20B

SA48-30B

SA48-35B

RSAJ7-0.5B

RSAJ7-0.5BDL

RSAJ7-10B

RSAJ7-20B

RSAK7-0.5B

RSAK7-0.5BDL

RSAK7-10B

RSAK7-10BD

RSAK7-20B

RSAK7-27B

### Introduction

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

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

### III. Initial Calibration

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 15.0% for each individual compound and less than or equal to 30.0% for calibration check compounds (CCCs).

In the case where %RSD was greater than 15.0%, the laboratory used a calibration curve to evaluate the compound. All coefficients of determination (r²) were greater than or equal to 0.990.

For the purposes of technical evaluation, all compounds were evaluated against the 30.0% (%RSD) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

Average relative response factors (RRF) for all semivolatile target compounds and system performance check compounds (SPCCs) were greater than or equal to 0.05 as required.

### 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).

For the purposes of technical evaluation, all compounds were evaluated against the 25.0% (%D) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

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 RRF values were greater than or equal to 0.05.

### 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
SBLK1	7/17/08	Butylbenzylphthalate Diethylphthalate Phenanthrene	2.8 ug/Kg 6.4 ug/Kg 1.6 ug/Kg	All samples in SDG R2844902
SBLK1RE	7/17/08	Phenanthrene	1.5 ug/Kg	All samples in SDG R2844902

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	Reported	Modified Final
	TIC (RT in minutes)	Concentration	Concentration
SA46-0.5B	Diethylphthalate	7.5 ug/Kg	7.5U ug/Kg
	Phenanthrene	2.5 ug/Kg	2.5U ug/Kg
SA46-10B	Diethylphthalate	9.0 ug/Kg	9.0U ug/Kg
	Phenanthrene	2.3 ug/Kg	2.3U ug/Kg
SA46-20B	Diethylphthalate	8.1 ug/Kg	8.1U ug/Kg
	Phenanthrene	2.6 ug/Kg	2.6U ug/Kg
SA46-30B	Diethylphthalate	15 ug/Kg	15U ug/Kg
	Phenanthrene	3.2 ug/Kg	3.2U ug/Kg
SA46-30BD	Phenanthrene	2.1 ug/Kg	2.1U ug/Kg
SA48-10B	Diethylphthalate	4.7 ug/Kg	4.7U ug/Kg
	Phenanthrene	1.8 ug/Kg	1.8U ug/Kg
SA48-30B	Diethylphthalate	7.1 ug/Kg	7.1U ug/Kg
	Phenanthrene	2.0 ug/Kg	2.0U ug/Kg
SA48-35B	Diethylphthalate	11 ug/Kg	11U ug/Kg
	Phenanthrene	2.5 ug/Kg	2.5U ug/Kg
RSAJ7-0.5B (10x)	Phenanthrene	25 ug/Kg	25U ug/Kg
RSAJ7-20B	Diethylphthalate	6.0 ug/Kg	6.0U ug/Kg
	Phenanthrene	2.1 ug/Kg	2.1U ug/Kg

Sample	Compound	Reported	Modified Final
	TIC (RT in minutes)	Concentration	Concentration
RSAK7-10B	Phenanthrene	1.5 ug/Kg	1.5U ug/Kg
RSAK7-10BD	Diethylphthalate	5.5 ug/Kg	5.5U ug/Kg
	Phenanthrene	1.7 ug/Kg	1.7U ug/Kg
RSAK7-20B	Diethylphthalate	6.4 ug/Kg	6.4U ug/Kg
	Phenanthrene	1.8 ug/Kg	1.8U ug/Kg
RSAK7-27B	Phenanthrene	2.3 ug/Kg	2.3U ug/Kg

No field blanks were identified in this SDG.

### VI. Surrogate Spikes

Surrogates were added to all samples and blanks as required by the method. Surrogate recoveries (%R) were not within QC limits for samples RSAJ7-0.5BDL and RSAK7-0.5BDL. Since these samples were diluted out, no data were qualified.

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Although the LCS/D percent recoveries (%R) were not within QC limits for several compounds and the relative percent difference (RPD) for one compound, the LCS and MS/MSD percent recoveries (%R) were within QC limits and no data were qualified.

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

All internal standard areas and retention times were within QC limits with the following exceptions:

Sample	Internal Standards	Area (Limits)	Compound	Flag	A or P
RSAK7-0.5BDL	Chrysene-d12	100763 (106904-427614)	Butylbenzylphthalate Pyrene Bis(2-ethylhexyl)phthalate Benzo(a)anthracene Chrysene	J (all detects) UJ (all non-detects)	A

### XI. Target Compound Identifications

Raw data were not reviewed for this SDG.

### XII. Project Quantitation Limit

All project quantitation limits were within validation criteria with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P
RSAJ7-0.5B RSAK7-0.5B	Hexachlorobenzene Octachlorostyrene	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	А

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R2844902	All compounds reported below the PQL.	J (all detects)	Α

Raw data were not reviewed for this SDG.

### XIII. Tentatively Identified Compounds (TICs)

Raw data were not reviewed for this SDG.

### XIV. System Performance

Raw data were not reviewed for this SDG.

### XV. Overall Assessment

The overall assessment of data was acceptable. In the case where more than one result was reported for an individual sample, the least technically acceptable results were rejected as follows:

Sample	Compound	Flag	A or P
RSAJ7-0.5B RSAK7-0.5B	Hexachlorobenzene Octachlorostyrene	X X	А
RSAJ7-0.5BDL RSAK7-0.5BDL	All TCL compounds except Hexachlorobenzene Octachlorostyrene	×	А

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

### XVI. Field Duplicates

Samples SA46-30B and SA46-30BD and samples RSAK7-10B and RSAK7-10BD were identified as field duplicates. No semivolatiles were detected in any of the samples with the following exceptions:

	Concentration (ug/Kg)			B		
Compound	SA46-30B	SA46-30BD	RPD (Limits)	Difference (Limits)	Flags	A or P
Diethylphthalate	15	250U	-	235 (≤250)	-	-
Bis(2-ethylhexyl)phthalate	200	220	-	20 (≤250)	-	-
Phenanthrene	3.2	2.1	-	1.1 (≤9.9)	-	-

	Concentra	Concentration (ug/Kg)		B		
Compound	RSAK7-10B	RSAK7-10BD	RPD (Limits)	Difference (Limits)	Flags	A or P
Diethylphthalate	190U	5.5	-	184.5 (≤190)	-	
Hexachlorobenzene	5.6	12	-	6.4 (≤7.2)	-	-
Phenanthrene	1.5	1.7	•	0.2 (≤7.2)	-	-

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG R2844902

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R2844902	RSAK7-0.5BDL	Butylbenzylphthalate Pyrene Bis(2-ethylhexyl)phthalate Benzo(a)anthracene Chrysene	J (all detects) UJ (all non-detects)	A	Internal standards (area) (i)
R2844902	RSAJ7-0.5B RSAJ7-10B	Hexachlorobenzene Octachlorostyrene	J (all detects) J (all detects)	Α .	Project Quantitation Limit (e)
R2844902	SA46-0.5B SA46-10B SA46-20B SA46-30B SA46-30BD SA48-0.5B SA48-10B SA48-20B SA48-30B SA48-35B RSAJ7-0.5B RSAJ7-0.5BDL RSAJ7-10B RSAJ7-20B RSAK7-0.5BDL RSAK7-0.5BDL RSAK7-0.5BDL RSAK7-0.5BDL RSAK7-10BD RSAK7-10BD RSAK7-20B	All compounds reported below the PQL.	J (all detects)	Α	Project Quantitation Limit (sp)
R2844902	RSAJ7-0.5B RSAK7-0.5B	Hexachlorobenzene Octachlorostyrene	X X	Α	Overall assessment of data (o)
R2844902	RSAJ7-0.5BDL RSAK7-0.5BDL	All TCL compounds except Hexachlorobenzene Octachlorostyrene	X	А	Overall assessment of data (o)

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG R2844902

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
R2844902	SA46-0.5B	Diethylphthalate Phenanthrene	7.5U ug/Kg 2.5U ug/Kg	А	bl
R2844902	SA46-10B	Diethylphthalate Phenanthrene	9.0U ug/Kg 2.3U ug/Kg	А	ld

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
R2844902	SA46-20B	Diethylphthalate Phenanthrene	8.1U ug/Kg 2.6U ug/Kg	А	bl
R2844902	SA46-30B	Diethylphthalate Phenanthrene	15U ug/Kg 3.2U ug/Kg	А	bl
R2844902	SA46-30BD	Phenanthrene	2.1U ug/Kg	А	bl
R2844902	SA48-10B	Diethylphthalate Phenanthrene	4.7U ug/Kg 1.8U ug/Kg	А	bl
R2844902	SA48-30B	Diethylphthalate 7.1U ug/l Phenanthrene 2.0U ug/l		А	bl
R2844902	SA48-35B	Diethylphthalate Phenanthrene	11U ug/Kg 2.5U ug/Kg	А	ld
R2844902	RSAJ7-0.5B (10x)	Phenanthrene	25U ug/Kg	А	bl
R2844902	RSAJ7-20B	Diethylphthalate Phenanthrene	6.0U ug/Kg 2.1U ug/Kg	А	bl
R2844902	RSAK7-10B	Phenanthrene	1.5U ug/Kg	А	bl
R2844902	RSAK7-10BD	Diethylphthalate 5.5U ug/Kg Phenanthrene 1.7U ug/Kg		А	ы
R2844902	RSAK7-20B	Diethylphthalate Phenanthrene			bl
R2844902	RSAK7-27B	Phenanthrene	2.3U ug/Kg	А	bl

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles- Field Blank Data Qualification Summary - SDG R2844902

No Sample Data Qualified in this SDG

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

SDG #:_	R2844902		
	<b>~</b>	A 1 41 1	<u> </u>

Stage 2B

Page: 1 of ) 2nd Reviewer:

Laboratory: Columbia Analytical Services

LDC #: 21257J2a

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

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

	Validation Area		Comments
l.	Technical holding times	Α	Sampling dates: 7/09-10/68
11.	GC/MS Instrument performance check	A	
III.	Initial calibration	A	2 RSD r~
IV.	Continuing calibration/ICV	A	COV/10V £ 25 2
V.	Blanks	SW	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	_SW)	
VIII.	Laboratory control samples	SW	VCS/D
IX.	Regional Quality Assurance and Quality Control	N	
Χ.	Internal standards	SW)	
XI.	Target compound identification	N	
XII.	Compound quantitation/CRQLs	MS	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	N	
XV.	Overall assessment of data	SW	
XVI.	Field duplicates	SW	D, = 4,5 D, = 17,/8
XVII.	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:

1	SA46-0.5B	11	RSAJ7-0.5B	21	SA48-35BMS	31	SBLKI
2	SA46-10B	12	RSAJ7-0.5BDL	22	SA48-35BMSD	32	SBUCI RE
3	SA46-20B	13	RSAJ7-10B	23		33	
4	SA46-30B <b>b</b> ,	14	RSAJ7-20B	24		34	
5	SA46-30BD 0,	15	RSAK7-0.5B	25		35	
6	SA48-0.5B	16	RSAK7-0.5BDL	26		36	
7	SA48-10B	17	RSAK7-10B D√	27		37	
8	SA48-20B	18	RSAK7-10BD ₽✓	28		38	
9	SA48-30B	19	RSAK7-20B	29		39	
10	SA48-35B	20	RSAK7-27B	30		40	

# **VALIDATION FINDINGS WORKSHEET**

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

A. Phenol**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenol**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	VV. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene™	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenoi**	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-Chioronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	III. Octachlosostyrene
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	uuu 1,4- Dioxane
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.

21257 129 SDG #: LDC#:

## VALIDATION FINDINGS WORKSHEET Blanks

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

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

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

Y N/A Y N/A

Was a method blank analyzed for each matrix? Was a method blank analyzed for each concentration preparation level?

Was a method blank associated with every sample?

Y/N N/A

Associated Samples: Vas the blank contaminated? If yes, please see qualification below. Blank analysis date: 7/27/6% Blank analysis date: 7/22/6%

**\*** 

7,1/4 5.0 0 4.7 8: (XX) 75 2.1/4 S Sample Identification 15/1 など 7 2.6 8.1 40.6 w, 7 2,5/W 51/ SOLKIRE Blank ID SELKI e, 4 **4** ∞ 444 22 Conc. units: 以久/ルヘ Compound

> h 7

Blank analysis date: Blank extraction date:\_

Ş Same

abore

Conc. units:

Associated Samples:

5x Phthalates 2x all others

different instrument 8 anadyzed SBUKIRE =

21 257 529 LDC #

25 7 SDG#:

VALIDATION FINDINGS WORKSHEET

Surrogate Recovery

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

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

d

2nd Reviewer:

Reviewer: 3/6 Page: of

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

XN N/A

N N/A

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

If any %R was less than 10 percent, was a reanalysis performed to confirm %R?

*	Date	Sample ID	Surrogate	%R (Limits)	Qualifications
1		(500x)	( A1)	(261-24) od	No ex
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				)	)
1					)
1				)	
C limits	* QC limits are advisory S1 (NBZ) = Nitrobenzene-d5	QC Limits (Soil) QC Limits (Water) 45 23-120 35-114		SS (2FP)= 2-Fluoranhend	OC Limits (Water)

QC Limits (Water) 21-100 10-123

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

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

43-116 33-141 10-94

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

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

33-110\* 16-110\*

LDC#: 2/257 J29 SDG#:\_

# VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Page: 2nd Reviewer: Reviewer:\_

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

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

Y N N/A

Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated

MS/MSD. Soil / Water. Y/N/N/A

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

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limite)	Associated Semiles	and the state of t
		21/22	EEE	165 ( )		44 ( 30 )	10	M. T. C. C. C. C. L.
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				( )	( )	( )		
				( )	( )	( )		
				( )	( )	)		
				(	( )	(		

	Compound	QC Limits (Soil)	RPD (Soil)	QC Umits (Water)	RPD (Water)		Compound	QC Limits (Solf)	RPD (Soll)	QC Limits	RPD
Α.	Phenol	26-90%	₹35%	12-110%	<u>≤ 42%</u>	99	Acenaphthene	31-137%	< 19%	46-118%	<u>&lt;</u> 31%
ပ	C. 2-Chlorophenol	25-102%	× 20%	27-123%	< 40%	·] =	4-Nitrophenol	11-114%	> 50%	10.80%	< 50%
шi	1,4-Dichlorobenzene	28-104%	< 27%	36-97%	< 28%	ξ	2.4-Dinitrotoluene	28-89%	< 47%	24-96%	× 38%
ㅋ	N-Nitroso-di-n-propylamine	41-126%	< 38%	41-116%	< 38%	1	Pentachlorophenol	17-109%	< 47%	9-103%	× 50%
ď	1,2,4-Trichlorobenzene	38-107%	< 23%	%86 <del>-</del> 6E	< 28%	13	Pyrene	35-142%	× 36%	26-127%	< 31%
,	4-Chloro-3-methylphenol	26-103%	×33%	23-97%	< 42%						

LDC# 2/357 J29 SDG#:

## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Page: \_\_lof\_\_/

Reviewer:

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

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

YANA Was a LCS required?

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

	Date	OI DESTEST	Compound	LCS %R (Limits)	ifts)	LCSD %R (Limits)		RPD (Limits)	Associated Samples	Outlifteethore	( <del></del>
Ш	Н	1 d/sn	××		ì	128 (50-120)	Ŕ	( )	All + Bike	No grad (166	
			hnn	64 (5	(0c) - 05)	47 (	_	•			\ \ \ \
			343		^ ,	270 ( J	<u> </u>	67 (36)	>	45W)	
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21257 524	Se Corre
LDC #:_	SDG #:

## VALIDATION FINDINGS WORKSHEET Internal Standards

Page: of Reviewer:\_ 2nd Reviewer.

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

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

Were all internal standard area counts within -50 to +100 of the associated calibration standard?

Were the retention times of the internal standards within +/- 30 seconds of the retention times of the associated calibration standard?

Qualifications	J/N5/A (i)	(grad, AAA ZZ, EEB												
RT (Limits)														
Area (Limits)	100763 (106904- 427614)													
Internal Standard	CRY	,												
Sample ID	16	-												
# Date														

\* QC limits are advisory IS1 (DCB) = 1,4-Dichlorobenzene-d4 IS2 (NPT) = Naphthalene-d8 IS3 (ANT) = Acenaphthene-d10

IS4 (PHN) = Phenanthrene-d10 IS5 (CRY) = Chrysene-d12 IS6 (PRY) = Perylene-d12

LDC#: 2/25/J2a

# VALIDATION FINDINGS WORKSHEET

Compound Quantitation and Reported CRQLs

Page:

2nd Reviewer:

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

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

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?

	(e)									
Qualifications	J dets/A									
Associated Samples	range	5								
Finding	SS TTT > cul 1	,								
Sample ID	1 5	,								
Date										
#										

Comments: See sample calculation verification worksheet for recalculations

SDG # 54 CS-

## **VALIDATION FINDINGS WORKSHEET** Overall Assessment of Data

Page: \_

Reviewer: 2nd Reviewer:

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

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

All available information pertaining to the data were reviewed using professional judgement to compliment the determination of the overall quality of the data.

Y N N/A

Was the overall quality and usability of the data acceptable?

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		1 15	55 TTT > Cal	(	(6) X/X
			,	D	
		2 16	All except SS. 7	77 dil	7
		-			
Comn	Comments:				

LDC #: 21 257 J29 SDG #: Sa (ore)

### VALIDATION FINDINGS WORKSHEET Field Duplicates

Page:_	<u>lof_//</u>
Reviewer:_	5/14
2nd reviewer	$\overline{N}$

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

$\langle \mathbf{Y} \rangle$	N	N/A
Y	N	N/A

Were field duplicate pairs identified in this SDG?
Were target compounds identified in the field duplicate pairs?

Compound	Concentration 4	ng lighter	Diff
LL	15	250 U	235 (4250)
EEE	200	220	20
UU	3, 2	2, ]	1.1 (=9.9)
·			

		Concentration ( 45/kg.)		Diff
Compound		17	18	₹₽D
	LL	190 U	5.5	184.5 (= 190)
	SS	5,6	12	6.4 (47.2)
	ии	1.5	1.7	0.2
				·

	Concentration ( )		·
Compound			RPD
	1		
	·		
		·	

	Concentration ( )	
Compound		RPD
·		
·		

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

**Project/Site Name:** 

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

Collection Date:

July 10 through July 11, 2008

LDC Report Date:

August 25, 2009

Matrix:

Soil

Parameters:

Semivolatiles

Validation Level:

Stage 2B

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2844922

### Sample Identification

RSAJ8-0.5B RSAK2-0.5B RSAJ8-0.5BDL RSAK2-10B RSAJ8-10B RSAK2-20B RSAJ8-20B RSAK2-20BD RSAJ8-30B RSAK2-20BDDL RSAJ8-33B RSAK2-30B RSAI7-0.5B RSAK2-35B RSAI7-10B RSA17-32B RSAI7-20B RSAL2-0.5BMS RSAI7-30B RSAL2-0.5BMSD

RSAL2-0.5B RSAL2-0.5BDL RSAL2-10B RSAL2-10BDL RSAL2-20B RSAL2-20BD RSAL2-20BDDL RSAL2-30B RSAL2-37B

RSAL2-40B

### Introduction

This data review covers 30 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

All samples were received in good condition with the following exceptions:

Sample	Compound	Finding	Flag	A or P
RSAJ8-0.5B RSAJ8-0.5BDL RSAJ8-30B	All TCL compounds	All jars possibly contaminated from water in the cooler. Water present in jars.	J (all detects) UJ (all non-detects)	А

One out of 3 jars possibly contaminated from water in the cooler. Water present in jars for samples RSAJ8-33B and RSAK2-0.5B .

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 15.0% for each individual compound and less than or equal to 30.0% for calibration check compounds (CCCs).

In the case where %RSD was greater than 15.0%, the laboratory used a calibration curve to evaluate the compound. All coefficients of determination (r²) were greater than or equal to 0.990.

For the purposes of technical evaluation, all compounds were evaluated against the 30.0% (%RSD) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

Average relative response factors (RRF) for all semivolatile target compounds and system performance check compounds (SPCCs) were greater than or equal to 0.05 as required.

### 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).

For the purposes of technical evaluation, all compounds were evaluated against the 25.0% (%D) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

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 RRF values were greater than or equal to 0.05.

### 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
SBLK1	7/18/08	Phenanthrene	1.4 ug/Kg	RSAJ8-0.5B RSAJ8-0.5BDL RSAJ8-10B RSAJ8-20B RSAJ8-30B RSAJ8-33B RSAJ7-0.5B RSAJ7-10B RSAJ7-20B RSAJ7-30B RSAL2-0.5B RSAL2-0.5B RSAL2-0.5BDL RSAL2-10BDL RSAL2-10BDL RSAL2-20BD RSAL2-20BD RSAL2-20BD RSAL2-37B RSAL2-37B RSAL2-40B RSAK2-10B RSAK2-10B RSAK2-10B RSAK2-10B RSAK2-20B

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	Reported	Modified Final
	TIC (RT in minutes)	Concentration	Concentration
RSAJ8-10B	Phenanthrene	1.6 ug/Kg	1.6U ug/Kg

Sample	Compound TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
RSAJ8-20B	Phenanthrene	1.9 ug/Kg	1.9U ug/Kg
RSAJ8-30B	Phenanthrene	2.0 ug/Kg	2.0U ug/Kg
RSAJ8-33B	Phenanthrene	1.5 ug/Kg	1.5U ug/Kg
RSAI7-0.5B	Phenanthrene	1.4 ug/Kg	1.4U ug/Kg
RSAI7-10B	Phenanthrene	2.1 ug/Kg	2.1U ug/Kg
RSAL2-0.5B (5x)	Phenanthrene	7.7 ug/Kg	7.7U ug/Kg
RSAL2-20BDDL (6x)	Phenanthrene	12 ug/Kg	12U ug/Kg
RSAL2-37B (6x)	Phenanthrene	16 ug/Kg	16U ug/Kg
RSAK2-0.5B (5x)	Phenanthrene	8.7 ug/Kg	8.7U ug/Kg
RSAK2-20B (5x)	Phenanthrene	10 ug/Kg	10U ug/Kg

No field blanks were identified in this SDG.

### VI. Surrogate Spikes

Surrogates were added to all samples and blanks as required by the method. Surrogate recoveries (%R) were not within QC limits for samples RSAJ8-0.5BDL and RSAL2-0.5BDL. Since these samples were diluted out, no data were qualified.

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

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

### XI. Target Compound Identifications

Raw data were not reviewed for this SDG.

### XII. Project Quantitation Limit

All project quantitation limits were within validation criteria with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P
RSAJ8-0.5B RSAL2-0.5B	Hexachlorobenzene Octachlorostyrene	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	А
RSAL2-10B RSAL2-20BD RSAK2-20BD	Di-n-butylphthalate	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	A

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R2844922	All compounds reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

### XIII. Tentatively Identified Compounds (TICs)

Raw data were not reviewed for this SDG.

### XIV. System Performance

Raw data were not reviewed for this SDG.

### XV. Overall Assessment

The overall assessment of data was acceptable. In the case where more than one result was reported for an individual sample, the least technically acceptable results were rejected as follows:

Sample	Compound	Flag	A or P
RSAJ8-0.5B RSAL2-0.5B	Hexachlorobenzene Octachlorostyrene	××	А
RSAJ8-0.5BDL RSAL2-0.5BDL	All TCL compounds except Hexachlorobenzene Octachlorostyrene	x	А
RSAL2-10B RSAL2-20BD RSAK2-20BD	Di-n-butylphthalate	х	А
RSAL2-10BDL RSAL2-20BDDL RSAK2-20BDDL	All TCL compounds except Di-n-butylphthalate	×	А

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

### XVI. Field Duplicates

Samples RSAL2-20B and RSAL2-20BD, samples RSAL2-20B and RSAL2-20BDDL, samples RSAK2-20B and RSAK2-20BDDL were identified as field duplicates. No semivolatiles were detected in any of the samples with the following exceptions:

	Concentration (ug/Kg)					
Compound	RSAL2-20B	RSAL2-20BD	RPD (Limits)	Difference (Limits)	Flags	A or P
Di-n-butylphthalate	560	660	-	100 (≤390)	-	
Butylbenzylphthalate	390U	30	-	360 (≤390)	•	-
Diethylphthalate	49	54	-	5 (≤390)	-	-
Dimethylphthalate	4.0	4.8	-	0.8 (≤390)	-	-
Fluoranthene	3.9	4.6	ı	0.7 (≤15)	-	-
Phenanthrene	15U	12	-	3 (≤15)	-	-
Pyrene	15U	1.9	-	13.1 (≤15)	-	•

	Concentration (ug/Kg)					
Compound	RSAL2-20B	RSAL2-20BDDL	RPD (Limits)	Difference (Limits)	Flags	A or P
Butylbenzylphthalate	390U	70	-	320 (≤390)	-	-
Di-n-butylphthalate	560	1800	-	1240 (≤1100)	J (all detects)	А
Diethylphthalate	49	1100U	-	1051 (≤1100)	-	-
Dimethylphthalate	4.0	1100U	-	1096 (≤1100)	-	-
Fluoranthene	3.9	43U	-	39.1 (≤43)	-	-
Phenanthrene	15U	12	-	3 (≤15)	-	-

	Concentration (ug/Kg)					
Compound	RSAK2-20B	RSAK2-20BD	RPD (Limits)	Difference (Limits)	Flags	A or P
Butylbenzylphthalate	64	62	-	2 (≤920)	-	-
Di-n-butylphthalate	1500	1000	-	500 (≤920)	-	-
Diethylphthalate	49	36	-	13 (≤920)	-	•
Dimethylphthalate	920U	3.4	-	916.6 (≤920)	-	-
Fluoranthene	36U	4.5	-	31.5 (≤36)	-	-
Phenanthrene	10	9.2	-	0.8 (≤36)	-	-

	Concentra	tion (ug/Kg)				
Compound	RSAK2-20B	RSAK2-20BDDL	RPD (Limits)	Difference (Limits)	Flags	A or P
Butylbenzylphthalate	64	130	•	66 (≤1800)	<u>.</u>	-
Di-n-butylphthalate	1500	3200	-	1700 (≤1800)	-	**
Diethylphthalate	49	1800U	-	1751 (≤1800)	-	-
Phenanthrene	10	71U	-	61 (≤71)	-	-

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG R2844922

SDG	Samuela	0			
R2844922	RSAJ8-0.5B RSAJ8-0.5BDL RSAJ8-30B	All TCL compounds	J (all detects) UJ (all non-detects)	A or P	Reason (Code) Sample condition (p)
R2844922	RSAJ8-0.5B RSAL2-0.5B	Hexachlorobenzene Octachlorostyrene	J (all detects) J (all detects)	А	Project Quantitation Limit (e)
R2844922	RSAL2-10B RSAL2-20BD RSAK2-20BD	Di-n-butylphthalate	J (all detects)	А	Project Quantitation Limit (e)
R2844922	RSAJ8-0.5B RSAJ8-0.5BDL RSAJ8-0.5BDL RSAJ8-10B RSAJ8-30B RSAJ8-30B RSAJ8-33B RSAI7-0.5B RSAI7-10B RSAI7-30B RSAL2-0.5B RSAL2-0.5BDL RSAL2-10B RSAL2-10BDL RSAL2-10BDL RSAL2-20BD RSAL2-20BDDL RSAL2-37B RSAL2-37B RSAL2-40B RSAK2-0.5B RSAK2-10B RSAK2-0.5B RSAK2-10B RSAK2-37B RSAK2-10B RSAK2-37B RSAK2-37B RSAK2-30B RSAK2-35B RSAK2-35B RSAK2-35B RSAK2-35B RSAK2-35B	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
R2844922	RSAJ8-0.5B RSAL2-0.5B	Hexachlorobenzene Octachlorostyrene	X X	Α	Overall assessment of data (o)
R2844922	RSAJ8-0.5BDL RSAL2-0.5BDL	All TCL compounds except Hexachlorobenzene Octachlorostyrene	X	Α	Overall assessment of data (o)
R2844922	RSAL2-10B RSAL2-20BD RSAK2-20BD	Di-n-butylphthalate	X	Α	Overall assessment of data (o)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R2844922	RSAL2-10BDL RSAL2-20BDDL RSAK2-20BDDL	All TCL compounds except Di-n-butylphthalate	х	A	Overall assessment of data (o)
R2844922	RSAL2-20B RSAL2-20BDDL	Di-n-butylphthalate	J (all detects)	A	Field duplicates (Difference) (fd)

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG R2844922

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
R2844922	RSAJ8-10B	Phenanthrene	1.6U ug/Kg	А	bl
R2844922	RSAJ8-20B	Phenanthrene	1.9U ug/Kg	А	bl
R2844922	RSAJ8-30B	Phenanthrene	2.0U ug/Kg	А	bl
R2844922	RSAJ8-33B	Phenanthrene	1.5U ug/Kg	Α	bl
R2844922	RSAI7-0.5B	Phenanthrene	1.4U ug/Kg	Α	bl
R2844922	RSAI7-10B	Phenanthrene	2.1U ug/Kg	А	bl
R2844922	RSAL2-0.5B (5x)	Phenanthrene	7.7U ug/Kg	Α	bl
R2844922	RSAL2-20BDDL (6x)	Phenanthrene	12U ug/Kg	А	bl
R2844922	RSAL2-37B (6x)	Phenanthrene	16U ug/Kg	А	bl
R2844922	RSAK2-0.5B (5x)	Phenanthrene	8.7U ug/Kg	А	bl
R2844922	RSAK2-20B (5x)	Phenanthrene	10U ug/Kg	А	bl

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles- Field Blank Data Qualification Summary - SDG R2844922

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson

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LDC #:	21257K2a	VALIDATION COMPLETENESS WORKSHEET
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SDG #: R2844922 Stage 2B Laboratory: Columbia Analytical Services

Page: of Reviewer: 2nd Reviewer:

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

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

	Validation Area		Comments
1.	Technical holding times	SW)	Sampling dates: 7/10 _ 11 /6 8
11.	GC/MS Instrument performance check	A	
III.	Initial calibration	A	2 RSD rx
IV.	Continuing calibration/ICV	A	2 RSD r2 car/101 = 252
V.	Blanks	SW	
VI.	Surrogate spikes	>W	
VII.	Matrix spike/Matrix spike duplicates	SN	
VIII.	Laboratory control samples	SW	VCS /3
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	¥	
XI.	Target compound identification	N	
XII.	Compound quantitation/CRQLs	SW	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	N	
XV.	Overall assessment of data	SW	
XVI.	Field duplicates	SW)	D = 15/16; 15/17; 23/24; 23/25
XVII.	Field blanks	N	

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

TB = Trip blank

EB = Equipment blank

Validated Samples:

Soil

	3071						
1	RSAJ8-0.5B	11	RSAL2-0.5B	21	RSAK2-0.5B	31	SBLKI
2	RSAJ8-0.5BDL	12	RSAL2-0.5BDL	22	RSAK2-10B	32	5 B 1 K 2
3	RSAJ8-10B	13	RSAL2-10B	23	RSAK2-20B D, D4	33	
4	RSAJ8-20B	14	RSAL2-10BDL	24	RSAK2-20BD	34	
5	RSAJ8-30B	15	RSAL2-20B	25	RSAK2-20BDDL D4	35	
6	RSAJ8-33B	16	RSAL2-20BD P	26	RSAK2-30B	36	
7	RSAI7-0.5B	17	RSAL2-20BDDL by	27	RSAK2-35B	37	
8	RSAI7-10B	18	RSAL2-30B	28	RSA17-32B	38	
9	RSAI7-20B	19	RSAL2-37B	29	RSAL2-0.5BMS	39	
10	RSAI7-30B	20	RSAL2-40B	30	RSAL2-0.5BMSD	40	

# **VALIDATION FINDINGS WORKSHEET**

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

A. Phenol**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenol**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	VV. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-butyiphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene⁴	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP, Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	TTT. Octachlorostyrune
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	uuu 1,4- Dioxane
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	W.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	WWW.

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

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### VALIDATION FINDINGS WORKSHEET Technical Holding Times

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Reviewer:	JV4
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All circled dates have exceeded the technical holding times.

METHOD : GC/N	Matrix	Preserved	Sampling Date	Extraction date	Analysis date	Total #	Qualifie
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### **TECHNICAL HOLDING TIME CRITERIA**

Water: Extracte

Extracted within 7 days, analyzed within 40 days.

Soil:

Extracted within 14 days, analyzed within 40 days.

21 2 S7 Kza SDG #: Sec Coner LDC #:

# VALIDATION FINDINGS WORKSHEET

**Blanks** 

Page: of ] 2nd Reviewer:\_ 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?

 $\sqrt[4]{N/A}$  Was the blank contaminated? If yes, please,see qualification below. Blank extraction date:  $\sqrt{1/3/08}$  Blank analysis date:  $\sqrt{1/2.2/08}$ 

(79)

Conc. units: we //c.			Associa	Associated Samples:	,	}				
mpour	Blank ID					Sample Identification	ítion			
	SBUK 1	8	4	2	ه	7	8	6	10	(xg) 11
7.4	<u>+</u> ,-	1.6/1	1.9 /4	2,0/4	1.5 /M	1.4/4	n/ I'c	(9.8)	(3)	1,7 h
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Biank extraction date:Conc. units:	_ biank analysis date:_	'sis date:	Associa	Associated Samples:_				(79)	()	
Compound	Blank ID				S	Sample Identification	ıtion			
	SBUKI	ટ	ة	(x9) L1	81	19 (6x)	20 (2x)	( ×5) lz	22 (2x)	23(30) 24 (2)
77	4.4	(8,5)	(11)	12/4	(8/	16/1		$8.7 / \mu$	7, 3)	(2.p) Wal
		)	)		)		)			)

5x Phthalates 2x all others

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VALIDATION FINDINGS WORKSHEET

Surrogate Recovery

Page: of

Reviewer: 2nd Reviewer:\_\_

Est. H SDG#:

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

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

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

*	Date	Sample ID	0	Surrogate	%R (Limits)	lits)	Qualifications
		2 (90x)	۲)	<i></i> 4⁄1	00	( K-155 )	NE gral
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* OC lir S1 (NBZ S2 (FBP S3 (TPH S4 (PHL	* QC limits are advisory S1 (NBZ) = Nitrobenzene-d5 S2 (FBP) = 2-Fluorobipheny S3 (TPH) = Terphenyl-d14 S4 (PHL) = Phenol-d5	QC Limits (Soil) 23-120 30-115 18-137 24-113	QC Limits (Water) 35-114 43-116 33-141 10-94	<i>w w w w</i>	5 (2FP)= 2-Fluorophenol 6 (TBP) = 2,4,6-Tribromophenol 7 (2CP) = 2-Chlorophenol-d4 8 (DCB) = 1,2-Dichlorobenzene-d4	QC Limits (Soil) 25-121 19-122 20-130*	QC Limits (Water) 21-100 10-123 33-110*

LDC# 2/251 RAN SDG #:

## VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Page: / of 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 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 Y N N/A

MS/MSD. Soil / Water.

,				MS	MSD				A
	Date	MS/MSD ID	Compound	%R (Limits)	%R (Limits)	RPD (Limits)	Associated Samples	Qualifications	
$\dashv$		29/30	444	(29-120)	651-05) 74	( )		No mal (L	. S.
		/ ,	X	1   1   1	230( )	( )		<i>-</i>	١,
Н			SS	896 ( )		( )			MSDin
			777	978 (	>78 ( )	( )			537
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	Compound	QC Umits (Soil)	RPD (Soll)	QC Limits (Water)	RPD (Water)		Compound	QC Limits (Soli)	RPD (Soll)	QC Limits (Water)	RPD (Water)
κ̈	Phenol	26-90%	~32%	12-110%	<u>≤ 42%</u>	ອອ	Acenaphthene	31-137%	<u>≤ 19%</u>	46-118%	<u>&lt;</u> 31%
ن	C. 2-Chlorophenol	25-102%	< 50%	27-123%	< 40%	=	4-Nitrophenol	11-114%	< 50%	10-80%	%09 ×
ш	1,4-Dichlorobenzene	28-104%	< 27%	36-97%	< 28%	KK.	2,4-Dinitrotoluene	78-83%	< 47%	24-96%	< 38%
-	N-Nitroso-di-n-propylamine	41-126%	< 38%	41-116%	< 38%	Ή.	Pentachlorophenol	17-109%	< 47%	9-103%	×09 >
α	1,2,4-Trichlorobenzene	38-107%	< 23%	39-98%	< 28%	72.	Pyrene	35-142%	× 36%	26-127%	< 31%
>	4-Chloro-3-methylphenol	26-103%	× 33%	23-97%	< 42%						

LDC# 21257 R 24 SDG #:

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

Reviewer: Page: 2nd Reviewer.

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

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

| N/A | N/A | Was a LCS required?

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

	•		801	283			
# Date	CS/CSD ID	Compound	%R (Limits)	%R (Limits)	RPD (Limits)	Associated Samples	Qualifications
	\c\>	PEF	158 (50-120)	( )	( )	11-25. SBUKI	No mass ( Mc/mchi.)
			( )	( )	( )		
			(	(	(		
			(	( )			
			( )	( )	( )		
			( )	( )	`		
			( )	( )	)		
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			`	( )	( )		
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			( )	( )	( )		
			( )	( )	( )		-
			( )	( )	(		
	-			,			

LDC #: 2/ 257 K2a

# Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

Page: 1 of Reviewer:

2nd Reviewer:

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

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

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

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		1 11	SS 777 > CON MAKE	9	Java/A (e)
		_	,		
		13, 16, 24	↑ XX		7
		=			
		:			

Comments: See sample calculation verification worksheet for recalculations

LDC# 21257 KZA

### **VALIDATION FINDINGS WORKSHEET** Overall Assessment of Data

Page: Reviewer:

2nd Reviewer:

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

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

All available information pertaining to the data were reviewed using professional judgement to compliment the determination of the overall quality of the data.

X/N N/A

Was the overall quality and usability of the data acceptable?

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		1, 11	> cul	hund	(6) X X
		2 2	#11 except SS, TTT	- 4:1	
		13 16 24	XX > cal range		
		14, 17, 25	A11 except xx dil		
Comm	Comments:				

LDC #:	21	257	K29
SDG #:	Su	Com	<b>√</b> `

### VALIDATION FINDINGS WORKSHEET Field Duplicates

Page:	lof/_
Reviewer:	SVE
2nd reviewer:	T

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

Y N N/A Y N N/A Were field duplicate pairs identified in this SDG? Were target compounds identified in the field duplicate pairs?

		Concentratio	ugks,		piff
Compound		15	16		-RPD
·	XX	560	660	100 (4	390)
	AAA	390 U	30 .	360	
	LL	49	54	5	
	CC	4.0	4.8	0.8	
	YY	3.9	4.6	0.7	£ 15).
	uu	15 U	12	3	
		Concentration	<b>U</b>		
Compound					RPD
	22	154	1.9	13.1	
				-	
		·			

	Concentratio	n ug/kg/	Dill Phrent
Compound	15	17	Biff my
AAA	3904	70	320 (= 390)
XX	560	180Q	1240 (£1100) J dets/4
. 1L	49	1100 U·	1051
CC	4:0	1	10.96
	3,9	43 u	39.1 (43)
ии	15 U	12	3 (415)
	Concentration	14	
: Compound			RPD
·			

LDC #:_	2125	7 K29
SDG #:_	Su	Coner

### VALIDATION FINDINGS WORKSHEET Field Duplicates

Page:_	⊋of_	$\nu$
Reviewer:	JI	74
2nd reviewer:		1

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

/Y	N	N/A
Y	N	N/A

Were field duplicate pairs identified in this SDG? Were target compounds identified in the field duplicate pairs?

	Concentration	ng ug/Eg,	Þiff	
Compound	23	24	-RPD	
AAA	64	62	2 (= 920)	
Χχ	1500	1000	500	
LL LL	49	36	13	
Ce	920 U	3.4	916,6	
	36 U	4.5	31.5 (= 36)	
ич	10	9.2	0.8	
	Concentratio	n 45/kg)		
Compound	23	25	RPD	
AAA	64	130	66 (= 1800)	
XX	1500	3200	1700 (= 1800)	
LL	49	1800 4	1751	
чч	10	71 U	61 (4717	
·				

	Concentratio	n ()	
Compound			RPD
	:		
	<u>.</u>		
·			·

	Concentration ( )		
Compound			RPD
·			

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

Project/Site Name:

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 11, 2008

LDC Report Date:

August 27, 2009

Matrix:

Soil

Parameters:

Semivolatiles

Validation Level:

Stage 2B

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2845025

Sample Identification

RSAI7-10B(119156) RSAI7-10B(119157)

### Introduction

This data review covers 2 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 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) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met with the following exceptions:

Sample	Compound	Total Days From Sample Collection Until SPLP Extraction	Required Holding Time (in Days) From Sample Collection Until SPLP Extraction	Flag	A or P
All samples in SDG R2845025	All TCL compounds	17	14	J- (all detects) UJ (all non-detects)	Р

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 15.0% for each individual compound and less than or equal to 30.0% for calibration check compounds (CCCs).

In the case where %RSD was greater than 15.0%, the laboratory used a calibration curve to evaluate the compound. All coefficients of determination (r²) were greater than or equal to 0.990.

For the purposes of technical evaluation, all compounds were evaluated against the 30.0% (%RSD) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

Average relative response factors (RRF) for all semivolatile target compounds and system performance check compounds (SPCCs) were greater than or equal to 0.05 as required.

### 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).

For the purposes of technical evaluation, all compounds were evaluated against the 25.0% (%D) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

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 RRF values were greater than or equal to 0.05.

### 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	Analysis Date	Compound TIC (RT in minutes)	Concentration	Associated Samples
SBLK1	7/31/08	Butylbenzylphthalate	0.28 ug/L	All samples in SDG R2845025
EQBLK1	7/31/08	Butylbenzylphthalate Diethylphthalate Bis(2-ethylhexyl)phthalate Naphthalene	0.35 ug/L 0.15 ug/L 0.28 ug/L 0.060 ug/L	All samples in SDG R2845025
EQBLK2	7/31/08	Butylbenzylphthalate Diethylphthalate Bis(2-ethylhexyl)phthalate	0.34 ug/L 0.14 ug/L 0.85 ug/L	All samples in SDG R2845025

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	Reported	Modified Final
	TIC (RT in minutes)	Concentration	Concentration
RSAI7-10B(119156)	Butylbenzylphthalate	0.30 ug/L	0.30U ug/L
	Diethylphthalate	0.33 ug/L	0.33U ug/L
	Bis(2-ethylhexyl)phthalate	0.98 ug/L	0.98U ug/L
	Naphthalene	0.051 ug/L	0.051U ug/L
RSAI7-10B(119157)	Butylbenzylphthalate	0.33 ug/L	0.33U ug/L
	Diethylphthalate	0.39 ug/L	0.39U ug/L
	Bis(2-ethylhexyl)phthalate	3.0 ug/L	3.0U ug/L
	Naphthalene	0.053 ug/L	0.053U ug/L

No field blanks were identified in this SDG.

### VI. Surrogate Spikes

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

### VII. Matrix Spike/Matrix Spike Duplicates

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 with the following exceptions:

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Flag	A or P
LCS/D1 (All samples in SDG R2845025)	Pyridine	40 (50-120)	15 (50-120)	91 (≤30)	J (all detects) UJ (all non-detects)	Р

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

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

### XI. Target Compound Identifications

Raw data were not reviewed for this SDG.

### XII. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R2845025	All compounds reported below the PQL.	J (ali detects)	А

Raw data were not reviewed for this SDG.

### XIII. Tentatively Identified Compounds (TICs)

Raw data were not reviewed for this SDG.

### XIV. System Performance

Raw data were not reviewed for this SDG.

### XV. Overall Assessment

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

### XVI. Field Duplicates

No field duplicates were identified in this SDG.

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Data Qualification Summary - SDG R2845025

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R2845025	RSAI7-10B(119156) RSAI7-10B(119157)	All TCL compounds	J- (all detects) UJ (all non-detects)	Р	Technical holding times (h)
R2845025	RSAI7-10B(119156) RSAI7-10B(119157)	Pyridine	J (all detects) UJ (all non-detects)	Р	Laboratory control samples (%R)(RPD) (I, Id)
R2845025	RSAI7-10B(119156) RSAI7-10B(119157)	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (sp)

### Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles - Laboratory Blank Data Qualification Summary - SDG R2845025

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P	Code
R2845025	RSAI7-10B(119156)	Butylbenzylphthalate Diethylphthalate Bis(2-ethylhexyl)phthalate Naphthalene	0.30U ug/L 0.33U ug/L 0.98U ug/L 0.051U ug/L	A	bl
R2845025	RSAI7-10B(119157)	Butylbenzylphthalate Diethylphthalate Bis(2-ethylhexyl)phthalate Naphthalene	0.33U ug/L 0.39U ug/L 3.0U ug/L 0.053U ug/L	- А	bl

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Semivolatiles- Field Blank Data Qualification Summary - SDG R2845025

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson**

_DC #:	21257L2a	VALIDATION COMPLETENESS WORKSHEET	
SDG #:_	R2845025	Stage 2B	
aborato	rv: Columbia Analytica	al Services	

Reviewer: 30 2nd Reviewer:

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

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

	Validation Area		Comments
I.	Technical holding times	SW	Sampling dates: 7/11 /08
11.	GC/MS Instrument performance check	A	
111.	Initial calibration	A	2 esp rr
IV.	Continuing calibration/ICV	A	2 esp r × cev / 1 cv ≤ 252
V.	Blanks	SW	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	N	Client spec
VIII.	Laboratory control samples	SN	us /o
IX.	Regional Quality Assurance and Quality Control	N	_
X.	Internal standards	A	
XI.	Target compound identification	N	
XII.	Compound quantitation/CRQLs	N	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	N	
XV.	Overall assessment of data	A	
XVI.	Field duplicates	N	
XVII.	Field blanks	Ŋ	

Note:

A = Acceptable N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate TB = Trip blank

EB = Equipment blank

Validated Samples:

Sail

	2011				
1	RSAI7-10B(119156)	11	21	3	31
2	RSAI7-10B(119157)	12	22		32
3	SBUK, (7/	n ) 13	23	3	33
4	EBBUKI	14	24	3	34
<b>4</b> 5	ERBIK!	15	25	3	35
6		16	26	3	36
7		17	27	3	37
8		18	28	3	38
9		19	29	3	39
10		20	30		40

# VALIDATION FINDINGS WORKSHEET

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

A. Phenol**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachiorophenol**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. 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-Dinitrophenoi*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY, Fluoranthene**	NNN, Aniline
G. 2-Methylphenol	V. 4-Chioro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	000. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethyiphthalate	AAA. Butylbenzylphthalate	PPP. Benzolc Acid
i. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chiorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	111.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	ກກກ
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	۷۸۷.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	www.

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

LDC #:	2/25	7129
SDG #:_	Ser	Circl

### VALIDATION FINDINGS WORKSHEET Technical Holding Times

Page:	of /
Reviewer:	JVZ
2nd Reviewer:	4

All circled dates have exceeded the technical holding times.

METHOD : GC	Matrix	Preserved	Sampling Date	CPLP  (Extraction date)	Analysis date	Total # of Days	Qualifier
1,2	5	N	7/11/08	7/28/08	7/31/08	17	5-/W/F
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### **TECHNICAL HOLDING TIME CRITERIA**

Water:

Extracted within 7 days, analyzed within 40 days.

Soil:

Extracted within 14 days, analyzed within 40 days.

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257	
7	
C #:	۱ ۲

### **VALIDATION FINDINGS WORKSHEET** Blanks

Page: \ of	Reviewer: 316	nd Reviewer:
		2nd

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

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

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

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

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

Was the blank contaminated? If yes, please see qualification below. In date: 7/31/3 Blank analysis date: N N A

Hank extraction date: 7/31/8 Blank analysis date: Conc. units:

(19) **→** 

Colle. dilles. The Ic			Assucial	Associated Salliples.				
Compound	Blank ID				Sa	Sample Identification		
	SBIKI	をおお水り	EQ BURY		2			
AAA	0.28 0.35	0.35	6.34	0.30/u 0.33/h	0.33 M			
77		0, 15	P10	W. 33/4 0.39/4	4/65.0			
337		0,28	58.0	48/4 3.0/V	h/0.8			
S		0.060		y/150.0	1/650.0 W/120.0			
					,			

Blank analysis date:\_ Blank extraction date:\_

Conc. units:

Associated Samples:

		_

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

2/22/12 SDG #: LDC#:

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

Page:

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

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

Y N N/A

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

Ŀ				SOT	CSD			
<u>.</u>	Cate	LCS/LCSD ID	-1r	%R (Limits)		RPD (Limits)	Associated Samples	Qualifications
			××	125 (50-120)		( )	All + Bikc	No gual (1650 il
			RKR	( † ) eh	(05/765) 51	( %) /b		1/11/6 11 H
				)	( )	( )		
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