

LABORATORY DATA CONSULTANTS, INC.

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

Northgate Environmental Management, Inc. 1100 Quail Street Ste. 102 New Port beach, CA 92660 ATTN: Ms. Cindy Arnold Februayr 3, 2010

SUBJECT: Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada, Data Validation

Dear Ms. Arnold,

Enclosed is the revised data validation report for the fractions listed below. The data validation was performed under Stage 2B & 4 guidelines. Please replace the previously submitted report with the enclosed revised report.

### LDC Project # 21768:

SDG # Fraction

R0904797 Chlorinated Pesticides, Cyanide, Gasoline Range Organics

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

Sincerely,

Erlinda T. Rauto Operations Manager/Senior Chemist

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

Project/Site Name: Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada

Collection Date: August 24 through August 26, 2009

LDC Report Date: February 2, 2010

Matrix: Soil

Parameters: Chlorinated Pesticides

Validation Level: Stage 2B & 4

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R0904797

### Sample Identification

SA154-0.5B\*\* SA154-10B\*\* SA154-33B RSAS3-0.5B RSAS3-0.5B RSAS3-10B RSAS3-10B RSAS3-25B RSAS3-25B RSAS3-44B SA154-0.5BMS SA154-0.5BMS RSAS3-0.5BMS

\*\*Indicates sample underwent Stage 4 review.

### Introduction

This data review covers 12 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8081A for Chlorinated Pesticides.

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

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### **II. GC/ECD Instrument Performance Check**

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

### III. Initial Calibration

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

The percent relative standard deviations (%RSD) were less than or equal to 20.0% for all compounds.

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

### IV. Continuing Calibration

Continuing calibration was performed at required frequencies.

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

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

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

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

### V. Blanks

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

Samples FB072909-SO (from SDG R0904226) and FB080309-SO (from SDG R0904279) were identified as field blanks. No chlorinated pesticide contaminants were found in these blanks with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB072909-SO	7/29/09	alpha-BHC	0.092 ug/L	SA154-0.5B** SA154-10B** SA154-33B

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

### VI. Surrogate Spikes

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

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
95021MB	Not specified	Tetrachloro-m-xylene	25 (40-140)	All TCL compounds	J- (all detects) UJ (all non-detects)	Ρ

### VII. Matrix Spike/Matrix Spike Duplicates

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Pesticide Cleanup Checks

### a. Florisil Cartridge Check

Florisil cleanup was not required and therefore not performed in this SDG.

### b. GPC Calibration

GPC cleanup was not required and therefore not performed in this SDG.

### XI. Target Compound Identification

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

### XII. Project Quantitation Limit

All project quantitation limits were within validation criteria for samples on which a Stage 4 review was performed.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R0904797	All compounds reported below the PQL.	J (all detects)	A

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

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

Samples RSAS3-0.5B and RSAS3009-0.5B were identified as field duplicates. No chlorinated pesticides were detected in any of the samples with the following exceptions:

	Concentrat	ion (ug/Kg)		D'11		
Compound	RSAS3-0.5B	RSAS3009-0.5B	(Limits)	Diπerence (Limits)	Flags	A or P
beta-BHC	1.6	2.0	-	0.4 (≤1.8)	-	-

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Chlorinated Pesticides - Data Qualification Summary - SDG R0904797

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R0904797	SA154-0.5B** SA154-10B** SA154-33B RSAS3-0.5B RSAS3-0.5B RSAS3-0.5B RSAS3-10B RSAS3-10B RSAS3-25B RSAS3-44B	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG R0904797

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG R0904797

No Sample Data Qualified in this SDG

Tronox Northgate Henderson	
VALIDATION COMPLETENESS WORKSHEE	T
Stage 2B / 4	

LDC #: 21768F3a

SDG #: <u>R0904797</u>

Laboratory: Columbia Analytical Services

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

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

			Comments
╘╼╾┥			3/24 24 ka
١.	Technical holding times	<u> </u>	Sampling dates: 0 / 24 - 26 /09
١.	GC/ECD Instrument Performance Check	A	
111.	Initial calibration	A	4 RSD E 20 D
IV.	Continuing calibration/ICV	<u>A</u>	Car/101 = 203
V.	Blanks	<u> </u>	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	SW	Las b
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	
Xb.	GPC Calibration	N	
XI.	Target compound identification	N	
XII.	Compound quantitation and reported CRQLs	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	D = 9,5
xv/	Field blanks	SW	FB= FB072901-50 (R0904226)
<u>لے کی ا</u>			+ FB 080309- 50 (R 09 09 279)

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:

anua	Soil			
1 1	SA154-0.5B ***	11 RSAS3-0.5BMS	211 95021 MB	31
2	SA154-10B **	12 RSAS3-0.5BMSD	22 7 95+17	32
3	SA154-33B	13	23	33
4 4	RSAS3-0.5B	14	24	34
5 I	RSAS3009-0.5B	15	25	35
6 1	RSAS3-10B	16	26	36
71	RSAS3-25B	17	27	37
8	RSAS3-44B	18	28	38
9	SA154-0.5BMS + 4	19	29	39
10	SA154-0.5BMSD 4+	20	30	40

Date: 10/23 Page: 1 of Reviewer: N 2nd Reviewer:

Page: 1 of 2 Reviewer: <u>JVC</u> 2nd Reviewer: <u>1</u>

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

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Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	/			
Cooler temperature criteria was met.				
II. GC/ECD Instrument performance check				
Was the instrument performance found to be acceptable?	/	ł		
III. Initial calibration		r		
Did the laboratory perform a 5 point calibration prior to sample analysis?	/_	-		
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations $(\%$ RSD) $\leq 20\%$ ?	/	-		
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?		_	Ł	
Did the initial calibration meet the curve fit acceptance criteria?			-	
Were the RT windows properly established?	$\leq$			
Were the required standard concentrations analyzed in the initial calibration?				
IV. Continuing calibration			r	
What type of continuing calibration calculation was performed?%D or%R	<			
Were Evaluation mix standards analyzed prior to the initial calibration and sample analysis?	/	-		
Were endrin and 4,4'-DDT breakdowns $\leq 5$ for individual breakdown in the Evaluation mix standards?	/			
Was a continuing calibration analyzed daily?				
Were all percent differences (%D) $\leq$ 20% or percent recovieries 80-120%?	/			
Were all the retention times within the acceptance windows?				
V Blanks				
Was a method blank associated with every sample in this SDG?	/	-		
Was a method blank analyzed for each matrix and concentration?	<			
Were extract cleanup blanks analyzed with every batch requiring clean-up?	$\checkmark$			
Was there contamination in the method blanks or clean-up blanks? If yes, please see the Blanks validation completeness worksheet.		/		
VI. Surrogate spikes				
Were all surrogate %R within the QC limits?		/		
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?		/		
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?				
VII. Matrix spike/Matrix spike duplicates				

### VALIDATION FINDINGS CHECKLIST

Page:_	2 of 2
Reviewer:	TVG
2nd Reviewer:	01

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Validation Area	Yes	No	NA	Findings/Comments
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			
Was a MS/MSD analyzed every 20 samples of each matrix?	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII. Laboratory control samples			r	
Was an LCS analyzed for this SDG?				
Was an LCS analyzed per extraction batch?				· · · · · · · · · · · · · · · · · · ·
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX: Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				·
Were the performance evaluation (PE) samples within the acceptance limits?				
X. Target compound identification		P	r	
Were the retention times of reported detects within the RT windows?				
XI. Compound quantitation/CRQLs	<b>I</b> 1		<b></b>	
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions, dry , weight factors, and clean-up activities applicable to level IV validation?				
XII: System performance				
System performance was found to be acceptable.		-		
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.	•			
Target compounds were detected in the field duplicates.				
XV. Field blanks				
Field blanks were identified in this SDG.	/			
Target compounds were detected in the field blanks.	/	· .		

VALIDATION FINDINGS WORKSHEET

METHOD: Pesticide/PCBs (EPASW 846 Method 8081/8082)

A alpha-BHC	l, Dieldrin	Q. Endrin katone	Y. Aroclor-1242	60.
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroelor-1248	HH.
c. detta-BHC	K. Endrin	8. aipha-Chiordane	AA. Aroolor-1254	
D. gamma-BHC	L. Endosuifan II	T. gamma-Chlordane	BB. Arocior-1260	JJ.
E. Heptachlor	M. 4,4'-DDD	U. Toxaphane	CC. DB 608	KK.
F. Aldrin	N. Endosulfan sulfate	V. Aroclar-1016	DD. DB 1701	LL.
G. Heptachior epoxide	Q. 4,4'-DDT	W. Aroclor-1221	EE. Hexachlorobenzene	MM.
H. Endosulfan I	P. Methoxychior	X. Aroclop-1232	Ť	NN.

Notes:

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LDC #: 21768 F34 SDG #: 24 64~			VALIDA.	TION FIND Field	INGS WOI Blanks	RKSHEET			Page: <u>of</u> of <u>N</u> Reviewer: <u>N</u>
METHOD: GC Pesticides/P <u>Y N N/A</u> Field blanks <u>V N N/A</u> Were target Blank units: <u>N 7</u> As	CBs (EPA SW s were identifi t compounds tsociated san	846 Method ed in this SD detected in t <b>nple units:</b>	8081) G. VS /S	ks?					2nd Reviewer:
Field blank type: (circle on	e) <sup>/</sup> Field Blank	/ Rinsate / C	)ther:		Associated {	Samples:	1- 3	(MD)	
Compound	Blank ID					Sample Identific	ation		
	FB673969-	8							
4	260'O								
CRal.									
Blank units: Asso	ciated sample	units:							
Sampling date: Field blank type: (circle one) F	-jeld Blank / Rir	sate / Other		Associat	ted Samples:				
Compound	Blank ID				0	ample identifica	ation		
CROL									
CIRCLED RESULTS WERE NOT OL	JALIFIED. ALL R	ESULTS NOT C	IRCLED WERE	QUALIFIED BY	THE FOLLOWIN	G STATEMENT			

Samples with compound concentrations within five times the associated field blank concentration are listed above, these sample results were qualified as not detected, "U".

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## VALIDATION FINDINGS WORKSHEET Surrogate Spikes

3 ð Page: Reviewer.\_\_\_\_\_ 2nd Reviewer.\_\_\_\_\_

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

Please see qualification below for all questions answered "N". Not applicable questions are identified as "N/A". <u>V N N/A</u> Were surrogates spiked into all samples, standards and blanks? <u>V N N/A</u> Did all surrogate percent recoveries (%R) meet the QC limits?

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Qualifications	(5/ 4/11	•																		Comments	
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%R (LIn	25																			Å	
Surrogate Compound	*																			tecovery QC Limits (Soil)	
Column	Net Sher.																				
Sample (D	15021 MB																			Surrogate Compound	Tetrachloro-m-xviene
Date																				ter Designation	A
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LDC # 21768 F34 SDG # 24 44

## VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

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

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". <u>N N/A</u> Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? <u>N N/A</u> Was a MS/MSD analyzed every 20 samples for each matrix or whenever a sample extraction was performed?

V N N/A	Were the MS/MSD	percent recover	ies (%R) and the relé	ative percent difference	SS (KPU) WILLIE LIE W		
			MS VD /1 imite1	MSD %R (1 Imits)	RPD (Limits)	Associated Samples	Qualifications
ة *	te MS/MSDID	nunoduo.			1 02 KBAL		No suce
	9/10	SCREA	(OMPONIDA)	MANE 16 R WA			
		tro	ide inits	( )	<b>^</b>		( ertner ms ; mb
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### COLUMBIA ANALYTICAL SERVICES, INC.

### QA/QC Report

Client:Northgate EnvironmentalProject:Tronox LLC Henderson/2027.001Sample Matrix:Soil

Service Request: R0904797 Date Collected: 8/24/09 Date Received: 8/25/09 Date Analyzed: 9/ 8/09 1.000

### Matrix Spike Summary Organochlorine Pesticides by Gas Chromatography

Sample Name:	SA154-0.5B	Units:	µg/Kg
Lab Code:	R0904797-009	Basis:	; Dry
Analytical Method:	8081A		

Prep Method: EPA 3541

rich memor.	Sample	N	1atrix Spike Q0908029-0	e 4	Duplic R	ate Matrix Q0908029-0	Spike 5	% Rec	מקמ	R	PD imit
Analyte Name	Result	Result	Amount	% Rec	Result	Amount	% Rec	Linus	NI D	,	
4,4'-DDD	ND	6.65	7.15	93	9.04	7.15	126 293	* 58 - 121 * 56 - 125	31 31	*	30 30
4,4'-DDE	ND	15.3	7.15	214 *	14 1	7 15	197	* 9 - 149	47	*	30
4,4'-DDT	ND	8.75	7.15	122	8 36	7 15	117	15 - 135	38	*	30
Aldrin	ND	5.68	7.15	/9	0.50	7.15	135	25 - 150	45	*	30
Dieldrin	ND	6.11	7.15	83 00	9.05 0.07	7.15	127	* 56 - 119	24		30
Endosulfan I	ND	7.11	7,15	97 104	10.9	7.15	152	* 65 - 127	36	*	30
Endosulfan II	ND	7.61	7.15	66	8 97	7.15	125	* 37 - 122	35	*	30
Endosulfan Sulfate	ND	6.29	7.15	00	10.0	7.15	140	28 - 143	39	*	30
Endrin	ND	6,73	7.15	75	8 90	7.15	124	18 - 135	49	*	30
Endrin Aldehyde	ND	5.39	7.15	86	9 25	7.15	129	* 57 - 123	40	*	30
Endrin Ketone	ND	0,18	7.15	94	9.32	7.15	130	* 35 - 127	32	*	30
Heptachlor	ND	7 70	7.15	109	10.8	7.15	151	* 61 - 120	32	*	30
Heptachlor Epoxide	ND	52 7	179	53	76.7	17.9	182	* 20 - 150	35	*	30
Hexachlorobenzene	44 NTD	33.7 A9 7	357	118	53,2	35.7	149	38 - 149	23		30
Methoxychlor	ND	5 03	7 15	83	8.65	7.15	121	53 - 130	37	*	30
aipha-BHC		5.03	7.15	83	8.72	7.15	122	27 - 130	38	Ŧ	30
alpha-Chiordane		121	7 15	170	* 16.3	7.15	227	* 35 - 142	29		30
beta-BHC		5 22	7 15	73	6,90	7.15	96	44 - 119	28	÷	30
delta-BHC		5.03	7 15	83	8.68	7.15	121	37 - 124	38	*	- 30
gamma-BHC (Lindand gamma-Chlordane	ND ND	8,90	7.15	124	12.2	7.15	171	* 38 - 127	31	¥	j0

Comments:

Printed 10/5/09 8:48 http://www.starturast.imsReps/MatrixSpike.ga Matrix Spike Summary

SuperSet Reference: 09-0000117998 rev 00

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	SDG #:

### VALIDATION FINDINGS WORKSHEET Laboratory Control Samples

No Page: 1 of Reviewer:

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

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". V N/A Were a laboratory control samples (LCS) and laboratory control sample duplicate (LCSD) analyzed for each matrix in this SDG? \_\_\_\_\_ V N/A Were the LCS percent recoveries (%R) and relative percent differences (RPD) within the QC limits? <u>Y N N/A</u> W <u>Y N N/A</u> W Level WR Only Ş

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sm)؛	-																·									
Qualification	Nº quel																									
Associated Samples	1-3,5-8,95021 MB			•																						
RPD (Limits)	( )		( )	( )		( )		( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(	(	( )	( )	( )	( )	( )
SD F	Rel-05	(	(	-		·	(	(	(	)	(	) (	•	(	(	-	(	(	(	(	(	(	)	) (	( )	
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LCS %R (Limits)	43 (20-130	)	>	}	1	~	~	-)	~	}	· ·	~	)	_	)	_	)	~	~	)	)	)	_	~	-	~
Compound		× ×																								
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LDC #:	21768	F 39
SDG #:	Su	Comor

### VALIDATION FINDINGS WORKSHEET Field Duplicates

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### METHOD: GC Pesticides/PCBs (EPA SW846 Method 8081/8082)

Y<u>N N/A</u> YN N/A Were field duplicate pairs identified in this SDG? Were target compounds detected in thie field duplicate pairs?

	Concentratio	ng has /kgg	
· Compound	4	5	RPD
<u></u>	). 6	2,0	0.4 ( ± 1.8 D ) -
		L	

	Concentration ()	
Compound		RPD

	Concentration	<u> </u>	
Compound			RPD
			······································
			·

	Concentration (	
Compound		RPD

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LDC #:	SDG #:

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

N Page: 1 of 1 2nd Reviewer: Reviewer:

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

The calibration factors (CF) and relative standard deviation (%RSD) were recalculated using the following calculations:

Average CF = sum of the CF/number of standards %RSD = 100 \* (S/X) CF = A/C

Where: A = Area of compound C = Concentration of compound S = Standard deviation of calibration factors X = Mean of calibration factors

					Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date		Compound	CF ( ) 0 std)	CF ( )0 std)	CF (initial)	CF (intial)	%RSD	%RSD
-	ILAL	8/20/20	#	STX-C1P1	2.094 67	2,094 e7	2.179 67	2. 179 e7	3.59	3.59
		holso /	d	(a) / [m]	0.975	0.9748	0.991	0 99)	1. 67	1.67
			Ħ		5.892	5 892	5.988	5.987	1-14	1.14
			4	(m) x X	7.430 1	2. 4303	2 412 4	2.492 V	3.15	3.18
2										
		<b></b> -								
3										
4										
		[]								

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.

INICLCrev.wpd

LDC #: 21 768 F 34 SDG #: 54 Cmm

VALIDATION FINDINGS WORKSHEET Continuing Calibration Results Verification

Page: of / Reviewer: <u>3v6</u> 2nd Reviewer: <u>\_\_\_\_</u>

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

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

Where: N = Initial Calibration Factor or Nominal Amount (ng) C = Calibration Factor from Continuing Calibration Standard or Calculated Amount (ng)

						Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date/Time	Compound		Average CF/ CCV Conc	CF/Conc CCV	CF/Conc CCV	Q%	<b>D%</b>
-	CCV 18A	6 / / a	×6 H	c1p)	21.794 26	22.08/ 00	22 .08 22	. 3	لا . ا
		1018011	d	•	9.914	9.673	9.673	2 ¢	メズ
			H	×	59.876	67.628	67,63	12.9	12.95
			4	7	24.917 V	25.539	25.39	ンン	とん
5	CCV21 A	on her has	#	<u> </u>		22. 120	22.13	۱. ۲	レー
			d			9.689	9.684	2,3	2, 3
			H	5		64.913	64.513	8. ¢	8.4
			4	~	<u> </u>	1 6 20.20	1 680 SC	0.7	0° 7
3									
4									

Comments: Refer to Continuing Calibration findings worksheet for list of gualifications and associated samples when reported results do not agree within 10.0% of the recalculated results. LDC #: 21768 F3( SDG #: Su Com

### VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page:_	1 of 1
Reviewer:	M.
2nd reviewer:	ŀ
	Ι

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

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

### Sample ID:

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene						
Tetrachloro-m-xylene	STX-CLP7	100	8.607 (10)	86	٤٢	0
Decachlorobiphenyl			9.628	96	96	
Decachlorobiphenyl			-			

### Sample ID:

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene						
Tetrachloro-m-xylene						
Decachlorobiphenyl						
Decachlorobiphenyl						

### Sample ID:

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene						
Tetrachloro-m-xylene						
Decachlorobiphenyl						
Decachlorobiphenyl						

### Sample ID:

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene						
Tetrachloro-m-xylene						
Decachlorobiphenyl						
Decachlorobiphenyl						

### Notes:

F34	Jus -
21768	ž
÷	#
Ĕ	SDC

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

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

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

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

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

SSC = Spiked sample concentration SA = Spike added

4

3

MS/MSD samples:

RPD = I MS - MSD I \* 2/(MS + MSD)

Where:

MS = Matrix spike percent recovery

MSD = Matrix spike duplicate percent recovery

SC = Concentration

Recalculated

Reported

Recalc.

**MS/MSD** RPD 38 47

4 38

	Ś	pike	Sample	Spiked	Sample	Matrix	r Spike	Matrix Spil	ke Duplicate
Compound	¥ (	deed (A)	Concentration ( uん / に)	Conce ( MS	Act)	Percent	Recovery	Percent	Recovery
	SM	0 MSD	<i>o</i> .	WS	0SM	Reported	Recalc.	Reported	Recalc
gamma-BHC	7. 15	212	٩	5.93	878	53	83	(7)	[2]
4,4'-DDT		<b>-</b>	-*	8.75	14.1	トイ	~~ (	167	197
Aroclor 1260				-	-			/	,

sociated samples when reported results do not agree within 10.0% of the recalculated results CONTINUENTS. RELET OF

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ETHOD: GC Pesticides/PCBs (EPA SW 846 Me re percent recoveries (%R) and Relative Percent mpounds identified below using the following ca Recovery = 100* (ssc-sc)/SA w No = 1LCs - LCSD 1* 2/(LCs + LCSD) D = 1LCs - LCSD 1* 2/(LCs + LCSD) CS/LCSD samples:	sthod 8081/ difference tlculation: sA =: SA =: LCS = Concentr	(B082) (RPD) of the = Spiked sample Spike added = Laboratory cont = Laboratory cont = Laboratory cont	laboratory contro concentration trol sample percent rec trol sample percent rec	sample and labo sovery LCSD = Lab ss tecovery	oratory control sa c = Concentration oratory control sample react	imple duplicate duplicate percent r <b>CSD</b> <b>Recovery</b>	were recalcu ecovery Reported R	lated for th LCSD PD Recalc.
the percent recoveries (%R) and Relative Percent mpounds identified below using the following ca Recovery = 100* (SSC-SC)/SA w D = I LCS - LCSD I * 2/(LCS + LCSD) CS/LCSD samples: לגר מין ארע לא SS/LCSD samples: לגר מין ארע לא Compound (גין לא לא) LCS LCSD	difference Ilculation: SA =: SA =: LCS = Concentr	(RPD) of the = Spiked sample Spike added = Laboratory cont = Laboratory cont = Laboratory cont	laboratory contro • concentration • trol sample percent rec trol sample percent rec • content rec	sample and laby every LCSD = Lab ss try	Dratory control sa c = Concentration oratory control sample <b>Percent</b>	imple duplicate duplicate percent r CSD Recovery Recover	were recalcu ecovery Reported	LCSD PD Recalc.
Recovery = 100° (SSC-SC)/SA W D = I LCS - LCSD 1* 2/(LCS + LCSD) SS/LCSD samples: うらっ レムンク SS/LCSD samples: うらっ レムシク Compound (いうん)	here: SSC SA =: SSC SA =: SC SA =: SC LCS = Concentr	= Spiked sample Spike added = Laboratory cont ample tation LCSD	trol sample percent rec Reported	sovery LCSD = Lab sovery LCSD = Lab ss ss fr	C = Concentration oratory control sample Percent	e duplicate percent n CSD Recovery Recalc.	ecovery Reported	LCSD PD Recalc.
D=ILCS-LCSD1*2/(LCS+LCSD) S/LCSD samples: S/LCSD samples: S/LCSD samples:	Spiked Si Spiked Si Concentr	= Laboratory cont ample ample 2.) LCSD	trol sample percent rec LC Reported C-4	overy LCSD = Lab S tecovery Cr	oratory control sample	o duplicate percent r CSD Recovery Recalc.	ecovery Reported	PD Recalc.
Compound (14/14) LCSD	Spiked Si Concentr (VK) /C	ample ration	LC Percent F Reported	iS tecovery Recalc.	Percent L	CSD Recovery Recalc.	LCS/ Reported	PD Recalc.
Compound (1/15/1k) Compound LCS CCSD	Concent CVC /C LCS	Z ) LCSD	Percent F Reported	tecovery Recalc. 57	Percent Reported	Recovery Recalc.	Reported R	PD Recalc.
	S LC	الدي کاريد	Reported ~ 4	Recalc. 54	Reported	Recalc.	Reported	Recalc.
	و	۲ ۲	5-4	25	1			1
amma-BHC 6.67 6-67 3		<u> </u>		-	<b>\</b> _>	Ľ,	- -	-
4-DDT 4 4 6	4.88	6. X	601	601	101	loi	4	$\boldsymbol{\lambda}$
roclor 1260								
mments: Refer to Laboratory Control Sample/Lat	boratory Co	IL Introl Sample	E Duolicate finding	s worksheet for li	st of gualification	s and associated	l l l	en renort
sults do not agree within 10.0% of the recalculate	ed results.							

V:\Validation Worksheets\Pesticides\LCSDCLC.wpd



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

N/A 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? STX-CPT

Example: Sample I.D. \_\_\_\_\_\_ EE: Conc. = (1219.9 ec) (10ml) (10)(0.984e8) (0.933) (309)= 44.29The A 4 ug Acy

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

Note:

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

Project/Site Name:	Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada				
Collection Date:	August 24, 2009				
LDC Report Date:	February 2, 2010				
Matrix:	Soil				
Parameters:	Cyanide				
Validation Level:	Stage 4				
Laboratory:	Columbia Analytical Services, Inc.				
Sample Delivery Group (SDG): R0904797					

### Sample Identification

SA154-0.5B

### Introduction

This data review covers one soil sample listed on the cover sheet. The analyses were per EPA SW 846 Method 9012A for Cyanide.

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

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section X.

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. Calibration

Calibration verification frequency and analysis criteria were met.

### III. Blanks

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

Samples FB072909-SO (from SDG R0904226) was identified as a field blank. No contaminant concentrations were found in this blank.

### IV. Matrix Spike/Matrix Spike Duplicates

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

### V. Duplicates

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

### VI. Laboratory Control Samples

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

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

All sample result verifications were acceptable.

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R0904797	All analytes reported below the PQL.	J (all detects)	А

### VIII. Overall Assessment

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

### IX. Field Duplicates

No field duplicates were identified in this SDG.

### Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Cyanide - Data Qualification Summary - SDG R0904797

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
R0904797	SA154-0.5B	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Cyanide - Laboratory Blank Data Qualification Summary - SDG R0904797

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Cyanide - Field Blank Data Qualification Summary - SDG R0904797

No Sample Data Qualified in this SDG

Laboratory: Columbia Analytical Services

LDC #: 21768F6 b

SDG #: R0904797

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

Stage 4

Date: 2-1-10 Page: Lof\_ Reviewer: C 2nd Reviewer:

### METHOD: (Analyte) Cyanide (EPA SW846 Method 9012A)

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
١.	Technical holding times	A	Sampling dates: 8124/09
lla.	Initial calibration	A	
lib.	Calibration verification	A	
111.	Blanks	A	
IV	Surrogate Spikes	$\mathbb{N}$	Not required
v	Matrix Spike/Matrix Spike Duplicates	N	Client specified
VI.	Duplicates	N	
VII	Laboratory control samples	A	LCS
VIII	Sample result verification	A	
	Overall assessment of data	A	
x	Field duplicates	$\overline{N}$	
	Field blanks	ND	FB=FB072909-SO (506# B0904226)

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.

Note:

					_	
1	SA154-0.5B	11	BBS	21	31	
2		12		22	32	
3		13		23	33	
4		14		24	34	
5		15		25	35	
6		16		26	36	
7		17		27	37	
8		18		28	38	
9		19		29	39	
10		20		30	40	

Notes:

LDC #: 2168

### VALIDATION FINDINGS CHECKLIST



Validation Area	Yes	s No	N.	A Findings/Comments
L'Technical holding times				
All technical holding times were met.		<u> </u>		-
Coolcr temperature criteria was met.	1	1		
II: Calibration				
Were all instruments calibrated daily, each set-up time?	14	1		
Were the proper number of standards used?	1-	1		
Were all initial calibration correlation coefficients > 0.995?	1-	1		
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?	-	1		
Were titrant checks performed as required? (Level IV only)	ļ	<u> </u>	1-	~
Were balance checks performed as required? (Level IV only)	11		<u> </u> -	t
In Blankston and State of Contract of Cont				
Was a method blank associated with every sample in this SDG?	14	ţ		·
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		~	1	
IV-Mator spike/Mator spike duplicates and Puplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.		-	╞	client specified
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.		-	-	
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq$ 20% for waters and $\leq$ 35% for soil samples? A control limit of $\leq$ CRDL( $\leq$ 2X CRDL for soil) was used for samples that were $\leq$ 5X the CRDL, including when only one of the duplicate sample values were $\leq$ 5X the CRDL.			~	
V Laboratore configs amples				
Was an LCS anayized for this SDG?	Ч			
Was an LCS analyzed per extraction batch?	1			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% (85-115% for Method 300.0) QC limits?	4	-		
VI. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?		-	-	
Nere the performance evaluation (PE) samples within the accentance limits?		Ī	H	

### Method: Inorganics (EPA Method Sectory)

WETC-EPA.IV version 1.0

LDC #: U768Fb SDG #: See caver

### VALIDATION FINDINGS CHECKLIST

Page: <u>Cof</u> Reviewer: <u>C</u> 2nd Reviewer: <u>W</u>

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

LDC #: 21768F6 SDG #: 560000	<u> </u>	tial and Cor	Validatin Fi <u>itinuing Ca</u> l	ndings Worksh <u>libration Calcu</u> l	ieet <u>ation Verifica</u>	tion	Page:of Reviewer:
Method: Inorganics, M	ethod <u>S</u>	Ravel					2nd Keviewer:
The correlation coefficient (	r) for the calibr	ation of $CN$	was recalc	ulated.Calibration c	late: 9/1/C	9	
An initial or continuing calit	oration verificat	ion percent reco	overy (%R) was	recalculated for ea	ch type of analysis	s using the followi	ng formula:
%R = <u>Found X 100</u> True		Where,	Found = conce True = conce	entration of each an entration of each an	alyte <u>measured</u> in alyte in the ICV or	the analysis of the CCV source	e ICV or CCV solution
				Q and a more a	Recalculated	Reported	Acceptable
Type of analysis	Analyte	Standard	Conc. (43/1)	Area	r or r <sup>2</sup>	r or r²	(X/N)
Initial calibration		s1	0	0.00824			
		s2	0.01	0.01763	0.999940	0.999940	
		s3	0.02	0.02561			_
		s4	0.05	0.05518			<u> </u>
	; ;	s5	0.1	0.10535			<del>723</del>
		s6	0.2	0.20435			
		s7	0.5	0.48191			
		s8	-	0.95541			
Calibration verification	CN	TCV	G,S	2,50202,0	101	I	
Calibration verification		CCV		D79P4.0	99	1	
Calibration verification	$\rightarrow$	CCV	)	118150	HOI		$\rightarrow$
Comments: Refer to Calibr. 10.0% of the recalculated re	ation Verificatic ssults.	n findings work	sheet for list o	f qualifications and	associated sample	es when reported	results do not agree within

:

LDC #: 22176875

VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

Page: 1 of Reviewer: C17 2nd Reviewer:

METHOD: Inorganics, Method Decover

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

concentration of each analyte <u>measured</u> in the analysis of the sample. For the matrix spike calculation, Found = SSR (spiked sample result) - SR (sample result). concentration of each analyte in the source. Found = True = Where, %R = Found x 100 True

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

 RPD = <u>[S-D]</u>
 × 100
 Where,
 S =
 Original sample concentration

 (S+D)/2
 D =
 Duplicate sample concentration

Acceptable (Y/N) %R / RPD Reported Receivelated **Udia / RPD** 5 ÷ True / D (units) 0.5 Found / S (units) L T (SSR-SR) Element С 2 ÷. Laboratory control sample Type of Analysis Matrix spike sample **Duplicate semple** Sample ID

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

TOTCLC.6

LDC # SDG

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

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\_reported with a positive detect were

METHOD: Inorganics, Method

SECON

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".  $\frac{V N N/A}{V N N/A}$  Have results been reported and calculated correctly?

Y N N/A Y N N/A

Are results within the calibrated range of the instruments? Are all detection limits below the CRQL?

Concentration =

**Recalculation:** 

Non t

#	Sample ID	Analyte	Reported Concentration ( )	Calculated Concentration ( )	Acceptable (Y/N)
					·
					·
				· · ·	

Note:

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

Project/Site Name:	Tronox LLC Facility, Henderson, Nevada	2009	Phase	В	Investigation,
Collection Date:	August 24 through Aug	gust 26	, 2009		

LDC Report Date: February 2, 2010

Matrix: Soil/Water

Parameters: Gasoline Range Organics

Validation Level: Stage 2B & 4

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R0904797

### Sample Identification

SA154-0.5B\*\* SA154-10B\*\* SA154-20B\*\* SA154-33B\*\* SA200-31B\*\* SA20009-31B SA200-10B\*\* SA200-20B\*\* SA200-31BMS SA200-31BMSD

\*\*Indicates sample underwent Stage 4 review.

### Introduction

This data review covers 10 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8015B for Gasoline Range Organics.

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 III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. Calibration

### a. Initial Calibration

Initial calibration of compounds was performed as required by the method.

The percent relative standard deviations (%RSD) of calibration factors for compounds were less than or equal to 20.0%.

### b. Calibration Verification

Calibration verification was performed at required frequencies. The percent differences (%D) of amounts in continuing standard mixtures were within the 20.0% QC limits.

### III. Blanks

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

Sample FB072909-SO (from SDG R0904226) was identified as a field blank. No gasoline range organic contaminants were found in this blank with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB072909-SO	7/29/09	Gasoline range organics	27 ug/L	All samples in SDG R0904797

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

### **IV. Accuracy and Precision Data**

### a. Surrogate Recovery

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

### b. 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.

### c. Laboratory Control Samples

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

### V. Target Compound Identification

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

### VI. Project Quantitation Limit

All project quantitation limits were within validation criteria for samples on which a Stage 4 review was performed.

Sample	Finding	Flag	A or P
All samples in SDG R0904797	All compounds reported below the PQL.	J (all detects)	A

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

### **VII. System Performance**

Raw data were not reviewed for this SDG.

### VIII. Overall Assessment of Data

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

### IX. Field Duplicates

Samples SA200-31B and SA200009-31B were identified as field duplicates. No gasoline range organics were detected in any of the samples.

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Gasoline Range Organics - Data Qualification Summary - SDG R0904797

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R0904797	SA154-0.5B** SA154-10B** SA154-20B** SA154-33B** SA200-31B** SA20009-31B SA200-10B** SA200-20B**	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Gasoline Range Organics - Laboratory Blank Data Qualification Summary - SDG R0904797

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Gasoline Range Organics - Field Blank Data Qualification Summary - SDG R0904797

No Sample Data Qualified in this SDG

**Tronox Northgate Henderson** VALIDATION COMPLETENESS WORKSHEET

Stage 2B /4

LDC #: 21768F7

SDG #: R0904797

Laboratory: Columbia Analytical Services

23/01 Date: 10 Page: 1 of Reviewer: JV6 2nd Reviewer:

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METHOD: GC Gasoline Range Organics (EPA SW 846 Method 8015B)

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

	Validation Area		Comments
	Technical holding times	A	Sampling dates: 8/24 - 26/09
lla.	Initial calibration	A	% KSD € 20 3
IIb.	Calibration verification/	A	Cau = 20 Z
111.	Blanks	A	
IVa.	Surrogate recovery	À	
IVb.	Matrix spike/Matrix spike duplicates	A	
IVc.	Laboratory control samples	A	465
V.	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	N	
VII).	Overall assessment of data	A	
IX.	Field duplicates	ND	D = 5,6
Х.	Field blanks	SW	FB = FB072909-50 (R0904226)

Note:

A = Acceptable N = Not provided/applicable ND = No compounds detected D = Duplicate

SW = See worksheet

R = Rinsate FB = Field blank

TB = Trip blank EB = Equipment blank

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2	Vali ¢	dat F	ed Samples: Level IV		Soil		
	1	١	SA154-0.5B	÷¥		11	167733MB
	2	1	SA154-10B	- ¥		12 Y	169325
	~		×	×			

1 1	SA154-0.5B	11 16/132MB	21	31
21	SA154-10B **	127 169325	22	32
31	SA154-20B * *	13	23	33
41	<b>★ ≭</b> SA154-33B	14	24	34
5 2	SA200-31B * * D	15	25	35
67	SA200009-31B D	16	26	36
<b>ر</b> 7	SA200-10B * ¥	17	27	37
8 2	SA200-20B *¥	18	28	38
9 7	SA200-31BMS	19	29	39
10 <b>~</b>	SA200-31BMSD	20	30	40

Notes:

Method: <u>C</u> GC HPLC				
Validation Area	Yes	No	NA	Findings/Comments
L Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.				
II. Initial calibration			<u> </u>	
Did the laboratory perform a 5 point calibration prior to sample analysis?	$\leq$			
Were all percent relative standard deviations (%RSD) $\leq$ 20%?	$\leq$			
Was a curve fit used for evaluation?				<u></u>
Did the initial calibration meet the curve fit acceptance criteria of $\geq$ 0.990?				
Were the RT windows properly established?				
IV. Continuing calibration		reality I	l I	and the second
Was a continuing calibration analyzed daily?				
Were all percent differences (%D) < 20%.0 or percent recoveries 80-120%?	Ľ			
Were all the retention times within the acceptance windows?				
V. Blanks		<u> </u>	1	
Was a method blank associated with every sample in this SDG?				
Was a method blank analyzed for each matrix and concentration?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
VI. Surrogate spikes			-	
Were all surrogate %R within the QC limits?				
If the percent recovery (%R) for one or more surrogates was out of QC limits, was a reanalysis performed to confirm samples with %R outside of criteria?			/	F
VII. Matrix spike/Matrix spike duplicates			1	
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			
Was a MS/MSD analyzed every 20 samples of each matrix?	$\overline{}$			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII. Laboratory control samples	г <u>т</u>	I T	1	
Was an LCS analyzed for this SDG?		<u> </u>		
Was an LCS analyzed per extraction batch?	$\vdash$	<u> </u>	<b> </b>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX. Regional Quality Assurance and Quality Control	1	r		
Were performance evaluation (PE) samples performed?	<u> </u>		1	¥
Were the performance evaluation (PE) samples within the acceptance limits?	<u> </u>	<u> </u>	<u> </u>	



Validation Area	Yes	No	NA	Findings/Comments
X. Target compound identification		teritori i		
Were the retention times of reported detects within the RT windows?				
XI. Compound quantitation/CRQLs	1.5			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?		1		
XII. System performance		4.		
System performance was found to be acceptable.				
XIII. Overall assessment of data			1	
Overall assessment of data was found to be acceptable.				
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.	/	-		
Target compounds were detected in the field duplicates.		/		
XV. Field blanks				
Field blanks were identified in this SDG.				
Target compounds were detected in the field blanks.				

ø

RETHOD:     GC     HPLC       Y     N N/A     Were field blanks identified in this SDG       Y     N N/A     Were target compounds detected in the stand blank type: (circle one) (Eleid Black / Trip Blank / Atmosing date:       Ield blank type:     L     Associated sample units:     US       Ield blank type:     Circle one) (Eleid Black / Trip Blank / Atmosing date:     US       Ield blank type:     Circle one) (Eleid Black / Trip Blank / Atmosing date:     US       Topound     Blank iD     Blank iD     Blank iD       Compound     Blank iD     Blank iD     Eleid blank / Atmosing date:       Carcol     A     A     Associated sample units:       Carcol     A     Associated sample units:     US       Blank unite:     Associated sample units:     Associated sample units:       Field blank type:     (Circle one) Field Blank / Trip Blank / Atmosing date:     Associated sample units:	Held blanks?	lent Blank Blank / Other:	Sample Ident	ssociated Samples:	Ϋ́Υ	2nd Reviewe	
The state / Equipment Rinsate	pheric Blank / Ambi	lent Blank / Other.	A. Sample Ident	ssociated Samples.	(II)	(av)	
Compound     Blank ID     Blank ID       C3 R0     37     99-SD       C3 R0     37     Associated sample units:       CROL     Associated sample units:       Sampling date:     Associated sample units:       Field blank type: (circle one) Field Blank / Trip Blank / Amo Rinsate / Equipment Rinsate / Equipment Compound			S ample Ident	tification			
G Rの マフ CRQL CRQL CRQL CRQL Associated sample units: Sampling date: Field blank type: (circle one) Field Blank / Trip Blank Atme Rinsate / Equipment Rinsate / Equip Compound Blank iD Blank iD							
CRQL CRQL Sampling date: ield blank type: (circle one) Field Blank / Trip Blank/ Atmo Rinsate / Equipment Rinsate / Equip Compound Blank ID Blank iD							
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CROL Slank units: Associated sample units: Sampling date: Associated sample units: ield blank type: (circle one) Field Blank / Trip Blank Atmo Rinsate / Equipment Rinsate / Equip Compound Blank ID Blank ID		· · ·				the second se	
cRoL Slank unite: Associated sample units: sampling date: Associated sample units: ield blank type: (circle one) Field Blank / Trip Blank/ Atmo ield blank type: (circle one) Field Blank / Trip Blank/ Atmo Compound Blank ID Blank ID Blank ID					_		
itank unite: Associated sample unite: ampling date:		-					
ield blank type: (circle one) Field Blank / Trip Blank/ Atmo Rinsate / Equipment Rinsate / Equip Compound Blank ID Blank ID	1						
Compound Blank ID Blank ID	pheric Blank/ Ambie	ent Blank	4	ssociated Samples:			
Compound Blank ID Blank ID	iani diank / Source	BIBNK / Other;					
			Semple Iden	utification			
CROL							

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LDC #: 21768 F7 SDG #: See Cover

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

lof 1/10 Page: 2nd Reviewer: Reviewer:

HPLC METHOD: GC

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

CF = A/C average CF = sum of the CF/number of standards %RSD = 100 • (S/X)

A = Area of compound,

C = Concentration of compound, S = Standard deviation of the CF X = Mean of the CFs

standard ID ICAL ICAL	Calibration Date	Compound Gaerline (le-le)	СF		A REAL PROPERTY AND A REAL OF A REAL AND A			
ICAL ICAL	7/04 /68	Gaucine (Ca-Ca)	(In std)	CF (Jvn std)	Average CF (initial)	Average CF (initial)	%RSD	%RSD
ICAL			0.9254	0.1234	0. 8576	6 8976	€0	8
Ical.	\ ``							
1041			0. 2107	0. 2107	0. 2058	0. 2058	5	Ь
	60/60/6							
						-		
1	I							
T	1							
<b>r</b>	I							
r	L							

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results.

SDG#: Ser Cover LDC # 2176 47

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

) of Page: 2nd Reviewer: Reviewer.

HPLC METHOD: GC The percent difference (%D) of the initial calibration average Calibration Factors (CF) and the continuing calibration CF were recalculated for the compounds identified below using the following calculation:

% Difference = 100 \* (ave. CF - CF)/ave. CF CF = A/C

CF = continuing calibration CF A = Area of compound C = Concentration of compound Where: ave. CF = initial calibration average CF

				Reported	Recalculated	Renoted	Recalculated
ndard ID	Calibration Date	Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc. CCV	Q%	Q%
7	8/27/69	Graphine (Ca-Go)	0. 8976	0. 6068	0.9068	B	
	- a hore ha		0.2050	0.2103	60120	$\boldsymbol{\lambda}$	Y
	62/02/						
0					•		
						-	
						•	

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 # 21768F7 Ser Cover SDG #:\_

## VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page:

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

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Sample ID:	-					
Surrogate	ColumhDetector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Perc
				Reported	Recalculated	
3- Fluero chlorabenzen	FID	at	26.913	90	eb .	(ر)

ent

Sample ID:

j		_	- 1	
Percent Difference				
Percent Recovery	Recalculated			-
Percent Recovery.	Reported			
Surrogate Found				
Surrogate Spiked				
Column/Detector				
Surrogate				

Sample ID:

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
P				Reported	Recalculated	

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LDC #: 2/20	SDG #: 0

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

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

METHOD: C GC HPLC The percent recoveries (%R) and relative percent differences (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

RPD =(({SCMS - SSCMSD} \* 2) / (SSCMS + SSCMSD))\*100

0 8 MS/MSD samples:

SSC = Spiked sample concentration SC = Sample concentration SA = Spike added Where

= Matrix spike ŚŴ

	D = Matrix spike duplicate	
ś	8	
-	-	

		Spik		Sample	Spike	Sample	Matrix	spike	Matrix Spike	e Duplicate	WS/N	SD
Compound		Pdde Adde	يد کھ ا	Cone.	Conce ( אק	htration /cc )	Percent	Recovery	Percent R	tecovery	RP	0
		WS	MsD	0	MS	DSM	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)		00219	Slam	۵	48600	43800	56	55	% (	8	<i>ح</i> ا	Q)
Diesel (8015)												
Benzene (8021B)												
Methane (RSK-1	175)											
2,4-D (8151)												
Dinoseb (8151)												
Naphthalene (8310)												
Anthracene (8310)												
HMX (8330)												
2,4,6-Trinitrotoluene (	8330)											
Comments: Refer to Ma	atrix Spi	ke/Matri>	< Spike D	uplicates fine	dings worksh	eet for list of (	qualifications.	and associate	d samples wh	nen reported	results do no	t agree withi
10.0% of the recalculate	ad result	y.										

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LDC #:	SDG #:

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

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

> GC HPLC **METHOD:**

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

% Recovery = 100\* (SSC-SC)/SA RPD = I LCS - LCSD I \* 2/(LCS + LCSD)

LCS/LCSD samples:\_

Where: SSC = Spiked sample concentration SA = Spike added LCS = Laboratory control sample percent recovery

SC = Concentration

LCSD = Laboratory control sample duplicate percent recovery

	5	pike	Spiked	Sample	
Compound	A (Lice	ided Asy	Conce ( 12	ntration らんこ)	
	rcs		LCS		Repo
Gasoline (8015)	کس ا	N.A	478	MA	16
Diesel (8015)					
Benzene (8021B)					
Methane (RSK-175)					

	S .	pike	Spiked	l Sample	L	CS	FC	SD	LCS/	-csD
Compound	A( 1/5	idea (	Conce	entration らんこ)	Percent	Recovery	Percent	Recovery	R	D O
	rcs		SCI		Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)	ans	ž	478	₩ <b>A</b>	36	96				
Diesel (8015)										
Benzene (8021B)										
Methane (RSK-175)										
2,4-D (8151)										
Dinoseb (8151)										
Naphthalene (8310)										
Anthracene (8310)										
HMX (8330)										
2,4,6-Trinitrotoluene (8330)										
	-									-
Comments: Refer to Labor	atory Control	Sample/Lab	oratory Contro	l Sample Dupl	icate findings w	orksheet for lis	st of qualifications	s and associate	d samples w	hen reporte

V:\Validation Worksheets\GC\LCSDCLC\_GC.wpd

results do not agree within 10.0% of the recalculated results.

LDC #: <u></u> SDG #: <u></u>	Coner 7	VALIDATI Sampl	ION FINDINGS WORKS e Calculation Verificati	HEET Ion	•	Page: <u>of 1</u> Reviewer: <u>MC</u>
METHOD:	GCHPLC	• •		•		Zind Noviewei.
X N N X	Were all reported re Were all recalculated	sults recalculated and verified fo d results for detected target com	rt all level IV samples? pounds within 10% of the re	ported results?		
Concentration= ( A= Area or height	(RF)(Vs or Ws)(%S/100) t of the compound to be mea	Example: ) Sample ID	Com	pound Name	A	
<ul> <li>Pilution Factor</li> <li>Dilution Factor</li> <li>RF= Average responsion</li> <li>In the initial colume</li> <li>VS= Initial volume</li> <li>WS= Percent Solid</li> </ul>	or extract or alibration of the sample of the sample	Concentration	8			
			·			
#	Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations (	· · ·	Qualifications
0						
Comments:						
					•	

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