

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson  
Data Validation Reports  
LDC #21495**

Chlorinated Pesticides

LDC

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

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

**Collection Date:** May 27 through June 4, 2009

**LDC Report Date:** September 28, 2009

**Matrix:** Water

**Parameters:** Chlorinated Pesticides

**Validation Level:** Stage 4

**Laboratory:** Columbia Analytical Services, Inc.

**Sample Delivery Group (SDG):** R0903006

**Sample Identification**

MC-3B  
EB052709  
M-127B  
FB060409

## **Introduction**

This data review covers 4 water 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.

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.
- UU 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.

## IV. Continuing Calibration

Continuing calibration was performed at required frequencies with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P
MC-3B EB052709	Hexachlorobenzene	Continuing calibration was not performed for this compound.	Continuing calibration must be performed for each compound.	J (all detects) UJ (all non-detects)	P

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 with the following exceptions:

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
6/18/09	FA581	STX-CLP2	Heptachlor Endrin aldehyde	25.2 21.8	FB060409 89250MB	J+ (all detects) J+ (all detects)	A

Retention times (RT) of all compounds in the calibration standards were within QC limits.

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

## V. Blanks

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

Sample EB052709 was identified as an equipment blank. No chlorinated pesticide contaminants were found in this blank.

Sample FB060409 was identified as a field blank. No chlorinated pesticide contaminants were found in this blank.

## VI. Surrogate Spikes

Surrogates were added to all samples and blanks as required by the method. Surrogate recoveries (%R) were not within QC limits for MC-3B and M-127B. Since these samples were diluted out, no data were qualified.

## 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
88638LCS/D (MC-3B EB052709 M-127B 88638MB)	Endrin aldehyde	22 (50-130)	27 (50-130)	-	J- (all detects) UJ (all non-detects)	P

## 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.

## **XII. Project Quantitation Limit**

All project quantitation limits were within validation criteria.

All compounds reported below the PQL were qualified as follows:

<b>Sample</b>	<b>Finding</b>	<b>Flag</b>	<b>A or P</b>
All samples in SDG R0903006	All compounds reported below the PQL.	J (all detects)	A

## **XIII. Overall Assessment of Data**

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

## **XIV. Field Duplicates**

No field duplicates were identified in this SDG.

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Data Qualification Summary - SDG R0903006**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R0903006	MC-3B EB052709	Hexachlorobenzene	J (all detects) UJ (all non-detects)	P	Continuing calibration (c)
R0903006	FB060409	Heptachlor Endrin aldehyde	J+ (all detects) J+ (all detects)	A	Continuing calibration (%D) (c)
R0903006	MC-3B EB052709 M-127B	Endrin aldehyde	J- (all detects) UJ (all non-detects)	P	Laboratory control samples (%R) (l)
R0903006	MC-3B EB052709 M-127B FB060409	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  
R0903006**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG R0903006**

No Sample Data Qualified in this SDG



Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 21495B3a

SDG #: R0903006

Laboratory: Columbia Analytical Services

Stage 4

Date: 9/16/09

Page: 1 of 1

Reviewer: JG

2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 5/27-28/09, 6/04/09
II.	GC/ECD Instrument Performance Check	A	
III.	Initial calibration	A	RSD
IV.	Continuing calibration/ICV	SW	CCV/ICV $\leq 20\%$
V.	Blanks	A	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	N	Client spec
VIII.	Laboratory control samples	SW	LCS B
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	
Xb.	GPC Calibration	N	
XI.	Target compound identification	A	
XII.	Compound quantitation and reported CRQLs	A	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	ND	EB = 2      FB = 4

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	MC-3B	11	88638 MB	21	31
2	EB052709	12	89250 ↓	22	32
3	M-127B	13		23	33
4	✓ FB060409	14		24	34
5		15		25	35
6		16		26	36
7		17		27	37
8		18		28	38
9		19		29	39
10		20		30	40

LDC #: 2495 B34  
 SDG #: Sue Conrad

**VALIDATION FINDINGS CHECKLIST**

Page: 1 of 2  
 Reviewer: ST  
 2nd Reviewer: [Signature]

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

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>II. GC/ECD instrument performance check</b>				
Was the instrument performance found to be acceptable?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>III. Initial calibration</b>				
Did the laboratory perform a 5 point calibration prior to sample analysis?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) $\leq$ 20%?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Did the initial calibration meet the curve fit acceptance criteria?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the RT windows properly established?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the required standard concentrations analyzed in the initial calibration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Continuing calibration</b>				
What type of continuing calibration calculation was performed? <input checked="" type="checkbox"/> %D or <input checked="" type="checkbox"/> %R	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were Evaluation mix standards analyzed prior to the initial calibration and sample analysis?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were endrin and 4,4'-DDT breakdowns $\leq$ 15%.0 for individual breakdown in the Evaluation mix standards?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a continuing calibration analyzed daily?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) $\leq$ 15%.0 or percent recoveries 85-115%?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all the retention times within the acceptance windows?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a method blank analyzed for each matrix and concentration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were extract cleanup blanks analyzed with every batch requiring clean-up?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was there contamination in the method blanks or clean-up blanks? If yes, please see the Blanks validation completeness worksheet.	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>VI. Surrogate spikes</b>				
Were all surrogate %R within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>VII. Matrix spike/Matrix spike duplicates</b>				

LDC #: 21495 B39  
 SDG #: San Carol

**VALIDATION FINDINGS CHECKLIST**

Page: 2 of 2  
 Reviewer: JV6  
 2nd Reviewer: J

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?			/	
<b>VIII. Laboratory control samples</b>				
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?			/	
<b>IX. Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?		/		
Were the performance evaluation (PE) samples within the acceptance limits?			/	
<b>X. Target compound identification</b>				
Were the retention times of reported detects within the RT windows?	/			
<b>XI. Compound quantitation/CRQLs</b>				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions, dry weight factors, and clean-up activities applicable to level IV validation?	/			
<b>XII. System performance</b>				
System performance was found to be acceptable.	/			
<b>XIII. Overall assessment of data</b>				
Overall assessment of data was found to be acceptable.	/			
<b>XIV. Field duplicates</b>				
Field duplicate pairs were identified in this SDG.		/		
Target compounds were detected in the field duplicates.			/	
<b>XV. Field blanks</b>				
Field blanks were identified in this SDG.	/			
Target compounds were detected in the field blanks.		/		

# VALIDATION FINDINGS WORKSHEET

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

A. alpha-BHC	I. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	GG.
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH.
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	II.
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	JJ.
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. DB 608	KK.
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. DB 1701	LL.
G. Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	EE. Hexachlorobenzene	MM.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	FF.	NN.

Notes:







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

**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:

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

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

#	Standard ID	Calibration Date	Compound	Reported		Recalculated		Reported		Recalculated					
				CF (10 <sup>6</sup> /100 std)	CF (10 <sup>6</sup> /100 std)	CF (Initial)	CF (Initial)	%RSD	%RSD						
1	1 CAL	6/02/09	H (STX-CUP1) 10	2.098 e7	2.038 e7	2.110 e7	2.110 e7	5.64	5.62						
				1.004	1.004	0.993	0.992	2.12	2.12						
				7.292	7.292	7.092	7.092	3.35	3.35						
				2.753	2.793	2.695	2.695	4.78	4.99						
2	1 CAL	6/17/09	H (STX-CUP1)	2.075 e7	2.075 e7	2.167 e7	2.167 e7	6.69	6.88						
				1.011	1.011	0.999	0.999	2.58	2.58						
				7.081	7.081	6.982	6.982	2.40	2.46						
				2.740	2.740	2.657	2.657	5.69	5.69						
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.



LDC #: 21445 B39  
 SDG #: SEA 6024

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

Page: 1 of 1  
 Reviewer: DZ  
 2nd Reviewer: S

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

The continuing calibration percent difference (%D) values were recalculated for \_\_\_\_\_ using the following calculation:

Percent difference (%D) =  $100 \cdot (N - C) / N$  Where: N = Initial Calibration Factor or Nominal Amount (ng)  
 C = Calibration Factor from Continuing Calibration Standard or Calculated Amount (ng)

Standard ID	Calibration Date/Time	Compound	Average CF/ CCV Conc	Reported		Recalculated		Reported		Recalculated	
				CF/Conc CCV	%D	CF/Conc CCV	%D	CF/Conc CCV	%D		
FA453	6/08/09	H STX-CP1	21.098 e6	22.692 e6	4.7	22.082 e6	4.7				
		P	9.926	9.834	0.9	9.834	0.9				
		H	70.916	70.756	0.2	70.756	0.2				
		P	26.949	26.290	2.4	26.290	2.4				
FA497	6/08/09	H STX-CP1	21.098 e6	22.213 e6	5.3	22.213 e6	5.3				
		P	9.926	9.799 e6	1.3	9.799	1.3				
		H	70.916	71.455	0.8	71.455	0.8				
		P	26.949	26.528	1.6	26.528	1.6				
	6/18/09	H STX-CP1	21.666 e6	23.175 e6	7.0	23.175 e6	7.0				
		P	9.994	10.796	8.0	10.796	8.0				
		H	69.818	77.900	11.6	77.900	11.6				
		P	26.526	28.835	8.8	28.835	8.8				

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 #: 21495 B3a  
 SDG #: Se Con

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

Page: 1 of 1  
 Reviewer: JV  
 2nd reviewer: J

**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: # 2

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene	STX-GLP1	100	47.89	48	48	0
Tetrachloro-m-xylene	↓	↓				↓
Decachlorobiphenyl			66.47	66	66	
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: \_\_\_\_\_

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

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

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

% Recovery =  $100 \cdot (SSC - SC) / SA$       Where: SSC = Spiked sample concentration      SC = Concentration  
 SA = Spike added

RPD =  $|LCS - LCSD| \cdot 2 / (LCS + LCSD)$       LCS = Laboratory control sample percent recovery      LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples: 88638 LCS/b

Compound	Spike Added (ug/L)		Spiked Sample Concentration (ug/L)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
gamma-BHC	0.200	0.200	0.172	0.187	86	86	93	93	9	8
4,4'-DDT	↓	↓	0.181	0.191	91	91	96	96	5	5
Aroclor 1260										

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

LDC #: 21495 B3c  
 SDG #: Sx Corv

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

Page: 1 of 1  
 Reviewer: [Signature]  
 2nd reviewer: [Signature]

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

Y N N/A  
 X N N/A

Were all reported results recalculated and verified for all level IV samples?

Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

Example:

87X-CLP1

Sample I.D. # 1 B :

$$\text{Conc.} = \frac{(417.526) (10\text{ml}) (100)}{(1.19227) (1050\text{ml})}$$

$$= 33.3 \text{ ug/L}$$

#	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:** July 1 through July 4, 2009

**LDC Report Date:** September 25, 2009

**Matrix:** Soil

**Parameters:** Chlorinated Pesticides

**Validation Level:** Stage 2B

**Laboratory:** Columbia Analytical Services, Inc.

**Sample Delivery Group (SDG):** R0903051

**Sample Identification**

RSA12-0.5B  
RSA12-0.5BDL  
RSAI3-0.5B  
RSAI3-0.5BDL  
RSAJ5-0.5B  
RSAJ5-0.5BDL  
RSAK5-0.5B  
RSAK5-0.5BDL  
RSAL3-0.5B  
RSAL3-0.5BDL  
RSAM3-0.5B  
RSAM2-0.5B  
RSAJ2-0.5B  
RSAJ2-0.5BDL  
RSAJ3-0.5B  
RSAI3-0.5BMS  
RSAI3-0.5BMSD  
RSAJ2-0.5BMS  
RSAJ2-0.5BMSD

## Introduction

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

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.
- UU 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.

## **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%.

## **V. Blanks**

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

Sample FB072109-SO (from SDG R0904016) was identified as a field blank. No chlorinated pesticide contaminants were found in this blank.

## **VI. Surrogate Spikes**

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



Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
88785MB	Not specified	Tetrachloro-m-xylene	8 (40-140)	All TCL compounds	J- (all detects) R (all non-detects)	P

## 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 and MSD percent recoveries (%R) were not within QC limits for all compounds and the MS/MSD relative percent differences (RPD) were not within QC limits for some compounds, the 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 percent recoveries (%R) and LCS/LCSD relative percent differences (RPD) were not within QC limits for some compounds, the LCSD percent recoveries (%R) were 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

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
RSA12-0.5B RSAI3-0.5B RSAJ5-0.5B RSAK5-0.5B RSAJ2-0.5B	Hexachlorobenzene	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	A
RSAL3-0.5B	4,4'-DDE	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 R0903051	All compounds reported below the PQL.	J (all detects)	A

Raw data were not reviewed for this SDG.

### XIII. Overall Assessment of Data

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
RSA12-0.5B RSAI3-0.5B RSAJ5-0.5B RSAK5-0.5B RSAJ2-0.5B	Hexachlorobenzene	X	A
RSA12-0.5BDL RSAI3-0.5BDL RSAJ5-0.5BDL RSAK5-0.5BDL RSAJ2-0.5BDL	All TCL compounds except Hexachlorobenzene	X	A
RSAL3-0.5B	4,4'-DDE	X	A
RSAL3-0.5BDL	All TCL compounds except 4,4'-DDE	X	A

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

#### **XIV. Field Duplicates**

No field duplicates were identified in this SDG.

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Data Qualification Summary - SDG R0903051**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R0903051	RSA12-0.5B RSAI3-0.5B RSAJ5-0.5B RSAK5-0.5B RSAJ2-0.5B	Hexachlorobenzene	J (all detects)	A	Project Quantitation Limit (e)
R0903051	RSAL3-0.5B	4,4'-DDE	J (all detects)	A	Project Quantitation Limit (e)
R0903051	RSA12-0.5B RSA12-0.5BDL RSAI3-0.5B RSAI3-0.5BDL RSAJ5-0.5B RSAJ5-0.5BDL RSAK5-0.5B RSAK5-0.5BDL RSAL3-0.5B RSAL3-0.5BDL RSAM3-0.5B RSAM2-0.5B RSAJ2-0.5B RSAJ2-0.5BDL RSAJ3-0.5B	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
R0903051	RSA12-0.5B RSAI3-0.5B RSAJ5-0.5B RSAK5-0.5B RSAJ2-0.5B	Hexachlorobenzene	X	A	Overall assessment of data (o)
R0903051	RSA12-0.5BDL RSAI3-0.5BDL RSAJ5-0.5BDL RSAK5-0.5BDL RSAJ2-0.5BDL	All TCL compounds except Hexachlorobenzene	X	A	Overall assessment of data (o)
R0903051	RSAL3-0.5B	4,4'-DDE	X	A	Overall assessment of data (o)
R0903051	RSAL3-0.5BDL	All TCL compounds except 4,4'-DDE	X	A	Overall assessment of data (o)

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG R0903051**

No Sample Data Qualified in this SDG

**Tronox Northgate Henderson**

LDC #: 21495C3a  
 SDG #: R0903051  
 Laboratory: Columbia Analytical Services

**VALIDATION COMPLETENESS WORKSHEET**  
 Stage 2B

Date: 9/16/09  
 Page: 1 of 1  
 Reviewer: JVG  
 2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: <u>6/01-04/09</u>
II.	GC/ECD Instrument Performance Check	A	
III.	Initial calibration	A	
IV.	Continuing calibration/ICV	<del>SWA</del>	<u>CV / ICV ≤ 20 %</u>
V.	Blanks	A	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	SW	<u>les / D</u>
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	SW	
XIII.	Overall assessment of data	SW	
XIV.	Field duplicates	N	
XV.	Field blanks	ND	<u>FB = 16</u> <u>FB = FB072109-S0 from R090406</u>

Note: A = Acceptable      ND = No compounds detected      D = Duplicate  
 N = Not provided/applicable      R = Rinsate      TB = Trip blank  
 SW = See worksheet      FB = Field blank      EB = Equipment blank

Validated Samples: Soil + Water

1	✓	RSA12-0.5B	S	11	✓	RSAM3-0.5B	S	21	<del>158256 MB</del>	31
2	✓	RSA12-0.5BDL		12	✓	RSAM2-0.5B		22	<u>88785 MB</u>	32
3	✓	RSAI3-0.5B		13	✓	RSAJ2-0.5B		23	<u>88986 MB</u>	33
4	✓	RSAI3-0.5BDL		14	✓	RSAJ2-0.5BDL		24		34
5	✓	RSAJ5-0.5B		15	✓	RSAJ3-0.5B		25		35
6	✓	RSAJ5-0.5BDL		<del>16</del>	<del>✓</del>	<del>FB080409</del>	<del>W</del>	26		36
7	✓	RSAK5-0.5B		17	✓	RSAI3-0.5BMS	S	27		37
8	✓	RSAK5-0.5BDL		18	✓	RSAI3-0.5BMSD		28		38
9	✓	RSAL3-0.5B		19	✓	RSAJ2-0.5BMS		29		39
10	✓	RSAL3-0.5BDL	✓	20	✓	RSAJ2-0.5BMSD	✓	30		40

(# 16 reported on R0903006)

# VALIDATION FINDINGS WORKSHEET

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

A. alpha-BHC	I. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	GG.
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH.
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	II.
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	JJ.
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. DB 608	KK.
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. DB 1701	LL.
G. Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	EE. <i>Hexachlorobenzene</i>	MM.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	FF.	NN.

Notes:

LDC #: 21 495 (31)  
 SDG #: See Cover

VALIDATION FINDINGS WORKSHEET  
Surrogate Spikes

Page: 1 of 2  
 Reviewer: DK  
 2nd Reviewer: R

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".  
 N/A Were surrogates spiked into all samples, standards and blanks?  
 Y/N/N/A Did all surrogate percent recoveries (%R) meet the QC limits?

#	Date	Sample ID	Column	Surrogate Compound	%R (Limits)	Qualifications
1		(100x)	Not spec	B	21065 (40-140)	No qual
				A	677 ( )	
2		(1000x)		A, B	0 ( )	
3		(10x)		B	1143 ( )	
4		(1000x)		A, B	0 ( )	
5		(10x)		A	383 ( )	
				B	1117 ( )	
6		(200x)		A	0 ( )	
				B	1366 ( )	
7		(10x)		B	1169 ( )	
8		(500x)		A, B	0 ( )	
9		(100x)				
10		(500x)				

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A	Tetrachloro-m-xylene			
B	Decachlorobiphenyl			

LDC #: 21415 C31  
SDG #: Sy Con

VALIDATION FINDINGS WORKSHEET  
Surrogate Spikes

Page: 2 of 2  
Reviewer: JK  
2nd Reviewer: S

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".  
 N N/A Were surrogates spiked into all samples, standards and blanks?  
 Y (N) N/A Did all surrogate percent recoveries (%R) meet the QC limits?

#	Date	Sample ID	Column	Surrogate Compound	%R (Limits)	Qualifications
		13 (10x)	Not Spec	B	1060 (40-140)	No qual
		14 (100x)		A	0	
				B	1060	
		15 (20x)		B	198	
		88785 MB	✓	A	8	J-R/P (S)

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A	Tetrachloro-m-xylene			
B	Decachlorobiphenyl			











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

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

**Collection Date:** June 9 through June 16, 2009

**LDC Report Date:** September 22, 2009

**Matrix:** Water

**Parameters:** Chlorinated Pesticides

**Validation Level:** Stage 2B

**Laboratory:** Columbia Analytical Services, Inc.

**Sample Delivery Group (SDG):** R0903243

**Sample Identification**

H-28AB  
AW-BW-02B  
M-142B  
M-130B  
M-29B  
M-29BRE

## Introduction

This data review covers 6 water 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.

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.
- UU 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.

## 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 with the following exceptions:

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
6/23/09	FA672	STX-CLP1	Hexachlorobenzene	31.5	M-130B M-29B	J+ (all detects)	A

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

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
6/18/09	FA581	STX-CLP2	Heptachlor Endrin aldehyde	25.2 21.8	H-28AB AW-BW-02B M-142B M-130B M-29B 89250MB 89410MB 89788MB	J+ (all detects) J+ (all detects)	A

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



## V. Blanks

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

Sample FB060409 (from SDG R0903006) was identified as a field blank. No chlorinated pesticide contaminants were found in this blank.

## VI. Surrogate Spikes

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

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
M-29B	Not specified	Decachlorobiphenyl	28 (40-140)	All TCL compounds	J- (all detects) UJ (all non-detects)	A

## VII. Matrix Spike/Matrix Spike Duplicates

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

## VIII. Laboratory Control Samples (LCS)

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

## IX. Regional Quality Assurance and Quality Control

Not applicable.

## X. 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

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 R0903243	All compounds reported below the PQL.	J (all detects)	A

Raw data were not reviewed for this SDG.

## XIII. Overall Assessment of Data

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-29B	All TCL compounds	X	A

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

## XIV. Field Duplicates

No field duplicates were identified in this SDG.

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Data Qualification Summary - SDG R0903243**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R0903243	M-130B M-29B	Hexachlorobenzene	J+ (all detects)	A	Continuing calibration (%D) (c)
R0903243	H-28AB AW-BW-02B M-142B M-130B M-29B	Heptachlor Endrin aldehyde	J+ (all detects) J+ (all detects)	A	Continuing calibration (ICV %D) (c)
R0903243	M-29B	All TCL compounds	J- (all detects) UJ (all non-detects)	A	Surrogate spikes (%R) (s)
R0903243	H-28AB AW-BW-02B M-142B M-130B M-29B M-29BRE	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
R0903243	M-29B	All TCL compounds	X	A	Overall assessment of data (o)

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG R0903243**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG R0903243**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 21495D3a

SDG #: R0903243

Laboratory: Columbia Analytical Services

Date: 9/17/09

Page: 1 of 1

Reviewer: JIC

2nd Reviewer: A

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/09/09 - 6/16/09
II.	GC/ECD Instrument Performance Check	A	
III.	Initial calibration	A	
IV.	Continuing calibration/ICV	SW	CCV/ICV ≤ 20%
V.	Blanks	A	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	N	Client spec
VIII.	Laboratory control samples	A	LCS/D
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	SW	
XIV.	Field duplicates	N	
XV.	Field blanks	ND	FB = FB060409 from R0903006

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	H-28AB	11	89250 MB	21	31
2	AW-BW-02B	12	89410 MB	22	32
3	M-142B	13	89788 MB	23	33
4	M-130B	14	89964 MB	24	34
5	M-29B	15		25	35
6	M-29BRE	16		26	36
7		17		27	37
8		18		28	38
9		19		29	39
10		20		30	40

LDC #: 21495 D39  
 SDG #: See Copy

VALIDATION FINDINGS WORKSHEET  
 Continuing Calibration

Page: 1 of 1  
 Reviewer: NG  
 2nd Reviewer: R

METHOD: GC Pesticides/PCBs (EPA SW 846 Method 8081/8092)

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

- N N/A What type or calibration verification calculation was performed? 3%D or    RPD
- N N/A Were Evaluation mix standards run before initial calibration and before samples?
- N N/A Were Endrin & 4,4'-DDT breakdowns acceptable in the Evaluation Mix standard ( $\leq 15.0\%$  for individual breakdowns)?
- Y (N) N/A Was at least one standard run daily to verify the working curve? 20
- Did the continuing calibration standards meet the percent difference (%D) / relative percent difference (RPD) criteria of  $\leq 15.0\%$ ?

Level I/II Only  
 Y N (N/A)

Were the retention times for all calibrated compounds within their respective acceptance windows?

#	Date	Standard ID	Column	Compound	%D (Limit $\leq 15.0\%$ )	RT (Limits)	Associated Samples	Qualifications
	6/18/09	FA 581 (CON)	5TX-CUP2	E (+) R (+)	25.2 21.8	( ) ( )	1-5 + A1 B1c5 except 89964 MB	J+dets/A (C)
	6/23/09	FA 672 (CON)	5TX-CUP1	EE (+)	31.5	( )	4 5	↓ ↓

A. alpha-BHC	E. Heptachlor	I. Dieldrin	M. 4,4'-DDD	Q. Endrin ketone	U. Toxaphene	Y. Aroclor-1242	CC. DB 608	GG.
B. beta-BHC	F. Aldrin	J. 4,4'-DDE	N. Endosulfan sulfate	R. Endrin aldehyde	V. Aroclor-1016	Z. Aroclor-1248	DD. DB 1701	HH.
C. delta-BHC	G. Heptachlor epoxide	K. Endrin	O. 4,4'-DDT	S. alpha-Chlordane	W. Aroclor-1221	AA. Aroclor-1254	EE. HP Xachlor Benzene	II.
D. gamma-BHC	H. Endosulfan I	L. Endosulfan II	P. Methoxychlor	T. gamma-Chlordane	X. Aroclor-1232	BB. Aroclor-1260	FF.	JJ.

LDC #: 21495 D34  
SDG #: See Cover

### VALIDATION FINDINGS WORKSHEET Surrogate Spikes

Page: 1 of 1  
Reviewer: JN  
2nd Reviewer: R

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".  
Y N N/A Were surrogates spiked into all samples, standards and blanks?  
Y N N/A Did all surrogate percent recoveries (%R) meet the QC limits?

#	Date	Sample ID	Column	Surrogate Compound	%R (Limits)	Qualifications
		5	Net Spec	B	28 (40-140)	J-N/A (S)
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A	Tetrachloro-m-xylene			
B	Decachlorobiphenyl			

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

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
		5	snr outside limits		X / A

Comments:

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

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

**Collection Date:** June 17 through June 24, 2009

**LDC Report Date:** September 22, 2009

**Matrix:** Water

**Parameters:** Chlorinated Pesticides

**Validation Level:** Stage 2B

**Laboratory:** Columbia Analytical Services, Inc.

**Sample Delivery Group (SDG):** R0903404

**Sample Identification**

M-78B  
M-128B  
H-38B  
M-19B  
M-34B  
M-125B  
M-22AB  
M-17AB  
M-125BMS  
M-125BMDS



## Introduction

This data review covers 10 water 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.

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.
- UU 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.

## 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 with the following exceptions:

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
6/23/09	FA672	STX-CLP1	Hexachlorobenzene	31.5	M-78B M-128B H-38B 89788MB	J+ (all detects)	A

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

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
6/18/09	FA581	STX-CLP2	Heptachlor Endrin aldehyde	25.2 21.8	M-78B M-128B H-38B 89788MB	J+ (all detects) J+ (all detects)	A

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

## **V. Blanks**

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

Sample FB060409 (from SDG R0903006) was identified as a field blank. No chlorinated pesticide contaminants were found in this blank.

## **VI. Surrogate Spikes**

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

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

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

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

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

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

Not applicable.

## **X. 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**

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 R0903404	All compounds reported below the PQL.	J (all detects)	A

Raw data were not reviewed for this SDG.

### **XIII. Overall Assessment of Data**

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

### **XIV. Field Duplicates**

No field duplicates were identified in this SDG.

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Data Qualification Summary - SDG R0903404**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R0903404	M-78B M-128B H-38B	Hexachlorobenzene	J+ (all detects)	A	Continuing calibration (%D) (c)
R0903404	M-78B M-128B H-38B	Heptachlor Endrin aldehyde	J+ (all detects) J+ (all detects)	A	Continuing calibration (ICV %D) (c)
R0903404	M-78B M-128B H-38B M-19B M-34B M-125B M-22AB M-17AB	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 R0903404**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG R0903404**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 21495E3a

SDG #: R0903404

Laboratory: Columbia Analytical Services

Stage 2B

Date: 6/17/09

Page: 1 of 1

Reviewer: JVG

2nd Reviewer: J

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/17-24/09
II.	GC/ECD Instrument Performance Check	A	
III.	Initial calibration	NEW A	
IV.	Continuing calibration/ICV	SW	CCV/ICV = 20%
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	LCS 1b
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	N	
XV.	Field blanks	ND	FB = FB060409 from R0903404

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-78B	11	89788 MB	21	31
2	M-128B	12	89964 MB	22	32
3	H-38B	13	90220 MB	23	33
4	M-19B	14		24	34
5	M-34B	15		25	35
6	M-125B	16		26	36
7	M-22AB	17		27	37
8	M-17AB	18		28	38
9	M-125BMS	19		29	39
10	M-125BMSD	20		30	40

**VALIDATION FINDINGS WORKSHEET**  
Continuing Calibration

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".

What type or calibration verification calculation was performed?     %D or     RPD  
 Were Evaluation mix standards run before initial calibration and before samples?      
 Were Endrin & 4,4'-DDT breakdowns acceptable in the Evaluation Mix standard ( $\leq 15.0\%$  for individual breakdowns)?      
 Was at least one standard run daily to verify the working curve?      
 Did the continuing calibration standards meet the percent difference (%D) / relative percent difference (RPD) criteria of  $\leq 15.0\%$ ?    

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

Level (WHS) Only  
 Y (N/A)

Were the retention times for all calibrated compounds within their respective acceptance windows?

#	Date	Standard ID	Column	Compound	%D (Limit $\leq 15.0$ )	RT (Limits)	Associated Samples	Qualifications
	<u>6/18/09</u>	<u>FA 581</u> <u>(10V)</u>	<u>STX-CLP2</u>	<u>E (+)</u>	<u>25.2</u>	( )	<u>1-3, 89788 MB</u>	<u>J + A U B / A</u> <u>(C)</u>
				<u>R (+)</u>	<u>21.8</u>	( )		
						( )		
	<u>6/23/09</u>	<u>FA 672</u> <u>(CV)</u>	<u>STX-CLP1</u>	<u>EE (+)</u>	<u>31.5</u>	( )		<u>   </u>
						( )		
						( )		
						( )		
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						( )		

A. alpha-BHC    E. Heptachlor    I. Dieldrin    M. 4,4'-DDD    Q. Endrin ketone    U. Toxaphene    Y. Aroclor-1242    CC. DB 608    GG.      
 B. beta-BHC    F. Aldrin    J. 4,4'-DDE    N. Endosulfan sulfate    R. Endrin aldehyde    V. Aroclor-1016    Z. Aroclor-1248    DD. DB 1701    HH.      
 C. delta-BHC    G. Heptachlor epoxide    K. Endrin    O. 4,4'-DDT    S. alpha-Chlordane    W. Aroclor-1221    AA. Aroclor-1254    EE. Hexachlorobenzene  
 D. gamma-BHC    H. Endosulfan I    L. Endosulfan II    P. Methoxychlor    T. gamma-Chlordane    X. Aroclor-1232    BB. Aroclor-1260    FF.      
 JI.



## Laboratory Data Consultants, Inc. Data Validation Report

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

**Collection Date:** June 5 through June 11, 2009

**LDC Report Date:** September 22, 2009

**Matrix:** Soil

**Parameters:** Chlorinated Pesticides

**Validation Level:** Stage 2B

**Laboratory:** Columbia Analytical Services, Inc.

**Sample Delivery Group (SDG):** R0903184

### Sample Identification

RSAJ6-0.5B  
RSAJ6-0.5BDL  
RSAK6-0.5B  
RSAK8-0.5B  
RSAL7-0.5B  
RSAL8-0.5B  
SA35-0.5B  
SA176-0.5B  
RSA03-0.5B  
SA182-0.5B  
SA166-0.5B  
RSAK4-0.5B  
RSAK4009-0.5B  
RSAJ6-0.5BMS  
RSAJ6-0.5BMSD  
SA35-0.5BMS  
SA35-0.5BMSD  
SA182-0.5BMS  
SA182-0.5BMSD

## Introduction

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

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.
- UU 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.

## 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 with the following exceptions:

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
6/18/09	FA581	STX-CLP2	Heptachlor Endrin aldehyde	25.2 21.8	RSAJ6-0.5BDL RSAK6-0.5B RSAK8-0.5B RSAL7-0.5B SA35-0.5B SA176-0.5B RSA03-0.5B SA182-0.5B SA166-0.5B RSAK4-0.5B RSAK4009-0.5B RSAJ6-0.5BMS RSAJ6-0.5BMSD SA35-0.5BMS SA35-0.5BMSD SA182-0.5BMS SA182-0.5BMSD 89340MB 89401MB	J+ (all detects) J+ (all detects)	A

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

## V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
89047MB	6/9/09	Hexachlorobenzene	2.0 ug/Kg	RSAJ6-0.5B RSAJ6-0.5BDL RSAK6-0.5B RSAK8-0.5B RSAL7-0.5B RSAL8-0.5B

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

Sample FB072109-SO (from SDG R0904016) was identified as a field blank. No chlorinated pesticide contaminants were found in this blank.

## VI. Surrogate Spikes

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

## VII. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. 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
SA182-0.5BMS/MSD (SA182-0.5B)	Endrin aldehyde	0 (18-135)	0 (18-135)	-	J- (all detects) R (all non-detects)	A

## 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
89401LCS/D (SA182-0.5B SA166-0.5B RSAK4-0.5B RSAK4009-0.5B 89401MB)	Endrin aldehyde	35 (50-130)	49 (50-130)	34 ( $\leq 30$ )	J (all detects) UJ (all non-detects)	P

## 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

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
RSAJ6-0.5B	Hexachlorobenzene beta-BHC	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	A

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R0903184	All compounds reported below the PQL.	J (all detects)	A

Raw data were not reviewed for this SDG.

### XIII. Overall Assessment of Data

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
RSAJ6-0.5B	Hexachlorobenzene beta-BHC	X X	A
RSAJ6-0.5BDL	All TCL compounds except Hexachlorobenzene beta-BHC	X	A

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

### XIV. Field Duplicates

Samples RSAK4-0.5B and RSAK4009-0.5B were identified as field duplicates. No chlorinated pesticides were detected in any of the samples with the following exceptions:

Compound	Concentration (ug/Kg)		RPD (Limits)	Difference (Limits)	Flags	A or P
	RSAK4-0.5B	RSAK4009-0.5B				
Hexachlorobenzene	170	250	38 ( $\leq 50$ )	-	-	-
beta-BHC	330	570	53 ( $\leq 50$ )	-	J (all detects)	A

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Data Qualification Summary - SDG R0903184**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R0903184	RSAJ6-0.5BDL RSAK6-0.5B RSAK8-0.5B RSAL7-0.5B SA35-0.5B SA176-0.5B RSA03-0.5B SA182-0.5B SA166-0.5B RSAK4-0.5B RSAK4009-0.5B	Heptachlor Endrin aldehyde	J+ (all detects) J+ (all detects)	A	Continuing calibration (ICV %D) (c)
R0903184	SA182-0.5B	Endrin aldehyde	J- (all detects) R (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R) (m)
R0903184	SA182-0.5B SA166-0.5B RSAK4-0.5B RSAK4009-0.5B	Endrin aldehyde	J (all detects) UJ (all non-detects)	P	Laboratory control samples (%R)(RPD) (l,ld)
R0903184	RSAJ6-0.5B	Hexachlorobenzene beta-BHC	J (all detects) J (all detects)	A	Project Quantitation Limit (e)
R0903184	RSAJ6-0.5B RSAJ6-0.5BDL RSAK6-0.5B RSAK8-0.5B RSAL7-0.5B RSAL8-0.5B SA35-0.5B SA176-0.5B RSA03-0.5B SA182-0.5B SA166-0.5B RSAK4-0.5B RSAK4009-0.5B	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
R0903184	RSAJ6-0.5B	Hexachlorobenzene beta-BHC	X X	A	Overall assessment of data (o)
R0903184	RSAJ6-0.5BDL	All TCL compounds except Hexachlorobenzene beta-BHC	X	A	Overall assessment of data (o)
R0903184	RSAK4-0.5B RSAK4009-0.5B	beta-BHC	J (all detects)	A	Field duplicates (RPD) (fd)



**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG  
R0903184**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG R0903184**

No Sample Data Qualified in this SDG

**Tronox Northgate Henderson**

**VALIDATION COMPLETENESS WORKSHEET**

LDC #: 21495F3a

SDG #: R0903184

Laboratory: Columbia Analytical Services

Stage 2B

Date: 9/17/09

Page: 1 of 1

Reviewer: JVL

2nd Reviewer: *[Signature]*

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/05 - 11/09
II.	GC/ECD Instrument Performance Check	A	
III.	Initial calibration	A	
IV.	Continuing calibration/ICV	SW	COV/ICV ≤ 20%
V.	Blanks	SW	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	SW	KCS / D
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	SW	
XIII.	Overall assessment of data	SW	
XIV.	Field duplicates	SW	D = 12, 13
XV.	Field blanks	ND	FB = FB-072109-50 from R0904016

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: *Soj1*

1	RSAJ6-0.5B	11	SA166-0.5B	21	89047 MB	31
2	RSAJ6-0.5BDL	12	RSAA4-0.5B	22	89340 MB	32
3	RSAA6-0.5B	13	RSAA4009-0.5B	23	89401 MB	33
4	RSAA8-0.5B	14	RSAJ6-0.5BMS	24		34
5	RSAL7-0.5B	15	RSAJ6-0.5BMSD	25		35
6	RSAL8-0.5B	16	SA35-0.5BMS	26		36
7	SA35-0.5B	17	SA35-0.5BMSD	27		37
8	SA176-0.5B	18	SA182-0.5BMS	28		38
9	RSA03-0.5B	19	SA182-0.5BMSD	29		39
10	SA182-0.5B	20		30		40

(#9 - RSA03  
↳ water not #)



LDC #: 21495 F3a  
SDG #: See Conv

**VALIDATION FINDINGS WORKSHEET**  
**Blanks**

Page: 1 of 1  
Reviewer: JVC  
2nd Reviewer: [Signature]

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

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

Y / N / N/A

Were all samples associated with a method blank?

Y / N / N/A

Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y / N / N/A

if extract clean-up was performed, were extract clean-up blanks analyzed at the proper frequencies?

X / N / N/A

Was there contamination in the method blanks? If yes, please see the qualifications below.

Blank extraction date: 6/09/09 Blank analysis date: 6/12/09

Associated samples: 1-6

Compound	Blank ID	Sample Identification
EE	89047 MB 2.0	(All > MB)

0.0

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

Associated samples: \_\_\_\_\_

Compound	Blank ID	Sample Identification

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:  
All contaminants within five times the method blank concentration were qualified as not detected, "U".

LDC #: 21495 F3a  
 SDG #: Sea Core

**VALIDATION FINDINGS WORKSHEET**  
Surrogate Spikes

Page: 1 of 1  
 Reviewer: DJK  
 2nd Reviewer: R

**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".

Y N N/A  
 Were surrogates spiked into all samples, standards and blanks?  
Y N N/A  
 Did all surrogate percent recoveries (%R) meet the QC limits?

#	Date	Sample ID	Column	Surrogate Compound	%R (Limits)	Qualifications
		1 (10x)	NOT SPEC	B	9667 (40-140)	No qual
				A	686 ( )	
		2 (500x)		A, B	0 ( )	
		4 (20x)		B	463 ( )	
		9 (10x)		B	231 ( )	
		10 (1000x)		B	4920 ( )	
				A	0 ( )	
		12 (10x)		B	272 ( )	
		13 (50x)	✓	B	400 ( )	
			✓	A	0 ( )	✓
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A	Tetrachloro-m-xylene			
B	Decachlorobiphenyl			

**VALIDATION FINDINGS WORKSHEET**  
**Matrix Spike/Matrix Spike Duplicates**

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

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

Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG?  
 Was a MS/MSD analyzed every 20 samples for each matrix or whenever a sample extraction was performed?  
 Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		14/15	All compounds have	( )	3 R outside	limits	1, 2	No qual (LCSB)
		16/17	Several compounds have	( )	3 R and	RPD's	7	
		18/19	All compounds except and 3 compounds have	( )	3 R RPD's	outside limits outside limits	10	
			R	0 (18-125)	0 (18-125)	( )		J-R/A (n)
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
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				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		

LDC #: 21415 Fax

SDG #: Sa Core

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".

N N/A

Y N/A

Level: IV/D Only

Y N/A

VALIDATION FINDINGS WORKSHEET

Laboratory Control Samples

Page: 1 of 1

Reviewer: JG

2nd Reviewer: R

Were a laboratory control samples (LCS) and laboratory control sample duplicate (LCSD) analyzed for each matrix in this SDG? \_\_\_\_\_  
 Were the LCS percent recoveries (%R) and relative percent differences (RPD) within the QC limits? \_\_\_\_\_

Was a LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed?

#	Date	LCS/LCSD ID	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		89047 LCS/D	R	( )	45 (50-130)	38 (30)	1-6, 89047 MB	No qual (LCS in)
				( )	( )	( )		
				( )	( )	( )		
		89340 LCS/D	R	40 (50-130)	( )	( )	7-9, 89340 MB	(LCSD in)
			EE	( )	( )	58 (30)	( )	(LCS/LCSD in)
				( )	( )	( )		
				( )	( )	( )		
		89401 LCS/D	R	35 (50-130)	49 (50-130)	34 (30)	10-13, 89401 MB	J/US/P (LCS in)
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
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				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		

LDC #: 21495 F3a  
SDG #: See Cover

**VALIDATION FINDINGS WORKSHEET**  
**Compound Quantitation and Reported CRQLs**

Page: 1 of 1  
Reviewer: D/K  
2nd Reviewer: R

**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".  
**Level IV/D Only**

Y N N/A Were CRQLs adjusted for sample dilutions, dry weight factors, etc.?

Y N N/A Did the reported results for detected target compounds agree within 10.0% of the recalculated results?

#	Sample ID Compound Name	Finding	Associated Samples	Qualifications
	1	EE, B > cal range		J Actb/A (e)

Comments: See sample calculation verification worksheet for recalculations



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

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

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	EE, B > w range		X/A (6)
		2	All except EE, B di		

Comments: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

LDC #: 21495 F 3A  
 SDG #: Su Comy

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

Page: 1 of 1  
 Reviewer: JVC  
 2nd reviewer: J

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

Y / N / N/A    Were field duplicate pairs identified in this SDG?  
Y / N / N/A    Were target compounds detected in this field duplicate pairs?

Compound	Concentration ( <u>ug/kg</u> )		RPD	Parent only
	12	13		
EE	170	250	38	-
B	330	570	53	JActB A (fd)

Compound	Concentration (            )		RPD

Compound	Concentration (            )		RPD

Compound	Concentration (            )		RPD

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

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

**Collection Date:** June 19 through June 24, 2009

**LDC Report Date:** September 22, 2009

**Matrix:** Soil

**Parameters:** Chlorinated Pesticides

**Validation Level:** Stage 2B

**Laboratory:** Columbia Analytical Services, Inc.

**Sample Delivery Group (SDG):** R0903443

**Sample Identification**

SA129-0.5B  
RSAN5-0.5B  
RSA06-0.5B  
RSAN5-0.5BMS  
RSAN5-0.5BMSD

## Introduction

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

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.
- UU 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.

## **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%.

## **V. Blanks**

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

No field blanks were identified in this SDG.

## **VI. Surrogate Spikes**

Surrogates were added to all samples and blanks as required by the method. Surrogate recoveries (%R) were not within QC limits for SA129-0.5B. 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. Although the MS and MSD percent recoveries (%R) and relative percent differences (RPD) were not within QC limits for some compounds, the MS 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 percent recovery (%R) and LCS/LCSD relative percent difference (RPD) were not within QC limits for one compound, the LCSD 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

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 R0903443	All compounds reported below the PQL.	J (all detects)	A

Raw data were not reviewed for this SDG.

## XIII. Overall Assessment of Data

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

#### **XIV. Field Duplicates**

No field duplicates were identified in this SDG.



**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Data Qualification Summary - SDG R0903443**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R0903443	SA129-0.5B RSAN5-0.5B RSAO6-0.5B	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 R0903443**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG R0903443**

No Sample Data Qualified in this SDG

**Tronox Northgate Henderson**

LDC #: 21495G3a

**VALIDATION COMPLETENESS WORKSHEET**

SDG #: R0903443

Stage 2B

Laboratory: Columbia Analytical Services

Date: 9/16/09

Page: 1 of 1

Reviewer: SVG

2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: <u>6/19 - 24/09</u>
II.	GC/ECD Instrument Performance Check	A	
III.	Initial calibration	A	
IV.	Continuing calibration/ICV	A	<u>COV/ICV ≤ 20%</u>
V.	Blanks	A	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	SW	<u>LCS/D</u>
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	N	
XV.	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:

501

1	SA129-0.5B	11	<u>9025 MB</u>	21		31	
2	RSAN5-0.5B	12		22		32	
3	RSAO6-0.5B	13		23		33	
4	RSAN5-0.5BMS	14		24		34	
5	RSAN5-0.5BMSD	15		25		35	
6		16		26		36	
7		17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	

(non-area)

# VALIDATION FINDINGS WORKSHEET

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

A. alpha-BHC	I. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	GG.
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH.
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	II.
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	JJ.
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. DB 608	KK.
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. DB 1701	LL.
G. Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	EE. <i>Hexachlorbenzene</i>	MM.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	FF.	NN.

Notes:

LDC #: 21495 G34  
SDG #: Su Gray

Page: 1 of 1  
Reviewer: JVC  
2nd Reviewer: R

### VALIDATION FINDINGS WORKSHEET

Surrogate Spikes

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".  
X N N/A Were surrogates spiked into all samples, standards and blanks?  
Y (N) N/A Did all surrogate percent recoveries (%R) meet the QC limits?

#	Date	Sample ID	Column	Surrogate Compound	%R (Limits)	Qualifications
		1 (1000x)	Not spec	A, B	0 (40-140)	No qual
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A	Tetrachloro-m-xylene			
B	Decachlorobiphenyl			

LDC #: 21495 G34  
SDG #: Se Cray

**VALIDATION FINDINGS WORKSHEET**  
**Matrix Spike/Matrix Spike Duplicates**

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".  
 N N/A Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG?  
 N N/A Was a MS/MSD analyzed every 20 samples for each matrix or whenever a sample extraction was performed?  
 Y(N) N/A 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 (Limits)	Associated Samples	Qualifications
		<u>4/5</u>	<u>L</u>	( )	<u>44</u> ( <u>65-127</u> )	<u>55</u> ( <u>30</u> )	<u>2</u>	<u>No qual (N.S.M.)</u>
			<u>N</u>	( )	<u>32</u> ( <u>37-122</u> )	<u>62</u> ( )		
			<u>R</u>	( )	( )	<u>46</u> ( )		
			<u>Q</u>	( )	<u>47</u> ( <u>57-123</u> )	<u>57</u> ( )		
			<u>P</u>	( )	<u>33</u> ( <u>38-149</u> )	<u>63</u> ( )		
			<u>β</u>	<u>162</u> ( <u>35-142</u> )	<u>0</u> ( <u>35-142</u> )	<u>32</u> ( )	<u>1</u>	<u>(LS/Din)</u>
				( )	( )	( )		
				( )	( )	( )		
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				( )	( )	( )		

### VALIDATION FINDINGS WORKSHEET Laboratory Control Samples

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

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

Y  N  N/A  
Were a laboratory control samples (LCS) and laboratory control sample duplicate (LCSD) analyzed for each matrix in this SDG?  
 Y  N  N/A  
Were the LCS percent recoveries (%R) and relative percent differences (RPD) within the QC limits?  
Level:  WHD  Only

Was a LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed?

#	Date	LCS/LCSD ID	Compound	LCS %R (Limits)	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		90255 LCS 1p	0	1574	(50-130)	( )	61	(30)	All + B1k No qual (LCSD in)
				( )	( )	( )	( )	( )	
				( )	( )	( )	( )	( )	
				( )	( )	( )	( )	( )	
				( )	( )	( )	( )	( )	
				( )	( )	( )	( )	( )	
				( )	( )	( )	( )	( )	
				( )	( )	( )	( )	( )	
				( )	( )	( )	( )	( )	
				( )	( )	( )	( )	( )	
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				( )	( )	( )	( )	( )	
				( )	( )	( )	( )	( )	
				( )	( )	( )	( )	( )	
				( )	( )	( )	( )	( )	
				( )	( )	( )	( )	( )	
				( )	( )	( )	( )	( )	
				( )	( )	( )	( )	( )	
				( )	( )	( )	( )	( )	

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

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

**Collection Date:** June 29 through June 30, 2009

**LDC Report Date:** September 22, 2009

**Matrix:** Soil

**Parameters:** Chlorinated Pesticides

**Validation Level:** Stage 2B

**Laboratory:** Columbia Analytical Services, Inc.

**Sample Delivery Group (SDG):** R0903615

**Sample Identification**

RSAO5-0.5B  
SA106-0.5B  
SA106-0.5BDL

## **Introduction**

This data review covers 3 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.

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.
- UU 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.

## **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%.

## **V. Blanks**

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

No field blanks were identified in this SDG.

## **VI. Surrogate Spikes**

Surrogates were added to all samples and blanks as required by the method. Surrogate recoveries (%R) were not within QC limits for SA106-0.5B and SA106-0.5BDL. Since the samples were diluted out, no data were qualified.

## VII. Matrix Spike/Matrix Spike Duplicates

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

## VIII. Laboratory Control Samples (LCS)

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

## IX. Regional Quality Assurance and Quality Control

Not applicable.

## X. 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

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
SA106-0.5B	Hexachlorobenzene	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 R0903615	All compounds reported below the PQL.	J (all detects)	A

Raw data were not reviewed for this SDG.

### XIII. Overall Assessment of Data

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
SA106-0.5B	Hexachlorobenzene	X	A
SA106-0.5BDL	All TCL compounds except Hexachlorobenzene	X	A

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

### XIV. Field Duplicates

No field duplicates were identified in this SDG.

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Data Qualification Summary - SDG R0903615**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R0903615	SA106-0.5B	Hexachlorobenzene	J- (all detects) UJ (all non-detects)	A	Project Quantitation Limit (e)
R0903615	RSA05-0.5B SA106-0.5B SA106-0.5BDL	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
R0903615	SA106-0.5B	Hexachlorobenzene	X	A	Overall assessment of data (o)
R0903615	SA106-0.5BDL	All TCL compounds except Hexachlorobenzene	X	A	Overall assessment of data (o)

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG R0903615**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG R0903615**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 21495H3a

SDG #: R0903615

Laboratory: Columbia Analytical Services

Stage 2B

Date: 9/18/09

Page: 1 of 1

Reviewer: SVB

2nd Reviewer: J

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/29-30/09
II.	GC/ECD Instrument Performance Check	A	
III.	Initial calibration	A	
IV.	Continuing calibration/ICV	A	CCV/ICV < 20%
V.	Blanks	A	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	N	client spec
VIII.	Laboratory control samples	A	LCS / D
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	SW	
XIII.	Overall assessment of data	SW	
XIV.	Field duplicates	N	
XV.	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	RSAO5-0.5B	11	90 767 MB	21		31	
2	SA106-0.5B	12		22		32	
3	SA106-0.5BDL	13		23		33	
4		14		24		34	
5		15		25		35	
6		16		26		36	
7		17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	

VALIDATION FINDINGS WORKSHEET

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

A. alpha-BHC	I. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	GG.
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH.
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	II.
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	JJ.
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. DB 608	KK.
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. DB 1701	LL.
G. Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	EE. <i>Hexachlorobenzene</i>	MM.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	FF.	NN.

Notes:

LDC #: 21495 H34  
 SDG #: SA Green

**VALIDATION FINDINGS WORKSHEET**  
**Surrogate Spikes**

Page: 1 of 1  
 Reviewer: DVG  
 2nd Reviewer: [Signature]

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

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

#	Date	Sample ID	Column	Surrogate Compound	%R (Limits)	Qualifications
2	(10x)	NAI Spike	A		0	(90-140)
3	(500x)		B			No qual

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A	Tetrachloro-m-xylene			
B	Decachlorobiphenyl			



# VALIDATION FINDINGS WORKSHEET

## Compound Quantitation and Reported CRQLs

**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".  
**Level IV/B Only**

Y N N/A  
Y N N/A

Were CRQLs adjusted for sample dilutions, dry weight factors, etc.?

Did the reported results for detected target compounds agree within 10.0% of the recalculated results?

#	Compound Name	Finding	Associated Samples	Qualifications
	EE	7 cm range	2	Jouts/A (e)

Comments: See sample calculation verification worksheet for recalculations

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

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

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
		2	EE - out range		X / A (0)
		3	All except EE ch1		↓

Comments: \_\_\_\_\_

## Laboratory Data Consultants, Inc. Data Validation Report

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

**Collection Date:** July 1 through July 2, 2009

**LDC Report Date:** September 22, 2009

**Matrix:** Soil/Water

**Parameters:** Chlorinated Pesticides

**Validation Level:** Stage 2B

**Laboratory:** Columbia Analytical Services, Inc.

**Sample Delivery Group (SDG):** R0903678

### Sample Identification

EB070109-SO1  
EB070109-SO1RE  
RSAN6-0.5B  
SA82-0.5B  
SA82-0.5BDL  
SA82-10B  
SA82-29B  
RSAL3-10B  
RSAL3-30B  
RSAK3-0.5B  
RSAK3-0.5BDL  
RSAK3-10B  
RSAK3-20B  
RSAK3-31B  
SA82-0.5BMS  
SA82-0.5BMSD  
RSAK3-31BMS  
RSAK3-31BMSD

## Introduction

This data review covers 16 soil samples and 2 water 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.

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.
- UU 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 Extraction	Required Holding Time (in Days) From Sample Collection Until Extraction	Flag	A or P
EB070109-SO1RE	All TCL compounds	15	7	J- (all detects) R (all non-detects)	A

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.

## 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%.

## V. Blanks

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

Samples EB070109-SO1 and EB070109-SO1RE were identified as equipment blanks. No chlorinated pesticide contaminants were found in these blanks.

Sample FB072109-SO (from SDG R0904016) was identified as a field blank. No chlorinated pesticide contaminants were found in this blank.

**VI. Surrogate Spikes**

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

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
EB070109-SO1	Not specified	Decachlorobiphenyl	26 (40-140)	All TCL compounds	J- (all detects) UJ (all non-detects)	A
EB070109-SO1RE	Not specified	Decachlorobiphenyl	36 (40-140)	All TCL compounds	J- (all detects) UJ (all non-detects)	A

**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 and MSD percent recoveries (%R) were not within QC limits for all compounds, the 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 percent recovery (%R) and LCS/LCSD relative percent difference (RPD) were not within QC limits for one compound, the LCSD 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**

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
RSAK3-0.5B	Hexachlorobenzene beta-BHC	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	A
SA82-0.5B	Hexachlorobenzene	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 R0903678	All compounds reported below the PQL.	J (all detects)	A

Raw data were not reviewed for this SDG.

## XIII. Overall Assessment of Data

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
EB070109-SO1RE	All TCL compounds	X	A
RSAK3-0.5B	Hexachlorobenzene beta-BHC	X X	A
RSAK3-0.5BDL	All TCL compounds except Hexachlorobenzene beta-BHC	X	A
SA82-0.5B	Hexachlorobenzene	X	A
SA82-0.5BDL	All TCL compounds except Hexachlorobenzene	X	A

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



#### **XIV. Field Duplicates**

No field duplicates were identified in this SDG.

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Data Qualification Summary - SDG R0903678**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R0903678	EB070109-SO1RE	All TCL compounds	J- (all detects) R (all non-detects)	A	Technical holding times (h)
R0903678	EB070109-SO1 EB070109-SO1RE	All TCL compounds	J- (all detects) UJ (all non-detects)	A	Surrogate spikes (%R) (s)
R0903678	RSAK3-0.5B	Hexachlorobenzene beta-BHC	J (all detects) J (all detects)	A	Project Quantitation Limit (e)
R0903678	SA82-0.5B	Hexachlorobenzene	J (all detects)	A	Project Quantitation Limit (e)
R0903678	EB070109-SO1 EB070109-SO1RE RSAN6-0.5B SA82-0.5B SA82-0.5BDL SA82-10B SA82-29B RSAL3-10B RSAL3-30B RSAK3-0.5B RSAK3-0.5BDL RSAK3-10B RSAK3-20B RSAK3-31B	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
R0903678	EB070109-SO1RE	All TCL compounds	X	A	Overall assessment of data (o)
R0903678	RSAK3-0.5B	Hexachlorobenzene beta-BHC	X X	A	Overall assessment of data (o)
R0903678	RSAK3-0.5BDL	All TCL compounds except Hexachlorobenzene beta-BHC	X	A	Overall assessment of data (o)
R0903678	SA82-0.5B	Hexachlorobenzene	X	A	Overall assessment of data (o)
R0903678	SA82-0.5BDL	All TCL compounds except Hexachlorobenzene	X	A	Overall assessment of data (o)

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG  
R0903678**

**No Sample Data Qualified in this SDG**

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG R0903678**

**No Sample Data Qualified in this SDG**

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 2149513a

SDG #: R0903678

Laboratory: Columbia Analytical Services

Stage 2B

Date: 9/18/09

Page: 1 of 1

Reviewer: JV

2nd Reviewer: Q

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

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

	Validation Area		Comments
I.	Technical holding times	SW	Sampling dates: 7/01-02/09
II.	GC/ECD Instrument Performance Check	A	
III.	Initial calibration	A	
IV.	Continuing calibration/ICV	A	CCV / ICV ≤ 20%
V.	Blanks	A	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	SW	LCS / 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	SW	
XIII.	Overall assessment of data	SW	
XIV.	Field duplicates	N	
XV.	Field blanks	ND	EB = 1, 2      FB = FB072109-SO from R0904016

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 + Soil

1	EB070109-SO1	W	11	RSK3-0.5BDL	S	21	90782 MB	31
2	EB070109-SO1RE	↓	12	RSK3-10B		22	91494 MB	32
3	RSAN6-0.5B	S	13	RSK3-20B		23	90767 MB	33
4	SA82-0.5B		14	RSK3-31B		24	91091 MB	34
5	SA82-0.5BDL		15	SA82-0.5BMS		25	<del>91494</del> MB	35
6	SA82-10B		16	SA82-0.5BMSD		26		36
7	SA82-29B		17	RSK3-31BMS		27		37
8	RSAL3-10B		18	RSK3-31BMSD	↓	28		38
9	RSAL3-30B		19			29		39
10	RSK3-0.5B	✓	20			30		40

# VALIDATION FINDINGS WORKSHEET

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

A. alpha-BHC	I. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	GG.
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH.
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	II.
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	JJ.
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. DB 608	KK.
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. DB 1701	LL.
G. Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	EE. <i>Hexachlorobenzene</i>	MM.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	FF.	NN.

Notes:

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LDC #: 21495 I 3a  
SDG #: Sea Cond

### VALIDATION FINDINGS WORKSHEET Technical Holding Times

Page: 1 of 1  
Reviewer: JVG  
2nd Reviewer: J

All circled dates have exceeded the technical holding times.  
Y/N/N/A. Were all cooler temperatures within validation criteria?

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

Sample ID	Matrix	Preserved	Sampling Date	Extraction date	Analysis date	Total # of Days	Qualifier
2	W	N	7/01/09	7/16/09	7/27/09	15	S-R/A (h)

#### TECHNICAL HOLDING TIME CRITERIA

Water: Extracted within 7 days, analyzed within 40 days.  
Soil: Extracted within 14 days, analyzed within 40 days.

LDC #: 21495 I 31  
 SDG #: Sea Army

VALIDATION FINDINGS WORKSHEET  
Surrogate Spikes

Page: 1 of 1  
 Reviewer: JLC  
 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".  
 N N/A Were surrogates spiked into all samples, standards and blanks?  
 N N/A Did all surrogate percent recoveries (%R) meet the QC limits?

#	Date	Sample ID	Column	Surrogate Compound	%R (Limits)	Qualifications
1			Not spec	B	26 (40-140)	J-MS/A (S) ↓
2					36 ( )	↓
3		(10X)			176 ( )	No peak
4		(10X)			178 ( )	
5		(50X)		A, B	0 ( )	
10		(10X)		B	3743 ( )	
				A	246 ( )	
11		(5000X)		A, B	0 ( )	↓
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	
					( )	

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A	Tetrachloro-m-xylene			
B	Decachlorobiphenyl			

**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".  
 **N** N/A Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG?  
 **N** N/A Was a MS/MSD analyzed every 20 samples for each matrix or whenever a sample extraction was performed?  
 **N** N/A 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 (Limits)	Associated Samples	Qualifications
		15/16	All compounds (see attached report)	R	R outside limits	( )	4, 5	No qual (LCSD in)
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
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				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		



COLUMBIA ANALYTICAL SERVICES, INC.

QA/QC Report

Client: Northgate Environmental  
 Project: Tronox LLC Henderson/2027.001  
 Sample Matrix: Soil

Service Request: R0903678  
 Date Collected: 7/1/09  
 Date Received: 7/2/09  
 Date Analyzed: 7/10/09

Matrix Spike Summary  
 Organochlorine Pesticides by Gas Chromatography

Sample Name: SA82-0.5B  
 Lab Code: R0903678-007

Units: µg/Kg  
 Basis: Dry

Analytical Method: 8081A  
 Prep Method: EPA 3541

Analyte Name	Sample Result	Matrix Spike RQ0905448-06			Duplicate Matrix Spike RQ0905448-07			% Rec Limits	RPD	RPD Limit
		Result	Amount	% Rec	Result	Amount	% Rec			
4,4'-DDD	ND	ND	6.85	0 *	ND	6.85	0 *	58 - 121	0	30
4,4'-DDE	ND	67.0	6.85	978 #	63.9	6.85	933 #	56 - 125	5	30
4,4'-DDT	ND	ND	6.85	0 *	ND	6.85	0 *	9 - 149	0	30
Aldrin	ND	ND	6.85	0 *	ND	6.85	0 *	15 - 135	0	30
Dieldrin	ND	ND	6.85	0 *	ND	6.85	0 *	25 - 150	0	30
Endosulfan I	ND	ND	6.85	0 *	ND	6.85	0 *	56 - 119	0	30
Endosulfan II	ND	ND	6.85	0 *	ND	6.85	0 *	65 - 127	0	30
Endosulfan Sulfate	ND	ND	6.85	0 *	ND	6.85	0 *	37 - 122	0	30
Endrin	ND	ND	6.85	0 *	ND	6.85	0 *	28 - 143	0	30
Endrin Aldehyde	ND	ND	6.85	0 *	ND	6.85	0 *	18 - 135	0	30
Endrin Ketone	ND	ND	6.85	0 *	ND	6.85	0 *	57 - 123	0	30
Heptachlor	ND	ND	6.85	0 *	ND	6.85	0 *	35 - 127	0	30
Heptachlor Epoxide	ND	ND	6.85	0 *	ND	6.85	0 *	61 - 120	0	30
Hexachlorobenzene	540	686	17.1	825 #	666	17.1	712 #	20 - 150	3	30
Methoxychlor	ND	ND	34.3	0 *	ND	34.3	0 *	38 - 149	0	30
alpha-BHC	ND	ND	6.85	0 *	ND	6.85	0 *	53 - 130	0	30
alpha-Chlordane	ND	ND	6.85	0 *	ND	6.85	0 *	27 - 130	0	30
beta-BHC	57	105	6.85	693 #	94.9	6.85	548 #	35 - 142	10	30
delta-BHC	ND	ND	6.85	0 *	ND	6.85	0 *	44 - 119	0	30
gamma-BHC (Lindane)	ND	ND	6.85	0 *	ND	6.85	0 *	37 - 124	0	30
gamma-Chlordane	ND	ND	6.85	0 *	ND	6.85	0 *	38 - 127	0	30

Comments:

**VALIDATION FINDINGS WORKSHEET**  
**Laboratory Control Samples**

LDC #: 21495 I-3a  
 SDG #: 5cc Conar  
 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".  
 Were a laboratory control samples (LCS) and laboratory control sample duplicate (LCSD) analyzed for each matrix in this SDG?  
 Were the LCS percent recoveries (%R) and relative percent differences (RPD) within the QC limits?

Y  N  N/A  
 Level IV/D Only

Was a LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed?  
 Y  N  N/A

#	Date	LCS/LCSD ID	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		91494 LCS/D	R	149 ( )	( )	52 ( 30 )	2, 91494 MB	No qual (LCSD)

LDC #: 21495 I32  
SDG #: Su Gray

# VALIDATION FINDINGS WORKSHEET

## Compound Quantitation and Reported CRQLs

Page: 1 of 1  
Reviewer: DJB  
2nd 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".  
Level I/V/D Only

Y  N  N/A  
 Y  N  N/A

Were CRQLs adjusted for sample dilutions, dry weight factors, etc.?  
Did the reported results for detected target compounds agree within 10.0% of the recalculated results?

#	Compound Name	Finding	Associated Samples	Qualifications
	EE, B	> cu range	10	J det A (e)
	EE	↓	4	↓

Comments: See sample calculation verification worksheet for recalculations

**VALIDATION FINDINGS WORKSHEET**  
Overall Assessment of Data

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

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
2			confirmation run for	sur outlier in #1	X/A (0)
10			EE, B	> cal range	
11			All except EE, B,	di	
4			EE	> cal range	
5			All except EE di		

Comments:

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

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

**Collection Date:** June 25 through July 1, 2009

**LDC Report Date:** September 24, 2009

**Matrix:** Water

**Parameters:** Chlorinated Pesticides

**Validation Level:** Stage 2B

**Laboratory:** Columbia Analytical Services, Inc.

**Sample Delivery Group (SDG):** R0903561

**Sample Identification**

M-75B  
M-13AB  
M-13009AB  
M-64B  
M-111AB  
EB062909-GW1  
M-25B  
M-12AB  
M-110B  
I-ARB

## Introduction

This data review covers 10 water 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.

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.
- UU 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.

## **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%.

## **V. Blanks**

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

Sample EB062909-GW1 was identified as an equipment blank. No chlorinated pesticide contaminants were found in this blank.

## **VI. Surrogate Spikes**

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



## VII. Matrix Spike/Matrix Spike Duplicates

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

## VIII. Laboratory Control Samples (LCS)

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

## IX. Regional Quality Assurance and Quality Control

Not applicable.

## X. 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

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 R0903561	All compounds reported below the PQL.	J (all detects)	A

Raw data were not reviewed for this SDG.

## XIII. Overall Assessment of Data

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

#### **XIV. Field Duplicates**

Samples M-13AB and M-13009AB were identified as field duplicates. No volatiles were detected in any of the samples.

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Data Qualification Summary - SDG R0903561**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R0903561	M-75B M-13AB M-13009AB M-64B M-111AB EB062909-GW1 M-25B M-12AB M-110B I-ARB	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 R0903561**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG R0903561**

No Sample Data Qualified in this SDG

**Tronox Northgate Henderson**

**VALIDATION COMPLETENESS WORKSHEET**

LDC #: 21495J3a

SDG #: R0903561

Laboratory: Columbia Analytical Services

Stage 2B

Date: 7/23/09

Page: 1 of 1

Reviewer: JLB

2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/25 - 7/01/09
II.	GC/ECD Instrument Performance Check	A	
III.	Initial calibration	A	
IV.	Continuing calibration/ICV	A	CCV/ICV ≤ 20 %
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	N	Client spec
VIII.	Laboratory control samples	A	LCS ID
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	ND	D = 2, 3
XV.	Field blanks	ND	EB = 6

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-75B	11	90395 MB	21	31
2	M-13AB	12	90700	22	32
3	M-13009AB	13	90782	23	33
4	M-64B	14		24	34
5	M-111AB	15		25	35
6	EB062909-GW1	16		26	36
7	M-25B	17		27	37
8	M-12AB	18		28	38
9	M-110B	19		29	39
10	I-ARB	20		30	40

## Laboratory Data Consultants, Inc. Data Validation Report

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

**Collection Date:** June 26, 2009

**LDC Report Date:** September 25, 2009

**Matrix:** Soil/Water

**Parameters:** Chlorinated Pesticides

**Validation Level:** Stage 2B

**Laboratory:** Columbia Analytical Services, Inc.

**Sample Delivery Group (SDG):** R0903584

### Sample Identification

RSAI3-10B  
RSAI3-10BDL  
RSAI3-20B  
RSAI3-32B  
RSAI2-10B  
RSAI2-10BDL  
RSAI2009-10B  
RSAI2009-10BDL  
RSAI2-20B  
RSAI2-31B  
RSAJ2-10B  
RSAJ2-20B  
RSAJ2-33B  
RSAJ2009-33B  
EB062609-SO  
RSAI3-10BMS  
RSAI3-10BMSD  
RSAI2-10BMS  
RSAI2-10BMSD

## Introduction

This data review covers 18 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8081A for Chlorinated Pesticides.

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

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.
- UU 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.

## 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%.

## V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
90348MB	6/26/09	Hexachlorobenzene	0.89 ug/kg	RSAI3-10B RSAI3-10BDL RSAI3-20B RSAI3-32B

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



Sample EB062609-SO was identified as an equipment blank. No chlorinated pesticide contaminants were found in this blank.

Sample FB072109-SO (from SDG R0904016) was identified as a field blank. No chlorinated pesticide contaminants were found in this blank.

## **VI. Surrogate Spikes**

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

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

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Although the MS and MSD percent recoveries (%R) were not within QC limits for all compounds and the relative percent differences (RPD) was not within QC limits for one compound, the LCS 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. Although the LCS percent recovery (%R) and LCS/LCSD relative percent difference (RPD) were not within QC limits for one compound, the LCSD 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**

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
RSAI3-10B RSAI2-10B RSAI2009-10B	Hexachlorobenzene	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 R0903584	All compounds reported below the PQL.	J (all detects)	A

Raw data were not reviewed for this SDG.

### XIII. Overall Assessment of Data

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
RSAI3-10B RSAI2-10B RSAI2009-10B	Hexachlorobenzene	X	A
RSAI3-10BDL RSAI2-10BDL RSAI2009-10BDL	All TCL compounds except Hexachlorobenzene	X	A

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

### XIV. Field Duplicates

Samples RSAI2-10B and RSAI2009-10B, samples RSAI2-10BDL and RSAI2009-10BDL, and samples RSAJ2-33B and RSAJ2009-33B were identified as field duplicates. No chlorinated pesticides were detected in any of the samples with the following exceptions:

Compound	Concentration (ug/Kg)		RPD (Limits)	Difference (Limits)	Flags	A or P
	RSAI2-10B	RSAI2009-10B				
4,4'-DDE	47	48	-	1 ( $\leq 36$ )	-	-

Compound	Concentration (ug/Kg)		RPD (Limits)	Difference (Limits)	Flags	A or P
	RSAI2-10B	RSAI2009-10B				
4,4'-DDT	71	66	-	5 ( $\leq 36$ )	-	-
Hexachlorobenzene	2100	2100	0 ( $\leq 50$ )	-	-	-
beta-BHC	120	100	18 ( $\leq 50$ )	-	-	-

Compound	Concentration (ug/Kg)		RPD (Limits)	Difference (Limits)	Flags	A or P
	RSAI2-10BDL	RSAI2009-10BDL				
Hexachlorobenzene	6700	7000	-	300 ( $\leq 1900$ )	-	-

Compound	Concentration (ug/Kg)		RPD (Limits)	Difference (Limits)	Flags	A or P
	RSAJ2-33B	RSAJ2009-33B				
Hexachlorobenzene	2.3	3.0U	-	0.7 ( $\leq 3.0$ )	-	-

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Data Qualification Summary - SDG R0903584**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R0903584	RSAI3-10B RSAI2-10B RSAI2009-10B	Hexachlorobenzene	J (all detects)	A	Project Quantitation Limit (e)
R0903584	RSAI3-10B RSAI3-10BDL RSAI3-20B RSAI3-32B RSAI2-10B RSAI2-10BDL RSAI2009-10B RSAI2009-10BDL RSAI2-20B RSAI2-31B RSAJ2-10B RSAJ2-20B RSAJ2-33B RSAJ2009-33B EB062609-SO	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
R0903584	RSAI3-10B RSAI2-10B RSAI2009-10B	Hexachlorobenzene	X	A	Overall assessment of data (o)
R0903584	RSAI3-10BDL RSAI2-10BDL RSAI2009-10BDL	All TCL compounds except Hexachlorobenzene	X	A	Overall assessment of data (o)

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG R0903584**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG R0903584**

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson

## VALIDATION COMPLETENESS WORKSHEET

LDC #: 21495K3a

SDG #: R0903584

Laboratory: Columbia Analytical Services

Stage 2B

Date: 9/21/09

Page: 1 of 1

Reviewer: JV6

2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: <u>6/26/09</u>
II.	GC/ECD Instrument Performance Check	A	
III.	Initial calibration	A	
IV.	Continuing calibration/ICV	A	<u>CCV/ICV ≤ 20%</u>
V.	Blanks	SW	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	SW	<u>US/D</u>
IX.	Regional quality assurance and quality control	N	
Xa.	Florisol cartridge check	N	
Xb.	GPC Calibration	N	
XI.	Target compound identification	N	
XII.	Compound quantitation and reported CRQLs	SW	
XIII.	Overall assessment of data	SW	
XIV.	Field duplicates	SW	<u>D<sub>1</sub> = 5, 7    D<sub>2</sub> = 6, 8    D<sub>3</sub> = 13, 14</u>
XV.	Field blanks	ND	<u>EB = 15    FB = FB072109 - So from R0904016</u>

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 + Soil

1	RSIAI3-10B	S	11	RSIAJ2-10B	S	21	90348 MB	31
2	RSIAI3-10BDL		12	RSIAJ2-20B		22	90455 MB	32
3	RSIAI3-20B		13	RSIAJ2-33B	D <sub>3</sub>	23	90581 MB	33
4	RSIAI3-32B		14	RSIAJ2009-33B	D <sub>3</sub>	24		34
5	RSIAI2-10B	D <sub>1</sub>	15	EB062609-SO	W	25		35
6	RSIAI2-10BDL	D <sub>1</sub>	16	RSIAI3-10BMS	S	26		36
7	RSIAI2009-10B	D <sub>1</sub>	17	RSIAI3-10BMSD		27		37
8	RSIAI2009-10BDL	D <sub>1</sub>	18	RSIAI2-10BMS		28		38
9	RSIAI2-20B		19	RSIAI2-10BMSD		29		39
10	RSIAI2-31B		20			30		40

# VALIDATION FINDINGS WORKSHEET

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

A. alpha-BHC	I. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	GG.
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH.
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	II.
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	JJ.
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. DB 608	KK.
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. DB 1701	LL.
G. Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	EE. Hexachlorobenzene	MM.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	FF.	NN.

Notes:

LDC #: 21495 k3a  
SDG #: Se Cony

# VALIDATION FINDINGS WORKSHEET

## Blanks

Page: 1 of 1  
Reviewer: JVC  
2nd Reviewer: Q

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

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

- Y N N/A Were all samples associated with a method blank?
- Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?
- Y N N/A If extract clean-up was performed, were extract clean-up blanks analyzed at the proper frequencies?
- Y N N/A Was there contamination in the method blanks? If yes, please see the qualifications below.

Blank extraction date: 6/29/04  
Blank analysis date: 7/16/04

Associated samples: 1-4

Compound	Blank ID	Sample Identification
EE	90348 MB 0.89	(All results > MB)

1-78

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

Associated samples: \_\_\_\_\_

Compound	Blank ID	Sample Identification

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:  
All contaminants within five times the method blank concentration were qualified as not detected, "U".

LDC #: 21415 K32  
SDG #: See below

**VALIDATION FINDINGS WORKSHEET**  
**Surrogate Spikes**

Page: 1 of 1  
Reviewer: SVL  
2nd Reviewer: Q

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".  
(Y) Were surrogates spiked into all samples, standards and blanks?  
(N) Did all surrogate percent recoveries (%R) meet the QC limits?

#	Date	Sample ID	Column	Surrogate Compound	%R (Limits)	Qualifications
		1 (10x)	Not spec	B	2116 (70-140)	No qual
		2, 6, 8 (1000x)		A, B	0	
		5 (10x)		B	903	
		7 (10x)	✓	B	867	✓

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A	Tetrachloro-m-xylene			
B	Decachlorobiphenyl			



**VALIDATION FINDINGS WORKSHEET**  
**Matrix Spike/Matrix Spike Duplicates**

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".  
 N N/A Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG?  
 N N/A Was a MS/MSD analyzed every 20 samples for each matrix or whenever a sample extraction was performed?  
 Y (N) N/A 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 (Limits)	Associated Samples	Qualifications
		16/17	All compounds have B (see attached report)	0% R outside limits	41 (30)	1, 2	↓	No qual (LCSD in) ↓
		18/19	All compounds have (see attached report)	%R outside limits	5, 6			No qual (LCSD in)

COLUMBIA ANALYTICAL SERVICES, INC.

QA/QC Report

Client: Northgate Environmental  
 Project: Tronox LLC Henderson/2027.001  
 Sample Matrix: Soil

Service Request: R0903584  
 Date Collected: 6/25/09  
 Date Received: 6/26/09  
 Date Analyzed: 7/13/09

Matrix Spike Summary  
 Organochlorine Pesticides by Gas Chromatography

Sample Name: RSAI3-10B  
 Lab Code: R0903584-004

Units: µg/Kg  
 Basis: Dry

Analytical Method: 8081A  
 Prep Method: EPA 3550C

Analyte Name	Sample Result	Matrix Spike RQ0905219-06			Duplicate Matrix Spike RQ0905219-07			% Rec Limits	RPD	RPD Limit
		Result	Amount	% Rec	Result	Amount	% Rec			
4,4'-DDD	ND	ND	7.28	0 *	ND	7.28	0 *	58 - 121	0	30
4,4'-DDE	ND	ND	7.28	0 *	ND	7.28	0 *	56 - 125	0	30
4,4'-DDT	ND	ND	7.28	0 *	ND	7.28	0 *	9 - 149	0	30
Aldrin	ND	ND	7.28	0 *	ND	7.28	0 *	15 - 135	0	30
Dieldrin	ND	ND	7.28	0 *	ND	7.28	0 *	25 - 150	0	30
Endosulfan I	ND	ND	7.28	0 *	ND	7.28	0 *	56 - 119	0	30
Endosulfan II	ND	ND	7.28	0 *	ND	7.28	0 *	65 - 127	0	30
Endosulfan Sulfate	ND	ND	7.28	0 *	ND	7.28	0 *	37 - 122	0	30
Endrin	ND	ND	7.28	0 *	ND	7.28	0 *	28 - 143	0	30
Endrin Aldehyde	ND	ND	7.28	0 *	ND	7.28	0 *	18 - 135	0	30
Endrin Ketone	ND	ND	7.28	0 *	ND	7.28	0 *	57 - 123	0	30
Heptachlor	ND	ND	7.28	0 *	ND	7.28	0 *	35 - 127	0	30
Heptachlor Epoxide	ND	ND	7.28	0 *	ND	7.28	0 *	61 - 120	0	30
Hexachlorobenzene	13000	21600	18.2	48760 #	28800	18.2	88440 #	20 - 150	29	30
Methoxychlor	ND	ND	36.4	0 *	ND	36.4	0 *	38 - 149	0	30
alpha-BHC	ND	ND	7.28	0 *	ND	7.28	0 *	53 - 130	0	30
alpha-Chlordane	ND	ND	7.28	0 *	ND	7.28	0 *	27 - 130	0	30
beta-BHC	ND	542	7.28	7450 *	819	7.28	11250 *	35 - 142	41 *	30
delta-BHC	ND	ND	7.28	0 *	ND	7.28	0 *	44 - 119	0	30
gamma-BHC (Lindane)	ND	ND	7.28	0 *	ND	7.28	0 *	37 - 124	0	30
gamma-Chlordane	ND	ND	7.28	0 *	ND	7.28	0 *	38 - 127	0	30

Comments:

**COLUMBIA ANALYTICAL SERVICES, INC.**

QA/QC Report

**Client:** Northgate Environmental  
**Project:** Tronox LLC Henderson/2027.001  
**Sample Matrix:** Soil

**Service Request:** R0903584  
**Date Collected:** 6/26/09  
**Date Received:** 6/27/09  
**Date Analyzed:** 7/14/09

**Matrix Spike Summary**  
**Organochlorine Pesticides by Gas Chromatography**

**Sample Name:** RSAI2-10B  
**Lab Code:** R0903584-013

**Units:** µg/Kg  
**Basis:** Dry

**Analytical Method:** 8081A  
**Prep Method:** EPA 3550C

Analyte Name	Sample Result	Matrix Spike RQ0905276-04			Duplicate Matrix Spike RQ0905276-05			% Rec Limits	RPD	RPD Limit
		Result	Amount	% Rec	Result	Amount	% Rec			
4,4'-DDD	ND	ND	7.26	0 *	ND	7.26	0	* 58 - 121	0	30
4,4'-DDE	ND	ND	7.26	0 *	ND	7.26	0	* 56 - 125	0	30
4,4'-DDT	ND	ND	7.26	0 *	ND	7.26	0	* 9 - 149	0	30
Aldrin	ND	ND	7.26	0 *	ND	7.26	0	* 15 - 135	0	30
Dieldrin	ND	ND	7.26	0 *	ND	7.26	0	* 25 - 150	0	30
Endosulfan I	ND	ND	7.26	0 *	ND	7.26	0	* 56 - 119	0	30
Endosulfan II	ND	ND	7.26	0 *	ND	7.26	0	* 65 - 127	0	30
Endosulfan Sulfate	ND	ND	7.26	0 *	ND	7.26	0	* 37 - 122	0	30
Endrin	ND	ND	7.26	0 *	ND	7.26	0	* 28 - 143	0	30
Endrin Aldehyde	ND	ND	7.26	0 *	ND	7.26	0	* 18 - 135	0	30
Endrin Ketone	ND	ND	7.26	0 *	ND	7.26	0	* 57 - 123	0	30
Heptachlor	ND	ND	7.26	0 *	ND	7.26	0	* 35 - 127	0	30
Heptachlor Epoxide	ND	ND	7.26	0 *	ND	7.26	0	* 61 - 120	0	30
Hexachlorobenzene	6700	6760	18.2	380 #	6630	18.2	-380 #	* 20 - 150	2	30
Methoxychlor	ND	ND	36.3	0 *	ND	36.3	0	* 38 - 149	0	30
alpha-BHC	ND	ND	7.26	0 *	ND	7.26	0	* 53 - 130	0	30
alpha-Chlordane	ND	ND	7.26	0 *	ND	7.26	0	* 27 - 130	0	30
beta-BHC	ND	ND	7.26	0 *	ND	7.26	0	* 35 - 142	0	30
delta-BHC	ND	ND	7.26	0 *	ND	7.26	0	* 44 - 119	0	30
gamma-BHC (Lindane)	ND	ND	7.26	0 *	ND	7.26	0	* 37 - 124	0	30
gamma-Chlordane	ND	ND	7.26	0 *	ND	7.26	0	* 38 - 127	0	30

Comments: \_\_\_\_\_

**VALIDATION FINDINGS WORKSHEET**  
**Laboratory Control Samples**

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".  
 Were a laboratory control samples (LCS) and laboratory control sample duplicate (LCSD) analyzed for each matrix in this SDG?  
 Were the LCS percent recoveries (%R) and relative percent differences (RPD) within the QC limits?  
 Level:  Y  N  N/A  
 Y  N  N/A  
 Level:  Y  N  N/A Only

Was a LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed?

#	Date	LCS/LCSD ID	Compound	LCS %R (Limits)	LCS D %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		90348 LCS/D	R	48 (50-120)	( ) ( )	39 (30)	1-4, 90348 MB	No qual (LCSD in)
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		

**VALIDATION FINDINGS WORKSHEET**  
**Compound Quantitation and Reported CRQLs**

**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".  
**Level IV/D Only**

Y N (N/A) Were CRQLs adjusted for sample dilutions, dry weight factors, etc.?  
Y N (N/A) Did the reported results for detected target compounds agree within 10.0% of the recalculated results?

#	Sample ID Compound Name	Finding	Associated Samples	Qualifications
	<u>1, 5, 7</u>	<u>EE &gt; cal range</u>		<u>J acts / A (e)</u>

Comments: See sample calculation verification worksheet for recalculations

LDC #: 21495 K91  
 SDG #: Sea Green

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

Page: 1 of 1  
 Reviewer: JV6  
 2nd Reviewer: [Signature]

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

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
		<u>1, 5, 7</u>	<u>EE &gt; cal range</u>		<u>X/A</u>
		<u>2, 6, 8</u>	<u>All except EE di</u>		<u>(o)</u>

Comments:

LDC #: 21995K 3a  
 SDG #: Su Com

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

Page: 1 of 1  
 Reviewer: JVG  
 2nd reviewer: JVG

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

Y N N/A Were field duplicate pairs identified in this SDG?  
 Y N N/A Were target compounds detected in the field duplicate pairs?

Compound	Concentration (ug/kg)		RPD
	5	7	
J	47	48	1 ( $\leq 36$ Diff)
O	71	66	5 ↓
EE	2100	2100	0 ( $\leq 50$ 2 RPD)
B	120	100	18 ↓

Compound	Concentration (ug/kg)		RPD
	6	8	
EE	6700	7000	310 ( $\leq 1900$ Diff)

Compound	Concentration (ug/kg)		RPD
	13	14	
EE	2.3	3.0 U	0.7 ( $\leq 3.0$ Diff)

Compound	Concentration ( )		RPD

## Laboratory Data Consultants, Inc. Data Validation Report

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

**Collection Date:** July 6 through July 7, 2009

**LDC Report Date:** September 27, 2009

**Matrix:** Soil

**Parameters:** Chlorinated Pesticides

**Validation Level:** Stage 2B

**Laboratory:** Columbia Analytical Services, Inc.

**Sample Delivery Group (SDG):** R0903729

### Sample Identification

RSAK4-10B  
RSAK4-20B  
RSAK4-31B  
RSAL4-0.5B  
RSAL4009-0.5B  
RSAL4-10B  
RSAL4-28B



## Introduction

This data review covers 7 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.

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.
- UU 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.

## **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%.

## **V. Blanks**

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

Sample FB072109-SO (from SDG R0904016) was identified as a field blank. No chlorinated pesticide contaminants were found in this blank.

## **VI. Surrogate Spikes**

Surrogates were added to all samples and blanks as required by the method. Surrogate recoveries (%R) were not within QC limits for RSAK4-10B and RSAK4-20B . Since the samples were diluted out, no data were qualified.

**VII. Matrix Spike/Matrix Spike Duplicates**

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

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

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

**IX. Regional Quality Assurance and Quality Control**

Not applicable.

**X. 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**

Raw data were not reviewed for this SDG.

**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 R0903729	All compounds reported below the PQL.	J (all detects)	A

Raw data were not reviewed for this SDG.

**XIII. Overall Assessment of Data**

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

#### XIV. Field Duplicates

Samples RSAL4-0.5B and RSAL4009-0.5B were identified as field duplicates. No chlorinated pesticides were detected in any of the samples with the following exceptions:

Compound	Concentration (ug/Kg)		RPD (Limits)	Difference (Limits)	Flags	A or P
	RSAL4-0.5B	RSAL4009-0.5B				
Hexachlorobenzene	43	59	31 ( $\leq 50$ )	-	-	-
beta-BHC	4.8	18U	-	13.2 ( $\leq 18$ )	-	-

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Data Qualification Summary - SDG R0903729**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
R0903729	RSAK4-10B RSAK4-20B RSAK4-31B RSAL4-0.5B RSAL4009-0.5B RSAL4-10B RSAL4-28B	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 R0903729**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada  
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG R0903729**

No Sample Data Qualified in this SDG

## Tronox Northgate Henderson

### VALIDATION COMPLETENESS WORKSHEET

LDC #: 21495L3a

SDG #: R0903729

Laboratory: Columbia Analytical Services

Stage 2B

Date: 9/23/09

Page: 1 of 1

Reviewer: JLB

2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 7/06 - 07/09
II.	GC/ECD Instrument Performance Check	A	
III.	Initial calibration	A	
IV.	Continuing calibration/ICV	A	COV/ICV $\leq 20\%$
V.	Blanks	A	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	N	Client Spec
VIII.	Laboratory control samples	A	LCS 1/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 = 4.5
XV.	Field blanks	ND	FB = FB072109-50 from R0904016

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	RSAK4-10B	11	91091 MB	21	31
2	RSAK4-20B	12		22	32
3	RSAK4-31B	13		23	33
4	RSAL4-0.5B <span style="margin-left: 20px;">D</span>	14		24	34
5	RSAL4009-0.5B <span style="margin-left: 20px;">b</span>	15		25	35
6	RSAL4-10B	16		26	36
7	RSAL4-28B	17		27	37
8		18		28	38
9		19		29	39
10		20		30	40

# VALIDATION FINDINGS WORKSHEET

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

A. alpha-BHC	I. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	GG.
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH.
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	II.
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	JJ.
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. DB 608	KK.
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. DB 1701	LL.
G. Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	EE. <i>Hexachloro benzene</i>	MM.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	FF.	NN.

Notes:

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LDC #: 2149513a  
SDG #: See Cons

VALIDATION FINDINGS WORKSHEET  
Surrogate Spikes

Page: 1 of 1  
Reviewer: SVL  
2nd Reviewer: R

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".  
 Y  N  N/A  
 Y  N  N/A  
Were surrogates spiked into all samples, standards and blanks?  
Did all surrogate percent recoveries (%R) meet the QC limits?

#	Date	Sample ID	Column	Surrogate Compound	%R (Limits)	Qualifications
		1 (10x)	Not spec	B	158 (40-140)	No qual
		2 (10x)	↓	↓	153 ( )	↓

Letter Designation	Surrogate Compound	Recovery QC Limits (Soil)	Recovery QC Limits (Water)	Comments
A	Tetrachloro-m-xylene			
B	Decachlorobiphenyl			

LDC #: 21495 L36  
 SDG #: See Cover

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

Page: 1 of 1  
 Reviewer: SVG  
 2nd reviewer: [Signature]

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

Y N N/A  
 Y N N/A

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

Compound	Concentration ( <u>ug/kg</u> )		RPD
	4	5	
EE	43	59	31 (≤ 50% RPD)
B	4.8	18 U	13.2 (≤ 18 Diff)

Compound	Concentration ( )		RPD

Compound	Concentration ( )		RPD

Compound	Concentration ( )		RPD