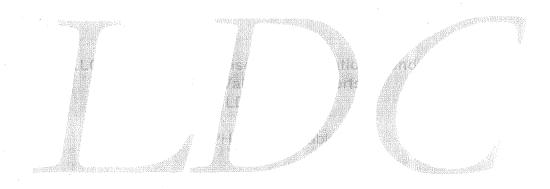
## Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Data Validation Reports LDC# 21257

TPH as Extractables



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

**Project/Site Name:** 

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

June 25 through June 26, 2008

LDC Report Date:

September 1, 2009

Matrix:

Soil

Parameters:

Total Petroleum Hydrocarbons as Extractables

Validation Level:

Stage 2B

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2844666

#### Sample Identification

SA87-0.5B

SA87-10B

SA87-20B

SA87-30B

SA87-25B

SA180-0.5B

SA180-0.5BD

SA180-10B

SA180-20B

SA180-30B

SA57-0.5B

SA57-0.3B SA57-10B

SA57-20B

SA57-30B

SA57-10BD

**SA87-10BMS** 

SA87-10BMSD

**SA180-10BMS** 

**SA180-10BMSD** 

#### 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 8015B for Total Petroleum Hydrocarbons (TPH) as Extractables.

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

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. Calibration

#### a. Initial Calibration

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

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

A curve fit, based on the initial calibration, was established for quantitation for selected compounds. The coefficient of determination  $(r^2)$  was greater than or equal to 0.990.

#### b. Calibration Verification

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

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

#### III. Blanks

Method blanks were reviewed for each matrix as applicable. No total petroleum hydrocarbons as extractable contaminants were found in the method blanks.

No field blanks were identified in this SDG.

#### IV. Accuracy and Precision Data

#### a. Surrogate Recovery

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

#### b. Matrix Spike/Matrix Spike Duplicates

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

#### c. Laboratory Control Samples

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

#### V. Target Compound Identification

Raw data were not reviewed for this SDG.

#### VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

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

Raw data were not reviewed for this SDG.

#### VII. System Performance

Raw data were not reviewed for this SDG.

#### VIII. Overall Assessment of Data

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

#### IX. Field Duplicates

Samples SA180-0.5B and SA180-0.5BD and samples SA57-10B and SA57-10BD were identified as field duplicates. No total petroleum hydrocarbons as extractables were detected in any of the samples.

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Data Qualification Summary - SDG R2844666

SDG	Sample	Compound	Flag	A or P	Reason
R2844666	SA87-0.5B SA87-10B SA87-20B SA87-30B SA87-25B SA180-0.5B SA180-0.5BD SA180-10B SA180-20B SA180-20B SA57-0.5B SA57-10B SA57-10B SA57-10B	All compounds reported below the PQL.	J (all detects)	Α	Project Quantitation Limit (sp)

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Laboratory Blank Data Qualification Summary - SDG R2844666

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Field Blank Data Qualification Summary - SDG R2844666

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

LDC #:_	21257C8	
SDG #:_	R2844666	

Stage 2B

Laboratory:	Columbia	Analytical	Services

Date: 8/14 / 6 9
Page: 1 of 1
Reviewer: 3/4
2nd Reviewer: 4

METHOD: GC TPH as Extractables (EPA SW 846 Method 8015B)

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6 /25 - 26 /6 8
lla.	Initial calibration	Å	2 RSD 6202 ry
IIb.	Calibration verification/ICV	A	100/ca £ 20 D
111.	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	Ä	
IVc.	Laboratory control samples	A	ics /D
V.	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	N	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	M	$D_{r} = 6.7$ $D_{r} = 12.15$
Χ.	Field blanks	ú	

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:

• anaa	ited Samples.	500			<u> </u>	
1	SA87-05B		11	SA57-0.5B	21	31
2	SA87-10B		12	SA57-10B D <sub>V</sub>	22	32
3	SA87-20B		13	SA57-20B	23	33
4	SA87-30B		14	SA57-30B	24	34
5	SA87-25B		15	SA57-10BD <b>0</b> √	25	35
6	SA180-0.5B	рı	16	SA87-10BMS	26	36
7	SA180-0.5BD	D,	17	SA87-10BMSD	27	37
8	SA180-10B		18	SA180-10BMS	28	38
9	SA180-20B		19	SA180-10BMSD	29	39
10	SA180-30B		20		30	40

Notes:			

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

June 30 through July 2, 2008

LDC Report Date:

August 18, 2009

Matrix:

Soil

Parameters:

Total Petroleum Hydrocarbons as Extractables

Validation Level:

Stage 4

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2844797

#### Sample Identification

SA207-0.5B

SA207-10B

SA207-20B

SA207-30B

SA207-40B

SA181-0.5B

SA181-10B

SA181-20B

SA181-30B

SA181-35B

SA207-30BMS

SA207-30BMSD

#### Introduction

This data review covers 12 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8015B for Total Petroleum Hydrocarbons (TPH) as Extractables.

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

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. Calibration

#### a. Initial Calibration

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

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

A curve fit, based on the initial calibration, was established for quantitation for selected compounds. The coefficient of determination  $(r^2)$  was greater than or equal to 0.990.

#### b. Calibration Verification

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

Date	Compound	%D	Affected Compound	Associated Samples	Flag	A or P
7/9/08	Oil range organics	27.6	Oil range organics	PBLK1	J+ (all detects)	А

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

#### III. Blanks

Method blanks were reviewed for each matrix as applicable. No total petroleum hydrocarbons as extractable contaminants were found in the method blanks.

No field blanks were identified in this SDG.

#### IV. Accuracy and Precision Data

#### a. Surrogate Recovery

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

#### b. Matrix Spike/Matrix Spike Duplicates

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

#### c. Laboratory Control Samples

Laboratory control samples were reviewed for each matrix as applicable. Although the LCS relative percent difference (RPD) was not within QC limits for diesel range organics, the LCS/D percent recoveries (%R) were within QC limits and no data were qualified.

#### V. Target Compound Identification

All target compound identifications were within validation criteria.

#### VI. Project Quantitation Limit

All compound quantitation and CRQLs were within validation criteria.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R2844797	All compounds reported below the PQL.	J (all detects)	А

#### VII. System Performance

The system performance was acceptable.

#### VIII. Overall Assessment of Data

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

#### IX. Field Duplicates

No field duplicates were identified in this SDG.

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Data Qualification Summary - SDG R2844797

SDG	Sample	Compound	Flag	A or P	Reason
R2844797	SA207-0.5B SA207-10B SA207-20B SA207-30B SA207-40B SA181-0.5B SA181-10B SA181-20B SA181-30B SA181-35B	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Laboratory Blank Data Qualification Summary - SDG R2844797

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Field Blank Data Qualification Summary - SDG R2844797

No Sample Data Qualified in this SDG`

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

	-			
SDG	#:	R284	44797	

LDC #: 21257E8

Stage 4

Laboratory: Columbia Analytical Services

Reviewer: 2nd Reviewer:

METHOD: GC TPH as Extractables (EPA SW 846 Method 8015B)

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

	Validation Area		Comments
l.	Technical holding times	A	Sampling dates: 6/20 - 7/62/08
lla.	Initial calibration	A	Sampling dates: $6/20 - 7/62/08$ $2 RSD \leq 20 D \qquad \Gamma^2$
llb.	Calibration verification/ICV	ς₩	ay/a ≤ 202
III.	Blanks	A	/
IVa.	Surrogate recovery	Ā	
IVb.	Matrix spike/Matrix spike duplicates	A	
IVc.	Laboratory control samples	SM	lcs/p
V.	Target compound identification	4	
VI.	Compound Quantitation and CRQLs	A	
VII.	System Performance	Δ	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N	
X.	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:

lia?

	3011			
1	SA207-0.5B	11 SA207-30BMS		31
2	SA207-10B	12   SA207-30BMSD	22 × PBUK2 (6/4)	32
3	SA207-20B	13	23	33
4 1	SA207-30B	14	24	34
<del>1</del> 5	SA207-40B	15	25	35
<b>6-2</b>	SA181-0.5B	16	26	36
7 >	SA181-10B	17	27	37
<u>- ۲</u>	SA181-20B	18	28	38
9	SA181-30B	19	29	39
10	SA181-35B	20	30	40

Notes:_	(4/12/19/X 14/19/)	
_	( Jan 1 gary may )	
	7	_

LDC #: 21257 E8 SDG #: See Cover

#### **VALIDATION FINDINGS CHECKLIST**

Page: 1 of 2
Reviewer: 76
2nd Reviewer: 1

Method: GC HPLC

Metnod: < GC HPLC				
Validation Area	Yes	No	NA	Findings/Comments
Ik-Technicaliholdingatimes 2				
All technical holding times were met.				
Cooler temperature criteria was met.				
i) Initial calibration (1)				
Did the laboratory perform a 5 point calibration prior to sample analysis?				
Were all percent relative standard deviations (%RSD) ≤ 20%?				
Was a curve fit used for evaluation?			. /	
Did the initial calibration meet the curve fit acceptance criteria of > 0.990?				
Were the RT windows properly established?				TOP TO SERVE
IV Continuing (cálibrátion):				
Was a continuing calibration analyzed daily?				
Were all percent differences (%D) ≤ 15%.0 or percent recoveries 85-115%?				
Were all the retention times within the acceptance windows?				
V≯Blanks				
Was a method blank associated with every sample in this SDG?	-			
Was a method blank analyzed for each matrix and concentration?	_			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
VI. Surrogate spikes				
Were all surrogate %R within the QC limits?		-		
If the percent recovery (%R) for one or more surrogates was out of QC limits, was				
a reanalysis performed to confirm samples with %R outside of criteria?				
VII Matrix spike/Matrix spike duplicates ************************************				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.				
Was a MS/MSD analyzed every 20 samples of each matrix?				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?		,		
VIII. Laboratory control samples			6 W 1 .	A Committee of the Comm
Was an LCS analyzed for this SDG?	/			
Was an LCS analyzed per extraction batch?	_			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?		/		
Were the performance evaluation (PE) samples within the acceptance limits?				

LDC#: 21257E8 SDG#: See Cover

#### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 2
Reviewer: 76
2nd Reviewer: 1

Validation Area	Yes	No	NA	Findings/Comments
X Taiget compound identification				
Were the retention times of reported detects within the RT windows?				
XI. Compound quantilation/GROLs				Market Control of
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII) System performances				ege to the second
System performance was found to be acceptable.				
XIII Overairassessment opdatā 14: 22 + 2 - 4: 2 - 4				
Overall assessment of data was found to be acceptable.				
XIV: Field duplicates 1.79				and the second s
Field duplicate pairs were identified in this SDG.			1	
Target compounds were detected in the field duplicates.			\	
XV-Field:blanks				
Field blanks were identified in this SDG.				
Target compounds were detected in the field blanks.				

SDG#: 2/257 E8

# VALIDATION FINDINGS WORKSHEET Continuing Calibration

Page: 1 of 1 Reviewer: 3VC

2nd Reviewer:

METHOD: \_\_GC\_\_ HPLC

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

Did the continuing calibration standards meet the %D / RPD validation criteria of <15.0%?

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

$\overline{}$	ভি	$\vdash$					<u> </u>																	=
Qualifications	J+404/A (																							
Associated Samples	PBURI																							
	-   ^	\ \ \	) [	(	)	\ \ 	)	)	) [	) [	)	)	)	)	)	)	)	)	)	)	)	)	)	
$\%D \mid RPD$ (Limit < 15:07( $\frac{4}{5}$ 20 $\lambda$ ) RT (limit)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	
%D / RPD (Limit < 15:0)																								
Compound	ORO (F)																		,					
Detector/ Column	28-5																							
Standard ID	AP347																							
Date	1/69/68																							
*																								

1DC # 21251 FB SDG # 54 Cone SDG#:

## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Reviewer:\_ Page: 2nd Reviewer:

METHOD: CGC HPLC

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

Y W 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 IV/D Only Y) N N/A Was an LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed?

			90 -		400-			
-	LCS/LCSD ID	Compound	%R (Limits)		LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
T	0'571 ·	pko	)		( )	( ct) ft	6-10, PBLKY	130 -020
$\exists$			`		( )	( ) /		(40/57)
				^	( )	)		
$\neg$			·	^	( )	)		
			•	^	( )	)		
			•	^	( )	)		
$\neg$				^	( )	)		
1			)					
一			)	^	(	( )		
$\exists$			)	î				
$\top$			· ·		( )	( )		
1				7		(		
$\top$			`	^	( )	( )		
$\top$			)		( )	( )		
十				^	( )	(		
1					(	,		
一			`		( )	(		
十			)		( )	( )		
1			)		( )	( )		
$\neg$			)	^	( )	( )		
十			)	_	( )	( )		
$\top$			,		( )	( )		
_			•	-	( )	( )		
1			,		(	(		

2125/E See Cover SDG #: LDC #:

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

1 of 1 Page: Reviewer: 2nd Reviewer:

> HPLC METHOD: GC\_

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

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

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

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound	CF (Shostd)	cF ( SPostd)	Average CF (initial)	Average CF (initial)	%RSD	%RSD
-	1521	20/21/2	DRO	1.19766	19766 1197468	1.184 26	1.184e6	196	3.60
				-					
2	15.41	1/1 1.4	+	1, 358 66 1,359.887	1.359887	1, 403 26	1. 403 26 1.403 ec	10.49	10,50
	<b>)</b>	24/08							
က							-		
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.

21 254 E8 See Cover LDC #: SDG #:

# Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

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

> HPLC METHOD: GC

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

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

Where: ave. CF = initial calibration average CF

CF = continuing calibration CF
A = Area of compound
C = Concentration of compound

					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc. CCV	%D	Q%
	COV 12	2/00/18	DRD	رمه	1094.392	1094. 548	4.4	4.4
							•	
					·			
7	CW 13	80/01/4		<b>\</b>	1017.885	1018,075	1.8	1.8
က	cev	7/14/62		2,000	1744	1744	12,8	12,8
		3						
							<b>*</b>	
4	733	7/10/64			2102	2012	9'0	
		3	<del>\</del>					

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.

2,19

262 269

CONCLC.18

LDC#: 21257 E-9

VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page: 2nd reviewer:

> METHOD: CG HPLC See Cover

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

<u>+</u> Sample ID:

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
0- Terpheny	28-5	100	66'08	80	80	۵
0 1						
		•				

(		
	9	Ì
	ĺ	
	Ċ	ĺ

Sample ID:						
Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	

Sample ID:

Ī				
	Percent Difference			
	Percent Recovery	Recalculated		
	Percent Recovery	Reported		
	Surrogate Found			
	Surrogate Spiked			
	Column/Detector			
Sample IU:	Surrogate			

LDC # 2/257 E8 SDG #: See Cover

# VALIDATION FINDINGS WORKSHEET

Matrix Spike/Matrix Spike Duplicates Results Verification

Page:\_\_

2nd Reviewer:\_\_ Reviewer:

> HPLC ပ္ပ METHOD:

The percent recoveries (%R) and relative percent differences (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using

RPD =(((SSCMS - SSCMSD) \* 2) / (SSCMS + SSCMSD))\*100 the following calculation: %Recovery = 100 \* (SSC - SC)/SA

MS/MSD samples:\_

SSC = Spiked sample concentration SA = Spike added MS = Matrix spike

SC = Sample concentration

MSD = Matrix spike duplicate

	Spike Added		Sample Conc.	Spike (	Spike Sample	Matri	Matrix spike	Matrix Spike Duplicate	9 Duplicate	QSW/SW	SD
Compound	143/4	K	( MS/R= )	(34)	E(	Percent	Percent Recovery	Percent Recovery	tecovery	RPD	٥
	MS	Msp.	0 -	MS	U MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recelo
Gasoline (8015)											
Diesel (8015)	28000 2	280 00C	ρ	(90 00)	20/ 200	87	8.9	72	7,	7	3
Benzene (8021B)						2				۵	١
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)											
Anthracene (8310)											
HMX (8330)											
2,4,6-Trinitrotoluene (8330)											
		Ī									

LDC #: 2/257 E8 SDG #: See Gover

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

Page: lof / Reviewer:\_\_\_

2nd Reviewer:

GC\_HPLC METHOD:

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

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

SC = Concentration

LCSD = Laboratory control sample duplicate percent recovery

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

LCS/LCSD samples:\_

	S	Spike	Spiked	Sample	רכ	SOT	רכ	CSD	/SOT	rcs/rcsp
Compound	(A)	1,450 (E.)	37 )	Concentration (124)	Percent l	Percent Recovery	Percent	Percent Recovery	~	RPD
	SOT	TCSD	SOT	C LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)										
Diesel (8015)	000 050	25000	220 000	210 000	88	8 8	84	84	5	7
Benzene (8021B)										
Methane (RSK-175)										
2,4-D (8151)										
Dinoseb (8151)										
Naphthalene (8310)										
Anthracene (8310)										
HMX (8330)										
2,4,6-Trinitrotoluene (8330)										
	-				-		-			

Comments: Refer to 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.

**SDC #: ス こま OSS** LDC#: 7 257 E8

# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page: 2nd Reviewer: Reviewer:

> CGC HPLC METHOD:

Y N N/A

Were all recalculated results for detected target compounds within 10% of the reported results? Were all reported results recalculated and verified for all level IV samples?

Concentration=

(A)(Fv)(Df)

(RF)(Vs or Ws)(%S/100)

A= Area or height of the compound to be measured

Fv≈ Final Volume of extract

RF= Average response factor of the compound Df= Dilution Factor

Vs= Initial volume of the sample Ws= Initial weight of the sample In the initial calibration

**%S= Percent Solid** 

Example:

Compound Name\_

#

Sample ID.\_

0 RD

(216 923 776) (1.016 e6)

Concentration =

2/3,5

final conc. = (213.5) (20 ml) (1ml) (1000)

(10.00) (5m1) (0.85)

100 471

Qualifications Recalculated Results Concentrations Concentrations Reported Compound Sample ID

Comments:	

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 7 through July 8, 2008

LDC Report Date:

August 18, 2009

Matrix:

Soil

Parameters:

Total Petroleum Hydrocarbons as Extractables

Validation Level:

Stage 2B

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2844862

#### Sample Identification

SA47-0.5B

SA47-0.5BRE

SA47-10B

SA47-20B

SA47-30B

SA47-35B

SA183-0.5B

RSAN2-0.5B

RSAN2-10B

RSAN2-20B

RSAN2-10BMS

RSAN2-10BMSD

#### Introduction

This data review covers 12 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8015B for Total Petroleum Hydrocarbons (TPH) as Extractables.

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

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. Calibration

#### a. Initial Calibration

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

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

#### b. Calibration Verification

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

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

#### III. Blanks

Method blanks were reviewed for each matrix as applicable. No total petroleum hydrocarbons as extractable contaminants were found in the method blanks.

No field blanks were identified in this SDG.

#### IV. Accuracy and Precision Data

#### a. Surrogate Recovery

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

Sample	Surrogate	%R (Limits)	Compound	Flag	A or P
SA47-0.5B	ortho-Terphenyl	47 (68-138)	TPH as extractables	J- (all detects) UJ (all non-detects)	Α
SA47-0.5BRE	ortho-Terphenyl	67 (68-138)	TPH as extractables	J- (all detects) UJ (all non-detects)	А

#### b. Matrix Spike/Matrix Spike Duplicates

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

#### c. Laboratory Control Samples

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

#### V. Target Compound Identification

Raw data were not reviewed for this SDG.

#### VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

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

Raw data were not reviewed for this SDG.

#### VII. System Performance

Raw data were not reviewed for this SDG.

#### VIII. 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
SA47-0.5BRE	TPH as extractables	×	А

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

#### IX. Field Duplicates

No field duplicates were identified in this SDG.

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Data Qualification Summary - SDG R2844862

SDG	Sample	Compound	Flag	A or P	Reason
R2844862	SA47-0.5B SA47-0.5BRE	TPH as extractables	J- (all detects) UJ (all non-detects)	А	Surrogate recovery (%R) (s)
R2844862	SA47-0.5B SA47-0.5BRE SA47-10B SA47-20B SA47-30B SA47-35B SA183-0.5B RSAN2-0.5B RSAN2-0.5B RSAN2-20B	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp#)
R2844862	SA47-0.5BRE	TPH as extractables	х	А	Overall assessment of data (o)

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Laboratory Blank Data Qualification Summary - SDG R2844862

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Field Blank Data Qualification Summary - SDG R2844862

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

LDC #:_	21257G8	 VALI
SDG #:_	R2844862	

Stage 2B

Laboratory: Columbia Analytical Services

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

METHOD: GC TPH as Extractables (EPA SW 846 Method 8015B)

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 7/67/08 - 7/08/08
IIa.	Initial calibration	A	2 GD = 20%
IIb.	Calibration verification/ICV	4	2 RCD = 20% CW/W = 20 %
111	Blanks	À	
IVa.	Surrogate recovery	SW	
IVb.	Matrix spike/Matrix spike duplicates	A	
IVc.	Laboratory control samples	A	US/p
V.	Target compound identification	N	,
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	N	
VIII.	Overall assessment of data	SW	
IX.	Field duplicates	N	
X.	Field blanks	7	

Note:

A = Acceptable

N = Not provided/applicable SW = See worksheet ND = No compounds detected

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

EB = Equipment blank

Validated Samples:

Soil

		' '					
1	SA47-0.5B	11	RSAN2-10BMS	21	PBULI	31	
2 7	SA47-0.5BRE	12	RSAN2-10BMSD	22 7	PB 162	32	
3	SA47-10B	13		23		33	
4	SA47-20B	14		24		34	
5	SA47-30B	15		25		35	
6	SA47-35B	16		26		36	
7	SA183-0.5B	17	, ,	27		37	
8	RSAN2-0.5B	18		28		38	*
9	RSAN2-10B	19		29		39	
10	RSAN2-20B	20		30	·	40	

Notes:				
_				

LDC#: 2125768 SDG #:

## VALIDATION FINDINDS WORKSHEET Surrogate Recovery

Page: Lof Reviewer.

METHOD: GC \_\_HPLC
Are surrogates required by the method? Yes \_\_ or No \_\_\_.
Rlease see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Were surrogates spiked into all samples and blanks? Y N N/A

Did all surrogate recoveries (%R) meet the QC limits?

#	Sample ID	Detector/ @Glumn	ctor/ mm	Surrogate Compound		%R (Limits)				Ö	Qualifications	
		2B-5	J	H		47	68-	(58)	4	/sn/	S) 4,	
								<u></u>		_		
	2	<b>\</b>				)		](				
						)						
								^				
					$\vdash$	)						
						)						
								^				
						)						
						)						
						)		) (				
								-				
						)						
						)		)				
						)		)				
						)		)				
						)		)				
						)		(				
	Surrogate Compound		Surrog	Surrogate Compound		Surrogate Compound		Surrogate Compound	punodwo			
4	Chlorobenzene (CBZ)	၅	ŏ	Octacosane	Σ	Benzo(e)Pyrene	s	1-Chloro-3-Nitrobenzene	itrobenzene	>	Tetrachloro-m- xylene	
60	4-Bromofluorobenzene (BFB)	Ŧ	ğ	Ortho-Terphenyl	z	Terphenyl-D14	-	3,4-Dinitratoluene	toluene			
٥	a,a,a-Trifluorotoluene	-	Fluore	Fluorobenzene (FBZ)	0	Decachlorobiphenyi (DCB)	Э	Tripentyltin	yltin			
a	Bromochiorobenene	4	-d	n-Triacontane	۵	1-methylnaphthalene	>	Tri-n-propyttin	povítin	1		
ш	1,4-Dichlorobutane	¥	I	Hexacosane	o	Dichlorophenyl Acetic Acid (DCAA)	3	Tributyl Phosphate	osphate			
Ē	1.4-Difluorobenzene (DFB)	_	Brc	Bromobenzene	R	4-Nitrophenol	I X	Triphenyl Phosphate	hosphate			

LDC # 21 25 7 68 SDG #: 54 Cm

# VALIDATION FINDINGS WORKSHEET Overall Assessment of Data

Page: Of Reviewer:

METHOD: \_\_GC \_\_HPLC

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?

L				
*	Jample ID Compound Name	Finding	Accordated Commiss	:
	7		Associated Samples	Ĕ  `
	8	contination Tun for sun	witstale lean to (in # 1)	への サ/×
Com	Comments:			

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 8 through July 9, 2008

LDC Report Date:

August 18, 2009

Matrix:

Soil

Parameters:

Total Petroleum Hydrocarbons as Extractables

Validation Level:

Stage 2B

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2844885

#### Sample Identification

RSAN2-30B

RSAN2-30BD

RSAN2-35B

RSAO2-0.5B

RSAO2-10B

**RSAO2-20B** 

RSAO2-20BD

**RSAO2-30B** 

RSAO2-33B

SA183-10B

SA183-10BD

SA183-20B

SA183-30B

SA183-33B

RSA04-0.5B

RSA04-10B

RSA04-20B

RSA04-30B

RSA04-36B

RSA04-36BMS

RSA04-36BMSD

#### Introduction

This data review covers 21 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8015B for Total Petroleum Hydrocarbons (TPH) as Extractables.

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

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. Calibration

#### a. Initial Calibration

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

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

A curve fit, based on the initial calibration, was established for quantitation for selected compounds. The coefficient of determination  $(r^2)$  was greater than or equal to 0.990.

#### b. Calibration Verification

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

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

#### III. Blanks

Method blanks were reviewed for each matrix as applicable. No total petroleum hydrocarbons as extractable contaminants were found in the method blanks.

No field blanks were identified in this SDG.

#### IV. Accuracy and Precision Data

#### a. Surrogate Recovery

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

#### b. Matrix Spike/Matrix Spike Duplicates

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

#### c. Laboratory Control Samples

Laboratory control samples were reviewed for each matrix as applicable. Although the LCSD percent recovery (%R) and relative percent difference (RPD) was not within QC limits for diesel range organics, the LCS percent recovery (%R) was within QC limits and no data were qualified.

#### V. Target Compound Identification

Raw data were not reviewed for this SDG.

#### VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

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

Raw data were not reviewed for this SDG.

#### VII. System Performance

Raw data were not reviewed for this SDG.

#### VIII. Overall Assessment of Data

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

#### IX. Field Duplicates

Samples RSAN2-30B and RSAN2-30BD, samples RSAO2-20B and RSAO2-20BD, and samples SA183-10B and SA183-10BD were identified as field duplicates. No total petroleum hydrocarbons as extractables were detected in any of the samples.

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Data Qualification Summary - SDG R2844885

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

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Laboratory Blank Data Qualification Summary - SDG R2844885

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Field Blank Data Qualification Summary - SDG R2844885

No Sample Data Qualified in this SDG

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

LDC #:_	2125/18
SDG #:	R2844885

Stage 2B

Laboratory: Columbia Analytical Services

Page: \ of Reviewer: 200 2nd Reviewer: 0

METHOD: GC TPH as Extractables (EPA SW 846 Method 8015B)

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 7/08- 09 /08
lla.	Initial calibration	A	3 RSD & 202 r
IIb.	Calibration verification/ICV	A	1 CV/CCV = 20 Z
111.	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	A	
IVc.	Laboratory control samples	SW	KS B
V	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	N	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	M	0, = 1.2 02 = 6,7 03 = 10,11
X.	Field blanks	N	

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected R = Rinsate

D = Duplicate

TB = Trip blank

FB = Field blank EB = Equipment blank

Validated Samples:

v anda	teu Gampies.	Sn')							
1 7	RSAN2-30B	DI	11	SA183-10BD 0,	21	RSA04-36BMSD	31	PBKI	7/16
2 1	RSAN2-30BD	DI	12	SA183-20B	22		32 7	PBLK2	7/3
3 1	RSAN2-35B		13	SA183-30B	23		33		
- <b>→</b>	RSAO2-0.5B		14	SA183-33B	24		34		
<b>1</b> 5	RSAO2-10B		+ » 15	RSA04-0.5B	25		35		
6	RSAO2-20B	$\mathcal{D}_{\gamma}$	16 1	RSA04-10B	26		36	•	
<b>→ y</b> 7	RSAO2-20BD	0√	17 1	RSA04-20B	27		37		
~ 2 8	RSAO2-30B		18	RSA04-30B	28		38		
19	RSAO2-33B		19 1	RSA04-36B	29		39		
10	SA183-10B	03	20	RSA04-36BMS	30		40		

Notes:	(no IW)	16/12	ICAL	WIW	)
_		7/24	IM	no la	<i>)</i>
		, , , , , ,		,	

100 # 2 | 257 IS SDG #:

# VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Page:

2nd Reviewer: Reviewer

METHOD: \_ GC \_ HPLC

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

X N/A N/A Were a laboratory control samples (LCS) and laboratory control sample duplicate (LCSD) analyzed for each matrix in this SDG?

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

Level IVID Only
Y. N. NIJA.) Was an LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed?

			SOT	9801			
	CS/LCSD ID	Compound	%R (Limits)	%R (Limits)	RPD (Limits)	Associated Samples	Qualifications
T	1 d/s)7	PRO	(	20	104 ( 30 )	9 2 9-11 14 16,17 19	N). m160
T			( )	( )	1 ~	90tk1	( 1, 83.)
			( )	(	( )		/ w 62
T			( )		( )		
$\neg$			( )	(	( )		
$\neg$			( )	( )	(		
			( )	)	)		
1			( )				
			( )	(	( )		
1							
$\neg$			•	( )	( )		
7							-
			( )	( )	(		
$\neg$			( )	( )			
$\neg$				(	( )		
1				( )			
_			( )	( )	( )		
			( )	( )	)		
_				( )	( )		
1			)	( )	( )		
$\neg$			( )	( )	( )		
_				( )	)		
1			( )	( )	(		
-1			(				

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 9 through July 10, 2008

LDC Report Date:

August 18, 2009

Matrix:

Soil

Parameters:

Total Petroleum Hydrocarbons as Extractables

Validation Level:

Stage 2B

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2844902

#### Sample Identification

SA46-0.5B

SA46-10B

SA46-20B

SA46-30B

SA46-30BD

SA48-0.5B

SA48-10B

SA48-20B

SA48-30B

SA48-35B

RSAJ7-0.5B

RSAJ7-10B

RSAJ7-20B

RSAK7-0.5B

RSAK7-10B

RSAK7-10BD

RSAK7-20B

RSAK7-27B

RSAK7-27BMS

RSAK7-27BMSD

#### Introduction

This data review covers 20 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8015B for Total Petroleum Hydrocarbons (TPH) as Extractables.

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

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. Calibration

#### a. Initial Calibration

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

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

#### b. Calibration Verification

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

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

#### III. Blanks

Method blanks were reviewed for each matrix as applicable. No total petroleum hydrocarbons as extractable contaminants were found in the method blanks.

No field blanks were identified in this SDG.

#### IV. Accuracy and Precision Data

#### a. Surrogate Recovery

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

#### b. Matrix Spike/Matrix Spike Duplicates

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

#### c. Laboratory Control Samples

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

#### V. Target Compound Identification

Raw data were not reviewed for this SDG.

#### VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R2844902	All compounds reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

#### VII. System Performance

Raw data were not reviewed for this SDG.

#### VIII. Overall Assessment of Data

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

#### IX. Field Duplicates

Samples SA46-30B and SA46-30BD and samples RSAK7-10B and RSAK7-10BD were identified as field duplicates. No total petroleum hydrocarbons as extractables were detected in any of the samples.

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Data Qualification Summary - SDG R2844902

SDG	Sample	Compound	Flag	A or P	Reason
R2844902	SA46-0.5B SA46-10B SA46-20B SA46-30BD SA46-30BD SA48-0.5B SA48-10B SA48-30B SA48-30B SA48-35B RSAJ7-10B RSAJ7-10B RSAJ7-10B RSAK7-10B RSAK7-10BD RSAK7-20B RSAK7-20B	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Laboratory Blank Data Qualification Summary - SDG R2844902

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Field Blank Data Qualification Summary - SDG R2844902

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

LDC #: 21257J8 SDG #: R2844902

Stage 2B

Laboratory: Columbia Analytical Services

Date:	0/14/0
Page:	of /
Reviewer:	347
2nd Reviewer:	9

METHOD: GC TPH as Extractables (EPA SW 846 Method 8015B)

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

	Validation Area		Comments
l.	Technical holding times	A	Sampling dates: 7/09 - 10 /08
lla.	Initial calibration	4	2 RSD € >0 2
IIb.	Calibration verification/ICV	A	2. RSD 6 20 2.
111.	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	A	
IVc.	Laboratory control samples	A	us/b
V.	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	N	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	ND	$D_{r} = 4.5$ $D_{r} = 15.16$
X.	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	SA46-0.5B	† 11 RSAJ7-0.5B	21 1 +BIKI	31
2	SA46-10B	12 RSAJ7-10B	22 V PBKY	32
3 <b>&gt;</b>	SA46-20B	13 RSAJ7-20B	23	33
<u>-</u> ٧	SA46-30B D	14 RSAK7-0.5B	24	34
5	SA46-30BD <b>b</b>	15 RSAK7-10B	25	35
6	SA48-0.5B	16 RSAK7-10BD <b>*</b>	26	36
7	SA48-10B	17 RSAK7-20B	27	37
8	SA48-20B	18 RSAK7-27B	28	38
9	SA48-30B	19 RSAK7-27BMS	29	39
10	SA48-35B	20 RSAK7-27BMSD	30	40

Notes:	3:		
-			

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

**Project/Site Name:** 

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 10 through July 11, 2008

LDC Report Date:

August 20, 2009

Matrix:

Soil

Parameters:

Total Petroleum Hydrocarbons as Extractables

Validation Level:

Stage 2B

RSAL2-0.5BMSD

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2844922

#### Sample Identification

RSAJ8-0.5B RSAK2-30B RSAJ8-10B RSAK2-35B RSAJ8-20B RSA17-32B RSAJ8-30B RSAJ8-20BMS RSAJ8-33B RSAJ8-20BMSD RSAJ7-0.5B RSAL2-0.5BMS

RSAI7-10B RSAI7-20B

RSAI7-30B

RSAL2-0.5B

RSAL2-10B

RSAL2-20B

RSAL2-20BD

RSAL2-30B

RSAL2-37B

RSAL2-40B

RSAK2-0.5B

RSAK2-10B

RSAK2-20B

RSAK2-20BD

#### Introduction

This data review covers 27 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8015B for Total Petroleum Hydrocarbons (TPH) as Extractables.

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

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

All samples were received in good condition with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P
RSAJ8-0.5B RSAJ8-30B	TPH as extractables	All jars possibly contaminated from water in the cooler. Water present in jars.	There should be no water in the sample containers.	J- (all detects) UJ (all non-detects)	А

One out of 3 jars possibly contaminated from water in the cooler. Water present in jars for samples RSAJ8-33B and RSAK2-0.5B.

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

#### II. Calibration

#### a. Initial Calibration

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

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

#### b. Calibration Verification

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

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

#### III. Blanks

Method blanks were reviewed for each matrix as applicable. No total petroleum hydrocarbons as extractable contaminants were found in the method blanks.

No field blanks were identified in this SDG.

#### IV. Accuracy and Precision Data

#### a. Surrogate Recovery

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

#### b. Matrix Spike/Matrix Spike Duplicates

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

#### c. Laboratory Control Samples

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

#### V. Target Compound Identification

Raw data were not reviewed for this SDG.

#### VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R2844922	All compounds reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

#### VII. System Performance

Raw data were not reviewed for this SDG.

#### VIII. Overall Assessment of Data

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

#### IX. Field Duplicates

Samples RSAL2-20B and RSAL2-20BD and samples RSAK2-20B and RSAK2-20BD were identified as field duplicates. No total petroleum hydrocarbons as extractables were detected in any of the samples.

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Data Qualification Summary - SDG R2844922

SDG	Sample	Compound	Flag	A or P	Reason
R2844922	RSAJ8-0.5B RSAJ8-30B	TPH as extractables	J- (all detects) UJ (all non-detects)	A	Sample condition (p)
R2844922	RSAJ8-0.5B RSAJ8-10B RSAJ8-20B RSAJ8-30B RSAJ8-33B RSAI7-0.5B RSAI7-20B RSAI7-30B RSAL2-10B RSAL2-10B RSAL2-20BD RSAL2-20BD RSAL2-30B RSAL2-30B RSAL2-37B RSAL2-37B RSAL2-40B RSAK2-10B RSAK2-10B RSAK2-10B RSAK2-20BD RSAK2-20BD RSAK2-20BD RSAK2-30B RSAK2-30B RSAK2-30B RSAK2-30B RSAK2-35B RSAK2-35B	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Laboratory Blank Data Qualification Summary - SDG R2844922

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Field Blank Data Qualification Summary - SDG R2844922

No Sample Data Qualified in this SDG

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

LDC #:_	2125/K8
SDG #:	R2844922

100 " 04057140

Stage 2B

Laboratory: Columbia Analytical Services

Date: 8/14/09 Page: \ of Reviewer: 2nd Reviewer: (

METHOD: GC TPH as Extractables (EPA SW 846 Method 8015B)

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

	Validation Area		Comments
l.	Technical holding times	SW	Sampling dates: 7/10 - 11 /0 8
lla.	Initial calibration	A	70 RSD 620 Z
IIb.	Calibration verification/ICV	A	20 RSD = 20 Z CCV/ICV = 20 Z
111.	Blanks	A	,
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	A	
IVc.	Laboratory control samples	A	us /b
V	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	N	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	M	$b_1 = 12.13$ $D_2 = 19, 20$
X.	Field blanks	Ŋ	1

Note:

A = Acceptable

N = Not provided/applicable SW = See worksheet

ND = No compounds detected

R = Rinsate FB = Field blank D = Duplicate

TB = Trip blank

EB = Equipment blank

Validated Samples:

500 RSAL2-10B 21 4 RSAK2-30B **3**个 PBIRI RSAJ8-0.5B 11 12 D PBKY 22 4 RSAK2-35B 32 RSAJ8-10B RSAL2-20B 33 D 9B 1 1 3 23 7 RSA17-32B RSAL2-20BD RSAJ8-20B 13 PBW4 RSAL2-30B 24 -RSAJ8-20BMS RSAJ8-30B 14 34 15 **4** RSAL2-37B RSAJ8-33B 25 RSAJ8-20BMSD 35 16 RSAL2-40B RSAI7-0.5B 26 RSAL2-0.5BMS 36 17 4 RSAK2-0.5B 27 RSAL2-0.5BMSD 37 RSAI7-10B <sub>18</sub>7 RSAK2-10B 28 38 8 RSAI7-20B p RSAK2-20B RSAI7-30B 29 39 D 10 / RSAL2-0.5B 20 RSAK2-20BD 30 40

Notes:_	 			 

LDC#: 21257 Kg SDG #:

# **VALIDATION FINDINGS WORKSHEET Technical Holding Times**

Page: 2nd Reviewer: Reviewer.

All circled dates have exceeded the technical holding times.

Sample D  Matrix Preserved Sampling Date  Extraction date  Analysis date  Total Sampling Date  Analysis date  Analysis date  Consider  Analysis date  Consider  Analysis date  Consider  Analysis date  Consider  Analysis date  Analysis date  Consider  Analysis date  Consider  Analysis date  A	METHOD/	METHOD / GC HPLC	٦٢c							
4 All jars possibly containinated from water in certer 5-145/4 ( 17 One out of 3 jars possibly containinated with from 17 one out of 3 jars possibly containinated with from 18 water in The Corter water present in jars	Sample ID	Matrix	Preserved	Sami	oling Date	Extraction date	Analysis date	Total # of Days	Qualifier	
17 one out of 5 jars possibly contaminated with from water in The Corter Water present in jars	4			6161y	contamin	rated from wa	٠٤.		J-/45/A	(a)
17 one out of 3 jars possibly contaminated with from water in The Corter Water present in jars		<b>*</b>		erent	2 . 2					(
17 One out of & jars possibly contaminated with from  water in The Corter Water present in jars			,		r					
17 one out of 3 jars possibly contaminated with from  water in jars  Mater present in jars										
in The Corter. Water pre	_	one o	مر	_	possibly	contaminated	my the		Text	
		True	SZ		Corter	Water pres	ant in lars			
				,			-			
	,									

# TECHNICAL HOLDING TIME CRITERIA VOLATILES: Water unpreserved: A

Aromatic within 7 days, non-aromatic within 14 days of sample collection.

Both within 14 days of sample collection. Both within 14 days of sample collection. Water preserved:

Soils: **EXTRACTABLES:** 

Water: Soil:

Extracted within 7 days, analyzed within 40 days. Extracted within 14 days, analyzed within 40 days.

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

Project/Site Name:

Tronox LLC Facility, 2008 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 11, 2008

LDC Report Date:

August 27, 2009

Matrix:

Soil

Parameters:

Total Petroleum Hydrocarbons as Extractables

Validation Level:

Stage 2B

Laboratory:

Columbia Analytical Services, Inc.

Sample Delivery Group (SDG): R2845025

Sample Identification

RSAI7-10B(119156) RSAI7-10B(119157)

#### Introduction

This data review covers 2 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8015B for Total Petroleum Hydrocarbons (TPH) as Extractables.

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

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met with the following exceptions:

Sample	Compound	Total Days From Sample Collection Until SPLP Extraction	Required Holding Time (in Days) From Sample Collection Until SPLP Extraction	Flag	A or P
All samples in SDG R2845025	TPH as extractables	17	14	J- (all detects) UJ (all non-detects)	Р

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

#### II. Calibration

#### a. Initial Calibration

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

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

#### b. Calibration Verification

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

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

#### III. Blanks

Method blanks were reviewed for each matrix as applicable. No total petroleum hydrocarbons as extractable contaminants were found in the method blanks.

No field blanks were identified in this SDG.

#### IV. Accuracy and Precision Data

#### a. Surrogate Recovery

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

#### b. Matrix Spike/Matrix Spike Duplicates

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.

#### c. Laboratory Control Samples

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

#### V. Target Compound Identification

Raw data were not reviewed for this SDG.

#### VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG R2845025	All compounds reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

#### VII. System Performance

Raw data were not reviewed for this SDG.

#### VIII. Overall Assessment of Data

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

#### IX. Field Duplicates

No field duplicates were identified in this SDG.

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Data Qualification Summary - SDG R2845025

SDG	Sample	Compound	Flag	A or P	Reason
R2845025	RSAI7-10B(119156) RSAI7-10B(119157)	TPH as extractables	J- (all detects) UJ (all non-detects)	Р	Technical holding times (h)
R2845025	RSAI7-10B(119156) RSAI7-10B(119157)	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (sp)

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Laboratory Blank Data Qualification Summary - SDG R2845025

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2008 Phase B Investigation, Henderson, Nevada Total Petroleum Hydrocarbons as Extractables - Field Blank Data Qualification Summary - SDG R2845025

No Sample Data Qualified in this SDG

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

LDC #:_	21257L8
SDG #:_	R2845025

Stage 2B

ahara	+~	'n			بات	imi	~i~	Λn	abo	tion	Cor	1000	_
₋abora	ιι	и ,	у.	V	UIL	<u> </u>	JIA	MIII	aly	<u>livai</u>	<u> </u>	VICE	<u> </u>

Page: 1 of 1 Reviewer: 2nd Reviewer:

METHOD: GC TPH as Extractables (EPA SW 846 Method 8015B)

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

	Validation Area		Comments
1,	Technical holding times	SW	Sampling dates: 7/n /o g
lla.	Initial calibration	A	Sampling dates: 7/11 /0 %  R K D ニンシ
Ilb.	Calibration verification/ICV	A	cen
III.	Blanks	A	
IVa.	Surrogate recovery	Ä	
IVb.	Matrix spike/Matrix spike duplicates	N	Ment spec
IVc.	Laboratory control samples	A	Count spec
V.	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	N	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N	
X.	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	RSAI7-10B(119156)	11	21	31	
2	RSAI7-10B(119157)	12	22	32	
3	PB261	13	23	33	
4	,	14	24	34	
5		15	25	35	
6		16	26	36	
7		17	. 27	37	
8		18	28	38	
9		19	29	39	
10		20	30	40	

Notes:_	SPLP			

LDC#: 2|257 L & SDG #: \_ Sa Con

# **VALIDATION FINDINGS WORKSHEET Technical Holding Times**

Page: Reviewer. 2nd Reviewer:

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

METHOD	METHOD GC HPLC	J.C					
Sample ID	Matrix	Preserved	Sampling Date	SPLP Extraction date	Analysis date	Total # of Days	Qualifler
2 1	S	×	7/11/08	80/80/1	8/64/08	17	J-/41/p (h)
•				,			
		-					
					-		
·							
•							

# **TECHNICAL HOLDING TIME CRITERIA**

Aromatic within 7 days, non-aromatic within 14 days of sample collection. Both within 14 days of sample collection. Both within 14 days of sample collection. Water unpreserved: VOLATILES:

Water preserved: Soils:

EXTRACTABLES:

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

HTNew.wpd