LDC Report# 23104A2b

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2010 Parcels, Henderson, Nevada

Collection Date:

April 6, 2010

LDC Report Date:

June 7, 2010

Matrix:

Soil/Water

Parameters:

Polynuclear Aromatic Hydrocarbons

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 280-2143-1

Sample Identification

Q3-PF-3-1-0.0**
Q3-PF-3-1-0.0FD
FB-PARCELS-032910
EB-PARCELS-032910
Q3-PF-3-1-0.0MS
Q3-PF-3-1-0.0MSD

^{**}Indicates sample underwent Stage 4 review

Introduction

This data review covers 4 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 8270C for Polynuclear Aromatic Hydrocarbons.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

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

I. Technical Holding Times

All technical holding time requirements were met.

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

II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

III. Initial Calibration

Initial calibration was performed using required standard concentrations.

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

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

IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

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

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

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

V. Blanks

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

Sample EB-PARCELS-032910 was identified as an equipment blank. No polynuclear aromatic hydrocarbon contaminants were found in this blank.

Sample FB-PARCELS-032910 was identified as a field blank. No polynuclear aromatic hydrocarbon 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 sample Q3-PF-3-1-0.0FD. Since only one surrogate was out for the acid compounds surrogates, no data were qualified. All other surrogate recoveries (%R) were within QC limits.

*Added text.

VII. Matrix Spike/Matrix Spike Duplicates

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

VIII. Laboratory Control Samples (LCS)

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

IX. Regional Quality Assurance and Quality Control

Not applicable.

X. Internal Standards

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

Sample	Internal Standards	Area (Limits)	Compound	Flag	A or P
Q3-PF-3-1-0.0**	Perylene-d12	374011 (820545-3282178)	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) R (all non-detects)	A
Q3-PF-3-1-0.0FD	Perylene-d12	357940 (820545-3282178)	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) R (all non-detects)	A

XI. Target Compound Identifications

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

*XII. Project Quantitation Limit

All project quantitation limits were within validation criteria for samples on which a Stage 4 review was performed with the following exceptions:

Sample	Compound	Finding	Flag	A or P
Q3-PF-3-1-0.0FD	Benzo(b)fluoranthene	Due to lack of resolution between these compounds in the samples, the laboratory	J (all detects) UJ (all non-detects)	Р
	Benzo(k)fluoranthene	performed the quantitation using the total peak area.	J (all detects) UJ (all non-detects)	

^{*}Added peak resolution qualification table.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 280-2143-1	All compounds reported below the PQL.	J (all detects)	А

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

XIII. Tentatively Identified Compounds (TICs)

Tentatively identified compounds were not reported by the laboratory.

XIV. System Performance

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

XV. Overall Assessment

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

XVI. Field Duplicates

Samples Q3-PF-3-1-0.0** and Q3-PF-3-1-0.0FD were identified as field duplicates. No polynuclear aromatic hydrocarbons were detected in any of the samples with the following exceptions:

	Concentrat	ion (ug/Kg)	, ppp	Difference		
Compound	Q3-PF-3-1-0.0**	Q3-PF-3-1-0.0FD	RPD (Limits)	(Limits)	Flags	A or P
Phenanthrene	370U	18	-	352 (≤370)	-	-
Pyrene	15	34	-	19 (≤370)	•	-
Benzo(b)fluoranthene	370U	110	-	260 (≤370)	-	-
Chrysene	370U	29	-	341 (≤370)	-	-
Fluoranthene	370U	49	_	321 (≤370)	-	-

*Tronox LLC Facility, 2010 Parcels, Henderson, Nevada Polynuclear Aromatic Hydrocarbons - Data Qualification Summary - SDG 280-2143-1

SDG	Sample	Compound	Flag	A or P	Reason (Code)
280-2143-1	Q3-PF-3-1-0.0** Q3-PF-3-1-0.0FD	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) R (all non-detects)	A	Internal standards (area) (i)
*280-2143-1	Q3-PF-3-1-0.0FD	Benzo(b)fluoranthene Benzo(k)fluoranthene	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Project Quantitation Limit (peak resolution) (o)
280-2143-1	Q3-PF-3-1-0.0** Q3-PF-3-1-0.0FD FB-PARCELS-032910 EB-PARCELS-032910	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (sp)

Tronox LLC Facility, 2010 Parcels, Henderson, Nevada Polynuclear Aromatic Hydrocarbons - Laboratory Blank Data Qualification Summary - SDG 280-2143-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2010 Parcels, Henderson, Nevada Polynuclear Aromatic Hydrocarbons - Equipment Blank Data Qualification Summary - SDG 280-2143-1

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2010 Parcels, Henderson, Nevada
Polynuclear Aromatic Hydrocarbons - Field Blank Data Qualification Summary - SDG
280-2143-1

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

LDC #:	23104A2 b	VALIDATION COMPLETENESS WORKSHEET
SDG #:	280-2143-1	Stage 2B/4
Laborator	ry: Test America	

Reviewer: 34 2nd Reviewer:

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

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 4 /6 6 /10
II.	GC/MS Instrument performance check	A	
111.	Initial calibration	A	J° K2D
IV.	Continuing calibration/ICV	A	CON /OU = 25]
V.	Blanks	A	÷
VI.	Surrogate spikes	SM	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	Α	ICS
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	WZ	
XI.	Target compound identification	A	Not reviewed for Stage 2B validation.
XII.	Compound quantitation/CRQLs	SW	Not reviewed for Stage 2B validation.
XIII.	Tentatively identified compounds (TICs)	N	Not reviewed for Stage 2B validation.
XIV.	System performance	A	Not reviewed for Stage 2B validation.
XV.	Overall assessment of data	A	
XVI.	Field duplicates	SW	D = 1, 7
XVII.	Field blanks	1,70	FB = 3 EB = 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: ** Indicates sample underwent State 4 validation

1	Q3-PF-3-1-0.0**	S	11	MB280-102521/1-A	21	31
2	Q3-PF-3-1-0.0FD		12	MB280-10253/1-A	22	32
3	FB-PARCELS-032910	W	13		23	33
1 7	EB-PARCELS-032910		14		24	34
5	Q3-PF-3-1-0.0MS	5	15		25	35
6	Q3-PF-3-1-0.0MSD		16		26	36
7			17		27	37
8			18		28	38
9			19		29	39
10			20		30	40

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
Reviewer: 10
2nd Reviewer: 10

Method: Semivolatiles (EPA SW 846 Method 8270C)

metriou. Serrivolatiles (EPA SVV 846 Metriou 8270C)	Ι.,	Γ	Ī	
Validation Area	Yes	No	NA	Findings/Comments
If Technical hading times All technical holding times were met.				
Cooler temperature criteria was met.				
U.G.S.M.S. (INSTRUMENTATION OF THE CONTROL OF THE C				
Were the DFTPP performance results reviewed and found to be within the specified criteria?	/			
Were all samples analyzed within the 12 hour clock criteria?			20 July 2011	
ation final realism and the second se				
Did the laboratory perform a 5 point calibration prior to sample analysis?	_			
Were all percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?				
Was a curve fit used for evaluation?				
Did the initial calibration meet the curve fit acceptance criteria of ≥ 0.990?				
Were all percent relative standard deviations (%RSD) \leq 30% and relative response factors (RRF) \geq 0.05?		_		
W. Communal Salibration				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?		-		
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?	/			
Were all percent differences (%D) ≤ 25% and relative response factors (RRF) ≥ 0.05?				·
Ye Bangs				
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.			-	
94 Starte 1308:				
Were all surrogate %R within QC limits?	***			
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?			M	A
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?				<u> </u>
Million and the Control of the Contr				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.				
Was a MS/MSD analyzed every 20 samples of each matrix?				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
Was an LCS analyzed for this SDG?	1			

LDC #: 23104 A26 SDG #: See Cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2 Reviewer: 3V6 2nd Reviewer: 1

Validation Area	Yes	. No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	/			
X (Regional Quality A surgice salet sugility Control 19				
Were performance evaluation (PE) samples performed?		_		
Were the performance evaluation (PE) samples within the acceptance limits?	14-27-T-2	75 A (M 1 V 2 2 2		
X liniánal státutants				
Were internal standard area counts within -50% or +100% of the associated calibration standard?	y AK	/		
Were retention times within ± 30 seconds from the associated calibration standard?		<u> </u>		
XI Farger compound then in cation 1. 20 Mar 18 May				
Were relative retention times (RRT's) within ± 0.06 RRT units of the standard?	/		L	
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	/		<u> </u>	
Were chromatogram peaks verified and accounted for?		11200010000	1 3 DK 2 DK 3	
Necomposition and the second s				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?		/		
XIII. Tenianian stantilang ropi sugas (IICs) ili 1903 ili				
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?	20.23.00.01		/	d St. 3 for the second
Were relative intensities of the major ions within ± 20% between the sample and the reference spectra?			/	
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?			-	
			SAMO SAMO	
AV/System detromates are as a second				
System performance was found to be acceptable.				
Metrine established				
Overall assessment of data was found to be acceptable.			(0.100)	
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.		·		
XVII.GERU ERIKA				
Field blanks were identified in this SDG.			_	
Target compounds were detected in the field blanks.				
Larger without his were detected in the neid blattics.			<u> </u>	

VALIDATION FINDINGS WORKSHEET

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

A Phenol**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenol**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	VV. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Anline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyi-phenyi ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol™	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	тт.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	ກດກ
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	w.
0. 2,4-Dimethytphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	WWW.

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

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VALIDATION FINDINGS WORKSHEET

Surrogate Recovery

Page:

2nd Reviewer: Reviewer:_

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

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

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

	# Date	Samule			
() ()		Campie ID	Surrogate	%R (Limits)	Qualifications
		7	N82	47 (52.12	No said
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S1 (NBZ) = Nitrobenzene-45 S2 (FBP) = 2-Fluorobipheny 3(S3 (TPH) = Terphenyl-d14 16 S4 (PHL) = Phenol-45 22

QC Limits (Water) 21-100

33-110*

20-130*

20-130*

OC Limits (Soil) 25-121 19-122

S5 (2FP)= 2-Fluorophenol S6 (TBP) = 2,4,6-Tribromophenol S7 (2CP) = 2-Chlorophenol-d4 S8 (DCB) = 1,2-Dichlorobenzene-d4

35-114 43-116 33-141 10-94

OC Limits (Soil) 23-120 1 30-115 18-137 24-113

16-110* 10-123

SUR.2S.wpd

LDC #: 23 10 4 4 26 SDG #:

VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Page: Reviewer:_ 2nd Reviewer:__

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

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

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

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?

				\n	Con			
*	Date	MS/MSD ID	Compound	%R (Limits)	%R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		9/5	666	347 (52420)	447 (52-120)	()		No mad (lism
		,	HHH	320 (54-120)	363 64-120)	()		
			111	157 (51-120)	(021-25) 161	()		
			KKK	256 (55-120)	289 (55-120)	()		-
			755	(°C1-h5) 94	()	()		(MSp in)
				()				
				()	()	()		
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				()	()	()		

	Compound	QC Limits (Soil)	RPD (Soil)	QC Limits (Water)	RPD (Water)		Compound	QC Limits (Soil)	RPD (Soil)	QC Limits (Water)	RPD (Water)
Ä	Phenol	26-90%	< 35%	12-110%	< 42%	99	Acenaphthene	31-137%	< 19%	46-118%	< 31%
ن	2-Chlorophenol	25-102%	< 50%	27-123%	< 40%	=	4-Nitrophenol	11-114%	< 50%	10-80%	< 50%
ш	1,4-Dichlorobenzene	28-104%	< 27%	36-97%	< 28%	<u> </u>	2,4-Dinitrotoluene	28-89%	< 47%	24-96%	< 38%
J.	N-Nitroso-di-n-propylamine	41-126%	< 38%	41-116%	< 38%	Ë.	Pentachlorophenol	17-109%	< 47%	9-103%	< 50%
œ	1,2,4-Trichlorobenzene	38-107%	< 23%	39-98%	< 28%	22.	Pyrene	35-142%	× 36%	26-127%	< 31%
>	4-Chloro-3-methylphenol	26-103%	< 33%	23-97%	< 42%						

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VALIDATION FINDINGS WORKSHEET Internal Standards

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

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

Were all internal standard area counts within -50 to +100 of the associated calibration standard? Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". X VA

3 333 KKK LLL ままエ Qualifications 999 No ouch ď Were the retention times of the internal standards within +/- 30 seconds of the retention times of the associated calibration standard? nel RT (Limits) 3 282 178 820545-Area (Limits) 292305 751868 374 011 357940 Internal Standard PRY アダイ Sample ID P Date Y(N)N/A # 27 32.50

* QC limits are advisory

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

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

LDC#: 27/04/A26 SDG#: 100 Com

VALIDATION FINDINGS WORKSHEET Compound Quantitation and CRQLs

Reviewer:

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

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

Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation? Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound? N N/A N/A

Qualifications	J/WJ/P										
Associated Samples	~		/								
Finding	669 HHH were us	(co-eluted									
Sample ID	7										
Date											
#											

Comments: See sample calculation verification worksheet for recalculations

LDC#: 23104A2b SDG#:See cover

VALIDATION FINDINGS WORKSHEET

Field Duplicates

Page:	<u> </u>
Reviewer:	376
2nd Reviewer:	1

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

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

	Conc (t	ıg/Kg)	RPD	Diff	Diff Limits	Quals
Compound Name	1 .	2	(≤50%)			(Parent Only)
Phenanthrene	370U	18		352	≤370	
Pyrene	15	34		19	≤370	,
Benzo(b)fluoranthene	370∪	110		260	≤370	
Chrysene	370U	29		341	≤370	
Fluoranthene	370U	49		321	≤370	

V:\FIELD DUPLICATES\23104A2b.wpd

LDC#: 73104 A36 SDG#:

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer: Reviewer:

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

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

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

 A_x = Area of Compound C_x = Concentration of compound,

A_{is} = Area of associated internal standard C_{is} = Concentration of internal standard X = Mean of the RRFs

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

#

S= Standard deviation of the RRFs,

			Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
	Calibration		RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
Standard ID	Date	Compound (Internal Standard)	(50 std)	(50 std)	(Initial)	(Initial)		
ICAL	3/18/10	Naphthalene (IS2)	1.1221	1.1221	1.1134	1.1134	3.0	3.0
MSSD		Fluorene (IS3)	1.3445	1.3445	1.3225	1.3225	5.0	5.0
	1	Phenanthrene (IS4)	1.1670	1.1670	1.1292	1.1292	6.7	6.7
		Chrysene (IS5)	1.0697	1.0697	1.0065	1.0065	8.2	8.2
		Benzo(a)pyrene (IS6)	1.1044	1.1044	1.0885	1.0885	6.7	6.7

		٩
Conc IS/Cpd	Area cpd	Area IS
40/50	1966829	1402196
40/50	1609531	957696
40/50	2338313	1602894
40/50	2492854	1864283
40/20	2472315	1790944

1					
Conc	Naphthalene	Fluorene	Phenanth	Chrysene	Benzo(a)py
4.00	1.1382	1.2049	1.2145	1.0249	0.9762
10.00	1.1444	1.2482	1.1130	1.0017	1.0038
20.00	1.1266	1.2972	1.1499	1.0484	1.0453
50.00	1.1221	1.3445	1.1670	1.0697	1.1044
80.00	1.0774	1.3440	1.1699	1.0741	1.1019
120.00	1.1406	1.3805	1.1804	1.0559	1.1505
160.00	1.1068	1.3878	1.0504	0.9457	1.1705
200.00	1.0507	1.3725	0.9882	0.8319	1.1554
×	1.1134	1.3225	1.1292	1.0065	1.0885
S	0.0335	0.0666	0.0754	0.0822	0.0728

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 # 29104 Ast SDG # See Cover

Continuing Calibration Results Verification VALIDATION FINDINGS WORSHEET

Page 1 of 1 Reviewer.

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

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

Where:

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

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

Ais = Area of associated internal standard Cx = Concentration of compound

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

Ax = Area of compound

Cis = Concentration of internal standard

# Standard ID Date Compound (Ref IS) (Initial RRF) (CC RRF) (CC RRF) %D			Calibration		Average RRF	Reported	Recalculated	Reported	Recalculated
D3873 4/12/10 Naphthalene (IS2) 1.113 1.176 1.176 5.6 Image: Example of the control	#	Standard ID	Date	Compound (Ref IS)	(Initial RRF)	(CC RRF)	(CC RRF)	%D	₩D
Fluorene (IS3) 1.323 1.380 1.380 4.3 Honanthrene (IS4) 1.129 1.184 1.184 4.8 Honanthrene (IS5) 1.007 1.089 1.089 8.2 Honanthrene (IS6) 1.007 1.089 1.089 8.2 Honanthrene (IS6) 1.089 1.178 8.2 Honanthrene (IS6) 1.089 1	٦	D3873			1.113	1.176	1.176	5.6	5.6
Phenanthrene (IS4) 1.129 1.184 4.8 4.8					1.323	1.380	1.380	4.3	4.3
Chrysene (1S6) 1.089 1.089 8.2 Renzo(a)pyrene (1S6) 1.089 1.178 1.178 8.2 Renzo(a)pyrene (1S6) 1.089 1.089 1.089 8.2 Renzo(a)pyrene (1S6) 1.089 1.178 1.178 8.2 Renzo(a)pyrene (1S6) 1.089 1.178 8.2 Renzo(a)pyrene (1S6) 1.089 1.089 8.2 Renzo(a)pyrene (1S6) 1.089 1.089 8.2 Renzo(a)pyrene (1S6) 1.089 1.089 1.089 1.089 Renzo(a)pyrene (1S6) 1.089					1.129	1.184	1.184	4.8	4.8
Benzo(a)pyrene (ISG)					1.007	1.089	1.089	8.2	8.2
3				Benzo(a)pyrene (IS6)	1.089	1.178	1.178	8.2	8.2
	2								
	3								

	CCV1		CCV2		ccv3	
Compound	Area Cpd	Area IS	Area Cpd	Area IS	Area Cpd	Area IS
Naphthalene	3148673	1338693				
Fluorene	2644619	958332				
Phenanthrene	3836885	1620877				
Chrysene	4002705	1837231				
Benzo(a)pyrene	3866113	1641089				

LDC #: 23104 A 26 SDG #: Sre Cover

VALIDATION FINDINGS WORKSHEET <u>Surrogate Results Verification</u>

Page:_	lof_1_
Reviewer:	M
2nd reviewer:	n

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

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

% Recovery: SF/SS * 100

Where: SF = Surrogate Found

SS = Surrogate Spiked

Sample ID: #1

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	100	52.4	53	5 3	O
2-Fluorobiphenyl		64.6	65	65	
Terphenyl-d14	ð	89.0	89	29	
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

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

Sample ID:

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

LDC#: 23104426 SDG#: See Gyer

VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates Results Verification

Page: 1 of 1 Reviewer: We 2nd Reviewer: 1

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

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

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

Where: SSC = Spiked sample concentration SA = Spike added

SC = Sample concentation

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

SA = Spike added MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples:

2/6

	So	\$	elameS	Spiked	Samole	Matrix Snike	Spike	Matrix Spike Duplicate	Duplicate	USW/SW	SD
Compound	Ade (SS)	Added (20 /F.)	Concentration ($V \leq / E_c$)	Concentration	tration (c.)	Percent Recovery	ecovery	Percent Recovery	ecovery	RPD	
	MS	U MSD	0	MS	/ MSD	Renorted	Recalc	Renorted	Recalc	Reported	Recalculated
Phenol											
N-Nitroso-di-n-propylamine											
4-Chloro-3-methylphenol											
Acenaphthene	2004	3016	0	(450	2260	ود	رد	752	15	15	ير
Pentachlorophenoi											
Pyrene	3000	30 m	51	. 0292	3010	89	81	66	66	\dot{z}	<u>></u>
								,			
			· · · · · · · · · · · · · · · · · · ·				-				

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

23104 A 26 SDG #: See Corer LDC#:

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification VALIDATION FINDINGS WORKSHEET

2nd Reviewer: 1 Reviewer: MC

Page: Lof 1

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

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 * (SC/SA

SSC = Spike concentration SA = Spike added Where:

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

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

LCS/LCSD samples:

286-10254

	S	ike	S	ike	31	SU	Ü	CSD	USD I/SD I	csp
Compound	A (الأو	Added (Wg/kg)	Conce (hk	Concentration (4人/内)	Percent Recovery	Recovery	Percent Recovery	Recovery	RPD	Q
	108	1 CSD	108	l CSD	Reported	Racalc	Reported	Racalc	Reported	Recalculated
Phenal										
N-Nitroso-di-n-propylamine										
4-Chloro-3-methylphenol										
Acenaphthene	26.50	¥2	19.70	Ą	74	74				1
Pentachlorophenol										
Pyrene	0.5 92	`	2600	,	98	86				

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

LDC #:_	2	304	A	26
SDG #	Con	Can	. /	

VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	<u>l</u> of 1
Reviewer:_	N
2nd reviewer:	V

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

/	Y	N	N/A
	Y	W	N/A

%S

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:

Concent	tration	= $(A_{\bullet})(L_{\bullet})(V_{\bullet})(DF)(2.0)$ $(A_{\bullet})(RRF)(V_{\bullet})(V_{\bullet})(\%S)$
A _x	=	Area of the characteristic ion (EICP) for the compound to be measured
A _{is}	=	Area of the characteristic ion (EICP) for the specific internal standard
l _s	=	Amount of internal standard added in nanograms (ng)
V _o	==	Volume or weight of sample extract in milliliters (ml) or grams (g).
V_i	=	Volume of extract injected in microliters (ul)
V,	=	Volume of the concentrated extract in microliters (ul)
Df	=	Dilution Factor.

Percent solids, applicable to soil and solid matrices only.

Sample I.D,UU	
Conc. = $(\frac{19937}{1957121})(\frac{40}{1.1292})(\frac{1}{36.29})(\frac{1000}{0.93})(\frac{1}{1000})$	
= 18,5 ug/zg	

2.0	= Factor of 2 to accoun	nt for GPC cleanup			
#	Sample ID	Compound	Reported Concentration ()	Calculated Concentration ()	Qualification
			·		
		4			
	4				
	:				
				-	
1 1			 		