

LABORATORY DATA CONSULTANTS, INC.

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August 15, 2008

ERM 2525 Natomas Park Drive, Suite 350

Sacramento, CA 95833

ATTN: Ms. Maria Barajas-Albalawi

SUBJECT: BRC Tronox Parcel G, Data Validation

Dear Ms. Barajas-Albalawi

Enclosed are the final validation reports for the fractions listed below. This SDG was received on July 28, 2008. Attachment 1 is a summary of the samples that were reviewed for each analysis.

LDC Project # 19188:

SDG#	<u>Fraction</u>
F8F120180	Volatiles, Semivolatiles, Chlorinated Pesticides, Polychlorinated Biphenyls, Metals, Wet Chemistry, Gasoline Range Organics, Diesel Range Organics, Polynuclear Aromatic Hydrocarbons, Dioxins/Dibenzofurans

The data validation was performed under EPA Level III and Level IV guidelines. The analyses were validated using the following documents, as applicable to each method:

- USEPA, Contract Laboratory Program National Functional Guidelines for Organic Data Review, October 1999
- USEPA, Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, October 2004
- EPA SW 846, Third Edition, Test Methods for Evaluating Solid Waste, update 1, July 1992; update IIA, August 1993; update II, September 1994; update IIB, January 1995; update III, December 1996; update IIIA, April 1998; IIIB, November 2004; Update IV, February 2007

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

Sincerely,

Erlinda T. Rauto

Operations Manager/Senior Chemist

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Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

BRC Tronox Parcel G

Collection Date:

June 11, 2008

LDC Report Date:

August 7, 2008

Matrix:

Soil/Water

Parameters:

Volatiles

Validation Level:

EPA Level III & IV

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

TSB-GJ-09-10'

TSB-GJ-09-20'**

TSB-GJ-09-30'

TSB-GJ-09-40'

TB-2 6/11/08

^{**}Indicates sample underwent EPA Level IV review

Introduction

This data review covers 4 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8260B for Volatiles.

This review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

Samples indicated by a double asterisk on the front cover underwent an EPA Level IV review. An EPA Level III review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Level III criteria since this review is based on QC data.

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- 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.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.

None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

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

II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

III. Initial Calibration

Initial calibration was performed using required standard concentrations.

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

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

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

Average relative response factors (RRF) for all volatile target compounds and system performance check compounds (SPCCs) were within method and validation criteria with the following exceptions:

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
6/9/08	Ethanol	0.00221 (≥0.05)	All soil samples in SDG F8F120180	J (all detects) UJ (all non-detects)	Α

IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

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

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

Date	Compound	%D	Associated Samples	Flag	A or P
6/19/08 (LCAL0317)	lodomethane	67.71684	All water samples in SDG F8F120180	J+ (all detects)	А

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

Date	Compound	%D	Associated Samples	Flag	A or P
5/28/08 (LICV9881)	lodomethane	31.67513	All water samples in SDG F8F120180	J+ (all detects)	А
5/28/08 (LICV9881)	2-Hexanone	25.04476	All water samples in SDG F8F120180	J- (all detects) UJ (all non-detects)	Α

All of the continuing calibration RRF values were within method and validation criteria with the following exceptions:

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
6/16/08 (FCAL1777)	Ethanol	0.00209 (≥0.05)	All soil samples in SDG F8F120180	J (all detects) UJ (all non-detects)	А

V. Blanks

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

Sample TB-2 6/11/08 was identified as a trip blank. No volatile contaminants were found in this blank with the following exceptions:

Trip Blank ID	Sampling Date Compound		Concentration	Associated Samples		
TB-2 6/11/08	6/11/08	Dichloromethane	0.47 ug/L	All soil samples in SDG F8F120180		

Sample concentrations were compared to concentrations detected in the field blanks. The sample concentrations were either not detected or were significantly greater (>10X for common contaminants, >5X for other contaminants) than the concentrations found in the associated field blanks.

VI. Surrogate Spikes

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

Sample	Surrogate	%R (Limits)	Compound	Flag	A or P
8172125MB	Bromofluorobenzene	117 (79-115)	All TCL compounds	J+ (all detects)	P

VII. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Percent recoveries (%R) and relative percent differences (RPD) were not within QC limits. Since there were no associated samples, no data were qualified.

VIII. Laboratory Control Samples (LCS)

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

IX. Regional Quality Assurance and Quality Control

Not applicable.

X. Internal Standards

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

XI. Target Compound Identifications

All target compound identifications were within validation criteria for samples on which an EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

XII. Compound Quantitation and CRQLs

All compound quantitation and CRQLs were within validation criteria for samples on which an EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III 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 an EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

XV. Overall Assessment of Data

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

XVI. Field Duplicates

No field duplicates were identified in this SDG.

BRC Tronox Parcel G Volatiles - Data Qualification Summary - SDG F8F120180

SDG	Sample	Compound	Flag	A or P	Reason
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	Ethanol	J (all detects) UJ (all non-detects)	A	Initial calibration (RRF)
F8F120180	TB-2 6/11/08	lodomethane	J+ (all detects)	А	Continuing calibration (%D)
F8F120180	TB-2 6/11/08	lodomethane	J+ (all detects)	А	Continuing calibration (ICV %D)
F8F120180	TB-2 6/11/08	2-Hexanone	J- (all detects) UJ (all non-detects)	A	Continuing calibration (ICV %D)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	Ethanol	J (all detects) UJ (all non-detects)	А	Continuing calibration (RRF)

BRC Tronox Parcel G Volatiles - Laboratory Blank Data Qualification Summary - SDG F8F120180

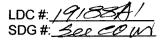
No Sample Data Qualified in this SDG

BRC Tronox Parcel G Volatiles - Field Blank Data Qualification Summary - SDG F8F120180

No Sample Data Qualified in this SDG

SDG ; Labor MET H	#: 19188A1 #: F8F120180 atory: Test America IOD: GC/MS Volatiles (E		L€ hod 8260E	evel III/I\ 3)	/		Rev 2nd Rev	
	ed validation findings wo		T	T				
	Validation	Area				Comments		
1.	Technical holding times		A	Sampling o	lates: 6/	11/08		
11.	GC/MS Instrument performa	nce check	A		· · · · · · · · · · · · · · · · · · ·	,		
111.	Initial calibration		M	tes	D. Y 2			
IV.	Continuing calibration/ICV		/w/	10V=	3 2570			
V.	Blanks		A		/			
VI.	Surrogate spikes		Ŵ					,
VII.	Matrix spike/Matrix spike dup	olicates	M	75B-0	€J-08-10	0'- No sq	plass'd	No Cenal
VIII.	Laboratory control samples		W/	100		/	· · · · · · · · · · · · · · · · · · ·	
IX.	Regional Quality Assurance	and Quality Control	N					
X.	Internal standards		4					
XI.	Target compound identificati	on	A	Not review	ed for Level III v	alidation.		
XII.	Compound quantitation/CRC		Â		ed for Level III v			
XIII.	Tentatively identified compo		11		ed for Level III v			
			 		ed for Level III v			
XIV.	System performance			Not review	red for Level III v			
XV.	Overall assessment of data		N.				****** *******************************	
XVI.	Field duplicates		N					
XVII.	Field blanks		\sim	TB=	5			
Note: Validat	A = Acceptable N = Not provided/applicable SW = See worksheet ed Samples: ** Indicates samp	R = Rin FB = Fi	eld blank	s detected	D = Dup TB = Tri EB = Ed			
1	TSB-GJ-09-10'	11 81702	91MB	21	(S)	31	1	
2	TSB-GJ-09-20'**	11 81702 12 817212 13 81723	SMB	22	4/	32		
3	TSB-GJ-09-30'	13 8/723	6/MF	3 23	(N)	33		
4	TSB-GJ-09-40'	14		24		34		
5	TB-2 6/11/08	15		25		35		
6	10 2 0/1/100	16		26		36		
7		17		27		37		

19188A1W.wpd

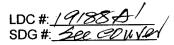


VALIDATION FINDINGS CHECKLIST

Page: __of _/ Reviewer: _____ 2nd Reviewer: _____

Method: Volatiles (EPA SW 846 Method 8260B)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.			The same of the sa	
III. GC/MS, instrument performance check		ı		
Were the BFB performance results reviewed and found to be within the specified criteria?		_		
Were all samples analyzed within the 12 hour clock criteria?				
III Initial calibration	T /			
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?	4			
Did the initial calibration meet the curve fit acceptance criteria of ≥ 0.990?	/			
Were all percent relative standard deviations (%RSD) ≤ 30% and relative response factors (RRF) ≥ 0.05?				
IV. Continuing calibration				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?				
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?		-		
Were all percent differences (%D) \leq 25% and relative response factors (RRF) \geq 0.05?				
V:Blanks				
Was a method blank associated with every sample in this SDG?				
Was a method blank analyzed at least once every 12 hours for each matrix and concentration?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
Mi Surrogate spikes				
Were all surrogate %R within QC limits?	K			
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?				
VIII-Maurix spike/Maurix spike/duplicates				erith.
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.		7		
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. Eaboratory control samples				
Was an LCS analyzed for this SDG?	/			



VALIDATION FINDINGS CHECKLIST

Page:—of— Reviewer:——— 2nd Reviewer:———

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per analytical batch?		,		
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX-Regional Quality Assurance and Quality Control			-	en Paris de la Companya de la Compa
Were performance evaluation (PE) samples performed?		/		
Were the performance evaluation (PE) samples within the acceptance limits?				
X Internal standards				
Were internal standard area counts within -50% or +100% of the associated calibration standard?				
Were retention times within ± 30 seconds of the associated calibration standard?				
XI. Target compound identification				Sec.
Were relative retention times (RRT's) within ± 0.06 RRT units of the standard?				
Did compound spectra meet specified EPA "Functional Guidelines" criteria?				
Were chromatogram peaks verified and accounted for?	/			
XII. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	/			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?		_		
XIII: Tentatively identified compounds (TIGs)				
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?				
Were relative intensities of the major ions within \pm 20% between the sample and the reference spectra?			/	
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?				
XIV. System performance				
System performance was found to be acceptable.				
XV ⊙verall assessment of data				terania de la companya de la company
Overall assessment of data was found to be acceptable.				
XVI : Flaid duplicates				
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.			/	
XVII» Field iblanks				
Field blanks were identified in this SDG.		/		
Target compounds were detected in the field blanks.	7			

TARGET COMPOUND WORKSHEET

METHOD: VOA (EPA SW 846 Method 8260B)

A. Chloromethane*	U. 1,1,2-Trichloroethane	OO. 2,2-Dichloropropane	III. n-Butylbenzene	CCCC.1-Chlorohexane
B. Bromomethane	V. Benzene	PP. Bromochloromethane	JJJ. 1,2-Dichlorobenzene	DDDD. Isopropyl alcohol
C. Vinyl choride**	W. trans-1,3-Dichloropropene	QQ. 1,1-Dichloropropene	KKK. 1,2,4-Trichlorobenzene	EEEE. Acetonitrile
D. Chloroethane	X. Bromoform*	RR. Dibromomethane	LLL. Hexachlorobutadiene	FFFF. Acrolein
E. Methylene chloride	Y. 4-Methyl-2-pentanone	SS. 1,3-Dichloropropane	MMM. Naphthalene	GGGG. Acrylonitrile
F. Acetone	Z. 2-Hexanone	TT. 1,2-Dibromoethane	NNN. 1,2,3-Trichlorobenzene	HHHH. 1,4-Dioxane
G. Carbon disulfide	AA. Tetrachloroethene	UU. 1,1,1,2-Tetrachloroethane	OOO. 1,3,5-Trichlorobenzene	IIII. Isobutyl alcohol
H. 1,1-Dichloroethene**	BB. 1,1,2,2-Tetrachloroethane*	VV. isopropylbenzene	PPP. trans-1,2-Dichloroethene	JJJJ. Methacrylonitrile
I. 1,1-Dichloroethane*	CC. Toluene**	WW. Bromobenzene	QQQ. cis-1,2-Dichloroethene	KKKK. Propionitrile
J. 1,2-Dichloroethene, total	DD. Chlorobenzene*	XX. 1,2,3-Trichloropropane	RRR. m,p-Xylenes	LLLL. Ethyl ether
K. Chloroform**	EE. Ethylbenzene**	YY. n-Propylbenzene	SSS. o-Xylene	MMMM. Benzyl chloride
L. 1,2-Dichloroethane	FF. Styrene	ZZ. 2-Chlorotoluene	TTT. 1,1,2-Trichloro-1,2,2-trifluoroethane	NNNN. 2,2-DiMethy Fortand
M. 2-Butanone	GG. Xylenes, total	AAA. 1,3,5-Trimethylbenzene	UUU. 1,2-Dichlorotetrafluoroethane	0000 Dimetly disultide
N. 1,1,1-Trichloroethane	HH. Vinyl acetate	BBB. 4-Chlorotoluene	VVV. 4-Ethyltoluene	- dddd
O. Carbon tetrachloride	II. 2-Chloroethyivinyl ether	CCC. tert-Butylbenzene	WWW. Ethanol	2000
P. Bromodichloromethane	JJ. Dichlorodifluoromethane	DDD. 1,2,4-Trimethylbenzene	XXX. Di-isopropyl ether	RRRR.
Q. 1,2-Dichloropropane**	KK. Trichlorofluoromethane	EEE. sec-Butylbenzene	YYY. tert-Butanol	SSSS.
R. cis-1,3-Dichloropropene	LL. Methyl-tert-butyl ether	FFF. 1,3-Dichlorobenzene	ZZZ. tert-Butyl alcohol	TTTT.
S. Trichloroethene	MM. 1,2-Dibromo-3-chloropropane	GGG. p-Isopropyltoluene	AAAA. Ethyl tert-butyl ether	ນນນນ.
T. Dibromochloromethane	NN. Methyl ethyl ketone	HHH. 1,4-Dichlorobenzene	BBBB. tert-Amyl methyl ether	ww.

^{* =} System performance check compounds (SPCC) for RRF; ** = Calibration check compounds (CCC) for %RSD.

LDC #: 19188#1 SDG #: 20 COVIUM

VALIDATION FINDINGS WORKSHEET Initial Calibration

Reviewer:

2nd Reviewer:

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

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

Did the laboratory perform a 5 point calibration prior to sample analysis?

Were percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCC's and SPCC's? Was a curve fit used for evaluation? If yes, what was the acceptance criteria used for evaluation?

Did the initial calibration meet the acceptance criteria? Y N N/A

Were all %RSDs and RRFs within the validation criteria of ≤30 %RSD and ≥0.05 RRF?

		Ī	Ī	Ī .									Γ	Γ	T
Qualifications	A/ 20/7														
Associated Samples	M 50, 5.	8/7029/MB													
Finding RRF (Limit: >0.05)	100000														
Finding %RSD (Limit: <30.0%)															
Compound	MMW														
Standard ID	1941								,						
Date	6/9/18														
*															

LDC #:19.8841 SDG #:200 COW

VALIDATION FINDINGS WORKSHEET Continuing Calibration

Reviewer: 2nd Reviewer:

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

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

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

Were all %D and RRFs within the validation criteria of ≤25 %D and ≥0.05 RRF?

	
Qualifications The factor of	
Associated Samples 5.817175 MB 8.7091 MB 6.817475 MB	
Finding RRF (Limit: ≥0.05)	
Finding %D (Limit: <25.0%) 3 / 67573 2 5 0 4 4 7 6	
Compound Codomethans AMN Codomethans	
Standard ID 1121/9881 (1CV) 201/203/7	
Date Date 52/2/8 54/2/8 54/2/8	
	

VALIDATION FINDINGS WORKSHEET Field Blanks

Reviewer: 2nd Reviewer:

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

A War fall black dangers in this OPCS	
Were lield planks identified in this SDG?	
Were target compounds detected in the field blanks?	
lank units: Mac Associated sample units: Mates	
ampling date: 6/11/0 8	
ield blank type; (circle one) Field Blank / Rinsate / Trip Blank) Other:	Associated Samples:

Compound	Blank ID		S	Sample Identification	ion		
	5						
Methylene chloride							
Acetone				•			
Chloroform							
Didloconethans 0.47	147						
сяаг							
Blank units: As	Associated sample units:	ple units:					

Sampling date: Field blank type: (circle one) Field Blank / Rinsa	e) Field Blank / Rinsa	/ Rinsate / Trip Blank / Other:	Associated Samples:
Compound	Blank ID		Sample Identification

Compound	Blank ID		Sam	Sample Identification	no		
Methylene chloride							
Acetone		-					
Chloroform							
CROL							

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:
Common contaminants such as Methylene chloride, Acetone, 2-Butanone and Carbon disultide that were detected in samples within ten times the associated field blank concentration were also qualified as not detected, "U". Other contaminants within five times the field blank concentration were also qualified as not detected, "U".

LDC #: 1918841

VALIDATION FINDINGS WORKSHEET Surrogate Spikes

Reviewer: 2nd 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 all surrogate %R within 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 out of outside

of criteria?

Qualifications	1 Hote A																			
mits)	(79715)	()	(()	()	()	()	<u> </u>	(()	 <u> </u>	((<u> </u>	()	<u> </u>	()	()	^ ·	()
%Recovery (Limits)	711																			
Surrogate	PFB																			
Sample ID	8172125 MB	,																		
Date																				
#																				

QC Limits (Water)

QC Limits (Soil)

81-117

88-110

86-115 80-120 86-118

74-121 80-120 80-120

SMC1 (TOL) = Toluene-d8 SMC2 (BFB) = Bromofluorobenzene SMC3 (DCE) = 1,2-Dichloroethane-d4 SMC4 (DFM) = Dibromofluoromethane

LDC #: 191887/ SDG #: 2000V

VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Reviewer: 2nd 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".

Y IN N/A

Was a LCS required? Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?

	11		$\widehat{=}$	\				,			,	-				_	_		_				,	
Qualifications	No lange	7	MS/NSD W	1																				
Associated Samples	8.817×XX	,																						
RPD (Limits)	(02×) c//	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()
LCSD %R (Limits)	()	(C+1-5+) 181	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()
LCS %R (Limits)	293 (42-140)	10domestand 16645-140)	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()
Compound	NN	ladomerka																						
TCS/TCSD ID	81721269	, D																						
. Date																								
*																								

LDC#.A SDG#:

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Reviewer:_ 2nd Reviewer:_ Page:

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

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

A_x = Area of compound,
C_x = Concentration of compound,
S = Standard deviation of the RRFs
X = Mean of the RRFs

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

 $RRF = (A_x)(C_{\bf k})/(A_{\bf k})(C_x)$ average RRF = sum of the RRFs/number of standards %RSD = 100 $^{\circ}$ (S/X)

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	RRF (Se std)	RRF (\$2\std)	Average RRF (initial)	Average RRF (initial)	%RSD	%RSD
-	1941	80/6/9	★ (1st internal standard)	126050	126050	1885.0 17p020 17p020	0.52831	2040,7 18040,7	7.0405
		///	4 (2nd internal standard)	- 85E 0	1.35512	255 1 35512 0 29404 0 2926 0	+oxb=0	07/201	127450 157450
				3.51047	3.5TB47	3.57047 3.57047 3.42549		1	135c X
2	Xe.	A/6//		P=1+70	18510 651 470 6-1450		178871		77952
		8/4/6	(802650	0.59203	0	0.55366	1347144	134718
	(+)		000 (3rd internal standard)	895/11:1	1.11568	1.11150	25111:1	2.41699	24169
က			(1st internal standard)					_	
			(2nd internal standard)						
			(3rd internal standard)						
4			(1st internal standard)						
			(2nd internal standard)						
			(3rd internal etandord)						

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 #: 19/98/4/ SDG #: 32/02/WY

VALIDATION FINDINGS WORKSHEET Continuing Calibration Results Verification

/of /	4	
Page:	Reviewer:	2nd Reviewer:_

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

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

% Difference = 100 * (ave. RRF - RRF)/ave. RRF RRF = $(A_x)(C_x)/(A_y)(C_x)$

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

Ax = Continuing calibration KKF
A_x = Area of compound,
C_x = Concentration of compound,

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

					7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7			
					керопео	Recalcillated	Keported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q%	Q %
-	FC41778 6/16/08	80/91/9	(1st internal standard)	1.5802.0	0.57288	88615.0 88515.0	0.89949 0.899	0.899
		`	(2nd internal standard)	potheo	2/0/8.0	8/2/8.0 Stel8.0	891919	61612
			\mathcal{ODP} (3rd internal standard)	34299 36503	36503	AA .	87826.5978	6.5978
2	F04-1777	Feb-1777 6/16/08	\mathcal{NNNN} (1st internal standard)	0.73871	45/62.0 45/6%		2.324-6 2 3244	2 3244
		\ \ \	000 0 (2nd internal standard)	0.55366	858/5.0	Π	35975	3.5.985
			000 (3rd internal standard)	171150	06401.1	1-10470	8119.0 551190	8119.0
က			(1st internal standard)					
			(2nd internal standard)					
	-		(3rd internal standard)					
4			(1st internal standard)					
			(2nd internal standard)					
			(3rd internal standard)					

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



VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page:_	
Reviewer:	9
2nd reviewer:	

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

The percent recoveries (%I	R) of surrogates were re	ecalculated for the compounds in	dentified below using the	following calculation
----------------------------	--------------------------	----------------------------------	---------------------------	-----------------------

% Recovery: SF/SS * 100

Where: SF = Surrogate Found

Sample ID:_

 \sim

Ο.		Cumogato	
SS	=	Surrogate	Spiked

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8	50	45-0289	90,	90	0
Bromofluorobenzene	1	42.1691	84	84	
1,2-Dichloroethane-d4	1,	45.1855	90	90	
Dibromofluoromethane	<i>Y</i>	44.0752	88	38	

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8					
Bromofluorobenzene					
1,2-Dichloroethane-d4					
Dibromofluoromethane					

Sample ID:_

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8					
Bromofluorobenzene					
1,2-Dichloroethane-d4					
Dibromofluoromethane					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8					
Bromofluorobenzene					
1,2-Dichloroethane-d4					
Dibromofluoromethane					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8					
Bromoffuorobenzene					
1,2-Dichloroethane-d4					
Dibromofluoromethane					

LDC#:1918841 SDG#:50c.com

VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Results Verification

Page: of Reviewer:

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

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

% Recovery = 100 * SSC/SA Where:

Where: SSC = Spiked sample concentration SA = Spike added

O COCOST CALLER COCOST COCOST

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

LCS ID: 817029/

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

CS/I CSD	RPD	Recalculated									
1 CS/I	RF	Reported									
	ecovery	Recalc									
LCSD	Percent Recovery	Reported									
S	ecovery	Recalc	96	66	88	201	(0)				
SD I	Percent Recovery	Reported	96	66	B	100	201				
ample	ration (O)	1 CSD	XX				\nearrow				
Spiked	Concentration	1.08	47.8	79.5	49.6	2.25	49.9				
ike	Added	I CSD	νŁ	,			<i>\</i>				
ďS	A A	LCS	25			,	/				
	Compound		1,1-Dichloroethene	Trichloroethene	Benzene	Toluene	Chlorobenzene				

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

LDC #: 191884
SDG #: Secous

VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	of
Reviewer:	4
2nd reviewer:	

METHOD:	GC/MS VO	(EPA SW	846 Method	8260B)
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Percent solids, applicable to soils and solid matrices

TY	N	N/A
∇	Ν	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?

Concer	tratio	$n = \frac{(A_{\bullet})(I_{\bullet})(DF)}{(A_{\bullet})(RRF)(V_{\bullet})(\%S)}$	Example:
A_{x}	=	Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D. 2, ND:
A_{is}	=	Area of the characteristic ion (EICP) for the specific internal standard	
l _s	=	Amount of internal standard added in nanograms (ng)	Conc. = () () () ()
RRF	=	Relative response factor of the calibration standard.	
V _o	=	Volume or weight of sample pruged in milliliters (ml) or grams (g).	=
Df	=	Dilution factor.	+

fication
_

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

BRC Tronox Parcel G

Collection Date:

June 11, 2008

LDC Report Date:

August 6, 2008

Matrix:

Soil

Parameters:

Semivolatiles

Validation Level:

EPA Level III & IV

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

Sample Identification

TSB-GJ-09-10'

TSB-GJ-09-20'**

TSB-GJ-09-30'

TSB-GJ-09-40'

^{**}Indicates sample underwent EPA Level IV review

Introduction

This data review covers 4 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.

None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

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

II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

III. Initial Calibration

Initial calibration was performed using required standard concentrations.

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

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

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

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
6/18/08	Phthalic acid	0.01422 (≥0.05)	All samples in SDG F8F120180	J (all detects) UJ (all non-detects)	А
	N-(Hydroxymethyl)phthalimide	0.04408 (≥0.05)		J (all detects) UJ (all non-detects)	

IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

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

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

Date	Compound	%D	Associated Samples	Flag	A or P
6/19/08	Phthalic acid	25.06878	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	J- (all detects) UJ (all non-detects)	A

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

All of the continuing calibration RRF values were within method and validation criteria with the following exceptions:

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
6/18/08	Phthalic acid N-(Hydroxymethyl)phthalimide	0.01330 (≥0.05) 0.04331 (≥0.05)	81 68439MB	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A
6/19/08	Phthalic acid N-(Hydroxymethyl)phthalimide	0.01066 (≥0.05) 0.04523 (≥0.05)	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	А

V. Blanks

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

No field blanks were identified in this SDG.

VI. Surrogate Spikes

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

VII. Matrix Spike/Matrix Spike Duplicates

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

VIII. Laboratory Control Samples (LCS)

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

IX. Regional Quality Assurance and Quality Control

Not applicable.

X. Internal Standards

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

Sample	Internal Standards	Area (Limits)	Compound	Flag	A or P
TSB-GJ-09-10'	Perylene-d12	198321 (281395-1125580)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) UJ (all non-detects)	A
TSB-GJ-09-20'**	Perylene-d12	191974 (281395-1125580)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) UJ (all non-detects)	A
TSB-GJ-09-30'	Perylene-d12	206248 (281395-1125580)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) UJ (all non-detects)	А
TSB-GJ-09-40'	Perylene-d12	212988 (281395-1125580)	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) UJ (all non-detects)	А

XI. Target Compound Identifications

All target compound identifications were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

XII. Compound Quantitation and CRQLs

All compound quantitation and CRQLs were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III 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 EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

XV. Overall Assessment of Data

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

XVI. Field Duplicates

No field duplicates were identified in this SDG.

BRC Tronox Parcel G Semivolatiles - Data Qualification Summary - SDG F8F120180

SDG	Sample	Compound	Flag	A or P	Reason
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	Phthalic acid N-(Hydroxymethyl)phthalimide	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	А	Initial calibration (RRF)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	Phthalic acid	J- (all detects) UJ (all non-detects)	А	Continuing calibration (%D)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	Phthalic acid N-(Hydroxymethyl)phthalimide	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	А	Continuing calibration (RRF)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene	J (all detects) UJ (all non-detects)	A	Internal standards (area)

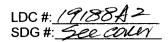
BRC Tronox Parcel G Semivolatiles - Laboratory Blank Data Qualification Summary - SDG F8F120180

No Sample Data Qualified in this SDG

BRC Tronox Parcel G Semivolatiles - Field Blank Data Qualification Summary - SDG F8F120180

No Sample Data Qualified in this SDG

SDG Labor	#: 19188A2 #: F8F120180 ratory: Test America	•		Le	evel III		SS WORKSHEET		Date: <u>8/5/0</u> 2 Page: _/of /_ Reviewer: 2nd Reviewer:
The s	amples listed below were ned validation findings wo	revie	ewed for ead		·	g val	idation areas. Validatior	n findir	ngs are noted in
	Validation	Area					Comme	nts	
1.	Technical holding times			A	Samplii	ng dat	es: 6/11/08		
11.	GC/MS Instrument performa	nce cl	neck	4					
111.	Initial calibration			W	ļ				
IV.	Continuing calibration/ICV			W	KEY	32	25/0		
V.	Blanks			4	ļ		/		
VI.	Surrogate spikes			1					
VII.	Matrix spike/Matrix spike du	plicate	s	\neq	TSI	3-6	EJ-08-10/		
VIII.	Laboratory control samples			W	10	2			
IX.	Regional Quality Assurance	and C	uality Control	N					
X.	Internal standards			SW					
XI.	Target compound identificat	ion		1	Not re	viewe	d for Level III validation.		
XII.	Compound quantitation/CRO	QLs		À	Not re	viewe	d for Level III validation.		
XIII.	Tentatively identified compo	unds (TICs)	$ \lambda $	Not re	viewe	d for Level III validation.		
XIV.	System performance			Ā	Not re	viewe	d for Level III validation.		
XV.				4					
XVI.	Field duplicates			N					
XVII	. Field blanks			N_					
Note: Valida	A = Acceptable N = Not provided/applicable SW = See worksheet ted Samples:		R = Rin	eld blank			D = Duplicate TB = Trip blank EB = Equipment blank dation		
	TOD 0.100.401		81684	39118		21		31	
1	TSB-GJ-09-10'		01007	- John		22		32	
2	TSB-GJ-09-20'**	12				23		33	
3	TSB-GJ-09-30'	13				24		34	
4	TSB-GJ-09-40' V	14				25		35	
5		15				26		36	
6		16						37	
7		17				27		38	
8		18	i			28		100	



VALIDATION FINDINGS CHECKLIST

Page: _/of _= Reviewer: ____ 2nd Reviewer: ____

Method: Semivolatiles (EPA SW 846 Method 8270C)

Validation Area	Yes	No	NA	Findings/Comments
J. Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.				
II. GCMS Instrument performance check				
Were the DFTPP performance results reviewed and found to be within the specified criteria?	1			
Were all samples analyzed within the 12 hour clock criteria?	Ľ			
III. Initial calibration				
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?	W			
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?		/		
IV. Continuing calibration				primit internal tracks for the second of the
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?				
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?				
Were all percent differences (%D) ≤ 25% and relative response factors (RRF) ≥ 0.05?				
V. Blanks				\mathcal{L}_{i}
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				And the second s
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?				
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?				
VII. Matrix spike/Matrix spike duplicates				Fig. 1. Probable CHINGS TWO THE STREET OF TH
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				Andrew Company of the
Was an LCS analyzed for this SDG?				



VALIDATION FINDINGS CHECKLIST

Page: of 2 Reviewer: 2nd Reviewer:

			T	· ·
Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?	/	<u> </u>	<u> </u>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX Regional Quality Assurance and Quality Control		4,55		and the state of t
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
X. Internal standards				And the second s
Were internal standard area counts within -50% or +100% of the associated calibration standard?	Ø			
Were retention times within ± 30 seconds from the associated calibration standard?	/			
XI. Target compound Identification				
Were relative retention times (RRT's) within ± 0.06 RRT units of the standard?	4			
Did compound spectra meet specified EPA "Functional Guidelines" criteria?				
Were chromatogram peaks verified and accounted for?				
XII. Compound quantitation/CRQLs		r		
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XIII. Tentatively identified compounds (TICs)				over the second of the second
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?				
Were relative intensities of the major ions within \pm 20% between the sample and the reference spectra?				
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?				
XIV _s System performance				and the second second second second
System performance was found to be acceptable.				
XV Overell assessment of data				
Overall assessment of data was found to be acceptable.	7			
XVI. Field duplicates (4. 32 v) halving the second				A programme a second
Field duplicate pairs were identified in this SDG.	ishiin ii iinn ii		-	
Target compounds were detected in the field duplicates.				
XVII, Field blanks				B THE STATE OF THE
				e la company de la company La company de la company d
Field blanks were identified in this SDG.		\mathcal{A}	_	
Target compounds were detected in the field blanks.				

VALIDATION FINDINGS WORKSHEET

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

A, Phenol:	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachiorophenoi**	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-Trichiorobenzene	GG. Acenaphthene**	W. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,l)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenoi*	XX. Di-n-butyiphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB, 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	TT.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	unu.
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	w.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	www. 4-Ch (No be wenth; e)
XXX. A-ft/johoxymethy AAAA 4-ch(notheny)	ly)>+h+halimide	XXX. Phenyl sultide	de 222. phenyl	olisul fide

VALIDATION FINDINGS WORKSHEET Initial Calibration

Page: Reviewer: 2nd Reviewer:

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

SDG #: See COUN

LDC #: ////

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

Did the laboratory conduct an acceptable 5 point calibration prior to sample analysis?

Were percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCC's and SPCC's? Was a curve fit used for evaluation? If yes, what was the acceptance criteria used for evaluation?

Did the initial calibration meet the acceptance criteria?

マ N N/A N/A Y/N N/A

N N/A

Were all %RSDs and RRFs within the validation criteria of ≤30 %RSD and ≥0.05 RRF ?

Qualifications	1,121														
Associated Samples	M+BACE														
Finding RRF (Limit: ≥0.05)	KC410.0	0.04408													
Finding %RSD (Limit: <30.0%)															
Compound	# HAThalic acid	XXX													
Standard ID	19/2														
Date,	6/8/8	\													
#															

VALIDATION FINDINGS WORKSHEET

LDC #: 19188.42

Continuing Calibration

2nd Reviewer:

pease see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". METHOD: GC/MS BNA (EPA SW 846 Method 8270)

Was a continuing calibration standard analyzed at least once every 12 hours of sample analysis for each instrument? Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's? Were all %D and RRFs within the validation criteria of ≤25 %D and ≥0.05 RRF?

X N N X N/N N/A

Qualifications Associated Samples MITNE Finding RRF (Limit: >0.05) (J.) 0433 0 (Limit: <25.0%) Finding %D 25.068 #HWIN acid Actualize aci Compound × 10405220 1CX-1519 Standard ID ∞ 0/6 Date #

LDC #:1918842 SDG #50e COWN

VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Reviewer: _ Page: 2nd Reviewer:

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

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

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

Qualifications	This is	Notheral	1	(No 05W/5W)																				
Associated Samples	7£+M																							
RPD (Limits)		()	()	()	()	()	()		()	(()	()	()	()	()	()	()	()	()	()	()	
LCSD %R (Limits)	()	()	()	()	()	()	()	()	()		()	()	()	()	()		()	()	()	()	()	()	()	
LCS %R (Limitş)	19 (54.90)	()	()		()	()	()	()	()						()		()	()	()	()	()	()	()	(
Compound) ## I																							
CS/ICSD ID	5076843918																							
# Date																								

SDG #: 280 (2011) LDC #: 0/884

VALIDATION FINDINGS WORKSHEET Internal Standards

Page: Reviewer: 2nd Reviewer:

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

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

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

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

1			Internal			
	Date	Sample ID	Standard	Area (Limits)	RT (Limits)	Qualifications
		/	PRY	0835211-5651800)185861	(885)	オートスト
			/ ,			
		7	FXY	191974 ()	
1.			`			
		8	PRY	206248(
						\
		7	PRY	2/2988C	(>
					The state of the s	(FF - 444)
				ć.		
ď						

* 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

SDG #: 200 COW

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: Reviewer: 2nd Reviewer:

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

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

 $\label{eq:RFF} $$RF = (A_{\mu})(C_{\mu})/(A_{\mu})(C_{\mu})$$ average RRF = sum of the RRFs/number of standards $$RSD = 100 * (S/X)$$$

A_x = Area of compound, C_x = Concentration of compound, S = Standard deviation of the RRFs,

 $A_{\mathbf{k}} = \text{Area of associated internal standard}$ $C_{\mathbf{k}} = \text{Concentration of internal standard}$ X = Mean of the RRFs

=									
_									
				Reported	Recalculated	Reported	Recalculated	Reported	Receivment
*	Standard ID	Calibration	Compound (Reference Internal Standard)	RRF	RRF	Average RRF	Average RRF	%RSD	%RSD
-	10/2	A / / /	Phenol (1st internal standard)	11 / 3/1	1 87 81d)	(initial)	(initial)		
\perp		00/2/0	Naphthalene (2nd internal standard)	109438	-+0-	100011	1,8653/	0/01	1.070
			Fluorene (3rd internal standard)	1.41778	14 TX0	14549	050	1.510	1.328
			Pentachlorophenol (4th internal standard)	02020	2000		_	0.5/3	0.573
			Bis(2-ethylhexyl)phthalate (5th Internal standard)	0 90762	0 001/2		0.19014	10.05	10.356
			Benzo(a)pyrene (6th internal standard)	$\Omega \mathbb{C}$	01.000	0.0042	0.8643	4.524	9524
Ø	1945	/~/~	Then of interest of the state o	0000	1.12800	11182	11182	6.486	6486
		80/8/08	tiend (19) internal standard)	0.57776	0.51926	0.57274	151274	112170	- 1 / 3 / 1
		\	Naphihalone (2nd internal standard) MM	1.20 177	1.20,77	18222	X CC X	2//2010	0.(151)
			Fluorene (3rd internal standard)				01:	non-	1.9366
			Pentachlorophenol (4th internal standard)				\		
		-	Bis(2-ethylhexyl)phthalate (5th internal standard)						
			Benzo(a)pyrene (6th internal standard)						
е	1941	8/7/	Phenol-(1st Internal standard) VVV	411091	7110/	1 27-43	- FT F.	100/	
		00/21/5	Naphthalene (2nd internal standard) ${\cal WW}$	0.33910	00000	3300	0000	1,0+0.	26427
			Elucrage (3rd internal standard)	13639	02/28	10000	0.53002	别	449540
			Pentachlorophenol (4th internal standard) 222	╁		2000	Ŋζ	$\sqrt{}$	12021
		_1	Bio(2 othylhoxyl)phithalete (5th internal standard)	0	NO TE	2426	寸		87548
			Benzo(a)pyrene (6th internal standard)	╁	2	20-10-1	0.59265	2,58836	18852

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

LDC #: 18188Az SDG #: Sec 20W

VALIDATION FINDINGS WORKSHEET Continuing Calibration Results Verification

Page: of / Reviewer: Cnd Reviewer:

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

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

% Difference = 100 * (ave, RRF - RRF)/ave, RRF RFF = $(A_{\nu})(C_{\mu})/(A_{\mu})(C_{\nu})$

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

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

 $A_{\mathbf{k}} = Area$ of associated internal standard $C_{\mathbf{k}} = Concentration$ of internal standard

5830 0450 Recalculated 1885 0101-1 588 2 ひとひとら 9784C 1480h Reported O% age 2.89 Recalculated 432 RRF (CC) 108 n Q 33952 20730 34 5915 Reported RRF (CC) 2016 80 Ţ, **∞** 8223 4122 8333 Average RRF 200 20 972 W 123xh 0.33002 (Initial) in Bis(2-ethylhexyl)phthalate (5th internal standard) Bis(2-ethylhexyl)phthalate (5th internal standard) Pentachlorophenol (4th internal standard) Bis(2-ethylhexyl)phthalate (5th internal standard) Naphthalene (2nd internal standard) $MM_{
m M}$ Naphithalene (2nd internal standard) $\mathcal{UU}($ Compound (Reference Internal Pentachlorophenol (4th internal standard) Fluorene (3rd internal standard) Pentachlorophenol (4th internal standard) Benzo(a)pyrene (6th internal standard) Benzo(a)pyrene (6th internal standard) Benzo(a)pyrene (6th internal standard) Naphthalene (2nd internal standard) Fluorene (3rd internal standard) Fluorene (3rd internal standard) Standard) Prenok (1st internal standard) Phenol (1st internal standard) Phenol (1st internal standard) 168) 8 Calibration Date 6 ā 6/1 575 JCA829 Standard ID 10.45=28 X *

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

LDC #: 19,1884> SDG #:

Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer: Reviewer:

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

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

% Difference = 100 * (ave, RRF - RRF)/ave, RRF RRF = $(A_{\nu})(C_{\nu})/(A_{\nu})(C_{\nu})$

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

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

 $A_{\mathbf{k}}=Area$ of associated internal standard $C_{\mathbf{k}}=Concentration$ of internal standard

					Reported	Receiculated	Bonorted	Latelization
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	RRF (CC)	RRF (CC)	Q%	necalculated %D
	JCAN 5195	8.0/81/2	Phenol (1st internal standard)	1.85537	1.8774	1.87174	0/288	- 00 0
	_	/ /	Naphthalene (2nd internal standard)	1.1090	1013	1.10130	OKAOTO	0 6000
			Fluorene (3rd internal standard)	1.41229	1.39801	1.34801	8000	20.0
			Pentachlorophenol (4th internal standard)	0.19634	0.20270	0.203/0	374085	>7477
	,		Bis(2-ethylhexyl)phthalate (5th internal standard)	0.86343	0.87788	0.87088	1'`	0 8658
			Benzo(a)pyrene (6th internal standard)	1.1118	1.11507	1.11507	0.2028	0 X X X
~	VCA15196	6/18/08	Phenol (1st internal standard)	0.51274	25/25.0	182187	- トレグル	1/1
		/ /	Naphthalene (2nd internal standard) UUU	Eze81.)	1.17316	1.17316	076746	0 767 19
			Fluorene (3rd internal standard)				\frac{1}{2}	6:6
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)		-			
			Benzo(a)pyrene (6th internal standard)					
ო	JCA46197	8/8/9	Phenol (1st internal standard) // //	1.67590	1.60400	1.60400	1.78300	17830
			Maphihelene (2nd internal standard) ${ m MMM}$	0.33002	0.33744	0.33744		7 200
			Fluorene (3rd internal standard)	1.02385	1.0336	0.880.		00000
			Pentachlorophenol (4th internal standard)	0.36637	0.38274	0.2834	74821	4112
			Bis(2-ethylhexyl)phthalate (5th interfallstandard)	0.39265	039671	0.39671	102201	0334
			Benzo(a)pyrene (6th internal standard)			<i>,</i> , , , , , , , , , , , , , , , , , ,		

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results. LDC #: 19188A> SDG #: <u>See COW</u>

VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page:	<u>/</u> of_/_
Reviewer:_	0
2nd reviewer:	

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

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

% Recovery: SF/SS * 100

Where: SF = Surrogate Found

SS = Surrogate Spiked

Sample ID:_____

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5		33.9134	65		
2-Fluorobiphenyl		35.9420	68		
Terphenyl-d14		39.8482	78		
Phenol-d5		52.05/37	66		
2-Fluorophenol		50,9295	65		
2,4,6-Tribromophenol		52.8840	69		
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID: -

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	50	32.5367	65	65	0
2-Fluorobiphenyl	1	33.9843	68	68	1
Terphenyl-d14		38.7625	78	78	
Phenol-d5	75	49.6403	66	66	
2-Fluorophenol		49.0421	65	65	
2,4,6-Tribromophenol		52.0744	69	69	
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					

SDG #: See Com LDC#: 01004>

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

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

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

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

% Recovery = 100 * (SC/SA

SSC = Spike concentration SA = Spike added Where:

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

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

LCS/LCSD samples: スルタスチュア

		و ا									
CS/I CSD	RPD	Recalculated									
I/SJ I	ià.	Renorted									
C)	ecovery	Recalc									
I CSD	Percent Recovery	Reported									
CS	Recovery	Recalc	12	77	77	75	79	70			
31	Percent Recovery	Reported	14	22	77	75	29	70			
ke	tration (カ)	I CSD	YN				,	\			
Spike	Concentration (人や) タ)	SOI	3356	0250	0956	0152	2240	2382			
Spike Added (ACK)		LCSD	ΝĂ					Ņ			
ds	Agi,)	SDT	33 70					<i>\</i>			
	Compound		Phenol	N-Nitroso-di-n-propylamine	4-Chloro-3-methylphenol	Acenaphthene	Pentachlorophenoi	Pyrene			

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 #: 19188A2 SDG #: Sec COWN

VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	(of
Reviewer:_	9
2nd reviewer:	

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

4	Y	N	N/A
-	V/		

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?

Conce	entratic	on = $\frac{(A_{\bullet})(I_{\bullet})(V_{\bullet})(DF)(2.0)}{(A_{\bullet})(RRF)(V_{\bullet})(V_{\bullet})(\%S)}$	Example:
A _x		Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D. Q., NO:
A _{is}	=	Area of the characteristic ion (EICP) for the specific internal standard	
l,	=	Amount of internal standard added in nanograms (ng)	Conc. = $()()()()()()()$
V _o	=	Volume or weight of sample extract in milliliters (ml) or grams (g).	
V_{t}	=	Volume of extract injected in microliters (ul)	=
V,	=	Volume of the concentrated extract in microliters (ul)	
Df	=	Dilution Factor.	
%S	=	Percent solids, applicable to soil and solid matrices only.	
20	=	Factor of 2 to account for GPC cleanup	

2.0	= Factor of 2 to accou	int for GPC cleanup									
#	Sample ID	Compound		Reported Concentration ()	Calculated Concentration ()	Qualification					
	·										
						L					

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

BRC Tronox, Parcel G

Collection Date:

June 11, 2008

LDC Report Date:

August 6, 2008

Matrix:

Soil

Parameters:

Chlorinated Pesticides

Validation Level:

EPA Level III & IV

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

Sample Identification

TSB-GJ-09-10'

TSB-GJ-09-20'**

TSB-GJ-09-30'

TSB-GJ-09-40'

^{**}Indicates sample underwent EPA Level IV review

Introduction

This data review covers 4 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8081A for Chlorinated Pesticides.

This review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- 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.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.

None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

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

II. GC/ECD Instrument Performance Check

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

III. Initial Calibration

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

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

A curve fit, based on the initial calibration, was established for quantitation for selected compounds. The coefficient of determination (r²) was greater than or equal to 0.990.

Retention time windows were evaluated and considered technically acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples on which a Level III review was performed.

IV. Continuing Calibration

Continuing calibration was performed at required frequencies.

The percent differences (%D) of calibration factors in continuing standard mixtures were within the 15.0% QC limits with the following exceptions:

Date	Standard	Channel	Compound	%D	Associated Samples	Flag	A or P
6/18/08	KCAL092	А	Toxaphene	15.2	TSB-GJ-09-30' TSB-GJ-09-40'	J+ (all detects)	A
6/18/08	KCAL095	А	2,4'-DDD	22.6	TSB-GJ-09-30' TSB-GJ-09-40'	J+ (all detects)	Р

The percent differences (%D) of the second source calibration standard were less than or equal to 15.0% for all compounds.

Retention times (RT) of all compounds in the calibration standards were within QC limits for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples on which a Level III review was performed.

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

V. Blanks

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

No field blanks were identified in this SDG.

VI. Surrogate Spikes

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

VII. Matrix Spike/Matrix Spike Duplicates

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

VIII. Laboratory Control Samples (LCS)

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

IX. Regional Quality Assurance and Quality Control

Not applicable.

X. Pesticide Cleanup Checks

a. Florisil Cartridge Check

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

b. GPC Calibration

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

XI. Target Compound Identification

All target compound identifications were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

XII. Compound Quantitation and Reported CRQLs

All compound quantitation and CRQLs were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

XIII. Overall Assessment of Data

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

XIV. Field Duplicates

No field duplicates were identified in this SDG.

BRC Tronox, Parcel G Chlorinated Pesticides - Data Qualification Summary - SDG F8F120180

SDG	Sample	Compound	Flag	A or P	Reason
F8F120180	TSB-GJ-09-30' TSB-GJ-09-40'	Toxaphene	J+ (all detects)	А	Continuing calibration (%D)
F8F120180	TSB-GJ-09-30' TSB-GJ-09-40'	2,4'-DDD	J+ (all detects)	Р	Continuing calibration (%D)

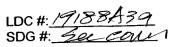
BRC Tronox, Parcel G Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG F8F120180

No Sample Data Qualified in this SDG

BRC Tronox, Parcel G Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG F8F120180

No Sample Data Qualified in this SDG

SDG # ₋abora	#:19188A3a VALIDA #:F8F120180 atory: <u>Test America</u> HOD: GC Chlorinated Pesticides (EF		evel III/IV	Date: 8/-/ Page:/of / Reviewer: 2nd Reviewer:	
	amples listed below were reviewed t tion findings worksheets.	for each of the f	following valida	ation areas. Validation	findings are noted in attache
****	Validation Area			Comme	nts
I.	Technical holding times	_ A	Sampling dates	: 6/11/08	<i>Y</i>
II.	GC/ECD Instrument Performance Check	A			
111.	Initial calibration	A ,	KSD.	y 2_	
IV.	Continuing calibration/ICV	/w/	CVE	1570	
V.	Blanks				
VI.	Surrogate spikes				
VII.	Matrix spike/Matrix spike duplicates	4	15B-G	1-08-10	
VIII.	Laboratory control samples		100		
IX.	Regional quality assurance and quality co	ntrol N			
Xa.	Florisil cartridge check	N			
Xb.	GPC Calibration	N			
XI.	Target compound identification	A	Not reviewed for	or Level III validation.	
XII.	Compound quantitation and reported CRC	QLs D	Not reviewed for	or Level III validation.	
XIII.	Overall assessment of data	4			
XIV.	Field duplicates	1			
XV.	Field blanks			 	
Note:	A = Acceptable N = Not provided/applicable F	ND = No compound R = Rinsate FB = Field blank Level IV validation		D = Duplicate TB = Trip blank EB = Equipment blank	
	·	10.11	B I I		
1	TSB-GJ-09-10' 3 11 8/a	68164 M	2 1	3	31
2	TSB-GJ-09-20'** 12		22	3	32
3	TSB-GJ-09-30' 13		23	3	33
4	TSB-GJ-09-40'		24	3	34
5	15		25	3	35
6	16		26	3	36



VALIDATION FINDINGS CHECKLIST

Page: /of 2
Reviewer:
2nd Reviewer:

Method:	/	GC	 HPLC

Method: GC HPLC		-		
Validation Area	Yes	No	NA	Findings/Comments
r-restricationing times				
All technical holding times were met.				
Cooler temperature criteria was met.				
If this callection Did the laboratory perform a 5 point calibration prior to sample analysis?				
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?	/			
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?				
Did the initial calibration meet the curve fit acceptance criteria?				
Were the RT windows properly established?				
IV Continuing calibration				
What type of continuing calibration calculation was performed?%D or %R				
Was a continuing calibration analyzed daily?				
Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?	Ĺ	\perp	<u> </u>	
Were all the retention times within the acceptance windows?		<u> </u>		
V-Blacks				
Was a method blank associated with every sample in this SDG?		1	<u> </u>	
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.		/		
V) Surrigate spikes				
Were all surrogate %R within the QC limits?	/	1		
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?				
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?				
VII. Matrix spike/Matrix spike duplicates				303
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?	17	1	1	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII*Laboratory.control/samples*				enter de la companya
Was an LCS analyzed for this SDG?	$ \downarrow $	4_	_	
Was an LCS analyzed per extraction batch?				



VALIDATION FINDINGS CHECKLIST

Page: <u>∂</u> of <u>→</u>
Reviewer:
2nd Reviewer:

Validation Area	Yes	No	NA	Findings/Comments
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	/			
IX: Regional Guality Assulaince and Guality Control 4				
Were performance evaluation (PE) samples performed?				/
Were the performance evaluation (PE) samples within the acceptance limits?	F32 +76-3800	*******************************		
X Target compound dentineation				
Were the retention times of reported detects within the RT windows?	O COLUMN TO	na de Andres en	122712324	
XI: Composind quantitation/CRQUs				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII System performance				
System performance was found to be acceptable.				
XIII)Overall assessment of data Sacrative Control of the Control o				Page 19
Overall assessment of data was found to be acceptable.				
XIV_Field doblicates as the second of the se				
Were field duplicate pairs identified in this SDG?				
Were target compounds idetected in the field duplicates?				
XV. Field planks				
Were field blanks identified in this SDG?			/	
Were target compounds detected in the field blanks?			/	

VALIDATION FINDINGS WORKSHEET

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

A. alpha-BHC	I. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	.96
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH.
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	II.
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	JJ.
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. DB 608	KK.
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. DB 1701	LL.
G. Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	EF.	MM.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	FF.	NN.

ition Worksheets/Pesticides/COMPLST-3S.wpd
V:\Validation Worl

Notes:

LDC#: 1958/139 SDG#: 1200WV

METHOD:

VALIDATION FINDINGS WORKSHEET Continuing Calibration

Page: __of__ Reviewer: _______

2nd Reviewer:_

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%?

Y M N/A Level IV Only Y N N/A

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

Qualifications	1+ Lety/																
Associated Samples	3-4	·															
RT (ilmit)	(()	()	(()	()			,		(())			
%D / RPD (Limit s 15.0)	3.3	0 .															
Compound	246.40	1991															
Detector/ Column	1 2 1	<i>y</i>															
Standard ID	KOHLO																
# Date 6/18/68																	

LDC #: 19188/139 SDG#: Secon

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Lot L	4	
Page:	Reviewer:	2nd Reviewer:
		2nd

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

CF # A/C

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

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

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound	CF 00 Std)	C. Sstd)	Average CF (initial)	Average CF (initial)	%RSD	%RSD
-	1346	80/21/9		3/366840	3/366840	3/366840 3/36840 3/378540 3/378560 1.30108 1.3011	3/3/28/26	80/08.1	1.301/
		,	F (ch. A)	21121/200	0025CH2520	21121/20 21121/2021/2021/8 2038021/8 21/0044 2.1004 4454220265545320 881332132 681332137 2.76188 2.7619	20380219	2.10044 2.76188	2.7619
2		7//	1) 0	2823380	8282850 C	28233860 28233800 30/26/26/26/26/26/26/26/26/26/26/26/26/26/	30/26/08	8x7=117	p2///
	10/2	81/91/9	F (ORB)	3635 /100	36359/000	0788. = 24388. = 12868/086 28868/088 000/1226 500/12686	381828d	2.83696	2.8370
			V 0	128/560	0/268/560	128/5600 1268/1400 133202078 133202078 8.79750 8.7920	13320208	8.79250	8.7925
ю									
4									

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

SDG #260 COM LDC #: (1/1/2/2)

Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

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

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

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

using the following calculation:

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%
-	404 LOBEL	84/8/19	F (ch. 4)	0.035	[250.0]	10.0357	9.8	8.6
			0.	1	20.0 120.0	1/20.0	5.0	25.0
l			24-DE (dr.f)	0.035	8500	0.038	33	3.7
7	CS07 123	4/0//	F (Ant)	0.035	0.0355	2550.0	8.	8 /
		0/10/10	0	/	1200		2.7	12
		`	2.4-00E		22550	0.0 25/5	2.2	رح زک
ო								
4								

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.

METHOD: __GC__ HPLC

VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page: Reviewer:_ 2nd reviewer:

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

% Recovery: SF/SS * 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Sample ID:

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
TONX	Ch. A	0000	0.01860	8	8	0
DC13	1	1	0.01958	l	8	

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

Sample ID:

	T	l	1	Ī
Percent Difference				
Percent Recovery	Recalculated			
Percent Recovery	Reported			
Surrogate Found				
Surrogate Spiked				
Column/Detector				
Surrogate				

LDC #: 1918 # 34 SDG # 346 @ WV

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

Page: of Reviewer: 2nd Reviewer:

METHOD: 4 GC HPLC

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

SSC = Spiked sample concentration SA = Spike added

Where:

SC = Concentration

RPD = I SSCLCS - SSCLCSD I * 2/(SSCLCS + SSCLCSD)

LCS/LCSD samples: 576 87

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

	S	Spike	Spiked	l Sample	1	rcs	dsol	ζΣ	/SOT	CS/LCSD
Compound			Sono:	Concentration (We 13)	Percent	Percent Recovery	Percent Recovery	ecovery	«	RPD
	TCS	LCSD	SOT	dson ,	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)										
Diesel (8015)										
Benzene (8021B)										
Methane (RSK-175)										
2,4-D (8151)										
Dinoseb (8151)										
Naphthalene (8310)										
Anthracene (8310)										
HMX (8330)										
2,4,6-Trinitrotoluene (8330)										
4	16.T	NA	0.51	NA	06	90				
0	1	//	8.91		101	- 0 -				

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

LDC #<u>/7/884</u>39 SDG #:\$<u>a_co</u>w/

VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

2

GC HF	Were all rep Were all rec
METHOD:	N/A N/A N/A

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

Concentration= (A)(Ev)(Df)	Example:		
(001/09/)(244-10-24)(34)	Sample II	Compound Name	6
A= Area or height of the compound to be measured Fv= Final Volume of extract			
Df= Dilution Factor			

Concentration =_

RF= Average response factor of the compound In the initial calibration Vs= Initial volume of the sample Ws= initial weight of the sample %S= Percent Solid

		 	 	_	
Qualifications					
Recalculated Results Concentrations					
Reported Concentrations					
Compound					
Sample ID					
#					

SAMPCALew.wpd

Somments:

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

BRC Tronox Parcel G

Collection Date:

June 11, 2008

LDC Report Date:

August 6, 2008

Matrix:

Soil

Parameters:

Polychlorinated Biphenyls

Validation Level:

EPA Level III & IV

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

Sample Identification

TSB-GJ-09-10'

TSB-GJ-09-20'**

TSB-GJ-09-30'

TSB-GJ-09-40'

^{**}Indicates sample underwent EPA Level IV review

Introduction

This data review covers 4 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8082 for Polychlorinated Biphenyls.

This review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.

None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

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

II. GC/ECD Instrument Performance Check

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

III. Initial Calibration

Initial calibration of multicomponent compounds was performed for the primary (quantitation) column as required by the method.

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

Retention time windows were evaluated and considered technically acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples on which a Level III review was performed.

IV. Continuing Calibration

Continuing calibration was performed at required frequencies.

The percent differences (%D) of calibration factors in continuing standard mixtures were within the 15.0% QC limits.

The percent differences (%D) of the second source calibration standard were less than or equal to 15.0% for all compounds.

Retention times (RT) of all compounds in the calibration standards were within QC limits for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples on which a Level III review was performed.

V. Blanks

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

No field blanks were identified in this SDG.

VI. Surrogate Spikes

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

VII. Matrix Spike/Matrix Spike Duplicates

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

VIII. Laboratory Control Samples (LCS)

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

IX. Regional Quality Assurance and Quality Control

Not applicable.

X. Pesticide Cleanup Checks

a. Florisil Cartridge Check

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

b. GPC Calibration

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

XI. Target Compound Identification

All target compound identifications were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

XII. Compound Quantitation and Reported CRQLs

All compound quantitation and CRQLs were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

XIII. Overall Assessment of Data

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

XIV. Field Duplicates

No field duplicates were identified in this SDG.

BRC Tronox Parcel G
Polychlorinated Biphenyls - Data Qualification Summary - SDG F8F120180

No Sample Data Qualified in this SDG

BRC Tronox Parcel G
Polychlorinated Biphenyls - Laboratory Blank Data Qualification Summary - SDG
F8F120180

No Sample Data Qualified in this SDG

BRC Tronox Parcel G
Polychlorinated Biphenyls - Field Blank Data Qualification Summary - SDG
F8F120180

No Sample Data Qualified in this SDG

LDC #: 19188A3b	VALIDATION COMPLETENESS WORKSHEET	
SDG #: F8F120180	Level III/IV	
Laboratory: Test America		
	LD:	2nd

Date:8/4/08
Page:
Reviewer:
2nd Reviewer:

METHOD: GC Polychlorinated Biphenyls (EPA SW 846 Method 8082)

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: 6/11/0-8
II.	GC/ECD Instrument Performance Check	N	/ /
111.	Initial calibration	A	
IV.	Continuing calibration/ICV	A	1CV=1570
V.	Blanks	A	
VI.	Surrogate spikes	1	
VII.	Matrix spike/Matrix spike duplicates	A	15B-61-08-10'
VIII.	Laboratory control samples	A	105
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	
Xb.	GPC Calibration	N	
XI.	Target compound identification	A	Not reviewed for Level III validation.
XII.	Compound quantitation and reported CRQLs	A	Not reviewed for Level III validation.
XIII.	Overall assessment of data		
XIV.	Field duplicates	N	
XV.	Field blanks		

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 Level IV validation

1	TSB-GJ-09-10'	11	3768762MB	21	31	
2	TSB-GJ-09-20'**	12	•	22	 32	
3	TSB-GJ-09-30'	/13		23	33	
4	TSB-GJ-09-40'	14		24	34	
5		15		25	 35	
6		16		26	36	
7		17		27	37	
8		18		28	38	
9		19		29	39	
10		20		30	40	

LDC #: 19188A3b SDG #: <u>5æ COW</u>

VALIDATION FINDINGS CHECKLIST

Page: _/of ___ Reviewer: _____ 2nd Reviewer:_____

Method:	V	GC	HPLC

Method: <u>// GCHPLC</u>				
Validation Area	Yes	No	NA	Findings/Comments
r Headical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.			W. W. W.	
p faila calbation				
Did the laboratory perform a 5 point calibration prior to sample analysis?				
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?				
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?				
Did the initial calibration meet the curve fit acceptance criteria?	<u> </u>		_	
Were the RT windows properly established?		******		
iV. Continuing calibration	<u> </u>		Tarky.	T
What type of continuing calibration calculation was performed?%D or				
Was a continuing calibration analyzed daily?	ν,	ļ	<u> </u>	
Were all percent differences (%D) ≤ 15%.0 or percent recoveries 85-115%?	/		-	
Were all the retention times within the acceptance windows?			Clare	
V»Blanks		1	T -	
Was a method blank associated with every sample in this SDG?	1	_	 	
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-Surrigate spikes				
Were all surrogate %R within the QC limits?	/	ļ	1	
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?				
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?			/	
VII. Mairix spike/Mardx 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?	1/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				NASARAN NEW TOWN THE PROPERTY OF THE PROPERTY
VIII*Laboratory control samples				The source of the second
Was an LCS analyzed for this SDG?	1	4_	$oldsymbol{\perp}$	
Was an LCS analyzed per extraction batch?	1/			



VALIDATION FINDINGS CHECKLIST

Page: of 2 Reviewer: 2nd Reviewer:

Validation Area	Yes	No	NA	Findings/Comments
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				
IX Regional Gualify Assurance and Gualify Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?	304-250-00-00	de a remoder		
X garget compound identification				
Were the retention times of reported detects within the RT windows?		ACTOR A SOCIETY OF	-511-26-11-28-34	
XI: Composind quantitation/CRQLS			7 72	
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII/SS/stem-periormance				
System performance was found to be acceptable.				
XIII. Toverall assessment of data: ***********************************	7			
Overall assessment of data was found to be acceptable.	/			
XIV Field triplicates				
Were field duplicate pairs identified in this SDG?				
Were target compounds idetected in the field duplicates?			/	
XV. Fjéld blánks				
Were field blanks identified in this SDG?				
Were target compounds detected in the field blanks?				

VALIDATION FINDINGS WORKSHEET

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

A. aipha-BHC	1. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	.99
В. beta-ВНС	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	÷
C. delta-BHC	K. Endrin	S. aipha-Chlordane	AA. Aroclor-1254	=
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	J.L.
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. DB 608	KK.
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. DB 1701	, LL.
G. Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	H.	MM.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	FF.	NN.

V:\Validation Worksheets\Pesticides\COMPLST-3S.wpd

Notes:

LDC #: 18788436 SDG #: 200

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

ot	4	
Page:	Reviewer:_	and Reviewer

METHOD: GC_

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

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

A = Area of compound,

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

Calibration Campound CF CF Average CF Average CF WRSD WRSD CA S S S C S S S S S S					Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
5/1/8 FB CAX-c2ps + 3 3154 33154 37077 120 FB C J II) 45676 45676 39164 3914 9582 B C J III) 45676 15677 120 B C J III) 45676 39164 39164 9582		Standard ID	Calibration Date	Compound	CF (<i>SD</i> std)	CF (572 std)	Average CF (initial)	Average CF (initial)	%RSD	%RSD
5/08 FB (1, II) 45676 39164 39164 9482	IL	10		BB (Ax-apost)	33154	33154	27977	27977	22)	(2.0
		1	20/12/5) ->	45676	 	39164	39164	= &ib	d.582
	1					J				
	IL									

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 #: 19 88 A.M. SDG#: Ser Cou

Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Page:of	Reviewer:	and Deviewer.
		\sim

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
Calibration Standard ID Date	Calibration Date		Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc. CCV	Q%	0%
1) SE 81/8/19 E80ADE			BB (XX-cufest)	0001	2061:256	9	8.7	4.8
2 present 6/8/08 1881 R	1/8/08 BBCK	\$38CK	1x-fegst)	(000)	937.3342	937.3	M. 0	6

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.

VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page: Reviewer: 2nd reviewer:

METHOD: VGC __ HPLC

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

p

Sample ID:

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
000	Ch. A.	20	85475	102	601	0
Sample ID:						
Surrogate	Column/Detector	Surrogate Spiked	Surrogate	Percent Recovery	Percent Recovery	Percent
				Reported	Recalculated	

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o de constant
Spiked

SDG #: See COWN LDC #: 19188A36

VALIDATION FINDINGS WORKSHEET

2nd Reviewer:

Laboratory Control Sample/Laboratory Control Sample Duplicate Results Verification

METHOD: VGC HPLC

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

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

Where:

SC = Concentration

RPD = I SSCLCS - SSCLCSD I * 2/(SSCLCS + SSCLCSD)

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

LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples: 8/6876

	S .	Spike	Spiked	Sample	PC	rcs	רכ	TCSD	/SOT	CS/LCSD
Compound	A Ac		Conce	Concentration	Percent Recovery	Recovery	Percent	Percent Recovery	<u>α</u>	RPD
	l.cs	TCSD	rcs	TCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)										
Diesel (8015)										
Benzene (8021B)										
Methane (RSK-175)										
2,4-D (8151)										
Dinoseb (8151)										
Naphthalene (8310)										
Anthracene (8310)										
HMX (8330)										
2,4,6-Trinitrotoluene (8330)										
88	191	NA	121	NA	501	102				
	•									

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

LDC #:/878843/ SDG #: 2000

VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page: Lof Reviewer: 🚣 2nd Reviewer:

> 77 GC HPLC METHOD:

Were all reported results recalculated and verified for all level IV samples? Y N N/A

Were all recalculated results for detected target compounds agree within 10% of the reported results?

Compound Name Sample ID. Example: (RF)(Vs or Ws)(%S/100) (A)(Fv)(Df) Concentration=

A= Area or height of the compound to be measured Fv= Final Volume of extract Df= Dilution Factor

RF= Average response factor of the compound in the initial calibration

Concentration =_

Vs= Initial volume of the sample Ws= Initial weight of the sample %S= Percent Solid

	i r —	T	 	 <u> </u>		
Qualifications						
Recalculated Results Concentrations (
Reported Concentrations						
Compound						
Sample ID						
#				-	_	

Somments:

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

BRC Tronox Parcel G

Collection Date:

June 11, 2008

LDC Report Date:

August 8, 2008

Matrix:

Soil

Parameters:

Metals

Validation Level:

EPA Level III & IV

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

Sample Identification

TSB-GJ-09-10'

TSB-GJ-09-20'**

TSB-GJ-09-30'

TSB-GJ-09-40'

^{**}Indicates sample underwent EPA Level IV review

Introduction

This data review covers 4 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Methods 6010B, 6020, and 7000 for Metals. The metals analyzed were Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Lithium, Magnesium, Manganese, Molybdenum, Mercury, Nickel, Niobium, Palladium, Phosphorus, Platinum, Potassium, Selenium, Silicon, Silver, Sodium, Strontium, Sulfur, Thallium, Tin, Titanium, Tungsten, Uranium, Vanadium, Zinc, and Zirconium.

This review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004) as there are no current guidelines for the methods 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.

Blanks are summarized in Section III.

Field duplicates are summarized in Section XIII.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- 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

An initial calibration was performed.

The frequency and analysis criteria of the initial calibration verification (ICV) and continuing calibration verification (CCV) were met.

III. Blanks

Method blanks were reviewed for each matrix as applicable. No contaminant concentrations were found in the initial, continuing and preparation blanks with the following exceptions:

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
ICB/CCB	Antimony Thallium Tungsten Vanadium Lithium Mercury	1.3 ug/L 1.1 ug/L 1.4 ug/L 2.7 ug/L 8.0 ug/L 0.1 ug/Kg	All samples in SDG F8F120180

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

Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-GJ-09-10'	Lithium	6.7 mg/Kg	26.6U mg/Kg
TSB-GJ-09-40'	Lithium Mercury	111 mg/Kg 22.0 ug/Kg	157U mg/Kg 52.4U ug/Kg

No field blanks were identified in this SDG.

IV. ICP Interference Check Sample (ICS) Analysis

The frequency of analysis was met.

The criteria for analysis were met.

V. Matrix Spike Analysis

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

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
TSB-GJ-08-10'MS/MSD (All samples in SDG F8F120180)	Sulfur Phosphorus	140.1 (75-125) 134.8 (75-125)	135.4 (75-125) -		J+ (all detects) J+ (all detects)	А
TSB-GJ-08-10'MS/MSD (All samples in SDG F8F120180)	Antimony Copper Silicon Vanadium Lithium Nickel Tungsten Zinc	55.2 (75-125) 72.5 (75-125) 65.4 (75-125) 68.4 (75-125) - - - -	39.4 (75-125) 60.9 (75-125) 44.6 (75-125) 56.0 (75-125) 69.8 (75-125) 71.1 (75-125) 60.6 (75-125) 62.2 (75-125)	- - - - - -	J- (all detects) UJ (all non-detects)	А
TSB-GJ-08-10'MS/MSD (All samples in SDG F8F120180)	Niobium	-	29.7 (75-125)	-	J- (all detects) R (all non-detects)	А

VI. Duplicate Sample Analysis

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable.

VII. Laboratory Control Samples (LCS)

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

VIII. Internal Standards (ICP-MS)

All internal standard percent recoveries (%R) were within QC limits for samples on which a Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

IX. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

X. ICP Serial Dilution

ICP serial dilution analysis was performed by the laboratory. The analysis criteria were met with the following exceptions:

Diluted Sample	Analyte	%D (Limits)	Associated Samples	Flag	A or P
TSB-GJ-08-10'L	Iron	10.4 (≤10)	All samples in SDG F8F120180	J (all detects)	А

XI. Sample Result Verification

All sample result verifications were acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

XII. Overall Assessment of Data

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

XIII. Field Duplicates

No field duplicates were identified in this SDG.

BRC Tronox Parcel G Metals - Data Qualification Summary - SDG F8F120180

SDG	Sample	Analyte	Flag	A or P	Reason
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	Sulfur Phosphorus	J+ (all detects) J+ (all detects)	А	Matrix spike/Matrix spike duplicates (%R)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	Antimony Copper Silicon Vanadium Lithium Nickel Tungsten Zinc	J- (all detects) UJ (all non-detects)	А	Matrix spike/Matrix spike duplicates (%R)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	Niobium	J- (all detects) R (all non-detects)	А	Matrix spike/Matrix spike duplicates (%R)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	Iron	J (all detects)	А	ICP serial dilution (%D)

BRC Tronox Parcel G Metals - Laboratory Blank Data Qualification Summary - SDG F8F120180

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8F120180	TSB-GJ-09-10'	Lithium	26.6U mg/Kg	Α
F8F120180	TSB-GJ-09-40'	Lithium Mercury	157U mg/Kg 52.4U ug/Kg	А

BRC Tronox Parcel G Metals - Field Blank Data Qualification Summary - SDG F8F120180

No Sample Data Qualified in this SDG

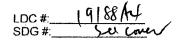
SDG # Labora	:: 19188A4 #: F8F120180 atory: <u>Test America</u>			Le	evel III/l'	ESS WORKSI	HEET	Date: 8/4/°) Page:(of Reviewer: 2nd Reviewer:
The sa	IOD: Metals (EPA SW 8 amples listed below were tion findings worksheets	e review			•	alidation areas. V	alidation findi	ngs are noted in attached
	Validation						Commonto	
		Alea		Δ	Sampling		<u>Comments</u>	
<u>l.</u>	Technical holding times Calibration			A	Sampling	dates. 7/1/08		
II. III.	Blanks			SW				
IV.	ICP Interference Check Sar	mnle (ICS)	Analysis	A				
V.	Matrix Spike Analysis	TIPIC (100)	Analysis	SW	749	/msp		
VI.	Duplicate Sample Analysis			N		1.4.2.2		
VII.	Laboratory Control Samples	s (LCS)		A	Les			
VIII.	Internal Standard (ICP-MS)			A-	r.t	berieved for	len 3	
IX.	Furnace Atomic Absorption			N	14	Mt. hize i		
X.	ICP Serial Dilution			3W	, ,	J. G. S. J. S.		
XI.	Sample Result Verification			A	Not revie	ved for Level III valida	tion.	
XII.	Overall Assessment of Data	a		À				
XIII.	Field Duplicates			ν,				
XIV.	Field Blanks			h				
Note: /alidate	A = Acceptable N = Not provided/applicable SW = See worksheet ed Samples: ** Indicates samp		R = Rins FB = Fie	eld blank	s detected	D = Duplicate TB = Trip bla EB = Equipm	nk	
	501)							
	TSB-GJ-09-10'	11			21		31	
	TSB-GJ-09-20'**	12			22		32	
	TSB-GJ-09-30'	13			23		33	
	TSB-GJ-09-40'	14			24		34	
5	<u> </u>	15			25 26		35 36	
7		16			27		37	
8		18			28		38	
9		19			29		39	
10		20			30		40	
Votes	,	<u>, * </u>					1.5 L	

VALIDATION FINDINGS CHECKLIST

Page: of A Reviewer: wu 2nd Reviewer:

Method: Metals (EPA SW 846 Method 6010/7000/6020)

	T.	T	Ī	
Validation Area	Yes	No	NA **	Findings/Comments
I. Technical holding times	41441 7	i i i i i i i i i i i i i i i i i i i		
All technical holding times were met.	1	<u> </u>		
Cooler temperature criteria was met.		1000 80000		
II. Galibration				
Were all instruments calibrated daily, each set-up time?	1			
Were the proper number of standards used?	/			
Were all initial and continuing calibration verification %Rs within the 90-110% (80- 120% for mercury and 85-115% for cyanide) QC limits?	/			
Were all initial calibration correlation coefficients ≥ 0.995? (Level IV only)	/			
III/Blanks				
Was a method blank associated with every sample in this SDG?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	/			
IV: IGI-state recrease Check Sample				
Were ICP interference check samples performed daily?	\			
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?				
IV-Matos spike/Matos spike/duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, Indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.		/		
Were the MS/MSD or duplicate relative percent differences (RPD) ≤ 20% for waters and ≤ 35% for soil samples? A control limit of +/- RL(+/-2X RL for soil) was used for samples that were ≤ 5X the RL, including when only one of the duplicate sample values were ≤ 5X the RL.		/		
V. Laboratory control samples:				
Was an LCS anayized for this SDG?				
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?	/			
VI; Furriace Atomic Absorption QC				
If MSA was performed, was the correlation coefficients > 0.995?			_	
Do all applicable analysies have duplicate injections? (Level IV only)				
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)				,
Were analytical spike recoveries within the 85-115% OC limits?	l			



VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
Reviewer: WM
2nd Reviewer: 0

Validation Area	Yes	No	NA	Findings/Comments
VILICA Senat Dilution	9 7 2 1 1			TENETOTISTICS TENEDOS TOTOS TOTOS
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the IDL?				
Were all percent differences (%Ds) < 10%?		1		
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.		/		
VIII. Internat Standards (EPA-SW-846-Method 6020)				
Were all the percent recoveries (%R) within the 30-120% of the intensity of the internal standard in the associated initial calibration?				
If the %Rs were outside the criteria, was a reanalysis performed?				
IX-Regional Quality Assurance and Quality Control t				
Were performance evaluation (PE) samples performed?			1	
Were the performance evaluation (PE) samples within the acceptance limits?			/	
X. Sample Result Verification (1988)				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
Xi Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			
XII Field duplicates				
Field duplicate pairs were identified in this SDG.		/		
Target analytes were detected in the field duplicates.				
XIII. Field blanks (s. 1997)				
Field blanks were identified in this SDG.		/		
Target analytes were detected in the field blanks.			7	

LDC #: 19188/14 SDG #: <u>Cel</u> comer

VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page:_	_of
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All circled elements are applicable to each sample.

Sample ID		Target Analyte List (TAL)
1-4	50,	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
1-4	50:1	(Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,)
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Analysis Method
ICP	·	Li. 8,
CP-MS		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
CP-MS		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Zr,)
GFAA		Al Sh As Ba Be Cd Ca Cr Co Cu Fe Ph Mg Mn Hg Ni K Se Ag Na Tl V Zn Mo B Si CN

Comments:	: Mercury by CVAA if performed	
Nb: Niobiun	um, Pd: Palladium, P: Phosphorus, Pt: Platinum, S: Sulfur, W: Tungsten, U: Uranium, Zr: Zirconium	1

SDG #: See Cover LDC #: 19188A4

Maximum PB^a mg/Kg

Analyte

S

≷ F

VALIDATION FINDINGS WORKSHEET

Reviewer: 2nd Reviewer: Sample Identification ₹ Associated Samples: PB/ICB/CCB QUALIFIED SAMPLES Soil preparation factor applied: 22.0 / 52.4 111 / 157 4 Sample Concentration units, unless otherwise noted: mg/Kg, except Hg ug/Kg 6.7 / 26.6 METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000) Blank Action 0.22 Maximum ICB/CCB^a 2.7 8.0 0.1 Maximum (1)011 PB_a

Hg (ug/Kg)

Samples with analyte concentrations within five times the associated ICB, CCB or PB concentration are listed above with the identifications from the Validation Completeness Worksheet. These sample results were qualified as not detected, "U".

Note: a - The listed analyte concentration is the highest ICB, CCB, or PB detected in the analysis of each element.

LDC #: 19188AT

Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

Reviewer:__ 2nd Reviewer:__ Page:_

METHOD: Trace metals (EPA SW 846 Method 6010/7000)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". Was a matrix spike analyzed for each matrix in this SDG?

Y 'N N/A

Were matrix spike percent recoveries (%R) within the control limits of 75-125? If the sample concentration exceeded the spike concentration by a factor

of 4 or more, no action was taken.

Were all duplicate sample relative percent differences (RPD) \leq 20% for water samples and \leq 35% for soil samples? Y (1) N/A WE LEVEL IV ONLY:

Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations. N N/A

84.4 34.4 34.4 66.4 66.4 60.6 60.6 60.6 60.6 60.6 7.7	_			MS	OSW	3,1		
50, 50 140, 134,4 M1 J+ 127 50, 50, 50, 140, 134,4 140, 134,4 140, 134,4 140, 134,4 140, 140, 140,6 140, 140,6 140, 140, 140,6 140, 140, 140,6 140		Watrix	Analyte	%Recovery	%Recovery	RPD (Limits)	Associated Samples	Qualifications
5h 55,2 34,4 Wh 40.6 29,7 V 134,8 Li 61,8 Will 60.6 Li 7,7 20.9 Will 7,7 20.9 Will 7,7 20.9 Li 1,7 20.9 L	8-10	10%	V	140.1	134.4		A1)	W+1+5
Ch 12-5 59.7 No 40.6 29.7 Si 65.4 44.6 Li 66.8 Ni Ni 60.6 So 20.2 So 3 No 4, Mn X 5y: T: 74.4		-	45	んだ	79.4		-	1-1 m 1/1
Nb			z	7246	6009	,		٦,
\$\partial \chi \chi \chi \chi \chi \chi \chi \chi			4 /4	40.6	29.7			J-/k/A
5; 65.4 44.6 7-141.0 14.6 14.6 14.6 14.6 14.6 14.6 14.6 14.6			đ	8761				4/+T+T
Li 65.4 56.0 Li Ail 69.8 Wi Fin 60.6 Li 60.6			\ \ \	4:59	9,45			J-/n1/4
Li 69.8 Ni All 60.6 En 60.0			Λ	カ・89	5610			
N; 9h, b 60, b 25, c 20, 9 1, 9cul			77		8.69			
2n			ίN		1 1 6			
20, 20, 33, 32, 4, 4, 4, 4, 4, 4, 4, 4, 4, 4, 4, 4, 4,			3		9.09			
Li Soig Wight of the stand of t			7n		2.29			<u></u>
Li Sa Sa Ca Ca Ca Ca Ca Ca Ca Ca Ca C						1		
56 Co. Co. Co. Ti. W W W W W W W W W W W W W			۲٦			2,00		-
Bec Co Co Tri W U U M W CA Tri Tri AX			5h			1.8 + X		
Co Ti W U U M Sr; Ti > TX			Be			29.32		
M U U Mg, My, K Sr; Tr, 74X			Z			>1, 4		
五: W U M K SY; 下: フザメ			Co			26.0		
W U Mg, Mu, K Sr; Tr, >4X			いた		:	5'92		4
Mg, Mu, \$ 5x; Tr, > 4x			Ň			280		/]
Mg, Mn, K Sr; F.			5			9.22	7	J
Mg, Mu, K Sr; Tr,								
, 119, M 3Y; 11,	,	⊣ I	1					
	الا الا الا	4	1	7	743			

19156/24 SDG #: LDC #:_

VALIDATION FINDINGS WORKSHEET ICP Serial Dilution

Page:__ Reviewer:_ 2nd Reviewer:

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

If analyte concentrations were > 50X the MDL (ICP) ,or >100X the MDL (ICP/MS), was a serial dilution analyzed? Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". Y) N/A

Y O N/A

Were ICP serial dilution percent differences (%D) <10%? Is there evidence of negative interference? If yes, professional judgement will be used to qualify the data.

CEVEL IV ONLY:

Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations.

	Qualifications	J1+/4												
vicio recalculated results acceptable : Ode Level IV Necesculation vicio recalculations	Associated Samples	Ā												
vecalculation vvo	%D (Limits)	4.0)												
מפפ רפאפו וא	Analyte	Fe												
acceptable:	Matrix	1305												
Vere recalculated result	Diluted Sample ID	158-67-0870												
	# Date													

-140 X Mr.

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\$

Comments:

LDC#: (9188A)

Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Reviewer: Page: 2nd Reviewer:__

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

An initial and continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:

Found = concentration (in ug/L) of each analyte measured in the analysis of the ICV or CCV solution True = concentration (in ug/L) of each analyte in the ICV or CCV source

Where, %R = Found × 100 True

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
7.6V	ICP (initial calibration)	S	42700	0000	8.401	3.90)	7
	GFAA (Initial calibration)						
Icv	CVAA (Initial calibration)	Hg	2,23	asi'z	43.2	43.2	۶
M	ICP (Continuing calibration)	, L,	4754	८००८	1-56	1-56	7
	GFAA (Continuing calibration)						
MO	CVAA (Continuing calibration)	145	867	2-0	3.18	966	7
MI	ICP/MS (Initial calibration)	کر	p.80)	(000)	8-201	8-00)	
Cov	ICP/MS (Continuing calibation)	B	908.3	رمور)	8.06	808	1

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

LDC#: (4189/M)
SDG#: 518 CONEN

VALIDATION FINDINGS WORKSHEET **Level IV Recalculation Worksheet**

Page: of 2nd Reviewer: C Reviewer: htt

METHOD: Trace Metals (EPA SW 846 Method 6010/7000)

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

%R = Found x 100 True

Where, Found = Concentration of each analyte <u>measured</u> in the analysis of the sample. For the matrix spike calculation, Found = SSR (spiked sample result) - SR (sample result).

True = Concentration of each analyte in the source.

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

RPD = $|S-D| \times 100$ (S+D)/2

S = Original sample concentration D = Duplicate sample concentration Where,

An ICP serial dilution percent difference (%D) was recalculated using the following formula:

%D = [I-SDR] × 100

Where, I = Initial Sample Result (mg/L) SDR = Serial Dilution Result (mg/L) (Instrument Reading x 5)

					Recalculated	Reported	
Sample ID	Type of Analysis	Element	Found / S / 1 (units)	True / D / SDR (units)	%R/RPD/%D	%R/RPD/%D	Acceptable (Y/N)
TUSAB	TUSAR ICP Interference check	4	(040)	مه)	∱ ∘1	40)	7
45	Laboratory control sample	43	896	(2)	46.2	76.2	
TSB-4J-08-10 Matrix spike	/ Matrix spike	22	(ssr-sr) $\langle \phi, \psi, \gamma \rangle$	60,(0)	97.3	91.3	
_	Duplicate	ζ	20.05	y in	- 2	1.0	
3	ICP serial dilution	TR	Z[19]	8959)	2.8	2.8	R

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

LDC #:	19188/AV
SDG #:	Su cour

Detected analyte results for ___

Dil

%S

VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	101/
Reviewer:	My
2nd reviewer:	

were recalculated and verified using the

METHOD: Trace Metals (EPA SW 846 Method 6010/7000)

M N N/A Have results been reported and calculated correctly?

N N/A Are results within the calibrated range of the instruments and within the linear range of the ICP?

Are all detection limits below the CRDL? 4) N N/A

Dilution factor

Decimal percent solids

followin	ıg equat	ion:		
Concenti	ration =	(RD)(FV)(Dil)	Recalculation:	
RD	=	(in. Vol.)(%S) Raw data concentration	S= 84.86 mg/ X0.05lx 5 X1000 g/mg	= 53291 mg/c
FV In. Vol.	=	Final volume (ml) Initial volume (ml) or weight (G)	0-58 X 0-9961	=3341.4/2

Sample ID	Analyte	Reported Concentration (Mg/Rg)	Calculated Concentration	Acceptable (Y/N)
7	S	I3300	t)300	Y
	Al	(0100	t)300 (0100	/i
	As Ba Be	27-6 64-6	27-6	
	Ba	646	64-6	
	l Be	0.64	0.64	
	Ca	15800	75800	
	CV	22.2	22,2	
	Co	2.7	5.6	
	Cu Fe	13-5	13-5	
	Fe	13200	13200	
	Pb	1.1	7.1	
	Mg	(8200	18200	
	My O	170	110	
	NI	14.7	14.6	
	p d		1.1	
	P	tis	tre	
	K	210	2/10	
	51	549	549	
	AS	0.14'	0,14	
	Va ⁰	944	943	
	5 _V	202	505	
	T	218	ts7	

LDC #:	19188/14
SDG #:	Set com

%S

VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	2017
Reviewer:	my
2nd reviewer:	V

METHOD: Trace Metals (EPA SW 846 Method 6010/7000)

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

N N/A Have results been reported and calculated correctly?

Are results within the calibrated range of the instruments and within the linear range of the ICP?

PN N/A Are all detection limits below the CRDL?

Decimal percent solids

	d analyt g equati	te results for ion:	<u> </u>	were recalculated and verified using the
Concentr	ation =	(RD)(FV)(Dil) (In. Vol.)(%S)	Recalculation:	10.15
RD	=	Raw data concentration	V= 4591 8/2X0.	= 57.67 mg/m
FV	=	Final volume (ml)	0.59 X	0.0661
In. Vol.	=	Initial volume (ml) or weight (G)	0.3 8 >	. *. 17"
Dil	=	Dilution factor	·	

Sample ID	Analyte	Reported Concentration (Mg/Kg)	Calculated Concentration (W. / //)	Acceptable (Y/N)
7	И	シ, つ	→, 'γ ⁰	Y
	V	37.7	サルケ	,
	Zh	91.5	91,2	
	2V	31-7	3/16	1
		/	,	
				·
				:

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

BRC Tronox Parcel G

Collection Date:

June 11, 2008

LDC Report Date:

August 7, 2008

Matrix:

Soil

Parameters:

Wet Chemistry

Validation Level:

EPA Level III & IV

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

Sample Identification

TSB-GJ-09-10'

TSB-GJ-09-20'**

TSB-GJ-09-30'

TSB-GJ-09-40'

^{**}Indicates sample underwent EPA Level IV review

Introduction

This data review covers 4 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 300.0 for Bromide, Bromine, Chlorate, Chloride, Chorine, Fluoride, Nitrate as Nitrogen, Nitrite as Nitrogen, Orthophosphate as Phosphorus, and Sulfate and EPA SW 846 Method 9071B for Oil & Grease.

The review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- 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

All criteria for the initial calibration of each method were met.

b. Calibration Verification

Calibration verification frequency and analysis criteria were met for each method when applicable.

III. Blanks

Method blanks were reviewed for each matrix as applicable. No contaminant concentrations were found in the initial, continuing and preparation blanks with the following exceptions:

Method Blank ID	Analyte	Concentration	Associated Samples
ICB/CCB	Orthophosphate as P	0.102 mg/L	All samples in SDG F8F120180

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

Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-GJ-09-20'**	Orthophosphate as P	1.5 mg/Kg	6.3U mg/Kg

No field blanks were identified in this SDG.

IV. Matrix Spike/Matrix Spike Duplicates

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

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
TSB-CJ-09-0'MS/MSD (All samples in SDG F8F120180)	Oil and grease	63 (75-125)	63 (75-125)	-	J- (all detects) UJ (all non-detects)	А

V. Duplicates

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable. Results were within QC limits.

VI. Laboratory Control Samples

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

VII. Sample Result Verification

All sample result verifications were acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

VIII. Overall Assessment of Data

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

IX. Field Duplicates

No field duplicates were identified in this SDG.

BRC Tronox Parcel G Wet Chemistry - Data Qualification Summary - SDG F8F120180

SDG	Sample	Analyte	Flag	A or P	Reason
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'	Oil and grease	J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)

BRC Tronox Parcel G Wet Chemistry - Laboratory Blank Data Qualification Summary - SDG F8F120180

SDG	Sample	Analyte	Modified Final Concentration	A or P
F8F120180	TSB-GJ-09-20'**	Orthophosphate as P	6.3U mg/Kg	А

BRC Tronox Parcel G
Wet Chemistry - Field Blank Data Qualification Summary - SDG F8F120180

No Sample Data Qualified in this SDG

DG#	: 19188A6 f: F8F120180 atory: <u>Test America</u>	F8F120180 Level III/IV							
/IETH EPA	OD: (Analyte) Bromic Method 300.0), O & G	de, Bromi 6 (EPA S'	ne, Chlorat W846 Meth	e, Chloride nod 9071B	e, Chorine, F	luoride, Nitrate-N, Nitri	te-N, Orthophosphate-P, Sulfate		
	amples listed below w tion findings workshee		wed for ea	ch of the f	ollowing val	idation areas. Validatio	n findings are noted in attached		
	Validatio	on Area				Comm	ents		
I.	Technical holding times			A	Sampling da	es: 6/11/8			
lla.	Initial calibration			A		, ·			
IIb.	Calibration verification			A					
III.	Blanks			SW					
IV	Matrix Spike/Matrix Spike	e Duplicate	s	SW	2 M	Jusp Dup			
٧	Duplicates			A	> '	/ / /			
VI.	Laboratory control samp	les		A	Les				
VII.	Sample result verification	n		A	Not reviewed for Level III validation.				
VIII.	Overall assessment of d	ata		A					
IX.	Field duplicates			N					
x	Field blanks		- 300	N					
lote: /alidate	A = Acceptable N = Not provided/applica SW = See worksheet ed Samples: **\indicates s		R = Rin FB = Fi	eld blank		D = Duplicate TB = Trip blank EB = Equipment blan	k		
	<u>Soi</u>	T							
	TSB-GJ-09-10'	11	.,		21		31		
2	TSB-GJ-09-20'**	12			22		32		
3	TSB-GJ-09-30'	13			23		33		
4	TSB-GJ-09-40'	14			24		34		
5	PB	15			25		35		
6		16			26		36		
7		17			27		37		
8		18			28		38		
9		19			29		39		
10		20			30		40		

Notes:_

LDC #:	19188	
SDG #:_	Sel	cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 1
Reviewer: WM
2nd Reviewer:

Method:Inorganics (EPA Method Su wyw

Method:Inorganics (EPA Method) W WY C	,		;	
Validation Area	Yes	No	NA	<u> </u>
Careting the company of the company		115		in the last
All technical holding times were met.	/			
Coolor temperature criteria was met.	/			
(Realization)	448	Hill		
Were all instruments calibrated daily, each set-up time?	/			
Were the proper number of standards used?				
Were all initial calibration correlation coefficients > 0.995?	/			
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?				
Were titrant checks performed as required? (Level IV only)			/	
Were balance checks performed as required? (Level IV only)	V		K	
Was a method blank associated with every sample in this SDG?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
TO COME PROPERTY OF THE PROPER				新國新四班中共共和國國
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.		/		
Were the MS/MSD or duplicate relative percent differences (RPD) \leq 20% for waters and \leq 35% for soil samples? A control limit of \leq CRDL(\leq 2X CRDL for soil) was used for samples that were \leq 5X the CRDL, including when only one of the duplicate sample values were \leq 5X the CRDL.	/			
Bucketting and San				
Was an LCS anaytzed for this SDG?				
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% (85-115% for Method 300.0) QC limits?				
VI. Regional Ocality Assirance and Quality Control 1882 252 252 252 252 252				
Were performance evaluation (PE) samples performed?			1	
Were the performance evaluation (PF) samples within the acceptance limits?			Δ	

LDC #:	19	188 A	Y6 ,
SDG #:	Ţ.,	J.	ww
	,		

VALIDATION FINDINGS CHECKLIST

Page: Vof Y Reviewer: M4 2nd Reviewer: V

	T	T	T-	The state of the s
Validation Area	Yes	No	NA	Findings/Comments
ML Sample Result Verification	1 × 1	terka		
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
Were detection limits < RL?	/			
A PROPERTY OF THE PROPERTY OF				Matrice Comments
Overall assessment of data was found to be acceptable.	7			
Field duplicate pairs were identified in this SDG.				
Target analytes were detected in the field duplicates.				
Field blanks were identified in this SDG.		/		
Target analytes were detected in the field blanks.			7	

LDC #: 19/88/Ab SDG #: ______ cover

VALIDATION FINDINGS WORKSHEET Sample Specific Analysis Reference

Page: ___of _/ Reviewer: ______ 2nd reviewer: ______

All circled methods are applicable to each sample.

Sample ID	Matrix	Parameter
1-4	Soil	Br Bromine Cl Chlorine F NO, NO, SO, O-PO, Chlorate ClO, O+G/TPH
	<i>r</i>	Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
·		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
	·	Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
		Br Bromine Cl Chlorine F NO ₃ NO ₂ SO ₄ O-PO ₄ Chlorate ClO ₄ O+G/TPH
	·	

Comments:	
F -	

LDC #: (9188A6

VALIDATION FINDINGS WORKSHEET

TO T	}	9
rage:	Reviewer:	2nd Reviewer:
		2nd

METHOD: Inorganics, Method

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

| Y | N | N/A | Were all samples associated with a given method blank?
| Y | N | N/A | Were any inorganic contaminants detected above the reporting limit in the method blanks? If yes, please see qualifications below.

Sample Identification Associated Samples: 1.5/6.3 Y Blank Action Limit Maximum ICB/CCB 40,0 Conc. units: Wg/kg Blank ID 0-por-p Analyte

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

(9188 AL LDC #:

Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

of	4	J
Page:	Reviewer:	2nd Reviewer:

METHOD: Inorganics, EPA Method_

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

Was a matrix spike analyzed for each matrix in this SDG?

Was a matrix spike analyzed for each matrix in this SDG?

Were matrix spike percent recoveries (%R) within the control limits of 75-125? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.

Were all duplicate sample relative percent differences (RPD) < 20% for water samples and <35% for soil samples?

Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations. EVEL IV ONLY: Y N/A

(S)	\vdash										,		
RPD (Limits)									,		,		
MSD %Recovery	63												
MS %Recovery									-				
1	0+6												
Matrix	ا (۲۰۵۶	,											
MS/MSD ID	TSB-CJ-09-6												

19188 AG LDC#:_

Initial and Continuing Calibration Calculation Verification Validatin Findings Worksheet

2nd Reviewer: Reviewer:___

Method: Inorganics, Method ___

8./37/9 _ was recalculated.Calibration date:_ The correlation coefficient (r) for the calibration of $- \ket{SY}$ An initial or continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:

%R = Found X 100

Found = concentration of each analyte measured in the analysis of the ICV or CCV solution

True = concentration of each analyte in the ICV or CCV source

					Recalculated	Reported	Acceptable
Type of analysis	Analyte	Standard	Conc. (ug/L)	Area	r or r²	r or r²	(Y/N)
Initial calibration		s1	250	0.02			
	Ā	s2	200	0.039	0.99997	0.99997	7
		83	1000	0.076			
		s4	2500	0.196			
		SS	2000	0.396			
$\mathcal{L}\mathcal{O}$ Calibration verification	LOS	4000	79.4		86	M	7
$\mathbb{C}_{\mathcal{N}}$ Calibration verification	1	000)	9495		9495	9495	-
$\mathcal{C} \sim \mathcal{N}$ Calibration verification	obut 600	8°e	1856		d8:m	9820	-)

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

DC#: 19188 A6 SDG#: See con-

VALIDATION FINDINGS WORKSHEET **Level IV Recalculation Worksheet**

2nd Reviewer:_ Page: Reviewer:

> 3 METHOD: Inorganics, Method_

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

Where, %R = Found x 100

Found =

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

True =

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

 $RPD = \underbrace{1S \cdot D!}_{(S+D)/2} \times 100 \text{ Where,}$

() () () ()

Original sample concentration Duplicate sample concentration

	-				Recalculated	Reported	
Sample ID	Type of Analysis	Element	Found / S (unite)	True / D (units)	%R / RPD	%R / RPD	Acceptable (Y/N)
	Laboratory control sample						
27		W2-14	Ţ	٠ ٩ ٩	9/8	86	>
	Matrix spike sample		(SSR-SR)				-
758-01-09-0	0-	019	2880	(390	6 >	63	
\rightarrow	Duplicate sample	10)	-	0471		~	B
r		7	000	0	<u>~</u>	^	

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

LDC #: (9 SDG #:	188/16 re com	VALIDATION FINDING Sample Calculation		Page:of Reviewer: 2nd reviewer:
METHOD: Inor	ganics, Method	Se our	-	
Please see que Y N N/A Y N N/A Y N N/A	Have results been r Are results within th	all questions answered "N". I reported and calculated corr ne calibrated range of the ins nits below the CRQL?	ectly?	s are identified as "N/A".
Compound (ar	nalyte) results for nd verified using the	following equation:	re	ported with a positive detect were
Concentration =		Recalculation:	200	
£103=-	Aven x 40ml	X6811 do3	0.079 x 45 x 0.	796=3.66 mg/vg

#	Sample ID	Analyte	Reported Concentration (Wg/kg)	Calculated Concentration	Acceptable (Y/N)
1	۷	0-p04-p Chlowty Cl Cl F	1.5	1-5	Y
		Chlority	3.7	3.7	
		cl	244	244	
		U2	488	488	
		F	0-58	0,59	
		102-N 504	5,3	2.3	
		7º 4	11600	11600	اد ا
<u></u>					
					
 					
				<u> </u>	
			<u> </u>	 	
 					
 			<u> </u>		
I				<u> </u>	

Note:	

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

BRC Tronox Parcel G

Collection Date:

June 11, 2008

LDC Report Date:

August 6, 2008

Matrix:

Soil

Parameters:

Gasoline Range Organics

Validation Level:

EPA Level III & IV

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

Sample Identification

TSB-GJ-09-10'

TSB-GJ-09-20'**

TSB-GJ-09-30'

TSB-GJ-09-40'

^{**}Indicates sample underwent EPA Level IV review

Introduction

This data review covers 4 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8015B for Gasoline Range Organics.

This review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- 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.
- 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 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 15.0% QC limits.

The percent differences (%D) of the second source calibration standard were less than or equal to 15.0% for all compounds.

III. Blanks

Method blanks were reviewed for each matrix as applicable. No gasoline range organic 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

All target compound identifications were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

VI. Compound Quantitation and CRQLs

All compound quantitation and CRQLs were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

VII. System Performance

The system performance was acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

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.

BRC Tronox Parcel G Gasoline Range Organics - Data Qualification Summary - SDG F8F120180

No Sample Data Qualified in this SDG

BRC Tronox Parcel G
Gasoline Range Organics - Laboratory Blank Data Qualification Summary - SDG
F8F120180

No Sample Data Qualified in this SDG

BRC Tronox Parcel G
Gasoline Range Organics - Field Blank Data Qualification Summary - SDG
F8F120180

No Sample Data Qualified in this SDG

Labora METH The sa	#: F8F120180 atory: Test America IOD: GC Gasoline Rang amples listed below were tion findings worksheets	e revie	•	SW846 N		i 80′	I5B)	as. Validatio	n finc	Page:of/_ Reviewer: 2nd Reviewer: dings are noted in attached
	Validation							Comm	ents	
l.	Technical holding times			4	Sampl	ling d	ates: 6	/11/08		
lla.	Initial calibration			A			7	/		
IIb.	Calibration verification/ICV			4	10	2V	£1570			
III.	Blanks			A						
IVa.	Surrogate recovery			lack						
IVb.	Matrix spike/Matrix spike du	uplicate	s	NA	4	100	d D	itid.	TSI	B4J-08-10'
IVc.	Laboratory control samples			\triangleleft	20		\triangleright			
V.	Target compound identifica	tion		4	Not re	•view	ed for Level III	validation.		
VI.	Compound Quantitation an	d CRQI	_S	4	Not re	eview	ed for Level III	validation.		
VII.	System Performance	•		\forall	Not re	eview	ed for Level III	validation.		
VIII.	Overall assessment of data	1		A						
IX.	Field duplicates			N		<u> </u>				
X.	Field blanks									
Note:	A = Acceptable N = Not provided/applicab SW = See worksheet		R = Rin	eld blank			TB = T	uplicate rip blank Equipment blanl	<	
П		11	1						24	
	1	1	816521	STAL		21			31	
	TSB-GJ-09-20'**	12				22			32	
	TSB-GJ-09-30'	/13				23			33 34	
5	TSB-GJ-09-40' ↓	15				24 25			35	
		16				<u>25 </u>			36	
7		17				20 27			37	
8		18				28			38	
9		19				<u>20</u> 29			39	
ا ا		1 3	 		-+				 	

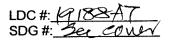
Notes:__

LDC#: 19188 \$7 SDG#: 20 COUN

VALIDATION FINDINGS CHECKLIST

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

Method: GC HPLC		 ,		
Validation Area	Yes	No	NA	Findings/Comments
f Technical holding times			- I	
All technical holding times were met.				
Cooler temperature criteria was met.	-	**********		
H*Initial calibration				
Did the laboratory perform a 5 point calibration prior to sample analysis?				
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?				
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?		/		
Did the initial calibration meet the curve fit acceptance criteria?				
Were the RT windows properly established?		and the second		
IV Continuing calibration	· · · · · ·	· · · · · · · · · · · · · · · · · · ·	r I	
What type of continuing calibration calculation was performed?%D or%R	/			
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?			100000000	
V.:Blanks	T	i I	T -	T
Was a method blank associated with every sample in this SDG?	/		<u> </u>	
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.				and the second s
VI: Surrogate spikes	T 7	T T		T
Were all surrogate %R within the QC limits?	/			
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?				
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?		riexavaaa		
VII. Matrix spike/Matrix spike duplicates	· /	T	T	
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?			1	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII*Laboratory/control samples :: 1.1				
Was an LCS analyzed for this SDG?		1		25
Was an LCS analyzed per extraction batch?	/			



VALIDATION FINDINGS CHECKLIST

Page: →of →
Reviewer:
2nd Reviewer:'

Validation Area	Yes	No	NA	Findings/Comments
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?	A CALL OF THE PARTY.			
X Target compound identification +				1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
Were the retention times of reported detects within the RT windows?				
XI: Compound quantitation/CROLs		r		
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII: System performance				
System performance was found to be acceptable.				
XIII Overall assessment of data (i.e., i.e.,				
Overall assessment of data was found to be acceptable.	/			
XIV. Field duplicates				
Were field duplicate pairs identified in this SDG?		/	1	
Were target compounds idetected in the field duplicates?			/	
XV: Field:blanks				1 - 200 CI
Were field blanks identified in this SDG?				
Were target compounds detected in the field blanks?			/	

SDG #: See COM LDC #: 191888

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

o o Reviewer: 2nd Reviewer:

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

					 ,	
Recalculated	%RSD	3915				
Reported	%RSD	3/68				
Recalculated	Average CF (initial)	1718=732				
Reported	Average CF (initial)	516 E 216 8 28/21/28/21/28/28/20/58/8/				
Recalculated	CF (Ø, /std)	1835.70				
Reported	CF (// . / std)	ades ESI				
	Compound	025				
	Calibration Date	81/6=/5				
	Standard ID	1940				
	#	-	2	е	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.

SDG #: Set COMM LDC #: 1918847

Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

/of /	3	
Page:	Reviewer:_	2nd Reviewer:_

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

Recalculated	Q %	6.5	do .		
Reported	%D	4	2		
Recalculated	CF/Conc. CCV	0.9982	7286.0		
Reported	CF/Conc. CCV	7	The 86.0		
	Average CF(Ical)/ CCV Conc.	0'1	0.7		
	Compound	₹R0	9RO		
	Calibration Date	042348 6/13/08	81/11/9 3904		
	Standard ID		Cho		
L_	#		72	<u>۳</u>	4

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

LDC #: 19/3847 SDG #: 500 conn

VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

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

% Recovery: SF/SS * 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Percent Difference

Recalculated Percent Recovery g Percent Recovery Reported X 03383 Surrogate Found Surrogate Spiked Column/Detector N Surrogate Sample ID:

l	
l	
ı	
	le ID:
	mple
	ιŭL

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

Sample ID:

t Percent ry Difference			
Percent Recovery	Recalculated		
Percent Recovery	Reported		
Surrogate Found			
Surrogate Spiked			
Column/Detector			
Surrogate			

LDC #: 91/284/ SDG #: 544 @W/

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

Reviewer:

METHOD: GC_HPLC

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

Where: SSC = Spiked sample concentration SA = Spike added

SC = Concentration

RPD = I SSCLCS - SSCLCSD I * 2/(SSCLCS + SSCLCSD)

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples: タ765269

	σ.	Spike	Spiked	Sample	רנ	rcs	วา	CSD	/SOT	TCS/TCSD
Compound	*) 	Added / WAAA		Concentration	Percent	Percent Recovery	Percent	Percent Recovery	~	RPD
	SDT	LCSD	rcs	rcsD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)	6.1	1.0	0.1	D.944	100	26/	76	76		8.8
Diesel (8015)										
Benzene (8021B)										-
Methane (RSK-175)										
2,4-D (8151)										
Dinoseb (8151)										
Naphthalene (8310)					1					
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.

VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

2nd Reviewer:

HPLC

ا ا	Were a Were a
	Y N N/A
	_

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

Example:		Sample ID.
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

Df= Dilution Factor

RF= Average response factor of the compound Vs= Initial volume of the sample Ws= Initial weight of the sample %S= Percent Solid In the initial calibration

Concentration =_

Compound Name_

	Ī	<u> </u>	Γ-	Γ	l l	
Qualifications						
Recalculated Results Concentrations (
Reported Concentrations (
Compound						
Sample ID						
#	-					

Comments:

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

BRC Tronox Parcel G

Collection Date:

June 11, 2008

LDC Report Date:

August 6, 2008

Matrix:

Soil

Parameters:

Diesel Range Organics

Validation Level:

EPA Level III & IV

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

Sample Identification

TSB-GJ-09-10'

TSB-GJ-09-20'**

TSB-GJ-09-30'

TSB-GJ-09-40'

^{**}Indicates sample underwent EPA Level IV review

Introduction

This data review covers 4 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8015B for Diesel Range Organics.

This review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- 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.
- 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 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 15.0% QC limits.

The percent differences (%D) of the second source calibration standard were less than or equal to 15.0% for all compounds.

III. Blanks

Method blanks were reviewed for each matrix as applicable. No diesel range organic 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) were within QC limits.

V. Target Compound Identification

All target compound identifications were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

VI. Compound Quantitation and CRQLs

All compound quantitation and CRQLs were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

VII. System Performance

The system performance was acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

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.

BRC Tronox Parcel G Diesel Range Organics - Data Qualification Summary - SDG F8F120180

No Sample Data Qualified in this SDG

BRC Tronox Parcel G Diesel Range Organics - Laboratory Blank Data Qualification Summary - SDG F8F120180

No Sample Data Qualified in this SDG

BRC Tronox Parcel G
Diesel Range Organics - Field Blank Data Qualification Summary - SDG
F8F120180

No Sample Data Qualified in this SDG

SDG Labor	#: 19188A8 #: F8F120180 ratory: Test America		Le	evel III/I\		Date:8/4/v Page: /of/ Reviewer: 2nd Reviewer:			
Γhe s	_					n findings are noted in attached			
	Validation A	\rea			Comme	ents			
ı.	Technical holding times		4	Sampling d	ates: 6/11/08				
lla.	Initial calibration		4						
IIb.	Calibration verification/ICV		A	K21 =	= 1570				
111.	Blanks		A		7				
lVa.	Surrogate recovery		4	,					
IVb.	Matrix spike/Matrix spike dupl	licates	HA	a le	ul perties	- TSB 41-08-10			
IVc.	Laboratory control samples		A	100	_				
V.	Target compound identification			Not review	ed for Level III validation.				
VI.	Compound Quantitation and CRQLs			Not reviewed for Level III validation.					
VII.	System Performance			Not reviewed for Level III validation.					
VIII.	Overall assessment of data		A						
IX.	Field duplicates		_ N						
X.	Field blanks								
lote:	A = Acceptable N = Not provided/applicable SW = See worksheet	R = Rinsate FB) = No compounds = Field blank	TE	D = Duplicate 3 = Trip blank EB = Equipment blank	(
/alidat	ed Samples: ** Indica		derwent Level IV	validation I					
1			= 29/MD	21		31			
2	TSB-GJ-09-20'**	12 <u>8170</u>	312 MB	22		32			
3 1	TSB-GJ-09-30' /	13		23		33			
4	TSB-GJ-09-40'	14		24	,	34			
5		15		25		35			
6		16		26		36			
7		17		27		37			
8		18		28		38			
9		19		29		39			
10		20		30		40			

Notes:_

LDC #: 19188 A8 SDG #: See cons

VALIDATION FINDINGS CHECKLIST

Page: /of // Reviewer: ______

	/ 00 1101.0				
Method:	Validation Area	Yes	No	NA	Findings/Comments
f Technical holding times	Validation Area	11001			
All technical holding times	were met.		/		
Cooler temperature criteria					
II Initial calibration					
	a 5 point calibration prior to sample analysis?				
	aluation? If yes, were all percent relative standard				
Was a curve fit used for evused?	aluation? If Yes, what was the acceptance criteria				
Did the initial calibration me	eet the curve fit acceptance criteria?				
Were the RT windows prop	perly established?				an a
IV: Continuing calibration		1		T	
What type of continuing ca %R	libration calculation was performed?%D or				
Was a continuing calibration	on analyzed daily?	/			
Were all percent difference	es (%D) < 15%.0 or percent recoveries 85-115%?	1/			
Were all the retention time	s within the acceptance windows?				
V. Blanks			I-	T ·	
Was a method blank asso-	ciated with every sample in this SDG?	1/		ļ	
Was a method blank analy	zed for each matrix and concentration?	1/			
Was there contamination i validation completeness w	n the method blanks? If yes, please see the Blanks orksheet.				OUR NORTH COMMITTEE COMMIT
VI. Surrogate spikes			F		
Were all surrogate %R wit	hin the QC limits?	1/		 	
If the percent recovery (%large) a reanalysis performed to	R) of one or more surrogates was outside QC limits, wa confirm %R?	ıs			
If any %R was less than 1	percent, was a reanalysis performed to confirm %R?		HALLAD DAVE		
VII. Matrix spike/Matrix spi	ke duplicates	- T		T -	
Were a matrix spike (MS) matrix in this SDG? If no, i MS/MSD. Soil / Water.	and matrix spike duplicate (MSD) analyzed for each ndicate which matrix does not have an associated			-	
	every 20 samples of each matrix?		/		
Were the MS/MSD percen (RPD) within the QC limits	t recoveries (%R) and the relative percent differences ?		No. of the Control of	/	
VIII. Laboratory control sa	mples The state of	jýr T	7 1		T
Was an LCS analyzed for	this SDG?	44	-	-	
Was an I CS analyzed per	extraction hatch?	-1/	1		

LDC#: 1918848 SDG#: <u>Sec CO W</u>

VALIDATION FINDINGS CHECKLIST

Page: <u>→</u> of <u>→</u>
Reviewer:
2nd Reviewer:

Validation Area	Yes	No	NA	Findings/Comments
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?	Section Sections			
X Target compound identification				
Were the retention times of reported detects within the RT windows?		e Zalechell (2)		
XI: Compound quantitation/CRQLS				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII; System performance			(1) (1) (1)	
System performance was found to be acceptable.				
XIII. Overall assessment of data and any first the second	Ż			
Overall assessment of data was found to be acceptable.				
XIV Field duplicates				grand and the second se
Were field duplicate pairs identified in this SDG?				
Were target compounds idetected in the field duplicates?			/	
XV. Field blanks		4		
Were field blanks identified in this SDG?				
Were target compounds detected in the field blanks?				

LDC #: 1918848 SDG # Secon

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

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Page:	Reviewer:	2nd Reviewer:

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

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

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

			Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
Standard ID	Calibration Date	Compound	CF (/ ^{OTStd})	CF (/estd)	Average CF (initial)	Average CF (initial)	%RSD	%RSD
	2/19/2	280	768.51	76851	16023		3.456	3.456
			-					

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 #: 191088-43 SDG#:2

Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

of	4	
Page:	Reviewer.	2nd 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

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

					Reported	Recalculated	Reported	Recalculated
	Standard ID	Calibration Date	Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc. CCV	Q %	Q%
	54574D	80/7/2 SESTA	ako.	0001	3/2	85966	0.3	0.0
2	44637	24537 6/17/08 DRO	DRO	0001	K95/7601	25/200)	3.5	15 'E
_								
						A THE STATE OF THE		

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.

8	3
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X	for
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METHOD: / GC __ HPLC

VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page: ___of___ Reviewer: _______ 2nd reviewer: _______

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

	Percent Difference		⊘		
	Percent Recovery	Recalculated	25		
	Percent Recovery	Reported	<i>S</i> 82		
1	Surrogate Found		10/5-12		
oo - ourrogate opiked	Surrogate Spiked		0.50		
	Column/Detector		\sqrt{s}		
Sample ID; 🔑	Surrogate		TFH		

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

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

SDG #: Sec Cours LDC #:/9/8348

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

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

METHOD:

GC HPLC

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

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

Where:

SC = Concentration

LCSD = Laboratory control sample duplicate percent recovery

RPD = I SSCLCS - SSCLCSD I * 2/(SSCLCS + SSCLCSD) LCS/LCSD samples: 3/6529

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

	dS	ike	Spiked	Sample	SOT	S	rcsd	D)	I/SOI	LCS/LCSD
Compound	Ad M	Added (M7/S)	Concel (M&	Concentration (MAS 13	Percent Recovery	tecovery	Percent Recovery	ecovery	R	RPD
	SOT	rcsD	rcs	CSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)										
Diesel (8015)	83.3	NA	68.9	NA	80	W W				
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.

LDC #: A B S 8

VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

2nd Reviewer:

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_	GC HP	
	тнор: 🗡	
	METH	

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

(A)(Fv)(Df)	(RF)(Vs or Ws)(%S/100)	
Concentration=	(R	

A= Area or height of the compound to be measured Fv= Final Volume of extract Df= Dilution Factor

RF= Average response factor of the compound In the initial calibration

Vs= Initial volume of the sample Ws= Initial weight of the sample %S= Percent Solid

Concentration =_

Compound Name

Sample ID.

Example:

#	Sample ID	Compound	Reported Concentrations (Recalculated Results Concentrations (Qualifications

Comments:

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

BRC Tronox Parcel G

Collection Date:

June 11, 2008

LDC Report Date:

August 8, 2008

Matrix:

Soil

Parameters:

Polynuclear Aromatic Hydrocarbons

Validation Level:

EPA Level III & IV

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

Sample Identification

TSB-GJ-09-10'

TSB-GJ-09-20'**

TSB-GJ-09-30'

TSB-GJ-09-40'

^{**}Indicates sample underwent EPA Level IV review

Introduction

This data review covers 4 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8310 for Polynuclear Aromatic Hydrocarbons.

This review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- 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) were less than or equal to 20.0% for all compounds.

Retention time windows were evaluated and considered technically acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples on which a Level III review was performed.

b. Calibration Verification

Calibration verification was performed at required frequencies.

The percent differences (%D) of calibration factors in continuing standard mixtures were within the 15.0% QC limits with the following exceptions:

Date	Detector	Compound	%D	Associated Samples	Flag	A or P
6/16/08	Not specified	Benzo(g,h,i)perylene	15.2	TSB-GJ-09-10' TSB-GJ-09-20'**	J+ (all detects)	А

The percent differences (%D) of the second source calibration standard were less than or equal to 15.0% for all compounds with the following exceptions:

Date	Detector	Compound	%D	Associated Samples	Flag	A or P
6/4/08	Not specified	Benzo(k)fluoranthene	16.6	All samples in SDG F8F120180	J+ (all detects)	А

Retention time windows were evaluated and considered technically acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples on which a Level III review was performed.

III. Blanks

Method blanks were reviewed for each matrix as applicable. No polynuclear aromatic hydrocarbon 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) were within QC limits.

V. Target Compound Identification

All target compound identifications were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

VI. Compound Quantitation and CRQLs

All compound quantitation and CRQLs were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

VII. System Performance

The system performance was acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

VIII. Overall Assessment of Data

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

IX. Field Duplicates

No field duplicates were identified in this SDG.

BRC Tronox Parcel G Polynuclear Aromatic Hydrocarbons - Data Qualification Summary - SDG F8F120180

SDG	Sample	Compound	Flag	A or P	Reason
F8F120180	TSB-GJ-09-10' TSB-GJ-09-20'**	Benzo(g,h,i)perylene	J+ (all detects)	А	Continuing calibration (%D)
F8F120180 TSB-GJ-09-10' TSB-GJ-09-20'** TSB-GJ-09-30' TSB-GJ-09-40'		Benzo(k)fluoranthene	J+ (all detects)	А	Continuing calibration (ICV %D)

BRC Tronox Parcel G
Polynuclear Aromatic Hydrocarbons - Laboratory Blank Data Qualification Summary
- SDG F8F120180

No Sample Data Qualified in this SDG

BRC Tronox Parcel G Polynuclear Aromatic Hydrocarbons - Field Blank Data Qualification Summary -SDG F8F120180

No Sample Data Qualified in this SDG

									-11/		
	#: <u>19188A9</u>	LIDATIO	I COMPLETENESS WORKSHEET				Date 8/4/0				
	#: F8F120180		Le	evel II	II/I\			Page: /of/			
Labo	ratory: Test America								Reviewer: 2nd Reviewer:		
METI	HOD: GC Polynuclear A	omati	c Hydrocarb	ons (EPA	SW 8	846 ľ	Method 8310)				
	amples listed below wer ation findings worksheets		ewed for eac	ch of the fo	ollowin	ng va	alidation areas. Validatio	n find	dings are noted in attached		
	Validation	Validation Area				Comments					
I.	Technical holding times			4	Sampling dates: 6/11/08						
Ila.	Initial calibration										
llb.	Calibration verification/ICV	Calibration verification/ICV			10	√ ≤	£1570				
111.	Blanks			A	<u>'</u>						
IVa.	Surrogate recovery			*							
IVb.	Matrix spike/Matrix spike d	uplicate	s	4	78B-GJ-08-10'						
IVc.	Laboratory control samples	3		4	209						
V.	Target compound identifica	Target compound identification			Not reviewed for Level III validation.						
VI.	Compound Quantitation ar	Compound Quantitation and CRQLs			Not reviewed for Level III validation.						
VII.	System Performance			4	Not reviewed for Level III validation.						
VIII.	Overall assessment of data	a		A							
IX.	Field duplicates	Field duplicates									
X.	Field blanks										
Note:	A = Acceptable N = Not provided/applicable SW = See worksheet	e	R = Rins	o compounds sate eld blank	s detect	ted	D = Duplicate TB = Trip blank EB = Equipment blank	ς.			
√alida	ted Samples: ** Indicates san	nple und	derwent Level I	IV validation							
1	TSB-GJ-09-10'	11	81681	58MV	3 1	21		31			
2	TSB-GJ-09-20'**	12			2	22		32			
3	TSB-GJ-09-30'	/ 13				23		33			
4	TSB-GJ-09-40'	14				24		34			
5	•	15				25		35			
6		16				26		36			
7		17				27		37			
8		18				28		38			
9		19				29		39			
10		20				30		40			

Notes:

LDC #: 1918889 SDG #: <u>Sa cow</u>

VALIDATION FINDINGS CHECKLIST

Page:_/of___ Reviewer:_____ 2nd Reviewer:_____

Method: GC HPLC				
Validation Area	Yes	No	NA	Findings/Comments
f Technical holding times			· I	A SHARE THE STATE OF THE STATE
All technical holding times were met.				
Cooler temperature criteria was met.			ar more than	
(Finitial calibration				
Did the laboratory perform a 5 point calibration prior to sample analysis?				
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) ≤ 20%?				
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?				
Did the initial calibration meet the curve fit acceptance criteria?				
Were the RT windows properly established?			· · · · · · · · · · · · · · · · · · ·	
IV Continuing calibration				
What type of continuing calibration calculation was performed?%D or %R				
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?		con S. Wall Day 17.	***	
M/Blanks			T	
Was a method blank associated with every sample in this SDG?			ļ	
Was a method blank analyzed for each matrix and concentration?			ļ	
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?	1	<u> </u>		
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?				
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?				

VIII: Laboratory control samples ***
Was an LCS analyzed for this SDG?

Was an LCS analyzed per extraction batch?

VII. Matrix spike/Matrix spike duplicates

MS/MSD. Soil / Water.

(RPD) within the QC limits?

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

Were the MS/MSD percent recoveries (%R) and the relative percent differences

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

LDC#: 1918849 SDG#: 2000

VALIDATION FINDINGS CHECKLIST

	Page:	<u></u> _of
	Reviewer:	9
2nd	Reviewer:	

Validation Area	Yes	No	NA	Findings/Comments
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		-		
IX Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
X. Target compound identification	ı			
Were the retention times of reported detects within the RT windows?		*645398261		
XI: Compound quantifation/CRQLs	T		į į į	
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII. System performance			450 100 100 100 100 100 100 100 100 100 1	Here is a second of the second
System performance was found to be acceptable.				
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			
XIV: Field duplicates				
Were field duplicate pairs identified in this SDG?				
Were target compounds idetected in the field duplicates?			/	
XV. Field blanks				
Were field blanks identified in this SDG?				
Were target compounds detected in the field blanks?			/	

VALIDATION FINDINGS WORKSHEET

METHOD: GC HPLC

8310	8330	8151	8444	× × × × × × × × × × × × × × × × × × ×	07000
				(1. uon): + : 0	00710
A. Acenaphthene	A. HMX	A. 2,4-D	A. Dichlorvos	V. Fensulfothion	V. Benzene
B. Acenaphthylene	B. RDX	B. 2,4-DB	B. Mevinphos	W. Bolstar	CC. Toluene
C. Anthracene	C. 1,3,5-Trinitrobenzene	C. 2,4,5-T	C. Demeton-O	X. EPN	EE. Ethyl Benzene
D. Benzo(a)anthracene	D. 1,3-Dinitrobenzene	D. 2,4,5-TP	D. Demeton-S	Y. Azinphos-methyl	SSS. O-Xylene
E. Benzo(a)pyrene	E. Tetryl	E. Dinoseb	E. Ethoprop	Z. Coumaphos	RRR. MP-Xylene
F. Benzo(b)fluoranthene	F. Nitrobenzene	F. Dichlorprop	F. Naled	AA. Parathion	GG. Total Xylene
G. Benzo(g,h,i)perylene	G. 2.4.6-Trinitrotoluene	G. Dicamba	G. Sulfotep	BB. Trichloronate	
H. Benzo(k)fluoranthene	H. 4-Amino-2,6-dinitrotoluene	H. Dalapon	H. Phorate	CC. Trichlorinate	
I. Chrysene	I. 2-Amino-4,6-dinitrotoluene	I. MCPP	I. Dimethoate	DD. Trifluralin	
J. Dibenz(a,h)anthracene	J. 2,4-Dinitrotolune	Ј. МСРА	J. Diazinon	EE. Def	
K. Fluoranthene	K. 2,6-Dinitrotoluene	K. Pentachlorophenol	K. Disulfoton	FF. Prowl	
L. Fluorene	L. 2-Nitrotoluene	L 2,4,5-TP (silvex)	L. Parathion-methyl	GG. Ethion	
M. Indeno(1,2,3-cd)pyrene	M. 3-Nitrotoluene	M. Silvex	M. Ronnel	HH. Tetrachlorvinphos	
N. Naphthalene	N. 4-Nitrotoluene		N. Malathion	II. Sulprofos	
O. Phenanthrene	О.		O. Chlorpyrifos		
P. Pyrene	.		P. Fenthion		
Ċ	8		Q. Parathion-ethyl		
 			R. Trichloronate		
ý.			S. Merphos		
			T. Stirofos		
			U. Tokuthion		

cmpd_list.wpd

SDG #: LELCON LDC #: 19188

METHOD:

VALIDATION FINDINGS WORKSHEET **Continuing Calibration**

Page:____Reviewer:___

2nd Reviewer:

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

Y N N/A

What type of continuing calibration calculation was performed? / %D or RPD Were continuing calibration standards analyzed at the required frequencies?

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

Level IV Only Y N N/A

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

Oualifications	1 to to to		1 1 7	Lasts 10													
Associated Samples	M+AC		/														
RT (limit)						() ,))	\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	(((((_
%D / RPD (Limit < 15.0)	16.6		15.2														
Compound	1		#	\ .													
Detector/ Column	\ \ \		NS		,												
Standard ID	101	,	AC4-2873	,													T
# Date	7		8/19/18														

SDG#: Secon LDC #: 19/887

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

/ ot/	d	
Page:_	Reviewer:_	and Beyjawar

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

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

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

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 #: 1718849 SDG#: \

Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

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

Recalculated	Q%	6.9	w W	6./	6 N					
Reported	Q%		W	6./	ė,					
Recalculated	CF/Conc. CCV	0.5344	0.4834	0.5307	728h.0					
Reported	CF/Conc. CCV		0.4834	0.5307	7867.0	, ,				
	Average CF(Ical)/ CCV Conc.	25.0	<i>></i>	0.50	1					
	Compound	V	A	9	b					
	Calibration Date	80/91/9		8/17/19						
	Standard ID	&cd/2862		2 824-873 61,61,8	/					
	#	-		7			9		4	

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

LDC#: 718849 SDG#: 54 COUN

VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Reviewer: C

METHOD: ___ GC __ HPLC

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

% Recovery: SF/SS * 100

4

Where: SF = Surrogate Found SS = Surrogate Spiked

Surrogate Surrogate Surrogate Percent Recovery Percent Difference 7PH NS NS	Sample ID:		30000	•			
.4 NS 25.0 18.35.28 7.3 7.3 1.3	Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
4 NS Q50 183528 73 73 T						Recalculated	
	<i>74</i> 74	NS	035.0	18.3528	ł	73	0

٥	1
9	b
3	2
E	=

Percent Difference			
Percent F Recovery Di	Recalculated		
Percent Recovery	Reported		
Surrogate Found			
Surrogate Spiked			
Column/Detector			
Surrogate			

Sample ID:

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

LDC #/9/8849

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

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

SSC = Spiked sample concentration Where:

SC = Concentration

RPD = I SSCLCS - SSCLCSD I * 2/(SSCLCS + SSCLCSD)

SA = Spike added LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples: \$768/58

	S.	oike	Spiked	Sample	רנ	SOT	rcsd	as	/SOT	TCS/TCSD
Compound	\$2)	Added Hales	Conce	Concentration	Percent I	Percent Recovery	Percent Recovery	ecovery	R	RPD
	CCS	LCSD	rcs	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)										
Diesel (8015)										
Benzene (8021B)										
Methane (RSK-175)										
2,4-D (8151)										
Dinoseb (8151)										
Naphthalene (8310)	1.79	47	9:25	NÅ	40	79				
Anthracene (8310)	/\		5/5	>	, 22	77				
HMX (8330)	,				1	,				
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.

LDC #: 10/88/4/

VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Reviewer: 2nd Reviewer:

> GC V HPLC METHOD

İ		
	A A	
<u> </u>	N N	

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

Example:	- -	Sample ID.
(A)(Fv)(Df)	(RF)(Vs or Ws)(%S/100)	
Concentration=	<u>R</u>	A -

Compound Name

A= Area or height of the compound to be measured Fv= Final Volume of extract Df= Dilution Factor

RF= Average response factor of the compound

Concentration =_

In the initial calibration
Vs= Initial volume of the sample
Ws= Initial weight of the sample
%S= Percent Solid

1	<u></u>	 		 	 	
	Qualifications					
	Recalculated Results Concentrations (
	Reported Concentrations (
	Compound					
	Sample ID					
	*					

Comments:

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

BRC Tronox Parcel G

Collection Date:

June 11, 2008

LDC Report Date:

August 8, 2008

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

EPA Level III & IV

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): F8F120180

Sample Identification

TSB-GJ-09-10'

TSB-GJ-09-20'**

TSB-GJ-09-30'

TSB-GJ-09-40'

^{**}Indicates sample underwent EPA Level IV review

Introduction

This data review covers 4 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

This review follows USEPA Contract Laboratory Program National Functional Guidelines for Polychlorinated Dioxins/Dibenzofurans Data Review (September 2005) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Samples indicated by a double asterisk on the front cover underwent EPA Level IV review. EPA Level III review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by EPA Level III criteria since this review is based on QC data.

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- 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.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- 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. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25%.

The exact mass of 380.9760 of PFK was verified. The static resolving power was at least 10,000 (10% valley definition) for samples on which EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by EPA Level III criteria.

III. Initial Calibration

A five point initial calibration was performed as required by the method.

Percent relative standard deviations (%RSD) were less than or equal to 20.0% for unlabeled compounds and less than or equal to 30.0% for labeled compounds.

The ion abundance ratios for all PCDDs and PCDFs were within validation criteria.

The minimum S/N ratio for each target compound was greater than or equal to 2.5 and and greater than or equal to 10 for each recovery and internal standard compound for samples on which EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by EPA Level III criteria.

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

All of the routine calibration percent differences (%D) between the initial calibration RRF and the routine calibration RRF were less than or equal to 20.0% for unlabeled compounds and less than or equal to 30.0% for labeled compounds with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
7/7/08	¹³ C-2,3,7,8-TCDF	37.2	TSB-GJ-09-40'	J+ (all detects)	Р

The ion abundance ratios for all PCDDs and PCDFs were within validation criteria.

V. Blanks

Method blanks were reviewed for each matrix as applicable. No polychlorinated dioxin/dibenzofuran contaminants were found in the method blanks.

No field blanks were identified in this SDG.

VI. 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.

VII. Laboratory Control Samples (LCS)

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

LCS ID	Compound	%R (Limits)	Associated Samples	Flag	A or P
8170493LCS	1,2,3,7,8,9-HxCDD OCDD	137 (71-129) 154 (74-144)	TSB-GJ-09-10' TSB-GJ-09-30' TSB-GJ-09-40' 8170493MB	J+ (all detects) J+ (all detects)	Р

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

All internal standard recoveries were within QC limits with the following exceptions:

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
TSB-GJ-09-30'	¹³ C-2,3,7,8-TCDF ¹³ C-1,2,3,7,8-PeCDF ¹³ C-1,2,3,7,8-PeCDD ¹³ C-1,2,3,4,7,8-HxCDD ¹³ C-1,2,3,6,7,8-HxCDD ¹³ C-1,2,3,4,6,7,8-HpCDF ¹³ C-1,2,3,4,6,7,8-HpCDD	38 (40-135) 26 (40-135) 27 (40-135) 18 (40-135) 21 (40-135) 11 (40-135) 16 (40-135) 9.7 (40-135)	1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,7,8-PeCDF 2,3,7,8-TCDF 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HyCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF 0CDF	J (all detects) UJ (all non-detects)	Р

X. Target Compound Identifications

All target compound identifications were within validation criteria for samples on which EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by EPA Level III criteria.

XI. Compound Quantitation and CRQLs

All compound quantitation and CRQLs were within validation criteria for samples on which EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by EPA Level III criteria.

XII. System Performance

The system performance was acceptable for samples on which EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by EPA Level III criteria.

XIII. Overall Assessment of Data

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

XIV. Field Duplicates

No field duplicates were identified in this SDG.

BRC Tronox Parcel G Dioxins/Dibenzofurans - Data Qualification Summary - SDG F8F120180

					<u> </u>
SDG	Sample	Compound	Flag	A or P	Reason
F8F120180	TSB-GJ-09-40'	2,3,7,8-TCDF	J+ (all detects)	Р	Routine calibration (%D)
F8F120180	TSB-GJ-09-10' TSB-GJ-09-30' TSB-GJ-09-40'	1,2,3,7,8,9-HxCDD OCDD	J+ (all detects) J+ (all detects)	Р	Laboratory control samples (%R)
F8F120180	TSB-GJ-09-30'	1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 2,3,7,8-TCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 0CDF	J (all detects) UJ (all non-detects)	Р	Internal standards (%R)

BRC Tronox Parcel G Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG F8F120180

No Sample Data Qualified in this SDG

BRC Tronox Parcel G Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG F8F120180

No Sample Data Qualified in this SDG

_DC #:19188A21 SDG #:F8F120180 _aboratory: Test America METHOD: HRGC/HRMS Diox The samples listed below were validation findings worksheets.	 ins/Dibenzofuran reviewed for eac	Leve es (EPA SW	el III/I\ 846 Me	thod 8290)		Date: 3/4/0 Page: of / Reviewer: 2nd Reviewer:
Validation	Area			Co	mments	
Technical holding times		A s	ampling d	ates: 4/11/0	8	
II. GC/MS Instrument performa	ince check	4		7 7		
III. Initial calibration		A				
IV. Routine calibration/I √		W				
V. Blanks		4		_	_	
VI. Matrix spike/Matrix spike du	plicates	N /	die	ent Det	'od	
VII. Laboratory control samples	,	m	20	9 1 1	-	
VIII. Regional quality assurance	and quality control	N				
IX. Internal standards		W				
X. Target compound identificat	ions	<u>#</u>	ot review	ed for Level III validation		
XI. Compound quantitation and		7		ed for Level III validation		
	System performance			ed for Level III validation		
XIV. Field duplicates		 			<u> </u>	
XV. Field blanks	*					
Note: A = Acceptable N = Not provided/applicable SW = See worksheet /alidated Samples: ** Indicates samp	R = Rin FB = Fid	eld blank	etected	D = Duplicate TB = Trip blank EB = Equipment	blank	
1 / TSB-GJ-09-10'	11 87704	93 ME	\$ 21		31	
2 TSB-GJ-09-20'**	12 8/7/5	9/MP	> 22		32	
3 / TSB-GJ-09-30'	/13		23		33	
4 TSB-GJ-09-40'	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:						



VALIDATION FINDINGS CHECKLIST

Page: /of 3
Reviewer: 9
2nd Reviewer:

Method: Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.				
IF GG/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?				
Were the retention time windows established for all homologues?				
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers \leq 25% ?				
is the static resolving power at least 10,000 (10% valley definition)?				
Was the mass resolution adequately check with PFK?				
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?				
III, Initial calibration				
Was the initial calibration performed at 5 concentration levels?				
Were all percent relative standard deviations (%RSD) \leq 20% for unlabeled standards and \leq 30% for labeled standards?	/			
Did all calibration standards meet the Ion Abundance Ratio criteria?	/			
Was the signal to noise ratio for each target compound \geq 2.5 and for each recovery and internal standard \geq 10?	/			
IV. Continuing calibration	,			
Was a routine calibration performed at the beginning and end of each 12 hour period?				
Were all percent differences (%D) \leq 20% for unlabeled standards and \leq 30% for labeled standards?		/		
Did all routine calibration standards meet the Ion Abundance Ratio criteria?				
V. Blanks				
Was a method blank associated with every sample in this SDG?				
Was a method blank performed for each matrix and concentration?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?				
VI. 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.		_		
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VII. Laboratory control samples	,			1
Was an LCS analyzed for this SDG?				

LDC #: 19188421 SDG #: <u>Sacouv</u>

VALIDATION FINDINGS CHECKLIST

Page:of
Reviewer: 😙
2nd Reviewer:

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?				
VIII. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
IX. Internal standards				
Were internal standard recoveries within the 40-135% criteria?				
Was the minimum S/N ratio of all internal standard peaks \geq 10?				
X. Target compound identification				
For 2,3,7,8 substituted congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard?				
For 2,3,7,8 substituted congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration?				
For non-2,3,7,8 substituted congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution?				
Did compound spectra contain all characteristic ions listed in the table attached?				
Was the Ion Abundance Ratio for the two quantitation ions within criteria?				
Was the signal to noise ratio for each target compound and labeled standard \geq 2.5?			/	
Does the maximum intensity of each specified characteristic ion coincide within \pm 2 seconds (includes labeled standards)?				
For PCDF identification, was any signal (S/N \geq 2.5, at \pm seconds RT) detected in the corresponding PCDPE channel?				
Was an acceptable lock mass recorded and monitored?				
XI. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII, System performance		-		
System performance was found to be acceptable.				
XIII. Overall assessment of data		-		
Overall assessment of data was found to be acceptable.				
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.		/		

LDC #: 19188 A > SDG #: See COUN

VALIDATION FINDINGS CHECKLIST

Page: <u>∂</u> o	<u>f.≥</u>
Reviewer:	
2nd Reviewer:	

Validation Area	Yes	No	NA	Findings/Comments
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.			7	

VALIDATION FINDINGS WORKSHEET

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

A. 2,3,7,8-TCDD	F. 1,2,3,4,6,7,8-HpCDD	K. 1,2,3,4,7,8-HxCDF	P. 1,2,3,4,7,8,9-HpCDF	U. Total HpCDD
B. 1,2,3,7,8-PeCDD	G. OCDD	L. 1,2,3,6,7,8-HxCDF	Q. OCDF	V. Total TCDF
C. 1,2,3,4,7,8-HxCDD	H. 2,3,7,8-TCDF	M. 2,3,4,6,7,8-HxCDF	R. Total TCDD	W. Total PeCDF
D. 1,2,3,6,7,8-HxCDD	I. 1,2,3,7,8-PeCDF	N. 1,2,3,7,8,9-HxCDF	S. Total PeCDD	X. Total HxCDF
E. 1,2,3,7,8,9-HxCDD	J. 2,3,4,7,8-PeCDF	O. 1,2,3,4,6,7,8-HpCDF	T. Total HxCDD	Y. Total HpCDF

Notes:

LDC #: 1918842/ SDG #: 22 20 W

VALIDATION FINDINGS WORKSHEET Routine Calibration

Reviewer: Cand Reviewer:

NOTIFIE CAIDIANT

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

N NA N NA

Were all percent differences (%D) of RRFs \leq 20% for unlabeled compounds and \leq 30% for labeled? Was a routine calibration was performed at the beginning and end of each 12 hour period?

Did all routine calibration standards meet the Ion Abundance Ratio criteria?

OR 108102 E	Compound		Finding Ion Abundance Ratio	Associated Samples	Qualifications
1	,	1.7		4	That I
	,				
	Selected ions (m/z)	Ion Abundance Ratio	PCDFs	Selected ions (m/z)	z) Ion Abundance Ratio
	M/M+2	0.65-0.89	Tetra-	M/M+2	0.65-0.89
	M+2/M+4	1.32-1.78	Penta-	M+2/M+4	1.32-1.78
	M+2/M+4	1.05-1.43	Hexa-	M+2/M+4	1.05-1.43
	M/M+2	0.43-0.59	Hexa-13C-HxCDF (IS) only	M/M+2	0.43-0.59
	M/M+2	0.37-0.51	Hepta-13C-HpCDF (IS) only		0.37-0.51
	M+2/M+4	0.88-1.20	Hepta-	M+2/M+4	0.88-1.20
	M+2/M+4	0.76-1.02	Octa-	M+2/M+4	0.76-1.02

VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

LDC #: 1918242

Reviewer:__ Page:

2nd Reviewer:

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

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

Was a LCS required?

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

N NA Y N NA

Qualifications Associated Samples Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits? RPD (Limits) LCSD %R (Limits) 521(74/44 137 (71-12) LCS %R (Limits) Compound W W Lab ID/Reference Date *

VALIDATION FINDINGS WORKSHEET Internal Standards

Page: Reviewer:

2nd Reviewer:

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

LDC #: 10/88/1-8

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". YANA Are all internal standard recoveries were within the 40-135% criteria?

Was the S/N ratio all internal standard peaks \geq 10?

Qualifications	JUJ (B-R)																				Check Standard Used									
% Recovery (Limit: 40-135%)	38 (40-135-))	37 ()	() &/) / ~) //	()) '7/	() Tib	())	()	()	()		(()	(()	()	()	Recovery Standards	K. 13C-1,2,3,4-TCDD	L. 1°C-1,2,3,7,8,9-HxCDD	M.	N.	O.	Ъ.	Ö	ď	
Internal Standard	*	U	R	7	7	4	++	77,													Check Standard Used									
Lab ID/Reference	X																				Internal Standards	JF	ac	eCDF	eCDD	HxCDF	HxCDD	8-HpCDF	8-HpCDD	
# Date														*								A. 13C-2,3,7,8-TCDF			D. 13C-1,2,3,7,8-Pe	E. 13C-1,2,3,6,7,8-HxCDF	H	G. 13C-1,2,3,4,6,7,8-HpCDF	H. ¹³ C-1,2,3,4,6,7,8-HpCDD	\dashv

LDC #: 19188424

VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: of L

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

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 = $(A_{\lambda})(C_{\kappa})/(A_{\mu})(C_{\lambda})$ average RRF = sum of the RRFs/number of standards %RSD = 100 * (S/X)

 $A_x = Area of compound,$ $A_k = C_x = C_x = C_x = C_x = S$ S = Standard deviation of the RRFs, X = P

 $A_{\bf k}=$ Area of associated internal standard $C_{\bf k}=$ Concentration of internal standard X= Mean of the RRFs

L									
				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF	RRF	6	
Ŀ	141		7378,100 (10,00,00)	000			(ma)	%RSD	%HSD
		8/2/1	(AO) 10' 10' 10' 10' 10' 10' 10' 10' 10' 10'	0.140	0.178	0.0	0.84	12.61	018
\int		00/11/	2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	5.63	0,913	083	noo	102	10 3
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1580	KXO	187	1x0	000%	1/1/
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.8det	July 0	880	000	N. C.	1.21
			OCDF (4°C-OCDD)	たん!	パルン	7.80	000	5.0	13:1
,						0	1	10:	16 3
~			2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)						
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)						
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)						
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8,-HpCDD)						
			OCDF (19c-OCDD)						
e			23.78-TODE (¹⁸ 0-23.78-TODE)						
		1	2,3,7,8-TCDD (13C-2,3,7,8-TCDD)						
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)						
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)						
			OCDF (4c-OCDD)						

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

· SDG #: SECCOMY LDC #: 1918842

Routine Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Page: Reviewer:

2nd Reviewer:_

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

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

% Difference = 100 * (ave. RRF - RRF)/ave. RRF RFF = $(A_{\nu})(C_{\nu})/(A_{\nu})(C_{\nu})$

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

 $A_{\rm s}=$ Area of associated internal standard $C_{\rm s}=$ Concentration of internal standard $A_x = Area$ of compound, $C_x = Concentration$ of compound,

L								
					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q%	0%
	SOM80NIJE	89/639	23.7,8-TCDF (3C.2,3,7,8-TCDF)	0.798	0.85	0.83	5.9	& 2
		20///	2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	0.913	0.82	0. XX	5 01	10.3
		1	1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1-8.0	0.94	0.93	9.11	11:11
			1,2,3,4,6,7,8-HpCDD (³ C-1,2,4,6,7,8,-HpCDD)	0.844	0.80	0.89	5.2	
			OCDF (3C-OCDD)	1.72/	1.60	1.64	AR	7
2			2,3,7,8-TCDF (13C-2,3,7,8-TCDF)					
		·	2,3,7,8-TCDD (13C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)					
			OCDF (13C-OCDD)					
က			2,3,7,8-TCDF (13C-2,3,7,8-TCDF)					
		<u></u>	2,3,7,8-TCDD (1°C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)					
		·	1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)					
			OCDF (13C-OCDD)					

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

LDC #: 1918842 SDG #: 26 COVE

Laboratory Control Sample Results Verification VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer: Reviewer:

METHOD: GC/MS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

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

% Recovery = 100 * SSC/SA

Where: SSC = Spiked sample concentration SA = Spike added

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: \$17159

RPD = I LCS - LCSD I * 2/(LCS + LCSD)

	ďS	ike	Spiked S	ample	รวา	S	I CSD	SD.	I/SD I	CS/I CSD
Compound	₽ ĝ	Added P9(4)	Concentration (PT)	tration	Percent Recovery	ecovery	Percent Recovery	ecovery	RPD	٥٠
	1.08	I CSD	SJI	l CSD	Reported	Recalc	Reported	Recalc.	Renorted	Recalculated
2,3,7,8-TCDD	20.0	NA	19.2	$\mathcal{N}^{\mathcal{A}}$	36	96				
1,2,3,7,8-PeCDD	001		701,		701	701				
1,2,3,4,7,8-HxCDD	1		90		R	126				
1,2,3,4,7,8,9-HpCDF	1		8.00		16	16				
	300	1	438	<u></u>	6/	6/				
							-			
	,				1	1				I

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

lons Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Ion ID Elemental Composition	Elemental Composition		Analyte	Descriptor	Accurate Mass ^(s)	Ol nol	Elemental Composition	Analyte
2 C ₁₂ H ₄ **Cl ₄ O C ₁₂ H ₄ **Cl ₄ **C10 '3C ₁₂ H ₄ **Cl ₄ O '3C ₁₂ H ₄ **Cl ₅ **ClO		100T 100T 100T	(S) (S)	4	407.7818 409.7788 417.8250 419.8220	M M X X X X X X X X X X X X X X X X X X	C ₁₂ H ²⁶ Cl ₆ 37ClO C ₁₂ H ²⁵ Cl ₅ 37Cl ₂ O 13C ₁₂ H ²⁵ Cl ₇ OClO	HPCDF HPCDF HPCDF (S)
C ₁₂ H, ³⁶ C ₁ O ₂ C ₁₂ H, ³⁶ C ₁ O ₂ 13C ₁₂ H, ³⁶ C ₁ O ₂		1000 1000 1000 1000 1000 1000 1000 100	(S)		423.7767 425.7737 435.8169	M M M + + + + + + + + + + + + + + + + +	C ₁₂ H ³⁶ Cl ₃ 7ClO ₂ C ₁₂ H ³⁶ Cl ₃ 7ClO ₂ C ₁₂ H ³⁶ Cl ₃ 7ClO ₂	Нрсор Нрсор Нрсор (S)
M+2 C ₁₂ H ₄ *Cl ₃ *ClO ₂ TCDD (S) M+2 C ₁₂ H ₄ *Cl ₅ *ClO HxCDPE LOCK C ₉ F ₁₃ PFK	2	HXCDD HXCDD PFK	<u>ெ</u> ப		437.8140 479.7165 [430.9728]	M M + 4 0 0 + 4 0 0 4 4	13C,2H ³⁵ Cl ₂ 37Cl ₂ O C,2H ³⁵ Cl ₂ 37Cl ₂ O C ₃ F ₁₇	HPCDD (S) NCDPE PFK
M+2 C ₁₂ H ₃ %Cl ₃ 70lO PeCDF M+4 C ₁₂ H ₃ %Cl ₃ 70l ₂ O PeCDF M+2 13C _{1,7} H ₃ %Cl ₃ 70ClO PeCDF		PecDF PecDF	Œ	ις	441.7428 443.7399 457.7377	ΣΣ + + -	C12**C15**C1C C12**C16**C1C	OCDF OCDF
10 C 12 4 20 10 10 10 10 10 10 10 10 10 10 10 10 10		Pecde (459.7348 469.7780 471.7750	Σ Σ Σ Σ Σ ; + + + + + ; - 4	C. aci, acio.	OCDD OCDD (S) OCDD (S)
		recup () PeCDD () HPCDPE	(i) (ii)		513.6775 [422.9278]	M+4 COCK	C,2*C ₂ °C ₂ O C,0 ^F 17	DCDPE PFK
M+2 C ₁₂ H ₂ **Cl ₃ **ClO HxCDF M+4 C ₁₂ H ₂ **Cl ₃ **Cl ₂ O HxCDF M 13C H **ClO		HXCDF						
12,12,0% 10,24,30,00 10,24,30,30,00 10,24,30,30,00		HXODE HXODE S(S)	ā 6					
19C ₁₂ H ₂ 35Cl ₃ 7ClO ₂ HXCDD 19C ₁₂ H ₂ 35Cl ₃ 7Cl ₂ O ₂ HXCDD C ₁₂ H ₂ 35Cl ₃ 7Cl ₂ O ₂ OCDPE	HXCDD HXCDD OCDPE	HXCDD (HXCDD (OCDPE	(S) (S)					
LOCK C ₀ F ₁ ,		PFK						

(a) The following nuclidic masses were used:

H = 1.007825 C = 12.000000 ¹³C = 13.003355 F = 18.9984

O = 15.994915 $^{36}CI = 34.968853$ $^{37}CI = 36.965903$

S = internal/recovery standard

LDC #: 1918847 SDG #: <u>See COV EN</u>

VALIDATION FINDINGS WORKSHEET

Sample Calculation Verification

Page:	of
Reviewer:	9
2nd reviewer:	

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

(Y)	N	N/A N/A	
/ /	N	N/A	

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

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

Conce	ntration	$a = \frac{(A_s)(I_s)(DF)}{(A_s)(RRF)(V_o)(\%S)}$
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).
RRF	=	Relative Response Factor (average) from the initial calibration
Df	****	Dilution Factor.
0/0		Danas and analysis and the state of the stat

Lample.			1	
Sample I.D.	a	_,	VD	:

=

#	Sample ID	Compound	Reported Concentration ()	Calculated Concentration ()	Qualification
	Sample ID	Compound		<u> </u>	
				<u> </u>	
	· · · · · · · · · · · · · · · · · · ·				
					,
		,			