

LABORATORY DATA CONSULTANTS, INC.

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Northgate Environmental Management, Inc. 1100 Quail Street Ste. 102 Newport Beach, CA 92660 ATTN: Ms. Cindy Arnold February 17, 2010

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

Dear Ms. Arnold,

Enclosed is the revised data validation report for the fraction listed below. The data validation was performed under Stage 2B/4 guidelines.

LDC Project # 21494:

SDG # Fraction

8304609 Organophosphorus Pesticides

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

Sincerely

Erlinda T. Rauto Operations Manager/Senior Chemist

Revision 2

LDC Report# 21494I17

Laboratory Data Consultants, Inc. Data Validation Report

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

Collection Date: June 29, 2009

LDC Report Date: February 17, 2010

Matrix: Water

Parameters: Organophosphorus Pesticides

*Validation Level: Stage 2B & 4

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 8304609

Sample Identification

M-111AB** EB062909-GW

**Indicates sample underwent Stage 4 review *Changed Validation Level from Stage 2B to Stage 4 for noted sample

Introduction

This data review covers 2 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8141A for Organophosphorus Pesticides.

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

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

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

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The following are definitions of the data qualifiers:

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

I. Technical Holding Times

All technical holding time requirements were met.

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

II. Calibration

a. Initial Calibration

Initial calibration of compounds was performed 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 (r^2) was greater than or equal to 0.990.

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

b. Calibration Verification

Calibration verification was performed at the required frequencies.

The percent differences (%D) of calibration factors in continuing standard mixtures were less than or equal to 20.0% with the following exceptions:

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
7/7/09	004F0401	1	Naled	44.4	All samples in SDG 8304609	J+ (all detects)	A
7/7/09	004F0401	2	Naled	24.0	All samples in SDG 8304609	J+ (all detects)	A

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

Revision 2

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
6/26/09	010F1001	1	Naled Disulfoton	40.1 20.6	All samples in SDG 8304609	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	Ρ
6/26/09	010F1001	2	Naled Malathion	47.6 23.2	All samples in SDG 8304609	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	Ρ

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

III. Blanks

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

Sample EB062909-GW was identified as an equipment bank. No organophosphorus pesticide contaminants were found in this blank.

IV. Accuracy and Precision Data

a. Surrogate Recovery

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

b. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there was insufficient sample volume for analysis of the matrix spike and matrix spike duplicate.

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 Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

VI. Project Quantitation Limit

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

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304609	All compounds reported below the PQL.	J (all detects)	A

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

VII. System Performance

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

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Data Qualification Summary - SDG 8304609

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304609	M-111AB EB062909-GW	Naled	J+ (all detects)	A	Continuing calibration (%D) (c)
8304609	M-111AB EB062909-GW	Naled Disulfoton Malathion	J- (all detects) UJ (all non-detects)	Ρ	Continuing calibration (ICV %D) (c)
8304609	M-111AB EB062909-GW	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (PQL) (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Laboratory Blank Data Qualification Summary - SDG 8304609

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304609

No Sample Data Qualified in this SDG

LDC #: 21494117 SDG #: 8304609 Laboratory: Test America

Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET Stage 2B 4

Date: 9/11/09
Page: \of
Reviewer: JVG
2nd Reviewer:

METHOD: GC Organophosphorus Pesticides (EPA SW 846 Method 8141A)

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

L	Validation Area		Comments
<u> </u>	Technical holding times	A	Sampling dates: 6/29/09
lla.	Initial calibration	A	2 RSD 5-202 rr
lib.	Calibration verification/ICV	<u>s</u> w	cav/1av = 202
111.	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	Ń	climit sher (insufficient sample)
IVc.	Laboratory control samples	A	ucs /p
V	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	N	
<u>_viii.</u>	Overail assessment of data	A	
IX.	Field duplicates	N	
X .	Field blanks	ND	EB = 2

A = Acceptable N = Not provided/applicable Note: SW = See worksheet

ND = No compounds detected R = Rinsate FB = Field blank

D = Duplicate TB = Trip blank EB = Equipment blank

Validated Samples: ¥

¥	Stage 4 Wa	ter			
1	M-111AB ¥¥	11	21	31	
2	EB062909-GW	12	22	32	
3	9182412 MB	13	23	33	
4		14	24	34	
5	······································	15	25	35	
6		16	26	36	
7		17	27	37	
8		18	28	38	
9		19	29	39	
10		20	30	40	

Notes:

21494117W.wpd

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

LDC #: 7 494 [17 SDG #: See Cover

Validation Area	Yes	No	NA	Findings/Comments
X. Target compound identification				
Were the retention times of reported detects within the RT windows?				
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		44.4		
System performance was found to be acceptable.		F		· · · · · · · · · · · · · · · · · · ·
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.			\square	
Target compounds were detected in the field duplicates.				
XV. Field blanks				
Field blanks were identified in this SDG.				
Target compounds were detected in the field blanks.				

VALIDATION FINDINGS WORKSHEET

METHOD: CC HPLC

0340					
0120	8330	8151	8141	R144.00 m	
A. Acenaphthene	A. HMX			1. uoci 1 + 1 o	8021B
B. Acenaphthylene		A. 6,940	A. Dichlarvas	V. Fensulfathion	V. Benzene
C. Anthracene	YAN G	B. 2/4-DB	B. Mevinphos	W. Bolatar	
	C. 1,3,5-Trinitrobenzene	C. 2,4,5-T	C. Demeton-O	X FPN	ut. I olutine
D. Benzo(a)anthracene	D. 1,3-Dinitrobenzene	D. 2,4,5-TP	D Denter e		EE. Eihyi Benzene
E. Benzo(a)pyrane	E. Tatryi		u. Varine(on-5	Y. Azinphos-methyl	SSS. O-Xylene
F. Benzo(b)fluoranthene			E. Ethoprop	Z. Coumaphos	RRR. MP-Xylerie
	· · Muroneuzene	F. Dichlorprop	F. Neled	AA. Parathion	GG. Total Xviana
	G. 2.4.6-Trinitrotoluene	G. Dicamba	G. Sulfatep	BB. Trichiomnete	
H. Benzo(k)fluoranthene	H. 4-Amino-2,6-dinitrotoluene	H. Dalapon	H. Phorate	Cc Table	
I. Chrysene	1. 2-Amina-4,6-dinitrotojuene	I. MCPP			
J. Dibenz(a,h)anthracane	J. 2.4-Dinitrataterea		I. Ultreundate	00. Trifluralin	
K. Fluorenthene		J. MCPA	J. Diazinon	EE Def	
	K. 2.6-Dinitratoluene	K. Pentachlorophenol	K. Disulfoton	FF. Prowl	
L. Fluorene	L. 2-Nitrotoluene	L 2,4,5-TP (silvex)	Partition anti-		
M. Indeno(1,2,3-cd)pyrene	M. 3-Nitrotoluene	M. Silvay		GG. Ethion	
N. Naphthalene	N AMINATION		M. Ronnel	HH. Tetrachlorvinphos	
	allenionistr		N. Melathion	ft. Suiprofos	
V. Thenanthrene	ö		0. Chiersvilles		
P. Pyrane	a.			J. Thiongzin	
ö	a		. Fentinon	kk. Phosmet	
Ŀ.			G. Parathion-ethyl	LL. 0.0.0-Tricthylphe	coharathia at.
			R. Trichloronate	MM. Homehur	
â			S. Merahas	Intra Intra	
				NN. Carbopheneth	. 40
			T. Stirofos	00. Carbophenoth	en - methul
			U. Tokuthion	~	
Votes:					

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LDC # 2494 I 17 SDG # <u>SCE Gury</u>

VALIDATION FINDINGS WORKSHEET **Continuing Calibration**

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METHOD: _____GC___HPLC

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

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	I Qualifications		J-LAT NP	1 4 10		1/W/- 2		J-J-J-D-		3- 11-10			17 Ath A	→														
secolated Samelar		+- b K							~					~	5													
																		-										
RT (limit)	· · · · · · · · · · · · · · · · · · ·) J							-		7		-				~							(
(1997)			+	_											_)							_)	
%D / RPD (Limit < 15,4	187 8		40.1	+	20.6	0.312	47.6	0		23. Y		4.44		٩ ۲														
Compound	C 63	4			k (-)	C U	ц Т			N F)	•	(+) #																
Detector/ Column	5.				*	Col.2				4		5	~ ~ ` `															
Standard ID	010 5 1001	していて										00170401																
Date	50/22/2											7/07/69																
*	Τ			Γ	T	1			Γ	T	T	1		Γ	Ť			T	t	1			-†		 \uparrow	\uparrow	╉	

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21494 117 SDG #: See Cover LDC #

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

- of 7ž Ø 2nd Reviewer:__ Reviewer: Page:

HPLO 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/Caverage CF = sum of the CF/number of standards %RSD = 100 $^{\circ}$ (S/X)

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

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Сотроила	CF (ンパ std)	CF (کرن std)	Average CF (initial)	Average CF (initial)	%RSD	%RSD
-		, , ,	(P; A (8141-1)	1.76366	1. 76 369	1.74977	1.74977	7. 99 554	7.99566
<u> </u>	L Z Z Z	borrer n		1. 82370	1. 82370	1. 81476	1-31476	5.6090)	5.60922
			V	Sre	rx c	rle.			
~			A (8141-2)	60561.5	2.17503	56510.c	7.01995	732345	7.32545
			+	1. 69691	1. 69691	1.76315	1. 76315	8.53946	8 53963
			N	1. 17724	1. 17724	1. 20369	1- 20369	3.60449	2 60 500
6					-				
4									
·									
	1								
	uments: Referto	<u>t</u>	on findings worksheet for list of gualificat	ions and assoc	iated samples	when reported re	sults do not agre	se within 10.0%	of the recalcu

ated results. S. . .

LDC # 21464]]7 Seelow SDG#

VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

5 Reviewer: <u>W</u> 2nd Reviewer: ____ Page: 🗡 of _

METHOD: GC EPA SW 846 Method 8141A

Parameter: Malathion

 		≻	×	X^2
Column	Compound	Area ratio	Conc ratio	
(8141A-1)	Malathion	0.14584	0.100	
		0.29331	0.250	
	I	0.55883	0.500	
		0.89027	1.000	
		1.76202	1.500	
		2.36769	2.000	
		2.77727	2.500	

Regression Output:			Reported	
Constant		-0.02062	Ш С	-0.02066
Std Err of Y Est		0.12319		
R Squared		0.99000	-2 -	0.99783
No. of Observations		7.00000		
Degrees of Freedom		5.00000		
X Coefficient(s)	1.1388	-0.002208	ŋ	1.14436E+000
Std Err of Coef.	0.054985	0.00		

IS = TOCP = 2.0ug/mL LAb used weighted linear regression

LDC #: 21494 II7 SDG #: See Cover

Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Page: 1 of Ĵ, 2nd Reviewer: Reviewer:

HPLC METHOD: GC

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

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

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

					Reported	Recalculated	Reported	Recalculated
* Standard ID	Calibration Date			Average CF(Ical)/ CCV Conc	CF/Conc. CCV	CF/Conc. CCV	0%	0%
1 004 FOGI	7 107 60	4	8141-1	5.50	2. 4145	5414.C	3,4	Z. ¢
	(a//_/.	.#			27 22.5	2. 5677	2,7	2.7
		Z	-~		2 - 5778.	2.5578	2,3	کر ج
2		4	8141-2		2.4117	2,4117	3,5	5
		Ŧ			2,4980	2,4980	0.1	6, 1
		Ν	*	λ	2.4450	2. 4454	2.2	ン、イ
<i>п</i>						•		
	I							
							-	
4								
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Comments: Refer to Continuing Calibration findings worksheet for list of gualifications and associated samples when reported results do not agree within 10.0% of the an crd Ł Y Ş 1 CP 322 recalculated results.

LDC #: 2 | 494 I 17 SDG #: See Cover METHOD: GC _____HPLC

VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page: 1 of 1

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

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	Percent Difference		01	\mathbf{A}		
	Percent Recovery	Recalculated	77	60		
	Percent Recovery	Reported	64	(ó		
	Surrogate Found		0.76690	0,60353		
	Surrogate Spiked		1.0	7		•
	Column/Detector		-			-
ŧŧ	Surrogate		TPP	Chluring fres		
Sample ID:						

Sample ID:

Sample ID:

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

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14	3
2140	205
LDC #:	SDG #:_

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

Page: lof I Reviewer: JK 2nd Reviewer: <u>&</u>

GC HPLC **METHOD:**

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

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

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

SC = Concentration

LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples: 9 / 8 24 / 2 LCS/D

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicate findings worksheet for list of qualifications and associated samples when reported Recalc. <u>v</u> LCS/LCSD RPD Reported 39 Þ Recalc. 6 1 Percent Recovery 80 LCSD Reported 57 S Recalc. Percent Recovery 5 6 LCS Reported 5 5 LCSD 3,23 5.08 Spiked Sample Concentration ЧЗ 2.09 3.64 LCS ද LCSD Spike Added (146 / L) ð LCS 4.00 2,4,6-Trinitrotoluene (8330) (8)41 Naphthalene (8310) Compound Methane (RSK-175) Anthracene (8310) Benzene (8021B) Gasoline (8015) Dinoseb (8151) Malatrian Dichloring Diesel (8015) 2,4-D (8151) HMX (8330)

V:\Validation Worksheets\GC\LCSDCLC_GC.wpd

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

ы v	DC #: 21 464 I 17 DG # See curer	VALIDATI <u>Sampl</u>	ION FINDINGS WORKSI e Calculation Verificati	HEET <u>on</u>	Page: 1 of 1 Reviewer: 30
Σ	ETHOD:GC HPLC	· ·		•	
\succ	N N/A N N/A Were all reported recolculate	esults recalculated and verified fo ed results for detected target com	or all level IV samples? Ipounds within 10% of the rep	oorted results?	
Ο ζτο	oncentration= <u>(A)(Fv)(Df)</u> (RF)(Vs or Ws)(%S/10(= Area or height of the compound to be me: /= Final Volume of extract f= Dilution Factor	0) Example: sasured Sample ID	Comp	ound Name	
£ ≥≥%	F= Average response factor of the compound In the initial calibration s= Initial volume of the sample /s= Initial weight of the sample S= Percent Solid	d Concentration =	11		
Ľ					
<u></u>	# Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations (Qualifications
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<u>باست.</u>					
للحص					
о С	mments:				

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