Laboratory Data Consultants, Inc.
Data Validation Report

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

Collection Date: July 14, 2009

LDC Report Date: February 18, 2010

Matrix: Water

Parameters: Organophosphorus Pesticides

***Validation Level:** Stage 4

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 8304614

Sample Identification

TR-8B

*Changed validation level from Stage 2B to Stage 4

Introduction

This data review covers one water sample 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.

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.

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%.

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:

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 8304614	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	P
6/26/09	010F1001	2	Naled Malathion	47.6 23.2	All samples in SDG 8304614	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	P

Retention time windows were evaluated and considered technically acceptable.

*Indicates change as the result of report review.

III. Blanks

Method blanks were reviewed for each matrix as applicable. No organophosphorus pesticide 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. 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.

VI. Project Quantitation Limit

All project quantitation limits were within validation criteria.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304614	All compounds reported below the PQL.	J (all detects)	A

VII. System Performance

The system performance was acceptable.

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 8304614

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304614	TR-8B	Naled Disulfoton Malathion	J- (all detects) UJ (all non-detects)	P	Continuing calibration (ICV %D) (c)
8304614	TR-8B	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 8304614

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304614

No Sample Data Qualified in this SDG

*Indicates change as the result of report review.

Tronox Northgate Henderson
VALIDATION COMPLETENESS WORKSHEET
Stage 2B

LDC #: 21494N17
SDG #: 8304614
Laboratory: Test America

Date: 9/11/09
Page: 1 of 1
Reviewer: JVC
2nd Reviewer: [signature]

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.

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 7/14/09
IIa.	Initial calibration	A	%RSD ≤ 20% r²
IIb.	Calibration verification/ICV	SW	CCV/ICV ≤ 20%
III.	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	N	Client spec (insufficient sample)
IVc.	Laboratory control samples	A	LCS/D
V.	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	N	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N	
X.	Field blanks	N	

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

Validated Samples: Water

1	TR-8B	11		21		31	
2	9198202-MB	12		22		32	
3		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: _____

LDC #: 21494N17
SDG #: See Cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 7
Reviewer: VG
2nd Reviewer: [illegible]

Method: ✓ GC _____ HPLC

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

LDC #: 21494N17
SDG #: See Cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
Reviewer: VV
2nd Reviewer:

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				
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.		✓		
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: ___/GC ___HPLC

8310	8330	8151	8141	8141(Con't)	8021B
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	J. MCPA	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	O.		O. Chlorpyrifos	JJ. Thionazin	
P. Pyrene	P.		P. Fenthion	kk. Phosmet	
Q.	Q		Q. Parathion-ethyl	LL. 0,0,0-Triethylphosphorothioate	
R.			R. Trichloronate	MM. Famphur	
S.			S. Merphos	NN. Carbophenothion	
			T. Stirofos	OO. Carbophenothion-methyl	
			U. Tokuthion		

Notes: _____

cmpd_list.wpd

LDC #: 2494 17
SDG #: See Cover

VALIDATION FINDINGS WORKSHEET
Continuing Calibration

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

METHOD: (GC) HPLC

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".
What type of continuing calibration calculation was performed? (%D) or RPD
(Y) N N/A Were continuing calibration standards analyzed at the required frequencies?
Y (N) N/A Did the continuing calibration standards meet the %D / RPD validation criteria of ~~≤15.0~~ ≤20%?
Level IV Only
Y N (N/A) Were the retention times for all calibrated compounds within their respective acceptance windows?

#	Date	Standard ID	Detector/ Column	Compound	%D / RPD (Limit ≤ ~~15.0~~ ≤20%)	RT (limit)	Associated Samples	Qualifications
	6/26/09	010F1001 (ICV)	Col. 1	~~C (+)~~	~~187.8~~	( )	All + Blk	~~J (all detects) (C)~~
				F (-)	40.1	( )		J-/UJ/P
				~~D (-)~~	~~83.1~~	( )		~~J (all detects)~~
				K (-)	20.6	( )		J-/UJ/P
			Col. 2	~~C (+)~~	~~215.0~~	( )		~~J (all detects)~~
				F (-)	47.6	( )		J-/UJ/P
				~~D (-)~~	~~85.9~~	( )		~~J (all detects)~~
				N (-)	23.7	( )		J-/UJ/P
						( )		
	7/21/09	~~010F1301~~ (~~CCV~~)				( )		
						( )		
						( )		
						( )		
						( )		
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LDC #: 21494 N17
SDG #: See Cover

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Page: 1 of 2
Reviewer: JM
2nd Reviewer:

METHOD: GC ✓ HPLC ____

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

#	Standard ID	Calibration Date	Compound	Reported CF (1 std)	Recalculated CF (1 std)	Reported Average CF (initial)	Recalculated Average CF (initial)	Reported %RSD	Recalculated %RSD
1	ICAL	6/26/09	A (8141-1)	1.76364	1.76369	1.74977	1.74977	7.99554	7.99566
			H	1.82370	1.82370	1.81476	1.81476	5.60901	5.60927
			N	See r.r calc.					
2			A (8141-2)	2.17503	2.17503	2.01995	2.01995	7.32345	7.32345
			H	1.69691	1.69691	1.76315	1.76315	8.53946	8.53963
			N	1.17724	1.17724	1.20369	1.20369	3.60449	3.60500
3									
4									

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

LDC # 21944N17
SDG# See Cover

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Reviewer: JVG
2nd Reviewer: [signature]

METHOD: GC EPA SW 846 Method 8141A

Parameter: Malathion

Date	Column	Compound	Y Area ratio	X Conc ratio	X^2
06/26/2009	(8141A-1)	Malathion	0.14584	0.100	
			0.29331	0.250	
			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	c =	-0.02066
Std Err of Y Est	0.12319		
R Squared	0.99000	r2 =	0.99783
No. of Observations	7.00000		
Degrees of Freedom	5.00000		
X Coefficient(s) 1.1388	-0.002208	a	1.14436E+000
Std Err of Coef. 0.054985	0.00		

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

LDC #: 21494 N17
SDG #: See Cover

VALIDATION FINDINGS WORKSHEET
Continuing Calibration Results Verification

Reviewer: JVG
2nd Reviewer: R

METHOD: GC ✓ HPLC _____

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

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

#	Standard ID	Calibration Date	Compound	Average CF(Ical)/ CCV Conc.	Reported CF/ Conc. CCV	Recalculated CF/ Conc. CCV	Reported %D	Recalculated %D
1	018F1801	7/21/09	A (8141-1)	2.500	2.5340	2.5340	1.4	1.4
			H		2.3783	2.3783	4.9	4.9
			N		2.2781	2.2781	8.9	8.9
2			A (8141-2)		2.6391	2.6391	5.6	5.6
			H		2.4548	2.4548	1.8	1.8
			N		2.2620	2.2620	9.5	9.5
3								
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 #: 21494N17
SDG #: See Cover
METHOD: GC HPLC

VALIDATION FINDINGS WORKSHEET
Surrogate Results Verification

Reviewer: VG
2nd reviewer: [illegible]

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

% Recovery: SF/SS * 100

Where: SF = Surrogate Found
SS = Surrogate Spiked

Sample ID: # 1

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
TPP	Col. 1	1.00	0.67409	67	67	0
Chlormefos	↓	↓	0.49078	49	49	↓

Sample ID:

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

Sample ID:

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

LDC #: 21494N17
SDG #: See Cover

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

Reviewer: JG
2nd Reviewer: R

METHOD: ✓ GC __HPLC

The percent recoveries (%R) and relative percent differences (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 SSC = Spiked sample concentration, SA = Spike added, SC = Sample concentration

RPD =(({SSCLCS - SSCLCSD} * 2) / (SSCLCS + SSCLCSD))*100 LCS = Laboratory Control Sample, LCSD = Laboratory Control Sample duplicate

LCS/LCSD samples: 9198202-LCS/D

Compound	Spike Added (ug/L)		Spike Sample Concentration (ug/L)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	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)										
Dichlorvos (8141)	4.00	4.00	3.557	3.224	89	89	48	81	9.8	9.8
Malathion ↓	↓	↓	2.87✓	2.665	72✓	72✓	67	67	7.5	7.5

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 #: 21494N17
SDG #: See Cover

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

Reviewer: JVG
2nd Reviewer: [illegible]

METHOD: ✓ GC __ HPLC

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

Concentration= $\frac{(A)(Fv)(Df)}{(RF)(Vs \text{ or } Ws)(\%S/100)}$

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

Example:

Sample ID._____ Compound Name ND

Concentration = _____

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

Comments: _____