

#### LABORATORY DATA CONSULTANTS, INC.

7750 El Camino Real, Suite 2L Carlsbad, CA 92009 Phone: 760/634-0437 Fax: 760/634-0439

Northgate Environmental Management, Inc.

October 16, 2009

1100 Quail Street Ste. 102 New Port beach, CA 92660 ATTN: Ms. Cindy Arnold

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

Data Validation

Dear Ms. Arnold,

Enclosed are the revised data validation reports for the fraction listed below. Please replace the previously submitted reports with the enclosed revised reports.

#### **LDC Project # 21494:**

SDG#

**Fraction** 

8304603, 8304604, 8304606 8304610, 8304621

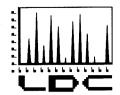
Organophosphorus Pesticides

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

Sincerely,

Erlinda T. Rauto

Operations Manager/Senior Chemist



#### LABORATORY DATA CONSULTANTS, INC.

7750 El Camino Real, Suite 2L Carlsbad, CA 92009 Phone: 760/634-0437 Fax: 760/634-0439

Northgate Environmental Management, Inc.

September 28, 2009

1100 Quail Street Ste. 102 New Port beach, CA 92660 ATTN: Ms. Cindy Arnold

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

**Data Validation** 

Dear Ms. Arnold,

Enclosed are the final validation reports for the fractions listed below. These SDGs were received on September 4, 2009. Attachment 1 is a summary of the samples that were reviewed for each analysis.

#### LDC Project # 21494:

SDG # Fraction

8304601, 8304602, 8304603, 8304604, 8304605, Organophosphorus Pesticides 8304606, 8304607, 8304608, 8304609, 8304610, Metals 8304611, 8304612, 8304614, 8304615, 8304616, 8304617, 8304619, 8304620, 8304621, 8304622, 8304623, 8304624

The data validation was performed under Stage 2B & 4 guidelines. The analyses were validated using the following documents, as applicable to each method:

- Standard Operating Procedures (SOP) 40, Data Review/Validation, BRC 2009
- Quality Assurance Project Plan Tronox LLC Facility, Henderson Nevada, June 2009
- NDEP Guidance, May 2006
- USEPA, Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, June 2008
- 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

Attachment 1

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LDC #: 21494

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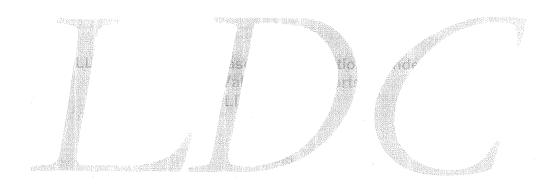
Page: 1 of 1 Reviewer: JE 2nd Reviewer: BC

#### Tronox Northgate Henderson Worksheet

EDD Area  1. Completeness	Yes	No	NA	Findings/Comments
Is there an EDD for the associated Tronox validation report?  II. EDD Qualifier Population	X			
Were all qualifiers from the validation report populated into the EDD?  III, EDD Lab Anomalies	X			
Were EDD anomalies identified?	X			
If yes, were they corrected or documented for the client?	Х			See EDD_discrepancy_ form_LDC21494_092409.doc
IV. EDD Delivery				
Was the final EDD sent to the client?	X			

#### Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Data Validation Reports LDC# 21494

Metals



# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

June 15 through June 19, 2009

LDC Report Date:

September 14, 2009

Matrix:

Water

Parameters:

Metals

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304607

#### Sample Identification

M-29B

M-130B

M-130BDISS

M-78B

M-128B

M-128BDISS

H-38B

M-19B

M-34B

M-29BMS

M-29BMSD

M-130BDISSMS

M-130BDISSMSD

#### Introduction

This data review covers 13 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Metals. The metals analyzed were Arsenic and Selenium.

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 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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

#### III. Calibration

An initial calibration was performed.

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

#### IV. 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	Selenium	0.887 ug/L	M-130BDISS M-128BDISS

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
M-128BDISS	Selenium	3.2 ug/L	5.0U ug/L

No field blanks were identified in this SDG.

#### V. ICP Interference Check Sample (ICS) Analysis

The frequency of analysis was met.

The criteria for analysis were met.

#### VI. 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.

#### VII. Duplicate Sample Analysis

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

#### VIII. Laboratory Control Samples (LCS)

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

#### IX. Internal Standards

Raw data were not reviewed for this SDG.

#### X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

#### XI. ICP Serial Dilution

ICP serial dilution analysis was performed by the laboratory. The analysis criteria were met.

#### XII. Sample Result Verification and Project Quantitation Limit

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304607	All analytes reported below the PQL.	J (all detects)	Α

Raw data were not reviewed for this SDG.

#### 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.

# Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Data Qualification Summary - SDG 8304607

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
8304607	M-29B M-130B M-130BDISS M-78B M-128B M-128BDISS H-38B M-19B M-34B	All analytes reported below the PQL.	J (all detects)	А	Sample result verification (PQL) (sp)

## Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Laboratory Blank Data Qualification Summary - SDG 8304607

SDG	Sample	Analyte	Modified Final Concentration	A or P	Code
8304607	M-128BDISS	Selenium	5.0U ug/L	А	bl

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Field Blank Data Qualification Summary - SDG 8304607

No Sample Data Qualified in this SDG

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

LDC #: 21494G4 SDG #: 8304607 Stage 2B Laboratory: Test America

	Date:	9/11/9
	Page:	Tot 1
	Reviewer:	+01
2nd	Reviewer:	

METHOD: As & Se (EPA SW 846 Method 6020)

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

	Validation Area		Comments
l.	Technical holding times	A	Sampling dates: 6/13/09 - 6/19/09
11.	ICP/MS Tune	A	
111.	Calibration	A	
IV.	Blanks	5m)	
V.	ICP Interference Check Sample (ICS) Analysis	A	
VI.	Matrix Spike Analysis	A	ymy/my
VII.	Duplicate Sample Analysis	Ń	, ,
VIII.	Laboratory Control Samples (LCS)	A	Les
IX.	Internal Standard (ICP-MS)	N	not haviend
X.	Furnace Atomic Absorption QC	N	wit while
XI.	ICP Serial Dilution	A	0. 1
XII.	Sample Result Verification	N	
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	N	
XV	Field Blanks	V	

Note:

Validated Samples:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

D = Duplicate

TB = Trip blank

FB = Field blank EB = Equipment blank

M-29B M-29BMSD 21 31 11 M-130B 12 M-130BDISSMS 22 32 M-130BDISS 13 M-130BDISSMSD 23 33 M-78B 14 24 34 M-128B 15 25 35 M-128BDISS 16 26 36 H-38B 17 27 37 M-19B 18 28 38 **M**-34B 19 29 39 M-29BMS 20 30 40

Notes:	
	-

LDC #: <u>>1494</u> SDG #: <u>See ar</u>e~

### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

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Reviewer:	
2nd reviewer:	

All circled elements are applicable to each sample.

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Sample ID	Matrix	Target Analyte List (TAL)
1-9	Aa	Al, Sb,(As) Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K,(Se,)Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN,
		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, CN,
UP,1)~	, M	Al, Sb, 🕢 Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, 😡 Ag, Na, Tl, V, Zn, Mo, B, Si, CN
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		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN
		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, CN,
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		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN <sup>-</sup> ,
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		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, CN <sup>-</sup> ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		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, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN,
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		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, CN,
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		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, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
	i	Analysis Method
ICP		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, CN,
ICP Trace		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, CN,
ICP-MS		Al, Sb.(As), Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se Ag, Na, Ti, V, Zn, Mo, B, Si, CN',
GFAA		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, CN',

Comments: Mercury by CVAA if performed

(84) Sample Identification VALIDATION FINDINGS WORKSHEET
PB/ICB/CCB QUALIFIED SAMPLES
Soil preparation factor applied: Associated Samples: 3, 6 3.2 / 5.0 ng/L ဖ METHOD: Trace Metals (EPA SW 846 Method 6020) Sample Concentration units, unless otherwise noted: Blank Action I imit Maximum ICB/CCB<sup>a</sup> (1/611) 0.887 Maximum Maximum PB<sup>a</sup> PB<sup>a</sup> (mg/Kg) (ug/l) LDC #: 21494G4 SDG #: See Cover

Analyte

21494G4.wpd

# Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** 

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

June 23 through June 25, 2009

LDC Report Date:

September 14, 2009

Matrix:

Water

Parameters:

Metals

Validation Level:

Stage 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304608

#### Sample Identification

M-125B

M-125BDISS

M-22AB

M-17AB

M-17ABDISS

M-64B

M-75B

M-13AB

M-13ABDISS

M-13009AB

M-13009ABDISS

M-125BMS

M-125BMSD

M-125BDISSMS

M-125BDISSMSD

M-22ABMS

M-22ABMSD

M-13ABDISSMS

M-13ABDISSMSD

#### Introduction

This data review covers 19 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Metals. The metals analyzed were Arsenic and Selenium.

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 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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

#### III. Calibration

An initial calibration was performed.

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

#### IV. Blanks

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

No field blanks were identified in this SDG.

#### V. ICP Interference Check Sample (ICS) Analysis

The frequency of analysis was met.

The criteria for analysis were met.

#### VI. 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
M-13ABDISSMS/MSD (M-13ABDISS)	Arsenic	•	132 (75-125)	-	J+ (all detects)	А

#### VII. Duplicate Sample Analysis

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

#### VIII. Laboratory Control Samples (LCS)

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

#### IX. Internal Standards

All internal standard percent recoveries (%R) were within QC limits.

#### X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

#### XI. ICP Serial Dilution

ICP serial dilution analysis was performed by the laboratory. The analysis criteria were met.

#### XII. Sample Result Verification and Project Quantitation Limit

All sample result verifications were acceptable.

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304608	All analytes reported below the PQL.	J (all detects)	А

#### XIII. Overall Assessment of Data

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

#### XIV. Field Duplicates

Samples M-13AB and M-13009AB and samples M-13ABDISS and M-13009ABDISS were identified as field duplicates. No metals were detected in any of the samples with the following exceptions:

	Concentration (ug/L)			D			
Analyte	M-13AB	M-13009AB	RPD (Limits)	Difference (Limits)	Flags	A or P	
Arsenic	120	120	-	0 (≤50)	-	-	

	Concentration (ug/L)			B			
Analyte	M-13ABDISS	M-13009ABDISS	RPD (Limits)	Difference (Limits)	Flags	A or P	
Arsenic	110	120	-	10 (≤50)	-	-	

# Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Data Qualification Summary - SDG 8304608

SDG	Sample	Analyte	Flag	A or P	Reason (Code)	
8304608	M-13ABDISS	Arsenic	J+ (all detects)	А	Matrix spike/Matrix spike duplicates (%R) (m)	
8304608	M-125B M-125BDISS M-22AB M-17AB M-17ABDISS M-64B M-75B M-13AB M-13ABDISS M-13009AB	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (PQL) (sp)	

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Laboratory Blank Data Qualification Summary - SDG 8304608

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Field Blank Data Qualification Summary - SDG 8304608

No Sample Data Qualified in this SDG

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

LDC #:\_\_\_ 21494H4 SDG #: 8304608 Stage 28 4 Laboratory: Test America

Date: <u><i>9/</i></u>	4/00
Page: <u>(</u> (	of_ <i></i> /
Reviewer:	<u> </u>
2nd Reviewer: 9	nA

METHOD: As & Se (EPA SW 846 Method 6020)

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 6/13/19 - 6/15/19
11.	ICP/MS Tune	Ä	, ,
111.	Calibration	A	
IV.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	A	
Vİ.	Matrix Spike Analysis	9W	2 ms/1000
VII.	Duplicate Sample Analysis	N	). 71.2
VIII.	Laboratory Control Samples (LCS)	A	Les
IX.	Internal Standard (ICP-MS)	A	
X.	Furnace Atomic Absorption QC	N	pit Worlynd
XI.	ICP Serial Dilution	A	4
XII.	Sample Result Verification	XX	
XIII.	Overall Assessment of Data	<b>A</b> "	
XIV.	Field Duplicates	SW	(8,00) (9,11)
XV	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:

	A>					
1	M-125B	11	M-13009ABDISS	21 MB	31	
2	M-125BDISS	12	M-125BMS	22	32	
3	M-22AB	13	M-125BMSD	23	33	
4	M-17AB	14	M-125BDISSMS	24	34	
5	M-17ABDISS	15	M-125BDISSMSD	25	35	·
6	M-64B	16	M-17ABMS	26	36	٥
7	M-75B	17	M-17ABMSD	27	37	
3 4 5 6 7 8	M-13AB	18	M-13ABDISSMS	28	38	
9	M-13ABDISS	19	M-13ABDISSMSD	29	39	
10	M-13009AB	20		30	40	

Notes:			
•			 
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LDC #: Magary
SDG #: Su w

#### **VALIDATION FINDINGS CHECKLIST**

Page: 1 of 2 Reviewer: 2014 2nd Reviewer: 2014

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

Wethod: Metals (EPA SW 846 Method 6010B/7000/6020)	T -	<del></del>	<del>1</del>	
Validation Area	Yes	No	NA	Findings/Comments
All technical holding times were met.	/			
Cooler temperature criteria was met.			1920	ASSESSMENT AS A CONTRACTOR AND A CONTRACTOR AS
Were all isotopes in the tuning solution mass resolution within 0.1 amu?	/			
Were %RSD of isotopes in the tuning solution ≤5%?				
Were all instruments calibrated daily, each set-up time?	/			
Were the proper number of standards used?	/			
Were all initial and continuing calibration verification %Rs within the 90-110% (80-120% for mercury) QC limits?	/			
Were all initial calibration correlation coefficients > 0.995?	/			
Was a method blank associated with every sample in this SDG?	1			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/		
Mais dileta de capación de la				
Were ICP interference check samples performed daily?	/			
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?				
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.		Mislandra		
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.		1		
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.		1221		
Was an LCS anayized for this SDG?		- CHRISTON	TOTAL PROPERTY.	
Was an LCS analyzed per extraction batch?	7		$\dashv$	
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?				

LDC #: 1 494 kg SDG #: Cu wie

#### **VALIDATION FINDINGS CHECKLIST**

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Reviewer:
2nd Reviewer: MR

N. F. A	V	<b>.</b>	Ī.,	Findings/Comments
Validation Area	Yes	No	NA MA	Findings/Comments
If MSA was performed, was the correlation coefficients > 0.995?	Abraitaci ta	antaits	_	and testing the manufacture of the state of
Do all applicable analysies have duplicate injections? (Level IV only)			1	
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)			/	
Were analytical spike recoveries within the 85-115% QC limits?				
		2112		
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the MDL (ICP)/>100X the MDL(ICP/MS)?	/			
Were all percent differences (%Ds) < 10%?	_			
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.			/	
Saman sa com membraka Min dilakan Basaba Salah Salah				
Were all the percent recoveries (%R) within the 30-120% (6020)/60-125% (200.8) of the intensity of the internal standard in the associated initial calibration?				
If the %Rs were outside the criteria, was a reanalysis performed?		riggerag		
ACCOMPGENICATION CONTRACTOR ASSESSMENT ASSES				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	1			
Overall assessment of data was found to be acceptable.				
Field duplicate pairs were identified in this SDG.	1			
Target analytes were detected in the field duplicates.				The control of the co
Field blanks were identified in this SDG.		$\sqrt{}$		,
Target analytes were detected in the field blanks.			V	

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# VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page: of Page: 2nd reviewer: MA

All circled elements are applicable to each sample.

<del></del>		
Sample ID	Matrix	Target Analyte List (TAL)
[-1]	Aa	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, CN,
		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, CN,
12-19	B	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, CN,
		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, CN,
		Ai, 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, CN,
		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, CN,
		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, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN',
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		Al. Sb. As. Ba. Be. Cd. Ca. Cr. Co. Cu. Fe. Pb. Mg. Mn. Hg. Ni. K, Se. Ag. Na. Ti, V, Zn. Mo. B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		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, CN,
		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, CN,
		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, CN.,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		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, CN,
		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, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Sì, CN <sup>-</sup> ,
		Analysis Method
ICP		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, CN,
ICP Trace		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, CN,
ICP-MS		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, CN,
GFAA		Ai, 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, CN,

Comments:	Mercury	by CVAA	if	perfo	rmed

1DC #: > 1494 (A)

# Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

Page: Reviewer:\_ 2nd Reviewer:

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".

| V | N/A | Was a matrix spike analyzed for each matrix in this SDG?
| Y | N/A | Were matrix spike percent recoveries (%R) within the control limits of 75-125?) If the sample control is the sample control limits of 75-125?

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?

CON N/A Wei

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

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	Qualifications	(五) 大村坊	1																					
	Associated Samples	6.X	}																					
	RPD (Limits)																							
	MSD %Recovery	(32																				-		
	ms %Recovery																						no Aven	
	Analyte	As	\ ,																				# 5 4 4	-
	Matríx	Ao																					1	
		51/8)														·			•				Comments: Cert	
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SDG#:	See Cover

#### **VALIDATION FINDINGS WORKSHEET** Field Duplicates

Page:_	<u></u>
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2nd Reviewer:	gn H

METHOD: Metals (EPA Method 6020)

YN NA AN NA

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

	Concentrat	ion (ug/L)	(≤30)	(ug/L)	(ug/L)	Qualifications
Compound	8	10	RPD	Difference	Limits	(Parent Only)
Arsenic	120	120		0	(≤50)	

	Concentrat	ion (ug/L)	(≤30)	(ug/L)	(ug/L)	Qualifications
Compound	9	11	RPD	Difference	Limits	(Parent Only)
Arsenic	110	120		10	(≤50)	

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LDC #: -1-31-75

# Initial and Continuing Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: of Reviewer: MY

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:

%R = Found × 100 True

Where, 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

					Receivulated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
725	ICF (Initial calibration)	43	39,-9	40	7612	46.2	À
	GFAA (Initial calibration)						
	CVAA (initial calibration)						
3	ICP (Continuing calibration)	Se	49,7	کر	18:4	4:36	<b>/</b>
	GFAA (Continuing calibration)						
	CVAA (Continuing calibration)						
	ICP/MS (Initial calibration)						
	ICP/MS (Continuing calibation)						

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 # 149417 SDG #: 22 CON

# **VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet**

2nd Reviewer:\_\_ Reviewer:\_\_ Page:

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 = <u>Found</u> x 100 True

Where, Found = Concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation, Found = SSR (spiked sample result).

True = Concentration of each analyte in the source.

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

RPD # IS-DI × 100 (S+D)/2

Where, S = Origina sample concentration

D = Duplicate sample concentration

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

%D = 1-SDR1 x 100

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

					Racalculated	Reported	
Sample ID	Type of Analysis	Element	Found / S / I (units)	True / D / SDR (units)	%R/RPD/%D	%R/RPD/%D	Acceptable (Y/N)
75 T	ICP interference check	M.	(حره)	<u>~</u>	(03.)	[~{a}]	Å
165	Laboratory control sample	73	78.5	400	76	96	_
1	Matrix spike	A	(ssr-sr) }8.7	40.0	46	43	
(V/) Dupticate	Duplicate	Z	へいまわ	8.58	(9)	61	,
	ICP serial dilution	M	6.2	81.9	4-5	4.6	1

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 #:	214941	44
SDG #:	Su	Com

#### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:_	<u> </u>
Reviewer:	5
2nd reviewer:_	MX

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

		is memo (El 7 Oll 040 Methor	u 0010/7000)	
Please N N N N	see qu N/A N/A N/A	alifications below for all question Have results been reported a Are results within the calibrate Are all detection limits below	ns answered "N". Not applicable on nd calculated correctly? ed range of the instruments and with the CRDL?	questions are identified as "N/A".
	ted analy	yte results fortion:	1,2	were recalculated and verified using the
Concen	tration =	(RD)(FV)(Dil) (In. Vol.)(%S)	Recalculation:	
RD	=	Raw data concentration	/ امده، / ۸	. 11041
FV	=	Final volume (ml)	M こんパイル×	10 = 61.8 y/
In. Vol.	=	Initial volume (ml) or weight (G)	17	` "
Dil	=	Dilution factor		

Sample ID	Analyte	Reported Concentration ( \( \forall \) \( \)	Calculated Concentration	Acceptable (Y/N)
	As	62	62	У
		_~		/
	As	5257	53	1
			·	/
			·	
			-	
			·	
		·		

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

June 29 through July 1, 2009

LDC Report Date:

September 14, 2009

Matrix:

Water

Parameters:

Metals

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304609

#### Sample Identification

M-111AB

M-110AB

M-110ABDISS

I-ARB

M-25B

M-12AB

M-12ABDISS

M-110ABMS

M-110ABMSD

M-12ABDISSMS

M-12ABDISSMSD

#### Introduction

This data review covers 11 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Metals. The metals analyzed were Arsenic and Selenium.

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 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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

#### III. Calibration

An initial calibration was performed.

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

#### IV. Blanks

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

No field blanks were identified in this SDG.

#### V. ICP Interference Check Sample (ICS) Analysis

The frequency of analysis was met.

The criteria for analysis were met.

#### VI. 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
M-110ABMS/MSD (M-110AB)	Selenium	-	126 (75-125)	-	J+ (all detects)	А
M-12ABDISSMS/MSD (M-12ABDISS)	Selenium	130 (75-125)	130 (75-125)	-	J+ (all detects)	А

#### VII. Duplicate Sample Analysis

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

#### VIII. Laboratory Control Samples (LCS)

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

#### IX. Internal Standards

Raw data were not reviewed for this SDG.

#### X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

#### XI. ICP Serial Dilution

ICP serial dilution analysis was performed by the laboratory. The analysis criteria were met.

#### XII. Sample Result Verification and Project Quantitation Limit

All analytes reported below the PQL were qualified as follows:

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

Raw data were not reviewed for this SDG.

#### 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.

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

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
8304609	M-110AB M-12ABDISS	Selenium	J+ (all detects)	A	Matrix spike/Matrix spike duplicates (%R) (m)
8304609	M-111AB M-110AB M-110ABDISS I-ARB M-25B M-12AB M-12ABDISS	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (PQL) (sp)

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

No Sample Data Qualified in this SDG

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

No Sample Data Qualified in this SDG

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

Stage 2B

Date: <u>9/14/</u> }
Page:of/
Reviewer:
2nd Reviewer: and

SDG #: 8304609 Laboratory: Test America

2149414

LDC #:

METHOD: As & Se (EPA SW 846 Method 6020)

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 6/-9/09 - 7/01/09
II.	ICP/MS Tune	A	' /
III.	Calibration	Á	
IV.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	A	
VI.	Matrix Spike Analysis	gW	\ms/m5/n50
VII.	Duplicate Sample Analysis	N	
VIII.	Laboratory Control Samples (LCS)	A	LUZ
IX.	Internal Standard (ICP-MS)	N	Int beviewal
X.	Furnace Atomic Absorption QC	N	pot beneared
XI.	ICP Serial Dilution	A	4
XII.	Sample Result Verification	N	
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	V	
ΧV	Field Blanks	M	

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

TB = Trip blank

FB = Field blank

EB = Equipment blank

D = Duplicate

Validated Samples:

	140-						
1	M-111AB	11	M-12ABDISSMSD	21	MR	31	
2	M-110AB	12		22	•	32	
3	M-110ABDISS	13		23		33	
4	I-ARB	14		24		34	
5	M-25B	15		25		35	
6	M-12AB	16		26		36	
7	M-12ABDISS	17		27		37	
8	M-110ABMS	18		28		38	•
9	M-110ABMSD	19		29		39	
10	M-12ABDISSMS	20		30		40	

Notes:			

LDC #: >149474 SDG #: <u>See ar</u>e

#### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page: \_\_\_\_of\_\_\_ Reviewer: \_\_\_\_am.R\_

All circled elements are applicable to each sample.

F		
Sample ID	Matrix	Target Analyte List (TAL)
1-7	AR	Al, Sb,(As) Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K,(Se,)Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN,
n811	Aa	Al, Sb, (s,)Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se) Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		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, CN,
		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, CN,
		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, CN,
		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, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN,
		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, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Π, V, Zn, Mo, B, Si, CN,
		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, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
		Al. Sb. As. Ba. Be, Cd. Ca. Cr. Co, Cu. Fe, Pb. Mg. Mn. Hg. Ni. K. Se, Ag, Na, Tl, V, Zh, Mo, B, Si, CN <sup>-</sup>
		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, CN <sup>-</sup> ,
		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, CN,
		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, CN,
		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, CN,
		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, CN <sup>*</sup> ,
		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, CN,
		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, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
	ı	Analysis Method
ICP		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, CN,
ICP Trace		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
ICP-MS		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, CN',
GFAA		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, CN,

Comments: Mercury by CVAA if performed

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# Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

Page: ( of Reviewer: 2nd Reviewer:

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".

YANA 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? K/N N/A

LEVEL IV ONLY:
Y N K/A Wel

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

		126	, ,		(30 30														In de	
		15																		
	MSD %Recovery	126			30															
	MS %Recovery				30	Winds													-दै	
	Analyte	Se		,	3														15 tot 5:	
	Matríx	*			g					·									ponest Smyle	,
	OI OSW/SW	\$613		JH 101															+	
l	*		1	1	4	1	1				1	1		1	1	$\pm$	1	1	Comments:	

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 6 through July 10, 2009

LDC Report Date:

September 14, 2009

Matrix:

Water

Parameters:

Metals

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304611

#### Sample Identification

M-117B

M-120B

M-103B

M-10B

M-10BDISS

M-121B

M-118B

M-117BMS

M-117BMSD

M-120BMS

M-120BMSD

M-10BDISSMS

M-10BDISSMSD

#### Introduction

This data review covers 13 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Metals. The metals analyzed were Arsenic and Selenium.

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 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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5% .

#### III. Calibration

An initial calibration was performed.

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

#### IV. Blanks

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

No field blanks were identified in this SDG.

#### V. ICP Interference Check Sample (ICS) Analysis

The frequency of analysis was met.

The criteria for analysis were met.

#### VI. 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.

#### VII. Duplicate Sample Analysis

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

#### VIII. Laboratory Control Samples (LCS)

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

#### IX. Internal Standards

Raw data were not reviewed for this SDG.

#### X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

#### XI. ICP Serial Dilution

ICP serial dilution analysis was performed by the laboratory. The analysis criteria were met.

#### XII. Sample Result Verification and Project Quantitation Limit

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304611	All analytes reported below the PQL.	J (all detects)	A

Raw data were not reviewed for this SDG.

#### 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.

## Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Data Qualification Summary - SDG 8304611

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
8304611	M-117B M-120B M-103B M-10B M-10BDISS M-121B M-118B	All analytes reported below the PQL.	J (all detects)	A	Sample result verification (PQL) (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Laboratory Blank Data Qualification Summary - SDG 8304611

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Field Blank Data Qualification Summary - SDG 8304611

No Sample Data Qualified in this SDG

#### **Tronox Northgate Henderson** ORKSHEET

LDC #:	21494K4	_ VALIDATION COMPLETENESS WO
SDG #:	8304611	Stage 2B
Laboratory	Test America	<del>-</del>

Date:	9/14/-9
Reviewer:	1 2
2nd Reviewer:	ma

METHOD: As & Se (EPA SW 846 Method 6020)

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

	Validation Area		Comments
l.	Technical holding times	A	Sampling dates: 7/6/09 _7 /(0/09
11.	ICP/MS Tune	A	, , , , ,
111.	Calibration	À	
. IV.	Blanks	À	
V.	ICP Interference Check Sample (ICS) Analysis		
VI.	Matrix Spike Analysis	A	2m5/450
VII.	Duplicate Sample Analysis	N	) // /
VIII.	Laboratory Control Samples (LCS)	A	Lez
IX.	Internal Standard (ICP-MS)	N	pit verseund
<u>X.</u>	Furnace Atomic Absorption QC	N	v.t uboly)
XI.	ICP Serial Dilution	A	•
XII.	Sample Result Verification	N	
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	M	
ΧV	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:

Valida	ated Samples:						
1	M-117B	11	M-120BMSD	21	IMB	31	
2	M-120B	12	M-10BDISSMS	22		32	
3	M-103B	13	M-10BDISSMSD	23		33	
4	M-10B	14		24		34	
5	M-10BDISS	15		25		35	
6	M-121B	16		26		36	
7	M-118B	17		27		37	
8	M-117BMS	18		28		38	
9	M-117BMSD	19		29		39	
10	M-120BMS	20		30		40	

Notes:				

LDC #: >1494 KJ SDG #: <u>See are</u>

### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page:	(of)
Reviewer:_	
2nd reviewer:	me

All circled elements are applicable to each sample.

	1	
Sample ID	Matrix	Target Analyte List (TAL)
1-7	Aa	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, CN,
		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, CN,
an 8-13	As-	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, CN,
		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, CN,
		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, CN,
		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, CN,
		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, CN,
		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, CN,
		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, CN,
		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, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN',
		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. CN
		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, CN <sup>-</sup> ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN,
		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, CN <sup>-</sup> ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		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, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
	<del> </del>	Analysis Method
ICP		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, CN,
ICP Trace		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
ICP-MS		Al, Sb,(As), Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se Ag, Na, Ti, V, Zn, Mo, B, Si, CN',
GFAA		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, CN,

Comments: Mercury by CVAA if performed

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** 

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 13 through July 15, 2009

LDC Report Date:

September 14, 2009

Matrix:

Water

Parameters:

Metals

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304614

#### Sample Identification

H-11B

H-11BDISS

TR-8B

TR-10B

M-92B

M-92BDISS

M-97B

H-11BMS

H-11BMSD

H-11BDISSMS

H-11BDISSMSD

TR-8BMS

TR-8BMSD

M-92BMS

M-92BMSD

M-92BDISSMS

M-92BDISSMSD

#### Introduction

This data review covers 17 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Metals. The metals analyzed were Arsenic and Selenium.

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 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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

#### III. Calibration

An initial calibration was performed.

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

#### IV. Blanks

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

No field blanks were identified in this SDG.

#### V. ICP Interference Check Sample (ICS) Analysis

The frequency of analysis was met.

The criteria for analysis were met.

#### VI. 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.

#### VII. Duplicate Sample Analysis

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

#### VIII. Laboratory Control Samples (LCS)

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

#### IX. Internal Standards

Raw data were not reviewed for this SDG.

#### X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

#### XI. ICP Serial Dilution

ICP serial dilution analysis was performed by the laboratory. The analysis criteria were met.

#### XII. Sample Result Verification and Project Quantitation Limit

All analytes reported below the PQL were qualified as follows:

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

Raw data were not reviewed for this SDG.

#### 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.

### Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Data Qualification Summary - SDG 8304614

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
8304614	H-11B H-11BDISS TR-8B TR-10B M-92B M-92BDISS M-97B	All analytes reported below the PQL.	J (all detects)	А	Sample result verification (PQL) (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Laboratory Blank Data Qualification Summary - SDG 8304614

No Sample Data Qualified in this SDG

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

No Sample Data Qualified in this SDG

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

Stage 2B

Date:	<u>9/141.</u>
Page:_	
Reviewer:	
2nd Reviewer:	om &

SDG #: 8304614 Laboratory: Test America

21494N4

LDC #:

METHOD: As & Se (EPA SW 846 Method 6020)

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: 1/13/09 - 7/15/09
II.	ICP/MS Tune	A	/ / /
111.	Calibration	A	
IV.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	A	
VI.	Matrix Spike Analysis	Ą	> m 5/1454
VII.	Duplicate Sample Analysis	N	7 / / / / / / / / / / / / / / / / / / /
VIII.	Laboratory Control Samples (LCS)	A	49
IX.	Internal Standard (ICP-MS)	N	Kit veriend
X.	Furnace Atomic Absorption QC	Ŋ	but while I
XI.	ICP Serial Dilution	A	O .
XII.	Sample Result Verification	N	
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	À	
XV	Field Blanks	μ	

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:

MR H-11B 11 H-11BDISSMSD 21 31 32 22 H-11BDISS 12 TR-8BMS TR-8BMSD 23 33 TR-8B 13 TR-10B 14 M-92BMS 24 34 35 25 M-92B 15 M-92BMSD M-92BDISS 16 M-92BDISSMS 26 36 37 M-97B 17 M-92BDISSMSD 27 28 38 H-11BMS 18 29 39 H-11BMSD 19 20 H-11BDISSMS 30 40

Notes:				
•				

LDC #: >1494N7 SDG #: <u>Sce cr</u>

#### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page: \_\_\_\_\_ of \_\_\_\_ Reviewer: \_\_\_\_\_\_ 2nd reviewer: \_\_\_\_\_\_

All circled elements are applicable to each sample.

,		
Sample ID	Matrix	Target Analyte List (TAL)
1-7	Aa	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, CN,
,		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, CN,
~814	m	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K(Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
,		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, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN,
·		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, CN,
		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, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN <sup>-</sup> ,
		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, CN,
		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. CN <sup>-</sup>
		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, CN <sup>-</sup> ,
		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, CN,
		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, CN,
		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, CN.,
		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, CN,
		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, CN,
		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, CN',
		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, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
		Analysis Method
ICP		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, CN <sup>-</sup> ,
ICP Trace		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
ICP-MS		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, CN <sup>-</sup> ,
GFAA		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, CN',

Comments: Mercury by CVAA if performed

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 20 through July 24, 2009

**LDC Report Date:** 

September 14, 2009

Matrix:

Water

Parameters:

Metals

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304615

#### Sample Identification

M-77B

M-77BDISS

M-33B

CLD-4RB

MW-6RB

M-35B

M-52B

M-77BMS

M-77BMSD

M-77BDISSMS

M-77BDISSMSD

M-33BMS

M-33BMSD

**CLD-4RBMS** 

CLD-4RBMSD

#### Introduction

This data review covers 15 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Metals. The metals analyzed were Arsenic and Selenium.

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 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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

#### III. Calibration

An initial calibration was performed.

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

#### IV. Blanks

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

No field blanks were identified in this SDG.

#### V. ICP Interference Check Sample (ICS) Analysis

The frequency of analysis was met.

The criteria for analysis were met.

#### VI. 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
M-77BMS/MSD (M-77B)	Arsenic	73 (75-125)	70 (75-125)	•	J- (all detects) UJ (all non-detects)	Α
,	Selenium	-	69 (75-125)	-	J- (all detects) UJ (all non-detects)	

#### VII. Duplicate Sample Analysis

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

#### VIII. Laboratory Control Samples (LCS)

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

#### IX. Internal Standards

Raw data were not reviewed for this SDG.

#### X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

#### XI. ICP Serial Dilution

ICP serial dilution analysis was performed by the laboratory. The analysis criteria were met.

#### XII. Sample Result Verification and Project Quantitation Limit

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304615	All analytes reported below the PQL.	J (all detects)	Α

Raw data were not reviewed for this SDG.

#### 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.

## Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Data Qualification Summary - SDG 8304615

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
8304615	М-77В	Arsenic Selenium	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	А	Matrix spike/Matrix spike duplicates (%R) (m)
8304615	M-77B M-77BDISS M-33B CLD-4RB MW-6RB M-35B M-52B	All analytes reported below the PQL.	J (all detects)	А	Sample result verification (PQL) (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Laboratory Blank Data Qualification Summary - SDG 8304615

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Field Blank Data Qualification Summary - SDG 8304615

No Sample Data Qualified in this SDG

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

Stage 2B

Date:	9/141
Page:_	
Reviewer:	
2nd Reviewer:	an O

METHOD: As & Se (EPA SW 846 Method 6020)

2149404

8304615 Laboratory: Test America

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: 1/20/09 - 1/24/09
II.	ICP/MS Tune	A	/
III.	Calibration	<u> </u>	
IV.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	A	
VI.	Matrix Spike Analysis	SW	3 M(/M)20
VII.	Duplicate Sample Analysis	<u>N</u>	)
VIII.	Laboratory Control Samples (LCS)	A_	Les
IX.	Internal Standard (ICP-MS)	N	pit venerus
X	Furnace Atomic Absorption QC	V	pot veneral Let Whitegel
XI.	ICP Serial Dilution	A_	3
XII.	Sample Result Verification	N	
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	μ	
XV	Field Blanks	N	

Note:

LDC #:

SDG #:

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:

1	M-77B	11	M-77BDISSMSD	21	Mrs	31	· · · · · · · · · · · · · · · · · · ·
2	M-77BDISS	12 ′	M-33BMS	22		32	
3	M-33B	13	M-33BMSD	23		33	
4	CLD-4RB	14	CLD-4RBMS	24		34	
5	MW-6RB	15	CLD-4RBMSD	25		35	
6	M-35B	16		26		36	
7	M-52B	17		27		37	
8	M-77BMS	18		28		38	
9	M-77BMSD	19		29		39	
10	M-77BDISSMS	20		30		40	

Notes:		

LDC #: >14940 f SDG #: <u>See ar</u>e

#### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page: \_\_\_\_of\_\_\_ Reviewer: \_\_\_\_\_\_ 2nd reviewer: \_\_\_\_\_A

All circled elements are applicable to each sample.

<u> </u>		
Sample ID	Matrix	Target Analyte List (TAL)
1-7	Aa	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, CN,
,		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, CN,
~8-15	Aa	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, CN,
		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, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN,
		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, CN,
		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, CN
		Al. Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Π, V, Zn, Mo, B, Si, CN,
		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, CN <sup>-</sup> ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		Al. Sb. As. Ba. Be, Cd. Ca. Cr. Co. Cu. Fe, Pb. Mg. Mn. Hg, Ni. K. Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN
		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, CN <sup>-</sup> ,
		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, CN,
		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, CN,
		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, CN,
		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, CN <sup>-</sup> ,
		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, CN,
		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, CN',
		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, CN,
		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, CN',
		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, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
	1	Analysis Method
ICP		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, CN,
ICP Trace		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
ICP-MS		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, CN',
GFAA		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,

Comments: Mercury by CVAA if performed

100 #: 21494 SDG #: 226 Contr

# Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

N X ō 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". (Y N N/A 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. Y NA V(N) N/A

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

LEVEL IV ONLY:

Y N (MA Wer

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

	Qualifications	J-42/4 (m)	//											
	Associated Samples													
	RPD (Limits)													
	MSD %Recovery	10	64											
	MS %Recovery	13												
	Analyte	A	X											
	Matrix	द												
	MS/MSD ID	8/9										-		
L	*										1	1	1	

Comments:

# Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** 

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 27, 2009

**LDC Report Date:** 

September 14, 2009

Matrix:

Water

Parameters:

Metals

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304619

#### Sample Identification

M-11B

M-11BDISS

M-11009B

M-11009BDISS

M-11BDISSMS

M-11BDISSMSD

M-11009BMS

M-11009BMSD

#### Introduction

This data review covers 8 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Metals. The metals analyzed were Arsenic and Selenium.

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 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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- 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. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

#### III. Calibration

An initial calibration was performed.

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

#### IV. Blanks

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

No field blanks were identified in this SDG.

#### V. ICP Interference Check Sample (ICS) Analysis

The frequency of analysis was met.

The criteria for analysis were met.

#### VI. 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.

#### VII. Duplicate Sample Analysis

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

#### VIII. Laboratory Control Samples (LCS)

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

#### IX. Internal Standards

Raw data were not reviewed for this SDG.

#### X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

#### XI. ICP Serial Dilution

ICP serial dilution analysis was performed by the laboratory. The analysis criteria were met.

#### XII. Sample Result Verification and Project Quantitation Limit

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304619	All analytes reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

#### XIII. Overall Assessment of Data

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

#### XIV. Field Duplicates

Samples M-11B and M-11009B and samples M-11BDISS and M-11009BDISS were identified as field duplicates. No metals were detected in any of the samples with the following exceptions:

	Concentra	tion (ug/L)		D!#		A or P	
Analyte	M-11B	M-11009B	RPD (Limits)	Difference (Limits)	Flags		
Arsenic	220	220	0 (≤30)	-	-	•	

	Concentra	tion (ug/L)		D.W.			
Analyte	M-11BDISS	M-11009BDISS	RPD (Limits)	Difference (Limits)	Flags	A or P	
Arsenic	190	200	5 (≤30)	-	-	-	

## Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Data Qualification Summary - SDG 8304619

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
8304619	M-11B M-11BDISS M-11009B M-11009BDISS	All analytes reported below the PQL.	J (all detects)	А	Sample result verification (PQL) (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Laboratory Blank Data Qualification Summary - SDG 8304619

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Field Blank Data Qualification Summary - SDG 8304619

No Sample Data Qualified in this SDG

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

Stage 2B

Date:_	9/14/ <sub>2</sub> 9
Page:_	_of/
Reviewer:_	<u>~</u>
2nd Reviewer:	ana

METHOD: As & Se (EPA SW 846 Method 6020)

21494S4

8304619 Laboratory: Test America

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: カットゥ
II.	ICP/MS Tune	À	/
III.	Calibration	À	
IV.	Blanks	Ά	
V.	ICP Interference Check Sample (ICS) Analysis	A	
VI.	Matrix Spike Analysis	A	lus/ puso
VII.	Duplicate Sample Analysis	N	
VIII.	Laboratory Control Samples (LCS)	A	w
IX.	Internal Standard (ICP-MS)	Ñ	Not beginned
X.	Furnace Atomic Absorption QC	N	but whiles
XI.	ICP Serial Dilution	A	
XII.	Sample Result Verification	N	
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	Sul	(1,3)(7,4)
ΧV	Field Blanks	V	

N 1 -	
INC	ote:

LDC #:

SDG #:

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:

	A>		,			
1	M-11B	11	MB	21	31	
2	M-11BDISS	12		22	32	
3	M-11009B	13		23	33	
4	M-11009BDISS	14		24	34	
5	M-11BDISSMS	15		25	35	
6	M-11BDISSMSD	16		26	36	
7	M-11009BMS	17		27	37	
8	M-11009BMSD	18		28	38	
9		19		29	39	
10		20		30	40	

Notes:				

LDC #: >149454 SDG #: See www

#### VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

All circled elements are applicable to each sample.

		Target Analyte List (TAL)
Sample ID	Matrix	
1-4	AQ	Al, Sb,(As) Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K,(Se,)Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN,
N5-8	p	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, CN,
	,	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN,
		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, CN,
		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, CN,
		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, CN,
		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, CN,
		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, CN
		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, CN,
		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, CN <sup>-</sup> ,
		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, CN,
		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. Sì. CN
		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, CN <sup>-</sup> ,
		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, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN',
		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, CN,
		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, CN,
		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, CN,
		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, CN,
	<b></b>	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, CN,
		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, CN',
	1	Analysis Method
ICP		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN',
ICP Trace		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, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
ICP-MS		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, CN',
GFAA		MI, SU, MS, DA, DE, CU, CA, CI, CU, CU, FE, FB, MI, MI, MI, MI, MI, N, GE, MG, MA, MA, M, GI, MG, D, GI, CA

Comments: Mercury by CVAA if performed

LDC#:_	2149	9484
SDG#:	See	Cover

# **VALIDATION FINDINGS WORKSHEET** Field Duplicates

	r 1	
Page: <u> </u>	_of	
Reviewer:	<u>رک</u>	
2nd Reviewer:	and	

METHOD: Metals (EPA Method 6020)

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

	Concentrat	ion (ug/L)	(≤30)	(ug/L)	(ug/L)	Qualifications
Compound	1	3	RPD	Difference	Limits	(Parent Only)
Arsenic	220	220	0			

	Concentrat	ion (ug/L)	(≤30)	(ug/L)	(ug/L)	Qualifications
Compound	2	4	RPD	Difference	Limits	(Parent Only)
Arsenic	190	200	5			

V:\FIELD DUPLICATES\FD\_inorganic\21494S4.wpd

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

August 3 through August 4, 2009

LDC Report Date:

September 24, 2009

Matrix:

Water

Parameters:

Metals

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304622

# Sample Identification

M-31AB

M-31ABDISS

M-50B

M-21B

FB080409-GW

M-31ABMS

M-31ABMSD

M-31ABDISSMS

M-31ABDISSMSD

M-21BMS

M-21BMSD

# Introduction

This data review covers 11 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6020 for Metals. The metals analyzed were Arsenic and Selenium.

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 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.

Blanks are summarized in Section IV.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

# I. Technical Holding Times

All technical holding time requirements were met.

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

### II. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

# III. Calibration

An initial calibration was performed.

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

### IV. Blanks

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

Sample FB080409-GW was identified as a field blank. No metals contaminants were found in this blank.

# V. ICP Interference Check Sample (ICS) Analysis

The frequency of analysis was met.

The criteria for analysis were met.

# VI. 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.

# VII. Duplicate Sample Analysis

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

# VIII. Laboratory Control Samples (LCS)

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

# IX. Internal Standards

Raw data were not reviewed for this SDG.

# X. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

# XI. ICP Serial Dilution

ICP serial dilution analysis was performed by the laboratory. The analysis criteria were met.

# XII. Sample Result Verification and Project Quantitation Limit

All analytes reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304622	All analytes reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

# 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.

# Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Data Qualification Summary - SDG 8304622

SDG	Sample	Analyte	Flag	A or P	Reason (Code)
8304622	M-31AB M-31ABDISS M-50B M-21B FB080409-GW	All analytes reported below the PQL.	J (all detects)	А	Sample result verification (PQL) (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Laboratory Blank Data Qualification Summary - SDG 8304622

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Metals - Field Blank Data Qualification Summary - SDG 8304622

No Sample Data Qualified in this SDG

# **Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET**

Stage 2B

Date: <u> </u>	0 0
Page: 1 of 1	_ /
Reviewer: 🛁	_
2nd Reviewer: MA	_

SDG #: 8304622 Laboratory: Test America

21494V4

LDC #:

METHOD: As & Se (EPA SW 846 Method 6020)

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

	Validation Area		Comments
l.	Technical holding times	A	Sampling dates: 8/3/59 - 8/4/69
<u>II.</u>	ICP/MS Tune	A	, ,
III.	Calibration	À	
IV.	Blanks	A	
V.	ICP Interference Check Sample (ICS) Analysis	A	
VI.	Matrix Spike Analysis	A	) m5/145p
VII.	Duplicate Sample Analysis	V	) 1
VIII.	Laboratory Control Samples (LCS)	A	Ly
IX.	Internal Standard (ICP-MS)	N	Vit varieurs
X.	Furnace Atomic Absorption QC	N	1. t utilizes
XI.	ICP Serial Dilution	A	
XII.	Sample Result Verification	N N	
XIII.	Overall Assessment of Data	A	
XIV.	Field Duplicates	2	
ΧV	Field Blanks	Np	FB=5

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: h

	1.3 0-					
1	M-31AB	11	M-21BMSD	21	31	
2	M-31ABDISS	12	MR	22	32	
3	M-50B	13	( )	23	 33	
4	M-21B	14		24	34	
5	FB080409GW	15		25	35	
6	M-31ABMS	16		26	36	
7	M-31ABMSD	17		27	37	
8	M-31ABDISSMS	18		28	38	
9	M-31ABDISSMSD	19		29	 39	
10	M-21BMS	20		30	 40	

Notes:				
			 •	

LDC#:<u>>1494</u> SDG#:<u>See</u> are~

# VALIDATION FINDINGS WORKSHEET Sample Specific Element Reference

Page: of Page: 2nd reviewer: 2nd reviewer:

All circled elements are applicable to each sample.

	<del></del>	
Sample ID	Matrix	Target Analyte List (TAL)
15	Aa	Al, Sb,(As) Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K,(Se)Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN,
V6-11	A	Al, Sb, 🕒 Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, 🔗 Ag, Na, Tl, V, Zn, Mo, B, Si, CN,
		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, CN
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN,
		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, CN,
		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, CN <sup>-</sup>
		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, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
		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, CN',
		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. CN <sup>-</sup>
		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, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		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, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
		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, CN',
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
		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, CN <sup>-</sup> ,
		Analysis Method
ICP		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, CN <sup>-</sup> ,
ICP Trace		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Si, CN,
ICP-MS		Al, Sb,(As), Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, & Ag, Na, Ti, V, Zn, Mo, B, Si, CN <sup>-</sup> ,
GFAA		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, CN,

Comments: Mercury by CVAA if performed	·

# Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Data Validation Reports LDC# 21494

Organophosphorus Pesticides



# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

May 27 through May 28, 2009

LDC Report Date:

September 18, 2009

Matrix:

Water

Parameters:

Organophosphorus Pesticides

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304601

Sample Identification

EB052709 MC-45B M-127B

## Introduction

This data review covers 3 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.

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

The following are definitions of the data qualifiers:

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

# I. Technical Holding Times

All technical holding time requirements were met.

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

# II. Calibration

## a. Initial Calibration

Initial calibration of compounds was performed 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.

# 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
6/5/09	023F1001	1	Fensulfothion	25.6	All samples in SDG 8304601	J- (all detects) UJ (all non-detects)	А
6/5/09	023F2501	2	Naled Azinphos-methyl	24.1 21.3	All samples in SDG 8304601	J+ (all detects) 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:

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
6/1/09	010F1001	1	Naled	29.0	All samples in SDG 8304601	J- (all detects) UJ (all non-detects)	Р
6/1/09	010F1001	2	Naled	35.8	All samples in SDG 8304601	J- (all detects) UJ (all non-detects)	Р

# III. Blanks

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

Sample EB052709 was identified as an equipment bank. No organophosphorus pesticide contaminants were found in this blank.

Sample FB060309-SO (from SDG 8304603) was identified as a field 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

Raw data were not reviewed for this SDG.

# VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304601	All compounds reported below the PQL.	J (all detects)	A

Raw data were not reviewed for this SDG.

# VII. System Performance

Raw data were not reviewed for this SDG.

# VIII. Overall Assessment of Data

Data flags 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 8304601

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304601	EB052709 MC-45B M-127B	Fensulfothion	J- (all detects) UJ (all non-detects)	А	Continuing calibration (%D) (c)
8304601	EB052709 MC-45B M-127B	Naled Azinphos-methyl	J+ (all detects) J+ (all detects)	А	Continuing calibration (%D) (c)
8304601	EB052709 MC-45B M-127B	Naled	J- (all detects) UJ (all non-detects)	Р	Continuing calibration (ICV %D) (c)
8304601	EB052709 MC-45B M-127B	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 8304601

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304601

No Sample Data Qualified in this SDG

SDG ; _abor <b>VIE</b> TH	#: 21494A17 #: 8304601 atory: Test America HOD: GC Organophosp	horus F	LIDATION Pesticides (I	N COMPI St EPA SW 84	age 2B 46 Method 8	S WC	ORKSHEET		F Rev 2nd Rev	/	
/alida	tion findings worksheet										Ī
	Validatio	n Area		Λ	Sampling date		Comm /27- 28 /				_
I. Ila.	Technical holding times  Initial calibration			A	r ~	<u> </u>	RSD	<u> </u>			-
IIb.	Calibration verification/ICV	······································		SW		/100					-
111.	Blanks			A		, <u>10 y</u>					
IVa.	Surrogate recovery	· · · · · · · · · · · · · · · · · · ·		A					<del></del>		
IVb.	Matrix spike/Matrix spike of	luplicates		N	Clie	nt s	vec		***************************************		
IVc.	Laboratory control sample		, . , . , . , . , . , . , . , . , . , .	A	LCS,	nt s					
V.	Target compound identific			N							
VI.	Compound Quantitation a		s	N							
VII.	System Performance			N							
VIII.	Overall assessment of dat	a		Α							
IX.	Field duplicates			N							
Х.	Field blanks			ND	EB =	. 1	753-7	<del>8</del> -	FB = F80	8304603	
Note: /alidat	A = Acceptable N = Not provided/applicab SW = See worksheet ed Samples: Water	ile	R = Rins	o compounds sate eld blank	detected	TB :	Duplicate = Trip blank = Equipment blar	nk	from	8304603	,
1-1	EB052709	11			21			31			
2	MC-45B	12			22			32			_
3	M-127B	13			23			33			_
4 1	9153088 MB	14			24			34			
5		15			25			35_			-
6		16			26			36			
7		17			27			<b>3</b> 7			
8		18			28			38			
9		19			29			39			
10		20			30			40			

Notes:\_\_

# VALIDATION FINDINGS WORKSHEET

METHOD: /GC HPLC

8310	8330	8151	(8141	8141(con't)	8021B
A. Acenaphthene	А. НМХ	A. 2,4-D	A. Dichlorvos	V. Fensulfothion	V. Benzene
B. Acenaphthylene	B. RDX	B. 2,4-DB	B. Mevinphos	W. Boistar	CC Tolliene
C. Anthracene	C. 1,3,5-Trinitrobenzene	C. 2,4,5-T	C. Demeton-O	X. EPN	
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	l. 2-Amino-4,6-dinitrotoluene	I. MCPP	I. Dimethoate	DD. Triffuralin	
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. Prowi	
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. Chlorpyrlfos	JJ. Thionazin	
P. Pyrene	F.		P. Fenthion	l	
ď	G		Q. Parathion-ethyl	LL. 0,00-Triethylphosphorothioate	os phorothio ate
.Y.			R. Trichloronate	ľ	
S.	÷		S. Merphos		100
			T. Stirofos	00. Carbophenothien	ion - methy/
			U. Tokuthion		

cmpd\_list.wpd

LDC#: 21494 A17 SDG#: 50 Con-1

# VALIDATION FINDINGS WORKSHEET Continuing Calibration

Page: of Reviewer:\_\_

2nd Reviewer:\_\_

METHOD: ZGC\_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 \_\_XN\_N/A Were continuing calibration standards analyzed at the required frequencies?

Were continuing calibration standards analyzed at the required frequencies? 20

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

C.d.,   -C. (+)   78,00     24,00     23,40     23,40     23,60       23,60       24,10     -   24,10     -   24,10     -   24,10     -   24,10     -   24,10         24,10       24,10       24,10       24,10       24,10       24,10       24,10       24,10       24,10       24,10         24,10 	#	Date	Standard ID	Detector/ Column	Compound	%D / RPD (Limit <-18:0) (	€ 20 & )RT (limit)		Associated Samples	Samples	Qualifications	<del></del> 7
$(iw) \qquad \downarrow \qquad $	l	66169	10014010	- - -	(+) -2	178.00	)	(	+	14	3+ dets/P(C)	
$(CA, 2) = \frac{284}{c}  (CA) = \frac$	ı		( Mr)		( <u>1</u> )		J	(			J-145/P	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	1			_	<del>6-1-4</del>		)	(			3-/8/5	T
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	I			١.	0		)	(			3+ dets/P	_
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	1				± €	3.56	)	(			J- /43/P	
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	ı			<b>\</b>	<del>(-) 4</del>	84.0	)	)			3-1R/P	
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	i i						)	)				
$0.23F_{250}I$ $C_{eff}I$ $V$ (-2) $3.5.6$ ( ) $(ccv)$ $c_{eff}I$ ( )       ( ) $V$ ( $ccv$ ) $V$ ( $v$ )	1						)	(				_
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	1	16/261	0 25F 250	5.1	(-) V	35.6	)	)			J- /MJ /A	1
( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( )	•		(ccv)	•	(F) 7	24,1	)	)			J+ d++/A	Ť
	,		\ \	7	(t) /	21.3	)	(				
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# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

June 1 through June 4, 2009

**LDC Report Date:** 

September 19, 2009

Matrix:

Soil

Parameters:

Organophosphorus Pesticides

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304602

Sample Identification

RSAM3-0.5B

RSAM2-0.5B

RSAJ3-0.5B

RSAM2-0.5BMS

RSAM2-0.5BMSD

## Introduction

This data review covers 5 soil 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.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- 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 (r²) was greater than or equal to 0.990.

# 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
6/19/09	051F5101	1	Dichlorvos Azinphos-methyl	24.2 30.9	All samples in SDG 8304602	J+ (all detects) J+ (all detects)	А
6/19/09	051F5101	1	Naled Disulfoton	38.2 26.8	All samples in SDG 8304602	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	Α .
6/19/09	051F5101	2	Naled	49.0	All samples in SDG 8304602	J- (all detects) UJ (all non-detects)	А
6/19/09	051F5101	2	Azinphos-methyl	35.3	All samples in SDG 8304602	J+ (all detects)	А

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
6/1/09	01 0F1 001	1	Naled	29.0	All samples in SDG 8304602	J- (all detects) UJ (all non-detects)	Р
6/1/09	01 0F1 001	2	Naled	35.8	All samples in SDG 8304602	J- (all detects) UJ (all non-detects)	Р

# III. Blanks

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

Sample FB072109-SO (from SDG 8304616) was identified as a field 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

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

Raw data were not reviewed for this SDG.

# VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304602	All compounds reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

# VII. System Performance

Raw data were not reviewed for this SDG.

# VIII. Overall Assessment of Data

Data flags 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 8304602

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304602	RSAM3-0.5B RSAM2-0.5B RSAJ3-0.5B	Dichlorvos Azinphos-methyl	J+ (all detects) J+ (all detects)	А	Continuing calibration (%D) (c)
8304602	RSAM3-0.5B RSAM2-0.5B RSAJ3-0.5B	Naled Disulfoton	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	А	Continuing calibration (%D) (c)
8304602	RSAM3-0.5B RSAM2-0.5B RSAJ3-0.5B	Naled	J- (all detects) UJ (all non-detects)	Р	Continuing calibration (ICV %D) (c)
8304602	RSAM3-0.5B RSAM2-0.5B RSAJ3-0.5B	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (PQL) (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Laboratory Blank Data Qualification Summary - SDG 8304602

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304602

No Sample Data Qualified in this SDG

# Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

LDC #: 21494B17	_ VALIDATION COMPLETENESS WORKSHEET	Date: <u>9/15/66</u>
SDG #: 8304602	Stage 2B	Page: Lof_/
Laboratory: Test America	<u> </u>	Reviewer: <u>JV</u>
,		2nd Reviewer:
METHOD: GC Organophospi	horus Pesticides (FPA 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
1.	Technical holding times	Α	Sampling dates: 6/01 - 04/09
Ila.	Initial calibration	Á	70 RSD & 20% r~
llb.	Calibration verification/ICV	Sw	COV/IW = 20 ]
	Blanks	A	
IVa.	Surrogate recovery	Â	
IVb.	Matrix spike/Matrix spike duplicates	A	
IVc.	Laboratory control samples	A	US
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	ND	FB = FB672109-SO from 8304616

Note: A = Acceptable

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

R = Rinsate

FB = Field blank

D = Duplicate

TB = Trip blank
EB = Equipment blank

Validated Samples:

Soil

	301	1				
* 1	RSAI2-0.5B	11	9166561 MB	21	31	
2	RSAM3-0.5B	12		22	32	· · · · · · · · · · · · · · · · · · ·
3	RSAM2-0.5B	13		23	33	
4	RSAJ3-0.5B	14		24	34	
5	RSAM2-0.5BMS	15		25	35	
6	RSAM2-0.5BMSD	16		26	36	
7		17		27	37	
8		18		28	38	
9		19		29	39	
10		20		30	40	

Notes: [#/	Not	Valid alid	per	list )	
	,		1	/	

# VALIDATION FINDINGS WORKSHEET

METHOD: /GC HPLC

A Accenaphthene         A HMX         A 2,4-D         A Dichlorvos           B. Accenaphthene         B. RDX         B. 2,4-DB         B. Mevinphos           C. Anthracene         C. 1,35-Trinitrobenzene         C. 2,45-T         C. Demeton-O           D. Benzo(a)muthracene         D. 0,1,3-Dinitrobenzene         C. 2,45-T         C. Demeton-O           D. Benzo(a)muthracene         E. Tetryl         E. Dichlorprop         E. Ethoprop           F. Benzo(a)muthracene         F. Nitrobenzene         F. Dichlorprop         F. Naled           G. Benzo(a)moranthene         H. A-Amino-2,6-dinitrotoluene         H. Dalapon         H. Phorate           H. Benzo(b)fluoranthene         H. A-Amino-A,6-dinitrotoluene         H. Dalapon         H. Phorate           I. Chrysene         I. Z-Amino-A,6-dinitrotoluene         H. Dalapon         H. Phorate           J. Dibenz(a,h)anthracene         I. Z-Amino-A,6-dinitrotoluene         H. Dalapon         H. Phorate           J. Leluorene         L. Z-Mintotoluene         K. Pentachlorophenol         H. Phorate           M. Indeno(1,2,3-cd)pyrene         M. 3-Mitrotoluene         M. Silvex         M. Roinel           O. Phenanthrene         P. Pyrene         P. Parathlon-tetryl         P. Parathlon-tetryl           O. O. Chiorpyrifos         P. Pyrene         P. P	A. 2,4-D A. B. 2,4-DB B. C. 2,4,5-T C.	3141	8141(con't)	8021B
A. HMX	A. 2,4-D A. B. 2,4-DB B. C. 2,4,5-T C.			
End EDX         B. 24-DB         F.           ene         D. 1,3-Trinitrobenzene         C. 24,5-TP         0           thene         E. Tetryl         E. Dinoseb         p           thene         F. Nitrobenzene         F. Dichlorprop         p           thene         F. Nitrobenzene         F. Dichlorprop         p           thene         F. Nitrobenzene         G. Dicamba         p           thene         H. 4-Amino-2,6-dinitrotoluene         H. Dalapon         p           i. 2-Amino-4,6-dinitrotoluene         I. MCPP         p           i. 2-Amino-4,6-dinitrotoluene         I. MCPP         p           K. 2,6-Dinitrotoluene         I. MCPP         p           K. 2,6-Dinitrotoluene         I. 2,4,5-TP (silvex)           N. 3-Nitrotoluene         M. Silvex           O.         p           P.         P	B. 2,4-DB B. C. 2,4,5-T C.		V. Fensulfothion	V. Benzene
C. 1,3,5-Trinitrobenzene       C. 2,4,5-T         chracene       D. 1,3-Dinitrobenzene       D. 2,4,5-TP       D         ene       E. Tetryl       E. Dinoseb       D         oranthene       F. Nitrobenzene       F. Dichlorprop       D         Operylene       G. 2,4,6-Trinitrotoluene       F. Dichlorprop       D         oranthene       H. 4-Amino-2,6-dinitrotoluene       H. Dalapon       D         ne       K. 2,4-Dinitrotoluene       I. MCPA       D         ne       K. 2,6-Dinitrotoluene       K. Pentachlorophenol       D         ne       N. 4-Nitrotoluene       M. 3livex         ne       O.       P.         P.       P.	C. 2,4,5-T	evinphos	W. Boistar	CC. Toluene
E. Tetryl E. Dinoseb B. 24,5-TP B. Tetryl E. Dinoseb B. C. 24,6-Trinitrotoluene B. Dicamba B. C. 24,6-Trinitrotoluene B. Dicamba B. C. 24,6-Trinitrotoluene B. Dicamba B. C. 24,6-Trinitrotoluene B. MCPP B. Dicamba B. C. 24,5-Trinitrotoluene B. M. Silvex B. C. 24,5-TP (silvex) B. 24,5-		-	X. EPN	EE. Ethyl Benzene
E. Tetryl E. Dinoseb I  F. Nitrobenzene F. Dichlorprop I  G. 2.4.6-Trinitrotoluene G. Dicamba H. 4-Amino-2,6-dinitrotoluene H. Dalapon I. 2-Amino-4,6-dinitrotoluene I. MCPP I. 2-Amino-4,6-dinitrotoluene I. MCPP K. 2,6-Dinitrotoluene K. Pentachlorophenol L. 2-Nitrotoluene M. 3-Nitrotoluene O. P. O.	D. 2,4,5-TP	emeton-S	Y. Azinphos-methyl	SSS. O-Xylene
F. Nitrobenzene F. Dichlorprop  G. 2.4.6-Trinitrotoluene G. Dicamba H. 4-Amino-2,6-dinitrotoluene H. Dalapon L. 2-Amino-4,6-dinitrotoluene I. MCPP K. 2,6-Dinitrotoluene K. Pentachlorophenol L. 2-Nitrotoluene K. Pentachlorophenol L. 2-Nitrotoluene M. Silvex  N. 4-Nitrotoluene O. P.	щ	thoprop	Z. Coumaphos	RRR. MP-Xylene
G. 2.4.6-Trinitrotoluene G. Dicamba H. 4-Arnino-2,6-dinitrotoluene H. Dalapon L. 2-Amino-4,6-dinitrotoluene I. MCPP L. 2-Anintrotoluene J. MCPA K. 2,6-Dinitrotoluene K. Pentachlorophenol L. 2-Nitrotoluene L 2,4,5-TP (silvex) ne M. 3-Nitrotoluene O. P.		aled	AA. Parathion	GG. Total Xylene
fluoranthene       H. 4-Amino-2,6-dinitrotoluene       H. Dalapon         1. 2-Amino-4,6-dinitrotoluene       I. MCPP         1. 2,4-Dinitrotoluene       J. MCPA         1. 2,6-Dinitrotoluene       K. Pentachlorophenol         1. 2-Nitrotoluene       L. 2,4,5-TP (silvex)         Intene       N. 4-Nitrotoluene         Intene       O.         P.       P.	G. Dicamba G.	ulfotep	BB. Trichloronate	
h)anthracene I. AcAmino-4,6-dinitrotoluene I. MCPP  J. 2,4-Dinitrotoluene J. MCPA  K. 2,6-Dinitrotoluene K. Pentachlorophenol  L. 2-Nitrotoluene L. 2,4,5-TP (silvex)  A. 3-Nitrotoluene M. Silvex  hrene O.  P.  Q  Q	H. Dalapon	horate	CC. Trichlorinate	
J. 2,4-Dinitrotolune J. MCPA  K. 2,6-Dinitrotoluene K. Pentachlorophenol  L. 2-Nitrotoluene M. Silvex  M. 3-Nitrotoluene M. Silvex  O.  P.	I. MCPP	methoate	DD. Triffuralin	
K. 2,6-Dinitrotoluene       K. Pentachlorophenol         L. 2-Nitrotoluene       L. 2,4,5-TP (silvex)         e       M. 3-Nitrotoluene         e       O.         P.       Q	J. MCPA	lazinon	EE. Def	
L. 2-Nitrotoluene L 2,4,5-TP (silvex) ,2,3-cd)pyrene M. 3-Nitrotoluene M. Silvex ene N. 4-Nitrotoluene O. P. P.	K. Pentachlorophenol	isulfoton	FF. Prowl	
M. 3-Nitrotoluene         M. Silvex         M.           N. 4-Nitrotoluene         N.           O.         P.         P.           Q.         Q.	rP (silvex)	arathion-methyl	GG. Ethion	
N. 4-Nitrotoluene     O.	M. Silvex	Ronnel	HH. Tetrachlorvinphos	
threne O. P. P. O.	N. N.	Malathion	II. Sulprofos	
.q. Q. u	0.0	Chlorpyrifos	JJ. Thionazin	
C G	P. F	-enthion	kk, Phosmet	
	o o	Parathion-ethyi	LL. O.O.O Triethylphosphorothioate	osphorothioate
	. <del>.</del> .	R. Trichloronate	MM. Famphur	
S. Merphos	·	Merphos	NN. Carbophenothlon	lon
T. Stirofos	. T. S.	ittrofos	00. Carbophenothlon - methy	ion - methyl
U. Tokuthion	0.1	Tokuthion		

cmpd\_list.wpd

Notes:

LDC #: 21494 B 17 SDG #: Sec Com

# VALIDATION FINDINGS WORKSHEET Continuing Calibration

Page: 1 of Reviewer: 3VC

2nd Reviewer:

METHOD: \_\_GC\_\_ HPLC

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

Did the continuing calibration standards meet the %D / RPD validation criteria of <20.0%?

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

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

sui	(2) ±		۵	6																				
Qualifications	Fracts /	J-/ M3/F	3/R/P	State of	J-145/P	J= /4/P			J+ 4cts /A	J-/MJ/A	,	Jt dots 1A	J-143/A	J+ dets A										
Samples	Bik																							
Associated Samples	A11 + B																							
	)	)	)	)	)	)	)	)	,	)	)	)	)	)	(	)	)	)	^	)	)	)	)	
≀T (limit)																								
202) F		)	)	)	)	)	)	)	)	)		)	)	)	)			)	)	)				
%D / RPD (Limit ≤ 45.0) RT (limit)	178.0	29.0	78.1	4524	35.8	89,0			24,2	38.2	26.8	90.9€	49.0	35,3										
Compound	( <del>+)</del>	T (F)		6 (4)	i i	Ш			A (+)	(J) #	Α (-)		F (-)	_										
Detector/ Column	- ਤੁ			3.4					Ch.)			->	Sei, 2											
Standard ID	010 F 1001	(S)							051F5101	(604)														
Date	661 101			-					6/6/2															
*	╫╴																							

(1W - AVE 720)

# LDC Report# 21494C17

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

June 1 through June 5, 2009

LDC Report Date:

October 6, 2009

Matrix:

Water

Parameters:

Organophosphorus Pesticides

**Validation Level:** 

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304603

# Sample Identification

M-7BB

M-5AB

FB060309

M-23B

M-23009B

PC-40B

PC-40BRE

PC-4009B

PC-4009BRE

# Introduction

This data review covers 9 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.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- Data are qualified as estimated, with a low bias likely to occur. False positives or false Jnegatives are unlikely to have been reported.
- Data are qualified as estimated; it is not possible to assess the direction of the potential J 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.
- Data are qualified as rejected. There is a significant potential for the reporting of false R negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- The analytical result may be a false positive totally attributable to blank contamination. В This qualifier is applicable to radiochemistry analysis only.
- The analytical result may be biased high and partially attributable to blank contamination. JB This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- The analytical result is not used for reporting because a more accurate and precise result Χ 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.
- Indicates the finding is based upon technical validation criteria. Α
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

# I. Technical Holding Times

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

Sample	Analyte	Total Days From Sample Collection Until Analysis	Required Holding Time (in Days) From Sample Collection Until Analysis	Flag	A or P
PC-40BRE PC-4009BRE	All TCL compounds	15	7	J- (all detects) R (all non-detects)	P

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 (r²) was greater than or equal to 0.990.

### 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
6/12/09	014F1401	1	Naled Azinphos-methyl	51.2 26.2	M-7BB M-5AB FB060309 M-23B M-23009B 9159499MB 9160222MB	J+ (all detects) J+ (all detects)	A

					Associated		
Date	Standard	Column	Compound	%D	Samples	Flag	A or P
6/12/09	014F1401	2	Naled Azinphos-methyl	60.9 31.0	M-7BB M-5AB FB060309 M-23B M-23009B 9159499MB 9160222MB	J+ (all detects) J+ (all detects)	А
6/21/09	003F0301	1	Dichlorvos	34.2	PC-40B PC-4009B 9159433MB	J- (all detects) UJ (all non-detects)	A
6/21/09	003F0301	1	Naled	34.0	PC-40B PC-4009B 9159433MB	J+ (all detects)	А
6/21/09	003F0301	2	Dichlorvos  Trichloronate	29.3 21.6	PC-40B PC-4009B 9159433MB	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	А
6/21/09	003F0301	2	Naled	45.1	PC-40B PC-4009B 9159433MB	J+ (all detects)	A
6/17/09	003F0301	1	Dichlorvos Mevinphos	29.1 20.2	PC-40BRE PC-4009BRE 9167134MB	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	А
6/17/09	003F0301	1	Naled	42.3	PC-40BRE PC-4009BRE 9167134MB	J+ (all detects)	Α
6/17/09	003F0301	2	Dichlorvos	24.7	PC-40BRE PC-4009BRE 9167134MB	J- (all detects) UJ (all non-detects)	А
6/17/09	003F0301	2	Naled	50.3	PC-40BRE PC-4009BRE 9167134MB	J+ (all detects)	А

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
6/1/09	010F1001	1	Naied	29.0	All samples in SDG 8304603	J- (all detects) UJ (all non-detects)	Р
6/1/09	010F1001	2.	Naled	35.8	All samples in SDG 8304603	J- (all detects) UJ (all non-detects)	Р

### III. Blanks

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

Sample FB060309 was identified as a field 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. Although the LCSD percent recoveries (%R) and the LCS/LCSD relative percent differences (RPD) were not within QC limits for some compounds, the LCS percent recoveries (%R) were within QC limits and no data were qualified.

# V. Target Compound Identification

Raw data were not reviewed for this SDG.

# VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

V:\LOGIN\TRONOXNG\21494C17.RV1

Sample	Finding	Flag	A or P
All samples in SDG 8304603	All compounds reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

## VII. System Performance

Raw data were not reviewed for this SDG.

## \*VIII. Overall Assessment of Data

The overall assessment of data was acceptable. In the case where more than one result was reported for an individual sample, the least technically acceptable results were rejected as follows:

Sample	Compound	*Flag	A or P
PC-40BRE PC-4009BRE	All TCL compounds	х	A

<sup>\*</sup>Corrected Flag in above table from "R" to "X".

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

## IX. Field Duplicates

Samples M-23B and M-23009B, samples PC-40B and PC-4009B, and samples PC-40BRE and PC-4009BRE were identified as field duplicates. No organophosphorus pesticides were detected in any of the samples.

## \*Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Data Qualification Summary - SDG 8304603

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304603	PC-40BRE PC-4009BRE	All TCL compounds	J- (all detects) R (all non-detects)	P	Technical holding times (h)
8304603	M-7BB M-5AB FB060309 M-23B M-23009B	Naled Azinphos-methyl	J+ (all detects) J+ (all detects)	А	Continuing calibration (%D) (c)
8304603	PC-40B PC-4009B PC-40BRE PC-4009BRE	Naled	J+ (all detects)	А	Continuing calibration (%D) (c)
8304603	PC-40B PC-4009B	Dichlorvos  Trichloronate	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	А	Continuing calibration (%D) (c)
8304603	PC-40BRE PC-4009BRE	Dichlorvos Mevinphos	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	А	Continuing calibration (%D) (c)
8304603	M-7BB M-5AB FB060309 M-23B M-23009B PC-40B PC-40BRE PC-4009B PC-4009BRE	Naied	J- (all detects) UJ (all non-detects)	P	Continuing calibration (ICV %D) (c)
8304603	M-7BB M-5AB FB060309 M-23B M-23009B PC-40B PC-40BRE PC-4009B PC-4009BRE	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (PQL) (sp)
*8304603	PC-40BRE PC-4009BRE	All TCL compounds	*X	А	Overall assessment of data (0)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Laboratory Blank Data Qualification Summary - SDG 8304603

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304603

No Sample Data Qualified in this SDG

## Tronox Northgate Henderson

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LDC #: 21494C17	VALIDATION COMPLETENESS WORKSHEET	Date: 9/10/09
SDG #: 8304603	Stage 2B	Page: <u>1</u> of <u>/</u>
Laboratory: Test America		Reviewer: <u>3V6</u>
•		2nd Reviewer:
METHOD: GC Organophosph	orus Pesticides (EPA SW 846 Method 8141A)	7

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	W2	Sampling dates: 6/02-01-05/09
lla.	Initial calibration	A	7 RSD 4 20 3 P
IIb.	Calibration verification/ICV	SW	CN/W € 20 3
101.	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	N	Client spec (insufficient sample LCS/D
IVc.	Laboratory control samples	SM	LCS/p
V.	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	N	
VIII.	Overall assessment of data	S₩	
IX.	Field duplicates	ND	$D_1 = 4.5$ $D_2 = 6.8$ $D_7 = 7.4$
X.	Field blanks	MD	TB = 3

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:

	Campica.	Water					
1 1	M-7BB		11 1	915 9499	MB	21	31
2 7	M-5AB		127	9160222	MB	22	32
3 7	FB060309		13 3	915 9433	MB	23	33
4 Y	M-23B	ρ,	14 4	9167134	MB	24	34
5 7	, M-23009B	Ď,	15			25	35
- 6 3	PC-40B	Dr	16			26	36
7 4	PC-40BRE	D <sub>3</sub>	17		·····	27	37
8 3	PC-4009B	<i>0</i> <sub>4</sub>	18			28	38
9 4	PC-4009BRE	I)3	19			29	39
10			20			30	40

Notes:			 

# VALIDATION FINDINGS WORKSHEET-

METHOD: /GC HPLC

				,	
8310	8330	8151	(8141	8141(Con't)	8021B
A. Acenaphthene	A. HMX	A. 2,4-D	A. Dichlorves	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,l)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. Triffuralin	
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. Ethlon	
M. Indeno(1,2,3-cd)pyrene	M. 3-Nitrotoluene	M. Silvex	M. Ronnel	HH. Tetrachlorvinphos	
N. Naphthalene	N. 4-Nitrotoluene		N. Malathion	II. Suiprofos	
O. Phenanthrene	.0		O. Chlorpyrifos	JJ. Thionazin	
P. Pyrene	Р,		P. Fenthion	kk, Phosmet	
Ġ	O		Q. Parathion-ethyl	LL. O.O.O. Triethylph	0,00 - Triethylphosphorothioate
R,			R. Trichloronate	MM. Famphur	
S.			S. Merphos	NN. Carbo pheno thion	hon
			T. Stirofos	00. Carbophenot	Carbo pheno then - methyl
	·		U. Tokuthion		

cmpd\_list.wpd

Notes:

750 225 21494617 SDG#: LDC#:

## VALIDATION FINDINGS WORKSHEET **Technical Holding Times**

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

All circled dates have exceeded the technical holding times.

METHOD GC HPLC	GCHF	orc.					
Sample ID	Matrix	Preserved	Sampling Date	Extraction date	Analysis date	Total # of Days	Qualifler
7 9	w/	N	6/01/09	6/11/9	6/17/09	15	5-18/P (h)
							•
					•		
·							

# TECHNICAL HOLDING TIME CRITERIA

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

Both within 14 days of sample collection.

Water: Soils: EXTRACTABLES:

Soil:

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

SDG #: Sec Cong C LDC #: 21494

**VALIDATION FINDINGS WORKSHEET** Continuing Calibration

Page: 1 of Reviewer: 316

2nd Reviewer.

METHOD: \_GC \_ HPLC

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

Did the continuing calibration standards meet the %D / RPD validation criteria of <20.0%? Y NIA

Level JA-Only

Were the retention times for all calibrated compounds within their respective acceptance windows? Y N(N/A)

Date		Detector/ Column		%D / RPD (Limit < 45.0)	%D / RPD (Limit s-45:07 4 20 2 ) RT (limit)	ssocia	1 1	
V	6 for for 010 F 1001	<u>.</u> उ		178.0	( ; )	Ail + Biles	5+ det (6)	
- 1	(IW)			29.0	(		J-/43/P	
- 1		->	4	78,T				
		51.2	6-(4)	. 6.22	( )		3+ drys / p-	
			(-) ±	35.8	(		0- /MJ /P	
		λ.	<del>(-) d</del>	89.0	( )	7	4-7K74	
					( )			
					(			
6/2/09	9 014F1401	CA. )	F (+)	51.2	( )	4W 66+6516'5-1	J+ 4cts/4	
	(35)		(+) K	26.2	(	9160222 MB		
ı	\ \ 	3,7	(+) H	60,09				
1			( <del>(</del> ) \	ه. اد	( )			
			,		(	<b>A</b>	<b>A</b>	
					( )			
7	6/12/69 003 FO301	F	A G	34.2	(	6, 8 9159433 MB	J-145/A	
- 1	(667)		F A)	34.0	(	}	J+ 4115 A	
	,	Ces. 7	A (-)	29.3	( )		D-101 A	
			F (+)	45.1	( )		3+ dets/A	
- 1			BB (-)	21.6		1	J-147/A	
17 61	21 002 F021	<u>_</u> ,	(-) V	29.1		7,9 9167134 MB	J- /NJ / A	
. ]			B (~)	20.2	( )			
		<b>→</b>		42,3			J+ Att 1A	
		د /م	A T	24.7		( George ras)	A 24/-12	_ \
ರ	CONCALNew-gc.wpd					,	* */ Sb * +7	

SDG #: SPE CONTY LDC #: 21 494C 17

## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

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

> GC HPLC METHOD:

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

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

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

Level 14/10 Only
Y N(N/A) Was an LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed?

20 (	
	+
( 98 ) 58 (	39 (
( ) (5(1-	( ) (5(1-09)
( )	( ) ( )
( )	( )
	( )
( )	
( ) (	( ) ( )
75	
	Z > 60
	Z > 60
	Z >- 60

LDC# 21494 C17 SDG# Sre Gary

# VALIDATION FINDINGS WORKSHEET Overall Assessment of Data

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

METHOD: CGC HPLC

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

All available information pertaining to the data were reviewed using professional judgement to compliment the determination of the overall quality of the data.

(Y) N/A

Was the overall quality and usability of the data acceptable?

	Finding	Associated Samples	Qualifications
6 7			(0) XXX

OVRNew.wpd

## LDC Report# 21494D17

## Laboratory Data Consultants, Inc. Data Validation Report

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

Henderson, Nevada

Collection Date: June 8 through June 12, 2009

LDC Report Date: October 22, 2009

Matrix: Water

Parameters: Organophosphorus Pesticides

Validation Level: Stage 2B

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 8304604

## Sample Identification

M-44B

M-44BRE

M-6AB

M-6ABRE

M-142B

## Introduction

This data review covers 5 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.

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

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

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

Sample	Analyte	Total Days From Sample Collection Until Analysis	Required Holding Time (in Days) From Sample Collection Until Analysis	Flag	A or P
M-44BRE	All TCL compounds	11	7	J- (all detects) UJ (all non-detects)	А
M-6ABRE	All TCL compounds	20	7	J- (all detects) R (all non-detects)	А

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.

## 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
6/17/09	003F0301	1	Dichlorvos	29.1	M-44B	J- (all detects)	А
			Mevinphos	20.2	9162333MB	UJ (all non-detects) J- (all detects) UJ (all non-detects)	
6/17/09	003F0301	1	Naled	42.3	M-44B 9162333MB	J+ (all detects)	А

		<del>7</del>		<del></del>		T	
Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
6/17/09	003F0301	2	Dichlorvos	24.7	M-44B 9162333MB	J- (all detects) UJ (all non-detects)	А
6/17/09	003F0301	2	Naled	50.3	M-44B 9162333MB	J+ (all detects)	А
6/24/09	027F2701	1	Fensulfothion	21.7	M-44BRE 9170431MB	J+ (all detects)	А
6/24/09	027F2701	2	Dichlorvos Thionazin Naled Demeton-S Chlorpyrifos Fenthion Tokuthion Parathion-methyl Famphur Azinphos-methyl Coumaphos	24.9 22.7 24.6 23.1 21.3 20.9 21.5 21.2 25.0 25.8 26.1	M-44BRE 9170431MB	J- (all detects) UJ (all non-detects)	A
7/2/09	003F0301	1	Phorate Naled	21.2 25.2	M-6ABRE 9181503MB	J+ (all detects) J+ (all detects)	А
7/2/09	003F0301	2	Dichlorvos Mevinphos Dimethoate Fensulfothion	21.1 45.9 49.7 44.0	M-6ABRE 9181503MB	J- (all detects) UJ (all non-detects)	А
7/2/09	003F0301	2	Naled EPN	28.9 21.2	M-6ABRE 9181503MB	J+ (all detects) J+ (all detects)	А

<sup>\*</sup>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
6/1/09	010F1001	1	Naled	29.0	M-44B 9162333MB	J- (all detects) UJ (all non-detects)	P
6/1/09	01 0F1 001	2	Naled	35.8	M-44B 9162333MB	J- (all detects) UJ (all non-detects)	Р
6/23/09	010F1001	1	Naled	43.3	M-44BRE 9170431MB	J- (all detects) UJ (all non-detects)	Р

Date	Standard	Column	Compound	%D	Associated Samples	Flag	AorP
6/23/09	010F1001	1	Naled Malathion Merphos	43.3 25.8 24.4	M-44BRE 9170431MB	J- (all detects) UJ (all non-detects)	Р
6/26/09	010F1001	1	Naled Disulfoton	40.1 20.6	M-6AB M-142B 9168145MB	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	Р
6/26/09	010F1001	2	Naled Malathion	47.6 23.2	M-6AB M-142B 9168145MB	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	Р

<sup>\*</sup>Added Malathion to table above for 010F1001 Column 2 on 6/26/09

## III. Blanks

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

Sample FB060309-SO (from SDG 8304603) was identified as a field 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 with the following exceptions:

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
9162333MB	Not specified	Triphenylphosphate Chlormefos	45 (60-154) 9.4 (49-171)	All TCL compounds	J- (all detects) R (all non-detects)	Р
M-6AB	Not specified	Triphenylphosphate Chlormefos	22 (60-154) 18 (49-171)	All TCL compounds	J- (all detects) UJ (all non-detects)	А

## 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. Although the LCS percent recovery (%R) was not within QC limits for one compound, the LCSD percent recovery (%R) was within QC limits and no data were qualified.

## V. Target Compound Identification

Raw data were not reviewed for this SDG.

## VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304604	All compounds reported below the PQL.	J (all detects)	Α

Raw data were not reviewed for this SDG.

## VII. System Performance

Raw data were not reviewed for this SDG.

## VIII. Overall Assessment of Data

The overall assessment of data was acceptable. In the case where more than one result was reported for an individual sample, the least technically acceptable results were rejected as follows:

Sample	Compound	Flag	A or P
M-44BRE M-6ABRE	All TCL compounds	×	А

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 8304604

				<del></del>	
SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304604	M-44BRE	All TCL compounds	J- (all detects) UJ (all non-detects)	А	Technical holding times (h)
8304604	M-6ABRE	All TCL compounds	J- (all detects) R (all non-detects)	Α	Technical holding times (h)
8304604	M-44B	Dichlorvos Mevinphos	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	А	Continuing calibration (%D) (c)
8304604	M-44B	Naled	J+ (all detects)	А	Continuing calibration (%D) (c)
8304604	M-44BRE	Fensulfothion	J+ (all detects)	Α	Continuing calibration (%D) (c)
8304604	M-44BRE	Dichlorvos Thionazin Naled Demeton-S Chlorpyrifos Fenthion Tokuthion Parathion-methyl Famphur Azinphos-methyl Coumaphos	J- (all detects) UJ (all non-detects)	Α	Continuing calibration (%D) (c)
8304604	M-6ABRE	Phorate Naled EPN	J+ (all detects) J+ (all detects) J+ (all detects)	A	Continuing calibration (%D) (c)
8304604	M-6ABRE	Dichlorvos Mevinphos Dimethoate Fensulfothion	J- (all detects) UJ (all non-detects)	A	Continuing calibration (%D) (c)
8304604	M-44B	Naled	J- (all detects) UJ (all non-detects)	Р	Continuing calibration (ICV %D) (c)
8304604	M-44BRE	Naled Malathion Merphos	J- (all detects) UJ (all non-detects)	Р	Continuing calibration (ICV %D) (c)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
*8304604	M-6AB M-142B	Naled Disulfoton Malathion	J- (all detects) UJ (all non-detects)	Р	Continuing calibration (ICV %D) (c)
8304604	M-6AB	All TCL compounds	J- (all detects) UJ (all non-detects)	А	Surrogate recovery (%R) (s)
8304604	M-44B M-44BRE M-6AB M-6ABRE M-142B	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (PQL) (sp)
8304604	M-44BRE M-6ABRE	All TCL compounds	Х	A	Overall assessment of data (0)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Laboratory Blank Data Qualification Summary - SDG 8304604

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304604

No Sample Data Qualified in this SDG

## **Tronox Northgate Henderson**

SDG	#: 21494D17 #: 8304604 ratory: <u>Test America</u>	_ VA	LIDATIO		PLETENE Stage 2B	SS WORKSH	EET	Date:9/o/ Page:1of_ Reviewer:2V/ 2nd Reviewer:4
MET	HOD: GC Organophosph	norus	Pesticides (	EPA SW	846 Method	l 8141A)		Zila Neviewei.
	samples listed below were ation findings worksheets		ewed for ea	ch of the f	ollowing va	lidation areas. Va	lidation fin	idings are noted in attache
	Validation	Area				с	omments	\
1.	Technical holding times			sw)	Sampling da	tes: 6/08-13	109	
ila	Initial calibration			A	70 RS	D € 20 2 r	~	
IIb.	Calibration verification/ICV			SW		NOW 420 %		
111.	Blanks			A				
IVa	. Surrogate recovery			SW				
IVb	. Matrix spike/Matrix spike du	plicate	s	N	Che	nt spec	(	insufficient un.)
IVc	Laboratory control samples			SW	us	nt spec		
V.	Target compound identification	tion		N				
VI.	Compound Quantitation and	CRQ	Ls	N				
VII.	System Performance			N				
VIII	. Overall assessment of data			SW				
IX.	Field duplicates			N				
Lx.	Field blanks			ND	FB=	FB060309	from	8304603
Note:	N = Not provided/applicable SW = See worksheet	•	R = Rins	o compound: sate eld blank	s detected	D ≖ Duplicate TB = Trip blank EB = Equipmen		
	Water	г			<del></del>		<del></del>	
1	M-44B	17 +	916233		21	<del></del>	31	
2 7	M-44BRE	12 >	917043		22	<del>-</del>	32	
3 3	M-6AB	13 3	916814		23		33	
4 4	M-6ABRE	14 4		0> MB	24	·	34	
5 3	M-142B	15	ક્ય .		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 WORKSHEET-

METHOD: / GC HPLC

8310	8330	8151	(8141	8141(Con't)	R024B
A. Acenaphthene	A. HMX	4 2.4-D			7.100
R Acessarking			A. Dichlorves	V. Fensutfothlon	V. Benzene
	B. RDX	B. 2,4-DB	B. Mevinphos	W. Bolster	Cr Tolinan
C. Antillacene	C. 1,3,5-Trinitrobenzene	C, 2,4,5-T	C. Demeton-O	X. EPN	FF Ethyl Densen
D. Benzo(a)anthracene	D. 1,3-Dinitrobenzene	D. 2,4,5-TP	D. Demeton-S	V Asimphot mother	בי ביולו הפוולפוים
E. Benzo(a)pyrene	E. Tetryl	E. Dinosab		. Attipuos-matuyi	SSS. O-Xylene
F. Benzolb/fluoranthana			E. Ethoprop	Z. Coumaphos	RRR. MP-Xylene
	r. Nicobenzene	F. Dichlorprop	F. Naled	AA. Parathion	GG. Total Xylene
G. Denzo(g.n.t)perylene	G. 2.4.6-Trinitrotokuene	G. Dicamba	G. Sulfotep	BB. Trichloronate	
H. Benzo(k)fluoranthene	H. 4-Amino-2,6-dinitrotoluene	H. Dalapon	H. Phorate	CC. Trichlorinate	
I. Chrysane	l. 2-Amino-4,8-dinitrotoluene	I. MCPP	1. Dimethoate	DD. Teffuratin	
J. Dibenz(a,h)anthracene	J. 2,4-Dinitrotolune	J. MCPA	J. Diezinon	EE. Def	
K. Fittoranthene	.K. 2,6-Dinitrotoluene	K. Pentachlorophenoi	K. Disulfoton	n Drysel	
L. Fluorene	L. 2-Nitrotoluene	L. 2,4,5-TP (silvex)	L. Parathion-methyl	- 141	
M. Indeno(1,2,3-cd)pyrene	M. 3-Nitrotoluene	M. Silvex	N Bonnes	oo. reilioi	
N. Naphthalene	N. 4-Nitrotoluene			nn. ieurchiorvinphos	
O. Phenanthrana			M. Malathion	II. Sulprofos	
4	Š		O. Chlorpyrifos	JJ. Thionezin	
r. ryrane	å		P. Fenthion	kk Phoemet	
Ċ	G	1.4.4. 1.4.4. 1.4.4. 1.4.4.	Q. Parathion-ethyi	ŀ,	1. 41.
œ.			R. Trichloronate	NA TOTAL	Sprioro inio Are
S.			S. Merphos		
			T. Strofos		00
			U. Tokuthion	hill us will stown as	1411111- 00

.

Notes:

cmpd\_fist.wpd

SDG#: See Con-LDC # 21 494 D17

## VALIDATION FINDINGS WORKSHEET **Technical Holding Times**

Page: 1 of 1 Reviewer 2nd Reviewer:

All circled dates have exceeded the technical holding times.

	METHOD:	METHOD: ZGC HPLC	<b>7</b> C		• • • • • • • • • • • • • • • • • • • •			
<u></u>	Sample ID	Matrix	Praserved	Sampling Date	Extraction data	Analysis data	Total	
<u> </u>	2 2	h,	, N	6/80/2	6/9/09	1 / Je / 69	l Clair # Cl Days	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
		-						
	4	<b>→</b>	<b>~</b>	69/01/9	70/05/2	7/02/09	22	T- 10 /+
								N/W/->
1								
1								

Aromatic within 7 days, non-aromatic within 14 days of sample collection. Both within 14 days of sample collection. Both within 14 days of sample collection. TECHNICAL HOLDING TIME CRITERIA VOLATILES: Water unpreserved: A Water unpreserved: Water preserved:

Soils:

EXTRACTABLES: Water: Soil:

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

HTNew.wpd

LDC # 21444 DI7 SDG #: Sec Com

VALIDATION FINDINGS WORKSHEET Continuing Calibration

Page: 1 of 3 Reviewer: JVC

2nd Reviewer:

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? 

XN N/A

Were continuing calibration standards analyzed at the required frequencies?

Y N/A N/A

Did the continuing calibration standards meet the %D/RPD validation criteria of <a href="#chargo: 20.0%">20.0%</a>?

Y N N/A Level JK-Only

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

		one one	4 0						1			A.	2	T	T	4	۵,	d		T	T			4			1	\ \	A A	_
		Qualifications	It dets 1p	/ CM /-	1		1 / 5/10 20	3-147 G	V=/K/P			5+ 401×10	7-7-1	7 4 4 4	V K	4-12-12-12	J-1400	4	2 4	2-143/P				J-/NJ/A		1 1	A T ACTS AS	A/5/1/2	J+ dcts /A	
		-	17	,			7	7	?										-	+	+	+	+	-				+	+	
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	(Ilmit)																													
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440,0	(Limit < 45:07 (4 20 2) RT (limit)			29.0	787	223.4	35.2	5 9	<u> </u>			74.8	43.3	7:58	244	1	12.3	99.6	25, 8	24.4			29.		20.2	42,3	7.7	50.3	-	
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CONCALNew-go-wpd

(1CV - AVE 720)

4 500

LDC# 21494 DIT

METHOD: GC HPLC

VALIDATION FINDINGS WORKSHEET Continuing Calibration

Page: 2 of 2 Reviewer: 3/6

2nd Reviewer:

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

Were continuing calibration standards analyzed at the required frequencies?

YN MA

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

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

L								
*	Date	Standard ID	Column	Compound	(Limit < 18:01420 2) RT (Ilmit)	2) RT (Imit)	Accordated Semulae	91
	6 24 69	027 F2701	CH. 1	(±) /	21.7		2 4174431 A.D	Qualifications (1)
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				F (-)	24.6			
				D (-)	23.1			
				6-) 0	21.3			
				P (-)	20.9			
				W C-)	21.5			
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				CH DAM	25.0			
				λ (-)	25.8			
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		<del>( (01</del> )			1			

LDC#: 21494 DI7 SDG #: 12 C2 METHOD: \_GC\_HPLC

## VALIDATION FINDINGS WORKSHEET Continuing Calibration

Page: 3 of 3 Reviewer. 3V6

2nd Reviewer:

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? — "N D O" — RPD

Were continuing calibration standards analyzed at the required frequencies?

Y N N/A

Did the continuing calibration standards meet the "ND / RPD validation criteria of <-15.0%?

V NIA V NIA Level IV Only Y N (N/A

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

	િ	F	F	F	F	F	F	1		Ī	T	Τ	Τ	T	T	T	Τ	Γ	Π	T	T	T	Ī	T
Qualifications	St det /A		4/ Tw/ √b		J+ 40te 14	7- /AT /A	1	3 + Arts/A																
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%D / RPD (Limit < 15.0)	21.7	25.7	71.15	45.9	28.9	44.7	44.0	7.12																
Compound	(+) H		(-) ¥		(+) <del> </del>	(-) I	(-) V	C+) X																
Detector/ Column	- 3		Z.X																					
Standard ID	0035030												(											
Date	7/62/69																							
**			_														ŀ		-					

See Gora LDC# 2/494 DIT SDG #:

# VALIDATION FINDINDS WORKSHEET

Surrogate Recovery

Reviewer

Page: 1 of

	Are surrogates required by the method? Yes Please see qualifications below for all question	the met	thod?	Yes Yes	or No inswered "N". Not	applic	Are sumogates required by the method? Yes or No	M, se pe	/ <b>A</b> .				
(eas													
<b>43</b>	N.M.A Were surro	gates si ogate re	piked	Into all sa tes (%R)	Were surrogates spiked into all samples and blanks? Did all surrogate recoveries (%R) meet the QC limits?	s? Its?							
*	Semple (I)		Column Column	<b>₽</b> \₫	Surrogate Compound		%R (Umits)	()			Quelli	Qualifications	
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8	4-Bromofluorobenzene (BFB)	FB)	I	ठ	Ortho-Terphenyl	z	Terphenyl-014	Ţ	3,4-Dinit		Z	Chlormetos	
٥	e.e.e-Triffuorotoluene	1	+	Fluore	Fluorobenzene (FBZ)	٥	Decachigrobiphenyl (DCB)	ס	Tripe	Tripentyllin			ļ
٩	Bromochlorobenene	+	1	Ä	n-Triecontene	٩	1-methylnaphthalene	>	In-0-5	Tri-n-propyttin			
	1,4-Dichlorobutane		×	Í	Hexacosane	٥	Dichlorophenyl Acetic Acid (DCAA)		Tributy	Tributy Phosphate			
F	1.4-Diffuorobenzene (DFB)	181	_	200	Bromobeazene	α	4-Nitrophenol	×	Trinhand	Trinhand Phosphate			

100 # 24 (pr)

# VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Reviewer: 306 2nd Reviewer: 200

METHOD: \_\_GC\_\_ HPLC

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

Were a laboratory control samples (LCS) and laboratory control sample duplicate (LCSD) analyzed for each matrix in this SDG? Were the LCS percent recoveries (%R) and relative percent differences (RPD) within the QC limits?

Level WID Only
Y N/N/A

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

7	Crapy		T	7	T	T	T	T	7	Т	Ŧ	_		<del>-</del>	-		7	<u> </u>	_	T .	<u> </u>	<del>-</del> -	T	T	7
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	P 58 (22-100) APD LIMITS Associated Samples	(3) P 58 (63-128) (1) (1) 1, 9162333 MB No 51.28	P 58 (43-/28) ( )	P 58 (63-128) ( ) ( ) 1 916.2339 MB No give ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) (	P 58 (63-728) ( ) 1,9162333/MB No guell ( ) ( ) ( ) 1,9162333/MB No guell ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) (	P 58 (63-128) ( ) 1 9162333 (Associated Samples Qualifications ( ) 1 9162333 (Associated Samples Associated Samples ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) (	P 58 (63-728) ( ) 1 9162393 Qualifications ( ) 1 9162393 MB No grade ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) (	P 58 (63-128) ( ) 1, 4162395 MB No grade (1) ( ) ( ) 1, 4162395 MB No grade (1) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( )	P 58 (63-128) ( ) 1, 9162399 MB No grade (1) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( )	P 58 (63-728) ( ) 1, 4162339 MB No state	P 58 (63-128) ( ) 1,9162393 Qualifications ( ) 1,9162393 MB No guard ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) (	P 58 (63-128)	P 58 (63-128) ( ) 1, 9162-395/MB No guze	P         58         (φ3-128)         ( )<	P 58 (69-128)	P 58 (49-)28) ( ) 1 9162-29-MB No gueral and a supplemental and a supp	P 58 (\$2-128) ( ) 1 4162-393 Mp No gued. ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( )	P         58         (φ3-12g)         ( )<	P 58 (\$9-128)	P 58 (63-128) (7 ) ( ) 1, 9 (62-25) MB Nu guest.  (	P         58         (φ3-12δ)         ( )<	P         5.8         (φ3-/32)         ( )	P 58 (43-128) ( ) 1 416.2.3.5.5 MB  Ns 91C.  ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) (	P         5.8         (φ2-128)         ( )	P 58 (63-128) ( ) 1 ( ) 1 (16.2-29-) MB No splitted families Qualitations ( ) 1 (16.2-29-) MB No splitted ( ) 1 (

LDC# 21494 by SDG# Su Cuny

# VALIDATION FINDINGS WORKSHEET Overall Assessment of Data

Reviewer: 3/2 2nd Reviewer: 2

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

All available information pertaining to the data were reviewed using professional judgement to compliment the determination of the overall quality of the data.

Was the overall quality and usability of the data acceptable?

Finding	Outside H.T.					
Sand le 1D Compound Name	2,4					Commenter

OVRNew.wpd

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

June 10 through June 11, 2009

LDC Report Date:

September 18, 2009

Matrix:

Soil

Parameters:

Organophosphorus Pesticides

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304605

Sample Identification

SA35-0.5B

SA176-0.5B

SA166-0.5B

SA182-0.5B

### 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 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.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- 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 (r²) was greater than or equal to 0.990.

### 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
6/19/09	051F5101	1	Dichlorvos Azinphos-methyl	24.2 30.9	All samples in SDG 8304605	J+ (all detects) J+ (all detects)	A
6/19/09	051F5101	1	Naled Disulfoton	38.2 26.8	All samples in SDG 8304605	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	А
6/19/09	051F5101	2	Naled	49.0	All samples in SDG 8304605	J- (all detects) UJ (all non-detects)	A
6/19/09	051F5101	2	Azinphos-methyl	35.3	All samples in SDG 8304605	J+ (all detects)	А

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
6/1/09	010F1001	1	Naled	29.0	All samples in SDG 8304605	J- (all detects) UJ (all non-detects)	Р
6/1/09	010F1001	2	Naled	35.8	All samples in SDG 8304605	J- (all detects) UJ (all non-detects)	Р

### III. Blanks

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

Sample FB072109-SO (from SDG 8304616) was identified as a field 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

Raw data were not reviewed for this SDG.

## VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304605	All compounds reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

## VII. System Performance

Raw data were not reviewed for this SDG.

## VIII. Overall Assessment of Data

Data flags 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 8304605

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304605	SA35-0.5B SA176-0.5B SA166-0.5B SA182-0.5B	Dichlorvos Azinphos-methyl	J+ (all detects) J+ (all detects)	А	Continuing calibration (%D) (c)
8304605	SA35-0.5B SA176-0.5B SA166-0.5B SA182-0.5B	Naled Disulfoton	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	A	Continuing calibration (%D) (c)
8304605	SA35-0.5B SA176-0.5B SA166-0.5B SA182-0.5B	Naled	J- (all detects) UJ (all non-detects)	Р	Continuing calibration (ICV %D) (c)
8304605	SA35-0.5B SA176-0.5B SA166-0.5B SA182-0.5B	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (PQL) (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Laboratory Blank Data Qualification Summary - SDG 8304605

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304605

No Sample Data Qualified in this SDG

## Tronox Northgate Henderson

	:21494E17	VA	LIDATIO			ESS WORK	SHEET	Date: 9/11/09
SDG #				S	Stage 2E	3		Page: 1 of 1
_abor	atory: Test America							Reviewer: 3/6 2nd Reviewer:
METH	IOD: GC Organophosph	orus P	esticides (E	EPA SW 84	46 Metho	d 81 <b>4</b> 1A)		
The s	amples listed helow were	revie	wed for eac	ch of the fo	ollowina v	alidation areas	Validation findi	ngs are noted in attached
	tion findings worksheets.	10010	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	511 01 410 10	morning v	andation arous.	vanadion in a	mgo aro notoa m attaonoa
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	Validation	Area	····				Comments	
I.	Technical holding times			A	Sampling of	dates: 6/10-11/	69	
lla.	Initial calibration			A	1	1SD = 202		
IIb.	Calibration verification/ICV			SN		cov/100 =:		
III.	Blanks			A				
IVa.	Surrogate recovery			Ä				
IVb.	Matrix spike/Matrix spike dup	licates		Ą	RSA	M2-0,5B	(from 830	4607)
IVc.	Laboratory control samples			A		(S	· · · · · · · · · · · · · · · · · · ·	
V.	Target compound identification	on		N-				
VI.	Compound Quantitation and			N				
VII.	System Performance			N				
VIII.	Overall assessment of data			A				
IX.	Field duplicates			N	<u> </u>	***************************************	_	
X.	Field blanks			ND	FB	= FB07210	9 - So (fro	n 8304616)
Vote:	A = Acceptable		ND = No	o compounds	·····		· · · · · · · · · · · · · · · · · · ·	
	N = Not provided/applicable SW = See worksheet		R = Rins			TB = Trip I	olank oment blank	
/alidata	ed Samples:			ora biarrit		22 2441		
alluate	Soi)							
1	SA35-0.5B	11			21		31	
2	SA176-0.5B	12			22		32	
2 1 3	SA166-0.5B	13			23		33	
_	SA182-0.5B	14			24		34	
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# VALIDATION FINDINGS WORKSHEET

METHOD: /GC HPLC

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A. Acenaphthene	А. НМХ	A. 2,4-D	A. Dichlorvos	V. Fensulfothion	V. Benzene
B. Acenaphthylene	B. RDX	B. 2,4-DB	B. Mavinohoe	W Delete	
C. Anthracene	C. 1,3,5-Trinitrobenzene	C. 2,4,5-T	C. Demeton-O	W. Doistar X FPN	CC. Toluene
D. Benzo(a)anthracene	D. 1,3-Dinitrobenzene	D. 2,4,5-TP	D. Demeton-S	Y. Azinnhos-mathyd	•
E. Benzo(a)pyrene	E. Tetryi	E. Dinoseb	E. Ethoprop	Z. Coumanhos	BRD MD Veloci
F. Benzo(b)fluoranthene	F. Nitrobenzene	F. Dichlorprop	F. Naled	AA. Parathion	GG. Total Xviene
G. Benzo(g,h,l)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	l. 2-Amino-4,6-dinitrotoluene	I. MCPP	1. 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. Chlorpyrlfos	JJ. Thionexin	
P. Pyrene	a.		P. Fenthion	1	
Ö.	ø		Q. Parathion-ethyl	0	or home this sto
ж.			R. Trichloronate	1	217011110114
S,			S. Merphos	1	NO.
			T. Stirofos		m - m++hu/
	·		U. Tokuthion		

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LDC#: 21494 E17 SDG #: Sec Cont

## VALIDATION FINDINGS WORKSHEET Continuing Calibration

Reviewer: 3VC Page: 1 of

2nd Reviewer:

METHOD: \_\_GC\_\_ HPLC

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

Did the continuing calibration standards meet the %D / RPD validation criteria of <20.0%? X M NA

Y(N/A)

Level JV-Only

Were the retention times for all calibrated compounds within their respective acceptance windows? Y N(N/A

								Γ	T										Г	Γ	Π		Г	ī
Qualifications	1+ dets 1p-1c	1-7 43/P	3-7K7p	3+ dkts/p	J-/WJ/P	3-7K7P-				J+ dets 1A	J-/NJ/A	J- MJ/A	5+ dets.A	J-/NJ/A	5+0145/A	•								
Associated Samples	A11 + 13/1K																							
RT (limit)	( )	( )	( )	( )	( )	( )	( )	·	) (	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	
%D/RPD (Limit ≤ 45:0) A 20 2) RT (limit)	178.6-	29.0	78.1	223.4	35.8	-89.0				24.2	38,2	26.8	30.9	0.64	35.03									
Compound	+	(-) F	(1) (4) (1)	(+) 7	(-) <del> </del>	(-) d				(+) A	F (う	(-) X	7 (+)	F (C)	(+) K									
Detector/ Column	[ CH. 1	1	<b>→</b>	Col. 2		<u>ر</u> ا				1.95				Ca1.2										
Standard ID	010 F1001	(M)								051 F5101	(CCV)													
# Date	19/19/9									6/9/01														
	1			l	L					L									L_				<u> </u>	<u> </u>

(1W - AVE 720)

#### LDC Report# 21494F17

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

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

Henderson, Nevada

Collection Date: June 16 through July 19, 2009

LDC Report Date: October 22, 2009

Matrix: Soil

Parameters: Organophosphorus Pesticides

Validation Level: Stage 2B

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 8304606

#### Sample Identification

SA85-0.5B

SA92-0.5B

SA86-0.5B

SA86-0.5BRE

SA129-0.5B

SA129-0.5BRE

SA85-0.5BMS

SA85-0.5BMSD

SA86-0.5BMS

SA86-0.5BMSD

#### Introduction

This data review covers 10 soil 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.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

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

Sample	Analyte	Total Days From Sample Collection Until Analysis	Required Holding Time (in Days) From Sample Collection Until Analysis	Flag	A or P
SA86-0.5BRE	All TCL compounds	20	14	J- (all detects) UJ (all non-detects)	Α
SA129-0.5BRE	All TCL compounds	19	14	J- (all detects) UJ (all non-detects)	А

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 .

#### \*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
7/3/09	019F1901	1	Mevinphos Fensulfothion Azinphos-methyl	22.3 22.9 25.9	SA86-0.5B SA129-0.5B SA86-0.5BMS SA86-0.5BMSD 9175172MB	J- (all detects) UJ (all non-detects)	А

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
7/3/09	019F1901	1	Merphos	27.1	SA86-0.5B SA129-0.5B SA86-0.5BMS SA86-0.5BMSD 9175172MB	J+ (all detects)	А
7/3/09	019F1901	2	Mevinphos Dimethoate Fensulfothion Azinphos-methyl Coumaphos	55.8 57.8 59.4 23.1 20.8	SA86-0.5B SA129-0.5B SA86-0.5BMS SA86-0.5BMSD 9175172MB	J- (all detects) UJ (all non-detects)	А
7/3/09	019F1901	2	Naled	25.6	SA86-0.5B SA86-0.5BRE SA129-0.5B SA86-0.5BMS SA86-0.5BMSD 9175172MB	J+ (all detects)	А
7/13/09	003F0301	1	Naled	33.1	SA86-0.5BRE SA129-0.5BRE 9189494MB	J+ (all detects)	А
7/13/09	003F0301	2	Naied	24.7	SA86-0.5BRE SA129-0.5BRE 9189494MB	J+ (all detects)	А

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
6/23/09	010F1001	1	Naled	43.3	SA85-0.5B SA92-0.5B SA85-0.5BMS SA85-0.5BMSD 9170419MB	J- (all detects) UJ (all non-detects)	P
6/23/09	010F1001	1	Naled Malathion Merphos	43.3 25.8 24.4	SA85-0.5B SA92-0.5B SA85-0.5BMS SA85-0.5BMSD 9170419MB	J- (all detects) UJ (all non-detects)	Р

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
6/26/09	010F1001	1	Naled Disulfoton	40.1 20.6	SA86-0.5B SA86-0.5BRE SA129-0.5B SA129-0.5BRE SA86-0.5BMS SA86-0.5BMSD 9175172MB 9189494MB	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	Р
6/26/09	010F1001	2	Naled Malathion	47.6 23.2	SA86-0.5B SA86-0.5BRE SA129-0.5B SA129-0.5BRE SA86-0.5BMS SA86-0.5BMSD 9175172MB 9189494MB	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	Р

<sup>\*</sup>Added Malathion to table above for 010F1001 Column 2 on 6/26/09

#### III. Blanks

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

Sample FB072109-SO (from SDG 8304616) was identified as a field 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 with the following exceptions:

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
SA129-0.5B	Not specified	Triphenylphosphate	28 (47-161)	All TCL compounds	J- (all detects) UJ (all non-detects)	А

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

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

Spike ID (Associated Samples)	Compound	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
SA86-0.5BMS/MSD (SA86-0.5B)	Dimethoate	0 (10-156)	0 (10-156)	<u>-</u>	J- (all detects) R (all non-detects)	А

#### 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 with the following exceptions:

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Flag	A or P
9175172LCS (SA86-0.5B SA129-0.5B 9175172MB)	Dimethoate	0 (10-156)	-	-	J- (all detects) R (all non-detects)	Р

#### V. Target Compound Identification

Raw data were not reviewed for this SDG.

#### VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304606	All compounds reported below the PQL.	J (all detects)	Α

Raw data were not reviewed for this SDG.

#### VII. System Performance

Raw data were not reviewed for this SDG.

#### VIII. Overall Assessment of Data

The overall assessment of data was acceptable. In the case where more than one result was reported for an individual sample, the least technically acceptable results were rejected as follows:

Sample	Compound	Flag	A or P
SA86-0.5B SA129-0.5B	Dimethoate	×	A
SA86-0.5BRE SA129-0.5BRE	All TCL compounds except Dimethoate	х	A

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 8304606

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304606	SA86-0.5BRE SA129-0.5BRE	All TCL compounds	J- (all detects) UJ (all non-detects)	А	Technical holding times (h)
8304606	SA86-0.5B SA129-0.5B	Merphos	J+ (all detects)	А	Continuing calibration (%D) (c)
8304606	SA86-0.5B SA129-0.5B	Mevinphos Dimethoate Fensulfothion Azinphos-methyl Coumaphos	J- (all detects) UJ (all non-detects)	А	Continuing calibration (%D) (c)
8304606	SA86-0.5B SA129-0.5B SA86-0.5BRE SA129-0.5BRE	Naled	J+ (all detects)	A	Continuing calibration (%D) (c)
8304606	SA85-0.5B SA92-0.5B	Naled Malathion Merphos	J- (all detects) UJ (all non-detects)	Р	Continuing calibration (ICV %D) (c)
*8304606	SA86-0.5B SA86-0.5BRE SA129-0.5B SA129-0.5BRE	Naled Disulfoton Malathion	J- (all detects) UJ (all non-detects)	Р	Continuing calibration (ICV %D) (c)
8304606	SA129-0.5B	All TCL compounds	J- (all detects) UJ (all non-detects)	А	Surrogate recovery (%R) (s)
8304606	SA86-0.5B	Dimethoate	J- (all detects) R (all non-detects)	А	Matrix spike/Matrix spike duplicates (%R) (m)
8304606	SA86-0.5B SA129-0.5B	Dimethoate	J- (all detects) R (all non-detects)	Р	Laboratory control samples (%R) (I)
8304606	SA85-0.5B SA92-0.5B SA86-0.5B SA86-0.5BRE SA129-0.5B SA129-0.5BRE	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (PQL) (sp)
8304606	SA86-0.5B SA129-0.5B	Dimethoate	х	А	Overall assessment of data (0)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304606	SA86-0.5BRE SA129-0.5BRE	All TCL compounds except Dimethoate	Х	А	Overall assessment of data (0)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Laboratory Blank Data Qualification Summary - SDG 8304606

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304606

No Sample Data Qualified in this SDG

#### Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEFT

SDG	#: 21494F17 #: 8304606 ratory: Test America	_ <b>V</b>			tage 2B	SS WORKSH	iee i	Page: 10f Reviewer: JV
	HOD: GC Organophosph	orus	Pesticides (	(EPA SW 8	346 Method	8141A)		2nd Reviewer:
The s	samples listed below wer ation findings worksheets	e revi	ewed for ea	ich of the f	ollowing vali	dation areas. Va	alidation find	ngs are noted in attach
	Validation	Area	<u> </u>				Comments	
1.	Technical holding times			SW	Sampling date	es: 6/6 -19	109	
Ila.	Initial calibration			A_	2 4	csp = 202	rv	
IIb.	Calibration verification/ICV			SW	cc	W/W £ 20	Σ	
111.	Blanks			A	<u> </u>	<del></del>		
IVa	Surrogate recovery			SW				
IVb.	Matrix spike/Matrix spike du	plicate	×	SW		****		
IVc.	Laboratory control samples			SN	us/	<u> &gt;</u>		
V.	Target compound identifica	tion		N				
V1.	Compound Quantitation and	CRQ	Ls	N		<del></del>		
VII.	System Performance			N				
VIII.	Overall assessment of data		<del></del>	SH				
IX.	Field duplicates			N.				
X.	Field blanks			ND	FB =	FB072109	-so from	n 8304616
Note:	A = Acceptable N = Not provided/applicable SW = See worksheet	•	R = Rins	o compounds sate eld blank	detected	D = Duplicate TB = Trip blanl EB = Equipme	k nt blank	11
Validat	Son Samples:			,				
1 1	SA85-0.5B	11	917041	9 MB	21		31	
2 1	SA92-0.5B	12	91751	72 MB	22		32	
3 7	SA86-0.5B	13 3	91894	94 MB	23		33	
4 3	SA86-0.5BRE	14		***	24		34	
1 -1 5 +	SA129-0.5B	15		······································	25		35	
	SA129-0.5BRE	16			26		36	
7	SA85-0.5BMS	17	······································		27		37	
8 1	SA85-0.5BMSD	18			28		38	
9 7	SA86-0.5BMS	19			29		39	
10	SA86-0.5BMSD	20			30		40	
Notes	:							

# VALIDATION FINDINGS WORKSHEET

METHOD: /GC HPLC

8310	8330	7470			
		6151	(8141	8141(con.1)	8024B
A. Acenaphthene	A. HMX	A. 2,4-D	A Distilian		01400
B. Acenaphthylene	B RDX		A. Dienoryon	V. Fensulfothlon	V. Benzene
C. Anthracene		B, 2,4-DB	B. Mevinphas	W. Boistar	CO Tollings
	C. 1,3,3-1 finitrobenzene	C. 2,4,5-T	C. Demeton-O	X. EPN	
V. Benzo(a)anthracene	D. 1,3-Dinitrobenzene	D. 2,4,5-TP	O Demetron 6		LL: Culyi Dunzene
E. Benzo(a)pyrene	E. Tetryl	E. Dinosah		Y. Azinphos-methyl	SSS. O-Xylene
F. Benzo(b)fluoranthene	F. Nitrobenzene	T. Pickie	E. Elhoprop	Z. Coumaphos	RRR. MP-Xylene
G. Benzo(a.h.l)perviene		r. oraniorprop	F. Naied	AA. Parathion	GG. Total Xylene
	G. K. J. G. I MILLOGOIGENE	G. Dicemba	G. Sulfotep	BB. Trichloronate	
n. Benzo(k)fluoranthene	H. 4-Amino-2, 8-dinitrotoluene	H. Dalapon	H. Phorate	C. Trophodost	
I. Chrysene	l. 2-Amino-4,8-dinitrotojuene	I. MCPP	1. Dimethoate	Co. Heading	
J. Dibenz(a,h)anthracene	J. 2,4-Dinitrotolune	J. MCPA		טט. ומומישווה	
K. Fluoranthene	K. 2.8-Dinitrateduses		J. Diszinon	EE. Def	
H (1000)		r. Pentachlorophenol	K. Disulfoton	FF. Prowl	
	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	14 O		
N. Naphthalene	Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z		m. Konnet	HH. Tetrachlorvinphos	
	wwirdelbene		N. Matathion	II. Suiprofes	
O. Phenanthrene	o.		O. Chlorovrifos		
P. Pyrene	Δ.		B Earthfor		
ď	0	***	r. renamion	kk, Phosmet	
ď			G. Parathlon-ethyl	LL. 0.00-Triethylphosphorothigate	sphoro this att
ú			R. Trichloronate	MM. Famphur	
			S. Merphos	ı	
			T. Stirofos		N.
			11 T-1(Edu-	00. Carbophenethen - methyl	m - methyl
			U. lokumion		

cmpd\_list.wpd

LDC#: 21 494 F17 SDG #: 54 Chr All circled dates have exceeded the technical holding times.

## VALIDATION FINDINGS WORKSHEET Technical Holding Times

Page: | of ) Reviewer: 3VC 2nd Reviewer:

Qualifier 5-145 Total # of Days 90 1 Analysis date 7/12/01 7 15/69 Extraction date 7 68/09 Sampiling Date 20/27 Preserved HPLC Matrix METHOD: GC Ś Semple ID

# TECHNICAL HOLDING TIME CRITERIA VOLATILES: Water unpreserved: A

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

Both within 14 days of sample collection.

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

EXTRACTABLES:
Water:
Soil:

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

HTNew.wpd

LDC#: 21494 F17

METHOD: GC HPLC

# VALIDATION FINDINGS WORKSHEET

Continuing Calibration

2nd Reviewer.

Page: of X Reviewer: 376

Were the retention times for all calibrated compounds within their respective acceptance windows? Level W.Only Y. N. N.A.

Standard ID Column OTO F.1 00 1 (1 (2)	/				_		
		Compound	(Umit = 45.0 (420 2)	RT (limit)	Associa	Associated Samples	Qualifications
\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\		C (+)	174.8	(	12.78	1,70419 MA	throp
->		(O) #	43.3	(		_	5-14-16
			85.7				To be to
7,3	۲,	6 0	244.0				2 7 87
		F(-)	43.3				7 - 7
			1	-			4 CM - 1
		(-)N	25.8				7 11 4
		(-)8	24.4				7/70/-
							A
010 F1001 CA.1		1	187.8		27/ 0	4.1.	7
- 3-		I	46		2 2 2	500 5 10 717 5173 MB	- STATE A
		4	73		1187464 MB	4 W P	2-/M/P
-		K	20.6				
412	_		4377				J-/WJ/P
	ہد	ij	47 (				****
		[Î   	65.5				J-/W/P
		5	23.2				
							2 -(M) /P
019F1901 CM.		B (-)	22.3		3.5.9 10	9175173 MB	1- 1/4T A
(S)	_	(-) A	22.9	· ·	ı	<u> </u>	4
		(-) X	25.9	)	(3,5)		1
		(±)	27.)	(			J+ det /4
			-				

LDC # 21494 FI7 SDG #: 54 Cm METHOD: <a href="CGC">CGC</a> HPLC

## **VALIDATION FINDINGS WORKSHEET** Continuing Calibration

Page: Yof Y Reviewer: JVC

2nd Reviewer:

Level IV Only
Y N (V/A)

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

	(0	F	Ŧ	F	Ŧ	Ŧ	F	F	T	Ť	¥	T	T	T	T	T	T	T	T	T	T	T	T
Qualifications	J-/M-/	Track A	J-64 7	200			7		7 1. F	77.00.4	7												
Associated Samples	3,5,9 10 7175172 MB								4 1 9189404 4.0	Ł													
RT (limit)	( )	(		-	,	,						_											
%0 / RPD (Limit < 150)	55.8	25,6	57.8	59.4	23.	20.8			73 -	7.7													
	J	用子	I C	\(\int\)	(-) k	(c) /z			(+) <u>+</u>	FG													
Detector/	7					_\			3	Carl. Y													
Standard ID	010 = 1901	( 200 )							003F0301	(60)	\ \											-	
Date	1/63/62							+	4/13/64														
**					- [			١						- 1	-[				-	- 1			

CONCALNew-gc.wpd

LDC#: 21494 F17 SDG #: Ca low

# **VALIDATION FINDINDS WORKSHEET**

Surrogate Recovery

Page: 1of 1 Reviewer: 5VZ 2nd Reviewer:

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

\*\*Note to the surrogate spiked into all samples and blanks?

\*\*Note to the surrogate recoveries (%R) meet the QC limits?

) *	Sample O	Detector	ritori ma	Surrogate Compound		%R (⊔mits)				Qualifications	
	5	Not Skuifiy	14.6.7	×		78	47 -16	-161	15	(N) A/ (N)	
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	Surrogete Compound		Surrogs	Surrogate Compound		Surrogate Compound		Surrogate	Surrogate Compound		
∢	Chlorobenzene (CBZ)	g	ŏ	Octacosane	2	Benzo(e)Pyrene	s	1-Chiaro-3	1-Chloro-3-Nitrobenzane Y	Tetrachloro-m-xylene	
æ	4-Bromofluorobenzene (BFB)	B) H	ð	Ortho-Terphany/	z	Terphenyl-D14	1	3,4-Dinit	3,4-Dinitrotoluene		
٥	a,a,a-Triffuorotoluene	-	Fluorol	Fluorobenzene (FBZ)	0	Decachlorobipheny (DCB)	n	Tripe	Tripentyttin		
٥	Bromochlorobenene		[-a	n-Triacontane	۵	1-methylnaphthalene	^	Tri-n-E	Tri-n-propyttin		
ш	1,4-Dichlorobutane	¥	Ĩ	Hexacosane	0	Dichlorophenyl Acetic Acid (DCAA)	≥	Tributy	Tributyl Phosphate		
ч	1.4-Diffuorobenzene (DFB)	1	Broi	Bromobenzens	ж	4-Nitrophenoi	×	Tripheny	Triphenyl Phosphate		

LDC#: 21494 F17 SDG# 54 Cony

## VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Reviewer: MZ 2nd Reviewer:

Page: 1 of 1

Was an MS/MSD analyzed every 20 samples for each matrix or whenever a sample extraction was performed? Were the MS/MSD percent recoveries (%R) and relative percent differences (RPD) within QC limits?

(177) Qualification J-181A No True No soul N Garal Associated Samples か RPD (Limits) ž لم ق (51-119) 1261-91 (511-25) (25-KZ) (६४८३) (pc1-05) (15-43) MSD %R (Limits) 3 4 48 0 (25-)47) ( 50-124) (47-123) (\$2-115) ( 51-119 ) ( क्टान्व ) (33-144) (15-143) MS %R (Limits) 8,3 なか 3  $\boldsymbol{c}$ £ ٥ Compound りり MM MS/MSD (D Ø σ

LDC # 3/444 F17 SDG #: 46 Carry

## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Page: 1 of 1 leviewer. Reviewer: 2nd Reviewer:\_\_

METHOD: \_GC\_ HPLC

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

Were a laboratory control samples (LCS) and laboratory control sample duplicate (LCSD) analyzed for each matrix in this SDG? Were the LCS percent recoveries (%R) and relative percent differences (RPD) within the QC limits?

Level IVID Only

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

(DC#: 31494 F17 SDG#: Set Cons

## VALIDATION FINDINGS WORKSHEET Overall Assessment of Data

Page: l of l Reviewer: 3/2 2nd Reviewer: 8

METHOD: CG HPLC

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

All available information pertaining to the data were reviewed using professional judgement to compliment the determination of the overall quality of the data.

Was the overall quality and usability of the data acceptable?

0 Qualifications Hun! Ws mind Grit Finding LCS entende morade MS MSD Compound Name All except Comments:

OVRNew.wpd

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

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

Henderson, Nevada

Collection Date: June 16 through June 19, 2009

LDC Report Date: October 22, 2009

Matrix: Water

Parameters: Organophosphorus Pesticides

Validation Level: Stage 2B

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 8304607

Sample Identification

M-39B M-123B

M-123009B

M-34B

#### 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 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.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. Calibration

#### a. Initial Calibration

Initial calibration of compounds was performed 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.

#### \*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
6/30/09	019F1901	1	Naled	21.6	All samples in SDG 8304607	J- (all detects) UJ (all non-detects)	А

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 8304607	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	Р
6/26/09	010F1001	2	Naled Malathion	47.6 23.2	All samples in SDG 8304607	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	Р

\*Added Malathion to table above for 010F1001 Column 2 on 6/26/09

#### III. Blanks

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

Sample FB060309-SO (from SDG 8304616) was identified as a field 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

Raw data were not reviewed for this SDG.

#### VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304607	All compounds reported below the PQL.	J (all detects)	A

Raw data were not reviewed for this SDG.

#### VII. System Performance

Raw data were not reviewed for this SDG.

#### VIII. Overall Assessment of Data

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

#### IX. Field Duplicates

Samples M-123B and M-123009B were identified as field duplicates. No organophosphorus pesticides were detected in any of the samples.

#### \*Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Data Qualification Summary - SDG 8304607

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304607	M-39B M-123B M-123009B M-34B	Naled	J- (all detects) UJ (all non-detects)	А	Continuing calibration (%D) (c)
*8304607	M-39B M-123B M-123009B M-34B	Naled Disulfoton Malathion	J- (all detects) UJ (all non-detects)	Р	Continuing calibration (ICV %D) (c)
8304607	M-39B M-123B M-123009B M-34B	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 8304607

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304607

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson**

LDC #: 21494G17	VALIDATION COMPLETENESS WORKSHEE
SDG #: 8304607	Stage 2B
Laboratory: Test America	
MFTHOD: GC Organophosph	orus Pesticides (EPA SW 846 Method 8141A)

Reviewer: 2nd Reviewer:

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: $6/16 - 19/09$ $2 RSD \leq 20 2 r^{2}$ $CU/W \leq 20 2$
IIa.	Initial calibration	A	2 RSD = 20 2 r2
IIb.	Calibration verification/ICV	SW	ca/a = 20 2
III.	Blanks	Α	
l∨a.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	2	Client spec (insufficient vol) LCS/D
IVc.	Laboratory control samples	Α_	us /p
V.	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	N	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	ND	p = 2,3
X.	Field blanks	ND	FB = FB060309 from 6304603

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:

brater

		NATOR			
1	M-39B	11	9173103 MB	21	31
2	M-123B	12		22	32
3	M-123009B	13		23	33
4	M-34B	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:			
<u> </u>			

# 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 Caralleanter	
B. Acenaphthylene	B. RDX	B 24.hB		v. rensullounon	V. Benzene
C. Anthracene		0.4.4	B. Mevinphos	W. Bolstar	CC. Toluene
	G. 1,3,5-Innitrobenzene	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-Xvlene
E. Benzo(a)pyrene	E. Tetryl	E. Dinoseb	E. Ethoprop	Z. Coumaphos	RRR. MP-Xylene
F. Benzo(b)fluoranthene	F. Nitrobenzene	F. Dichlorprop	F. Nated	AA. Parathion	GG. Total Xviene
G. Benzo(g,h,f)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. Trichiorinate	
1. Chrysene	1. 2-Amino-4,6-dinitrotoluene	I. MCPP	I. Dimethoate	DD. Trifluratin	
J. Dibenz(a,h)anthracene	J. 2,4-Dinitrotolune	J. MCPA	J. Diazinon	EE. Def	
K. Fluoranthene	K. 2,6-Dinitrotoluene	K. Pentachlorophenol	K. Disulfaton	FF. Prowl	
L. Fluorene	L. 2-Nitrotoluene	L., 2,4,5-TP (slivex)	L. Parathion-methyl	GG. Ethlon	
M. Indeno(1,2,3-cd)pyrene	M. 3-Nitrotoluene	M. Silvex	M. Ronnel	HH. Tetrachiorvinphos	
N. Naphthalene	N. 4-Nitrotoluene		N. Malathion	II. Sulprofos	
O. Phenanthrene	0,		O. Chlorpyrifos	J.T. This are in	
P. Pyrene	ď		P. Fenthion		
ď	o		Q. Parathion-ethyl	1	7
ж.			R. Trichloronate	MM. Hamphur	25 Prior 0 1110 41€
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Notes:

**VALIDATION FINDINGS WORKSHEET** Continuing Calibration

Reviewer: 3/2 Page: of

2nd Reviewer:

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

METHOD: \_\_GC\_\_ HPLC

LDC# 2494 17 SDG# 500 Gury

What type of continuing calibration calculation was performed? \_\_\_\_ RD or \_\_\_ RPD \_\_\_\_ N/A \_\_\_\_ Were continuing calibration standards analyzed at the required frequencies?

Did the continuing calibration standards meet the %D / RPD validation criteria of ≤₹€00%?

Y N NIA Y N NIA Level IV Only Y N (N/A)

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

*	a) e C	Standard ID	Detector/	Compound	%D/RPD	10 / RPD (1 / 15 / 15 / 15 / 15 / 15 / 15 / 15 /	g.	Asse	Associated Samples	Qualifications
	1/20/02	016 1 1001	3	6	87.8	)	(	I V	+ Bik	J. Thurs Le
		(3)		(-) <u>+</u>	40.)	)	)	1 1		J-105 1P
		\ \		4	80.4	)	)			148 P
			1	(-) y	20.6	)	(			5-/45/2
			CA.2	643	215.0	•	(			Jet Total
				F (-)	47.0	_	(			5-145A
				\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	85.7	~	^			A ME A
Γ				(-) N	23.7	_	(			J-/NJ/P
							(			
	69/06/0	019 1991	Z.	(-) #	21.0	)	)			J-/MJ/A
	1	$(c \omega )$		•		)	(			
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#### LDC Report# 21494H17

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

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

Henderson, Nevada

Collection Date: June 23, 2009

LDC Report Date: October 22, 2009

Matrix: Water

Parameters: Organophosphorus Pesticides

Validation Level: Stage 4

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 8304608

Sample Identification

M-125B M-125BMS M-125BMSD

#### Introduction

This data review covers 3 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.

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	All samples in SDG 8304608	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	Р
6/26/09	010F1001	2	Naled Malathion	47.6 23.2	All samples in SDG 8304608	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	Р

<sup>\*</sup>Added Malathion to table above for 010F1001 Column 2 on 6/26/09

Retention times (RT) of all compounds in the calibration standards were within QC limits.

#### 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

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.

#### VI. Project Quantitation Limit

All project quantitation limits PQLs were within validation criteria.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304608	All compounds reported below the PQL.	J (all detects)	Α

#### 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 8304608

SDG	Sample	Compound	Flag	A or P	Reason (Code)
*8304608	M-125B	Naled Disulfoton Malathion	J- (all detects) UJ (all non-detects)	Р	Continuing calibration (ICV %D) (c)
8304608	M-125B	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (PQL) (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Laboratory Blank Data Qualification Summary - SDG 8304608

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304608

No Sample Data Qualified in this SDG

SDG	#: <u>21494H17</u> #: <u>8304608</u> ratory: <u>Test America</u>	_ <b>V</b>	Tro ALIDATIO		rthgate PLETE Stage 2	NESS	WORKSH	IEET	Date: <u>ዓ/ክ/o</u> Page: <u> </u> of <u> </u> Reviewer: <u></u> У 6
	H <b>OD:</b> GC Organophosp						·		2nd Reviewer:
The s	samples listed below we ation findings worksheet	re rev s.	riewed for ea	ch of the f	ollowing	valida	tion areas. Va	alidation f	indings are noted in attached
	Validation	n Are:	a					Commen	te
J	Technical holding times			A	Sampling	dates:	-		
lla.	Initial calibration			A			£ 26 ℃		
IIb.	Calibration verification/ICV	,		SW			1CV = 20		
111.	Blanks			A					
IVa.	Surrogate recovery			A					
IVb.	Matrix spike/Matrix spike d	uplicate	es	A					
IVc.	Laboratory control samples	<u> </u>		Α	LC.	Sta			
V.	Target compound identification	ation		мA					
VI.	Compound Quantitation an	d CRQ	Ls	A 4.					
VII.	System Performance			NA					
VIII.	Overall assessment of data	1		<u>A</u>					
IX.	Field duplicates			N					
Χ.	Field blanks			QN	F.B	- F	B060309	from	8304603
Note: /alidate	A = Acceptable N = Not provided/applicable SW = See worksheet ed Samples:	e	ND = No R = Rins FB = Fie		detected		D = Duplicate TB = Trip blant EB = Equipme		
- 1	Water	T		-		<del>T</del>			
	M-125B	11		<del>-</del>	21	ļ		31	
	M-125BMS	12			22	-		32	
_	M-125BMSD	13			23	ļ		33	
4	9177142-MB	14			24	<del> </del>		34	
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7		16		· · · · · · · · · · · · · · · · · · ·	26	<b> </b>		36	
,		17 I			1 27	ī			1 !!

Notes:			
10	20	30	40
			[39

# VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
Reviewer: 576
2nd Reviewer:

Method: GC HPLC				
Validation Area	Yes	No	NA	Findings/Comments
EP pagenting a following to a company of the pagenting and the pag				
All technical holding times were met.	4	—	ـ	
Cooler temperature criteria was met.				
Elizabilitat zilipizitisi				
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?	+	<del> </del>	┢	
	+	<del>                                     </del>		
Did the initial calibration meet the curve fit acceptance criteria of > 0.990?  Were the RT windows properly established?	+	<del>                                     </del>	<del> </del>	
W relatification in the stabilished?			22.74 2.04 2.04 3.04 3.04	[
Was a continuing calibration analyzed daily?				
Were all percent differences (%D) < 15%:0 or percent recoveries 85-125%?		<u> </u>	<del>                                     </del>	
Were all the retention times within the acceptance windows?	+	<b>-</b>	_	
WHanks V				
Was a method blank associated with every sample in this SDG?		228 (030)		
Was a method blank analyzed for each matrix and concentration?	1			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
WESHGERIGERIKES 4				
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?				
Mic data kad kad kad kad kad kad bija legies				
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?				
uli satoratolykona samplas				
Nas an LCS analyzed for this SDG?				and the state of t
Nas an LCS analyzed per extraction batch?				
Nere the LCS percent recoveries (%R) and relative percent difference (RPD) vithin the QC limits?  X:Regionals:ualiy/Assurance and Quality control.				
Vere performance evaluation (PE) samples performed?				
Vere the performance evaluation (PE) samples within the acceptance limits?		-	7	

LDC# 214aq H17 SDG# See Cover

# **VALIDATION FINDINGS CHECKLIST**

Page:  $\frac{2}{J\sqrt{t}}$  Reviewer:  $\frac{2}{J\sqrt{t}}$  2nd Reviewer:

Validation Area	Yes	No	NA.	Flading (0
28 Talent verden in annult allement				Findings/Comments
Were the retention times of reported detects within the RT windows?				
XX. scingount straintistic//ctXqts		N SAL		
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
M Signar Caper garden				
System performance was found to be acceptable.		1		
XIII (eXalallessa-sama), quadra (exalallessa-sam				
Overall assessment of data was found to be acceptable.		· .		
ANY RELOGUED COME THAT				
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.				
XXV: replacing the second of t				
Field blanks were identified in this SDG.				
Target compounds were detected in the field blanks.			7	

# VALIDATION FINDINGS WORKSHEET

METHOD: / GC HPLC

cmpd\_list.wpd

Notes:

LDC # 2494 H 17

METHOD: \_GC\_ HPLC

# VALIDATION FINDINGS WORKSHEET Continuing Calibration

Page: of

Reviewer:

2nd Reviewer:

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

Level IX Only X N (N/A)

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

	7	Mons	<b>4</b> (C)	9	3		2	*	1.4	1	Y		-									Ī			
		August 1	12. E	トイノーク	1	,	W/- 0	- tests	J-145/2	a)		74/-	•												
	Associated Samples	211 1 P 11	TILL TOLK							->															
	(Limit < 15.0) ( 202) RT (limit)				(					(				(			,		(	(			7	(	
naa/u%	(Limit < 15.07	8 28	9	700	22.	7 02	215	47 (		25	23.7														
	Compound	4	Щ	4		k (-)	3	4		1	(-) N												†-		-
Detector/	Column	3				7	52.2				4														***************************************
	Standard ID	016 F 1001	3																						
	# Date	50/92/3	,																		+	+			

LDC # 21494 117 SDG #: See Cover

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer: Reviewer:\_

> HPLC METHOD: GC\_

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

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

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

L									
				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound	CF ( 2_ std)	ر ق	Average CF (initial)	Average CF (initial)	%RSO	CV CV
-	78-	6/20/3	Dichler	1.76366 1.76369	1.76369	1.74977	1.74977	7.7300.7 USSP P. 5	7.4957.6
				1.82370	1.82370 1.82570	7-618-1	181476	10609.5	5,609,2
			Malathia 1	7,60	12	20			
2		<u>-</u> -	Dichlarys (814-2)	2, 17503 2, 17503	2,17503	2,01915	2,01345	7.323W	7.323W 7.323VC
			Phone	1.69491	1.63631	51691.	1.76315	8,53546	6,5344 B.53 913
			malatrian 7	1.17724	1, 17724	1.20369	1.20369	2.60489	3.60 yys 3.6.500
ო									
4									
		1							
		·							

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.

LDC # 21 499 #17 SDG# Su Con

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: of 2 

> GC EPA SW 846 Method 8141A METHOD:

Malathion Parameter: X^2 Conc ratio 0.100 0.250 0.500 1.000 1.500 2.000 2.500 Area ratio 0.29331 0.89027 1.76202 2.36769 2.77727 0.14584 0.55883 Compound Malathion (8141A-1) Column 06/26/2009 Date

Regression Output:			Reported	77
Constant		-0.02062	= 0	-0.02066
Std Err of Y Est		0.12319		
R Squared		0.99000	12 =	0.99783
No. of Observations		7.00000		
Degrees of Freedom		5.00000		
\\\-\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\	120			
A Coellicient(s)	1.1388	-0.002208	ю	1.14436E+000
Std Err of Coef.	0.054985	00.0		

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

LDC # 21 494 #17 SDG#: See Cover

# Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Page: 1 of / Reviewer: TVC 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

					Reported	Recalculated	Reported	Recalcidated
*	Standard ID	Calibration Date	Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc.	۵%	Q%
-	016 F1601 6/20/69	60/06/0	Pichlaryous (8191-1)	3,00	2.6730	2,6730	10, 9	6,01
		-	phorace		2.9763	2, 1700	K 2	0.7
			Malatina		3.0648	3.0648	7	۲.۲
7			Dichluross (8191-2)		2.6533	2.6533	11,6	7 111
			Phorate		3.1758	3.1758	6,0	7 5
			Modethim }		3.0056	3.00.5	9.9	- 6
ဗ						,		<i>\</i>
4								
T								

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

# H17	Cre
2149	25
LDC#:	SDG#

# VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page: of 2nd reviewer: Reviewer:

> The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation: METHOD: \_\_GC \_\_ HPLC

% Recovery: SF/SS \* 100

#

Sample ID:

Where: SF = Surrogate Found SS = Surrogate Spiked

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent	Percent
				Reported	Receiption	
TPP	3	1.00	0.59488	77	77	0.
Ch women for	<b>\</b>	ì	0,7671	24	24	-
		-				
Sample ID:		·				
Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent	Percent	Percent
				Proceed and	, second	Mileterice
				nahoj jed	Vecaliculated	
Sample ID:						
Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent	Percent
				Reported	Receiculated	

LDC# 21494 HI7 SDG #: See Cone

# Matrix Spike/Matrix Spike Duplicates Results Verification VALIDATION FINDINGS WORKSHEET

Page: Reviewer: 2nd Reviewer:

METHOD: \_\_\_\_GC\_\_\_HPLC
The percent recoveries (%R) and relative percent differences (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation: %Recovery = 100 \* (SSC - SC)/SA

Where

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

SC = Sample concentration

RPD =(((SSCMS - SSCMSD) \* 2) / (SSCMS + SSCMSD))\*100

2/2

MSD = Matrix spike duplicate

MS/MSD samples:\_

	Spike		Sample	Spike (	Spike Sample	Matri	Matrix spike	Matrix Soike Dunitate	Publicate	1000	
Compound	Depoy	<u></u>	Conc.	Concer	ıtration			Y TO Y	Cupilicate	COMOCI	John Community of the C
	1 45 1/2	1	1 23 (4.)	5g)	(1-)	Percent	Percent Recovery	Percent Recovery	Recovery	2	
	MS M	MSD		WS	QSW	Reported	Becalc	1	,		п
Gasoline (8015)							Sign	Nepocren	Recalc.	Reported	Recalc.
Diesel (8015)		T									
Benzene (8021B)		1									
Methane (RSK-175)		†									
2,4-D (8151)		<del> -</del>									
Dinoseb (8151)											
Naphthalene (8310)											
Anthracene (8310)											
HMX (8330)											
2,4,6-Trinitrotoluene (8330)											
Dichlmorg (8141)	286 3.9	63	0	2.87	2.97	74	74	7/	FC	2	-
Malattin	<u> </u>		<b>→</b>	2.75	2.99	7	77	70	76	6 2	2,1
									<b>)</b>	)	

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

LDC#: 2494 417 SDG# See and

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

Page: lof / Reviewer:\_

2nd 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 RPD = 1 LCS - LCSD 1 \* 2/(LCS + LCSD)

SC = Concentration

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

Z

9177142

LCS/LCSD samples:\_

LCSD = Laboratory control sample duplicate percent recovery

	8	pike	Spiked	Spiked Sample		SOT	TCSD	SD	lCS/	TCS/TCSD
Compound	* શ	Maded ( )	Concentrati	intration	Percent	Percent Recovery	Percent Recovery	Recovery	۵	uaa
	SOT	TCSD	SOT	CSD	Reported	Recalc	Reported	Dacalc		1
Gasoline (8015)									periodes	אפלפולי.
Diesel (8015)										
Benzene (8021B)										
Methane (RSK-175)										
2,4-D (8151)										
Dinoseb (8151)										
Naphthalene (8310)										
Anthracene (8310)										
HMX (8330)										
2,4,6-Trinitrotoluene (8330)										
Dichlara [814)	4.07)	Ž	2-78	\$	82	8 9				
Marthin &	-6	_	11.5		66	66				

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.

LIH 464 HIJ SDG #: See Cores

# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page: \\_of\_\_ Reviewer: JA

METHOD: CGC\_HPLC

(A)	N/A	<b>)</b>
z	Z	

Were all reported results recalculated and

		5
? if the reported results?		Compound Name
were all recalculated results for detected target compounds, within 10% of the reported results?  Were all recalculated results for detected target compounds, within 10% of the reported results?	Example:	Sample ID.
Were all recalculated results reca	(A)(Fy)(Df) (RF)(Vs or Ws)(%S/100)	
X N N X	Concentration≈ (	•

Compound Name

Concentration =\_\_

A= Area or height of the compound to be measured
Fv= Final Volume of extract
Of= 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

#	Sample ID	Сотроила	Reported Concentrations	Recalculated Results Concentrations	Qualifications
			-		
Comments:	ants:				

# LDC Report# 21494|17

# 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: October 22, 2009

Matrix: Water

Parameters: Organophosphorus Pesticides

Validation Level: Stage 2B

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 8304609

Sample Identification

M-111AB

EB062909-GW

# 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.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- 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.
- Pata 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.

# \*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)	А
7/7/09	004F0401	2	Naled	24.0	All samples in SDG 8304609	J+ (all detects)	А

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	40.1	All samples in	J- (all detects)	Р
			Disulfoton	20.6	SDG 8304609	UJ (all non-detects) J- (all detects) UJ (all non-detects)	,

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

<sup>\*</sup>Added Malathion to table above for 010F1001 Column 2 on 6/26/09

# 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

Raw data were not reviewed for this SDG.

# VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Finding		
	Flag	I Aor P
reported helewah - DOI		A
	reported below the PQL. J (all	reported below the PQL. J (all detects)

Raw data were not reviewed for this SDG.

# VII. System Performance

Raw data were not reviewed for this SDG.

# VIII. Overall Assessment of Data

Data flags 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)	А	Continuing calibration (%D) (c)
*8304609	M-111AB EB062909-GW	Naled Disulfoton Malathion	J- (all detects) UJ (all non-detects)	Р	Continuing calibration (ICV %D) (c)
8304609	M-111AB EB062909-GW	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Lim (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

# **Tronox Northgate Henderson**

LDC #: 21494I17	VALIDATION COMPLETENESS WORKSHEET
SDG #:8304609	Stage 2B
Laboratory: Test America	
METHOD: GC Organophosp	horus Pesticides (EPA SW 846 Method 8141A)

Date: 9/11/09
Page: <u> </u> of <u> </u>
Reviewer: JV6
2nd Reviewer:

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 6/29/09
lla.	Initial calibration	A	2 RSD 5-20 7 VY
lib.	Calibration verification/ICV	SW)	ca/ 10 = 20 ]
111.	Blanks	A	,
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	N	Client sher (insufficient sample)
IVc.	Laboratory control samples	A	Client spec (insufficient sample)
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	
Χ.	Field blanks	Νb	EB = 2

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:

	wa	er			
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 7 8		16	26	36	
7		17	27	37	
1		18	28	38	
9		19	29	39	
10		20	30	40	

Notes:	

# VALIDATION FINDINGS WORKSHEET

METHOD: /GC HPLC

8310	8330	8151	(8141	8141Con'th	90240
A. Acenaphthene	A. HMX	A 2.4-D		h upoli e i o	90718
B. Acenaphthylene	>00 e		A. Uichioryos	V. Fensulfathion	V. Benzene
C. Anthracene		B. 2,4-DB	B. Mevfnphos	W. Boistar	CC. Toltrena
	C. 1, 2, 2-1 Initrobenzene	C. 2,4,5-T	C. Demeton-O	X. EPN	EE. Ethyl Benzene
	D. 1,3-Dinitrobenzene	D. 2,4,5-TP	D. Demeton-S	Y. Azinphos-methyl	- A O 333
E. Benzo(a)pyrene	E. Tetryi	E. Dinoseb	E. Ethoprop	Z. Coumaphos	SSS. C-Aylene
F. Benzo(b)fluoranthene	F. Nitrobenzene	F. Dichlorprop	F. Naled	AA Derathics	AKK. MP-Aylene
G. Benzo(g,h,l)peryfene	G. 2.4.6-Trinitrotoluene	G. Dicamba	0.00		GG. Total Xylene
H. Benzo(k)fluoranthene	H. 4-Amino.2 & disitratellises		e. Sulfotep	BB. Trichioronate	
I. Chrysene	eueniolo nulliporte citatione	H. Dalapon	H. Phorate	CC. Trichlorinate	
	L. Z-Amino-4,8-dinitrotoluene	I. MCPP	l. Dimethoate	DD. Triffuralin	
J. Dibenz(a,h)anthracene	J. 2,4-Dinitrotolune	J. MCPA	J. Diazinon	EE. Def	
K. Fluoranthene	K. 2,6-Dinitrotoluene	K. Pentachiorophenoi	K. Disulfoton	FF Drawi	
L. Fluorene	L. 2-Nitrotoluene	1. 2.4 5.TP (ellow)			
M. Indeno(1,2,3-cd)pyrana		Yanale land	L. Parathlon-methyl	GG. Ethion	
	m. J-Mrrotoluene	M. Slivex	M. Ronne	HH. Tetrachiorvinphos	
v. raphinalene	N. 4-Nitrotoluene		N. Malathion	II. Sulprofos	
O. Phenanthrene	o'		O. Chlorpyrifos		
P. Pyrane	ď		P. Fenthion		
Ö	ø			AK, Phosmet	
ĸ.			u. rarathion-ethyi	11. 0.00-Tricthylphosphorothiogte	sephoro this ate
S			R. Trichloronate	MM. Famphur	
			S. Merphos	NN. Carbo pheno thou	S
			T. Stirofos	l	
			U. Tokuthlon	vi - methor - methol	en - metaul

Notes:

SDG # See Gury LDC # 2494 I 17

# VALIDATION FINDINGS WORKSHEET

Continuing Calibration

Page: 1 of 1

Reviewer:

d

2nd Reviewer:

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

Were continuing calibration standards analyzed at the required frequencies?

MN/A

Did the continuing calibration standards meet the %D / RPD validation criteria of 3460%?

Y N N/A Y N N/A Level IX Only

Were the retention times for all calibrated compounds within their respective acceptance windows? Y N N/A

		<u>ত</u>	_	T	T	Ţ	T			T	Ţ	Ţ	Ī	<b>~</b>		T	T		_	Γ	T	T	Τ	T	T	7	
	Cuaimcanons	- STATES	C/ +1/-10	7 40 40	3, 2, 7, 7	1/ M/- 2	1200	ウーイルグ	The state of	3- /hr /p		F	2+ 9475/A	,													
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# LDC Report# 21494J17

# Laboratory Data Consultants, Inc. Data Validation Report

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

Henderson, Nevada

Collection Date: June 26 through June 1, 2009

LDC Report Date: October 22, 2009

Matrix: Soil/Water

Parameters: Organophosphorus Pesticides

Validation Level: Stage 2B

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 8304610

# Sample Identification

EB062609-SO

EB062609-SORE

SA106-0.5B

SA82-0.5B

SA82-10B

SA82-29B

SA106-0.5BMS

SA106-0.5BMSD

SA82-0.5BMS

**SA82-0.5BMSD** 

## Introduction

This data review covers 8 soil samples and 2 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 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.

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

The following are definitions of the data qualifiers:

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

# I. Technical Holding Times

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

Sample	Analyte	Total Days From Sample Collection Until Analysis	Required Holding Time (in Days) From Sample Collection Until Analysis	Flag	A or P
EB062609-SORE	All TCL compounds	12	7	J- (all detects) UJ (all non-detects)	А

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 (r²) was greater than or equal to 0.990.

### \*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
7/2/09	003F0301	1	Dichlorvos Mevinphos Dimethoate Fensulfothion	21.1 45.9 49.7 44.0	EB062609-SO 9180507MB	J- (all detects) UJ (all non-detects)	А
7/2/09	003F0301	1	Naled EPN	28.9 21.2	EB062609-SO 9180507MB	J+ (all detects) J+ (all detects)	A
7/2/09	003F0301	2	Mevinphos Fensulfothion Azinphos-methyl	22.3 22.9 25.9	EB062609-SO 9180507MB	J- (all detects) UJ (all non-detects)	А

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
7/2/09	003F0301	2	Merphos	27.1	EB062609-SO 9180507MB	J+ (all detects)	А
7/13/09	010F1001	1	Naled	43.2	SA106-0.5B SA82-0.5B SA82-10B SA82-29B SA106-0.5BMS SA106-0.5BMSD SA82-0.5BMSD SA82-0.5BMSD 9188427MB	J+ (all detects) J+ (all detects)	А
7/13/09	010F1001	2	Naled	32.5	SA106-0.5B SA82-0.5B SA82-10B SA82-29B SA106-0.5BMS SA106-0.5BMSD SA82-0.5BMS SA82-0.5BMSD 9188427MB	J+ (all detects) J+ (all detects)	А

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	All samples in SDG 8304610	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	P
6/26/09	010F1001	2	Naled Malathion	47.6	All samples in SDG 8304610	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	P

<sup>\*</sup>Added Malathion to table above for 010F1001 Column 2 on 6/26/09

## III. Blanks

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

Samples EB062609-SO and EB062609-SORE were identified as equipment banks. No organophosphorus pesticide contaminants were found in these blanks.

Sample FB072109-SO (from SDG 8304616) was identified as a field 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 with the following exceptions:

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
EB062609-SO	Not specified	Triphenylphosphate Chlormefos	46 (60-154) 37 (49-171)	All TCL compounds	J- (all detects) UJ (all non-detects)	A
9180507MB	Not specified	Triphenylphosphate Chlormefos	25 (60-154) 30 (49-171)	All TCL compounds	J- (all detects) UJ (all non-detects)	Р

# b. Matrix Spike/Matrix Spike Duplicates

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

# c. Laboratory Control Samples

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

## V. Target Compound Identification

Raw data were not reviewed for this SDG.

## VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304610	All compounds reported below the PQL.	J (all detects)	Α

Raw data were not reviewed for this SDG.

# VII. System Performance

Raw data were not reviewed for this SDG.

## VIII. Overall Assessment of Data

The overall assessment of data was acceptable. In the case where more than one result was reported for an individual sample, the least technically acceptable results were rejected as follows:

Sample	Compound	Flag	A or P
EB062609-SORE	All TCL compounds	x	А

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 8304610

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304610	EB062609-SORE	All TCL compounds	J- (all detects) UJ (all non-detects)	Α	Technical holding times (h)
8304610	EB062609-SO	Dichlorvos Mevinphos Dimethoate Fensulfothion Azinphos-methyl	J- (all detects) UJ (all non-detects)	A	Continuing calibration (%D) (c)
8304610	EB062609-SO	Naled EPN Merphos	J+ (all detects) J+ (all detects) J+ (all detects)	А	Continuing calibration (%D) (c)
8304610	SA106-0.5B SA82-0.5B SA82-10B SA82-29B	Naled	J+ (all detects) J+ (all detects)	А	Continuing calibration (%D) (c)
*8304610	EB062609-SO EB062609-SORE SA106-0.5B SA82-0.5B SA82-10B SA82-29B	Naled Disulfoton Malathion	J- (all detects) UJ (all non-detects)	P	Continuing calibration (ICV %D) (c)
8304610	EB062609-SO	All TCL compounds	J- (all detects) UJ (all non-detects)	А	Surrogate recovery (%R) (s)
8304610	EB062609-SO EB062609-SORE SA106-0.5B SA82-0.5B SA82-10B SA82-29B	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (PQL) (sp)
8304610	EB062609-SORE	All TCL compounds	х	А	Overall assessment of data (0)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Laboratory Blank Data Qualification Summary - SDG 8304610

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304610

No Sample Data Qualified in this SDG

	21494J17 VALIDATI	ON COMP	thgate Hen		SHEET		9/15/09
SDG # Labora	: 8304610 tory: Test America		Stage 2B			;Page Reviewer 2nd Reviewer	
METH	OD: GC Organophosphorus Pesticide	s (EPA SW	846 Method 8	141A)	•		
	imples listed below were reviewed for ion findings worksheets.	each of the t	following valida	ation areas	. Validation fi	ndings are noted in	attached
	Validation Area				Comment	s	
l.	Technical holding times	SM	Sampling dates	6/26	- 7/01/6	1	
IIa.	Initial calibration	A		0 = 20			
IIb.	Calibration verification/ICV	SW	cw/	iw = 2	0 2		
10.	Blanks	Á					
IVa.	Surrogate recovery	SW					
IVb.	Matrix spike/Matrix spike duplicates	SW					
IVc.	Laboratory control samples	A	us /D				
· V.	Target compound identification	N					
VI.	Compound Quantitation and CRQLs	N					
VII.	System Performance	N					
VIII.	Overall assessment of data	SW	ļ <u></u> .				
IX.	Field duplicates	N			·		
X	Field blanks	ND	EB =	1, 7	ŦB=	FB072109-50	from 8304611
Note:	N = Not provided/applicable: R =	= No compound Rinsate = Field blank	ds detected	D = Dupli TB = Trip EB = Equ			i e
Validate	d Samples: Water + Soil						
T 1 1	EB062609-SO W 11 1 9 1 5	30507 MB	21		31		
2 7	EB062609-SORE 172 7 918	945) MI	22		32		
3 3 3	SA106-0.5B S 13 3 91	88427 M	B 23		33		

	Wat	er +	Soil			
<u>- 1</u>	EB062609-SO	W 17	1 9180507 MB	21	31	
2 3	EB062609-SORE	1 72	7 9189451 MD	22	32	
3 2	SA106-0.5B	S 13	91 88427 MB	23	33	
4 3	SA82-0.5B	14	' '	24	34	
5 3	SA82-10B	15		25	35	
6 3	SA82-29B	16		26	36	
7 3	SA106-0.5BMS	17		27	37	
8	SA106-0.5BMSD	18		28	38	
9 ,	SA82-0.5BMS	19		29	39	
10	SA82-0.5BMSD	20		30	40	

Notes:			
			 -
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# VALIDATION FINDINGS WORKSHEET

METHOD: / GC HPLC

8310	8330	8151	8141	8141(con't)	8021B
A. Acenaphthene	A. HMX	A 2,4D	A. Dichlarvos	V. Fensulfothlan	V. Benzene
B. Acenaphthylene	B. RDX	B. 2,4-DB	B. Mevinphos	W. Boistar	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	888. O-Xylene
E. Benzo(a)pyrene	E. Tetryl	E. Dinoseb	E, Ethoprop	Z. Coumphos	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	SB. Trichloronate	
H. Benzo(k)fluoranthene	H. 4-Amine-2,6-dinitrotoluene	H. Dalapon	H. Phorate	CC. Trichlorinate	
I. Chrysens	1. 2-Amino-4,6-dinitrotoluene	I. MCPP	1. Dimethoate	DD. Triffuralin	
J. Dibenz(a,h)anthracene	J. 2,4-Dinitrotolune	J. MCPA	J. Diazinon	EE. Def	
K. Fluoranthane	K. 2,6-Dinitrotoluene	K. Pentachlorophenol	K. Disulfoton	FF. Prowl	
L. Fluorene	L. 2-Mitrotoluene	L 2,4,6-TP (slivex)	L. Parathion-methyl	GG. Ethion	
M. Indeno(1,2,3-cd)pyrene	M. 3-Nitrotoluene	M. Silvex	M. Ronnel	HH. Tetrachiowinphos	
N. Naphthalene	N. 4-Nifrotoluene		N. Malathlon	II. Sulprofos	
O. Phenanthrene	0,		O. Chlorpyrifos	JJ. Thiongrin	
P. Pyrene	Р.		P. Fenthion	kk, Phosmet	
Q.	a		Q. Parathion-ethyl	LL. O.O.O. Triethylphosphorothia 412	osphoro thio ate
Ж,		·	R. Trichloronate	MM. Famphur	
S,			S. Merphos	NN. Carbo pheno thion	ion.
			T. Stirofos	00. Carbo phenothon - methy	im -methy/
			U. Tokuthion		

cmpd\_list.wpd

Notes:

LDC# 2/494 J17 SDG #: 24 Can

# VALIDATION FINDINGS WORKSHEET **Technical Holding Times**

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

All circled dates have exceeded the technical holding times.

METHOD	METHOD GC HPLC	ĽC					
Gi elames	Matrix	pevies	Sampling Date	Extraction data	Analysis date	Total # of Days	Qualifler
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				·			

# TECHNICAL HOLDING TIME CRITERIA

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

Both within 14 days of sample collection. Both within 14 days of sample collection.

Water: Soil: Soils: EXTRACTABLES:

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

HTNew.wpd

LDC #: 2494 17

# VALIDATION FINDINGS WORKSHEET Continuing Calibration

2nd Reviewer:\_\_

Page: Lof Seviewer: 3/2

Reviewer

METHOD: GC HPLC

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

Y N N/A Level IV Onty Y N NIA

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

	3	CI brahasi	Detector/ Column	Compound	(J.mit s.48:0) (2.8.2.)	202) RT (Ilmit)	Associated Samples	Qualifications
			1 170	7.5	4 7 8		M. + 12.11	James de C
	13/97/2	100 1100	-	1	- 01/			J-145 1
				رجوا ر دوا ر	1.4	_		+18.72.
			,	65.4		`		5-/45/19
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	76-64	ののよまのない	1	A E)	21.1		1 9130567 MB	J-/WIA
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				×	ν <u>'</u> 'ν			J+005A
			7.3	BC)		(		3-/M/A
				ΛC		)		
				(J)K				
			/	(+) <i>\$</i>	122			S+4447
			•					
	7/2/09	010 F 1001	ed.)	<b>も</b>	43.7		3-10, 9188427MB	J+ AMA
l		(cc1)	र क	F(t)	32.5			
		\ \						
					18.50			
						)		

VALIDATION FINDINDS WORKSHEET Surrogate Recovery

Reviewer:

LDC# 2/4/4 J17 SDG#:

Are surrogates required by the method? Yes or No Plasse see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". Plasse see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". Were surrogates spiked into all samples and blanks?

Y N N/A Did all surrogate recoveries (%R) meet the QC limits?

Y N AVA		3		Did all suitages ( co. )								=
,		8	Detector	Surrogata		V.R (Llmfts)				Qualifications	tions	
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		te.	Not Steel had		1	3	200	+	1			Γ
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L	Chlorobenzene (CBZ)	-	0	Octacosane	2	Benzo(e)Pyrene	S	1-Chloro-3-Nftrobenzens	$\dashv$	>	Tetrachloro-m- xylene	
6	4-Bromofluorobenzene (BFB)	-		Ortho-Temphenyl	z	Terphenyl-D14	-	3,4-Dinitrotokuene	$\dagger$	 	Chlorme fos	
٥	a.a.a.Trifluorobluena		_	Fluorobenzene (FBZ)	٥	Decachlorobiphenyl (DCB)	ס	Tripentyffin	tytti.	+		
١	Bromochforabenene	•		n-Triecontane	۵	1-methylnaphthalana	<b>&gt;</b>	Tri-n-propyffin	opytho	$\frac{1}{1}$		
_	1,4-Dichlorobutane		Ж	Hexacosane	σ	Dichlorophenyl Acetic Acid (DCAA)	*	Tributyl Phosphate	nosphate	+		
u	1.4-Diffuorobenzene (DFB)	DFB)		Bromobenzene	B	4-Nitrophenol	X	Tripheny Phosphate	hosphata	-		

LDC # 2/494517 SDG # Sec Cons

## Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

Page: 1 of 1 Reviewer: 3/6 2nd Reviewer:

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

Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG?

Was an MS/MSD analyzed every 20 samples for each matrix or whenever a sample extraction was performed?

Were the MS/MSD percent recoveries (MS) and relative percent differences (RPD) within QC limits?

ξ.	2		4 02	OSH SX	2	MSD		17 17			
	7/8	7	0	, b-15L )	0	1251-0	O'N	(Simila)	Associated Samples	No AAA	
$\vdash$		^	36	(47-123)	91	(123)	75	49.		10 HOV 108	\ \ <u>\</u>
		9	0	(15-143)	8.8	(15-143)	42	20/			12
		λ		^		-	8	- 63			
		0		(	25	(511-25)		-			
П		_ ヮ		î	48	(49-122)		(			
		N		( )	46	(4d-124)					
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		I	7.7	(10-156)		( )	107	(86)		or LCS in	
		>	44	( 47-133		( )	,	(			
		Z	48	( 44-124		( )		(			
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LDC# 31494 317 SDG#: See Come

## VALIDATION FINDINGS WORKSHEET **Overall Assessment of Data**

Page: 1 of 1 

METHOD: \_GC \_ HPLC

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

All available information pertaining to the data were reviewed using professional judgement to compliment the determination of the overall quality of the data.

(X) N/A

Was the overall quality and usability of the data acceptable?

# Compound theme 2 Outried H.T.	Associated Samples Gualifications	$\times \mathcal{K}/\wedge (\mathfrak{o})$								
	Sample 1D Compount Hane									

Comments:

OVRNew.wpd

#### LDC Report# 21494L17

## Laboratory Data Consultants, Inc. Data Validation Report

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

Henderson, Nevada

Collection Date: July 9 through July 10, 2009

LDC Report Date: October 22, 2009

Matrix: Soil/Water

Parameters: Organophosphorus Pesticides

Validation Level: Stage 2B

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 8304612

#### Sample Identification

RSAM2-10B RSAM2-35B SA35-10B SA35-32B SA35009-32B SA85-33B

EB071009-SO

#### Introduction

This data review covers 6 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 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.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- 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.

#### \*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
7/24/09	003F0301	1	Dichlorvos	20.6	RSAM2-10B 9203338-MB	J- (all detects) UJ (all non-detects)	А
7/24/09	003F0301	1	Naled	32.6	RSAM2-10B 9203338-MB	J+ (all detects)	А
7/24/09	003F0301	2	Naled	34.7	RSAM2-10B 9203338-MB	J+ (all detects)	A
7/16/09	019F1901	2	Naled	28.1	RSAM2-35B SA35-10B SA35-32B SA35-32B SA85-33B 9194431-MB	J- (all detects) UJ (all non-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:

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 8304612	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 8304612	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	Р

<sup>\*</sup>Added Malathion to table above for 010F1001 Column 2 on 6/26/09

#### III. Blanks

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

Sample EB071009-SO was identified as an equipment bank. No organophosphorus pesticide contaminants were found in this blank.

Sample FB072109-SO (from SDG 8304616) was identified as a field 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

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

#### c. Laboratory Control Samples

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

#### V. Target Compound Identification

Raw data were not reviewed for this SDG.

#### VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304612	All compounds reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

#### VII. System Performance

Raw data were not reviewed for this SDG.

#### VIII. Overall Assessment of Data

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

#### IX. Field Duplicates

Samples SA35-32B and SA35009-32B were identified as field duplicates. No organophosphorus pesticides were detected in any of the samples.

#### \*Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Data Qualification Summary - SDG 8304612

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304612	RSAM2-10B	Dichlorvos	J- (all detects) UJ (all non-detects)	А	Continuing calibration (%D) (c)
8304612	RSAM2-10B	Naled	J+ (all detects)	А	Continuing calibration (%D) (c)
8304612	RSAM2-35B SA35-10B SA35-32B SA35009-32B SA85-33B	Naled	J- (all detects) UJ (all non-detects)	А	Continuing calibration (%D) (c)
*8304612	RSAM2-10B RSAM2-35B SA35-10B SA35-32B SA35009-32B SA85-33B EB071009-SO	Naled Disulfoton Malathion	J- (all detects) UJ (all non-detects)	Р	Continuing calibration (ICV %D) (c)
8304612	RSAM2-10B RSAM2-35B SA35-10B SA35-32B SA35009-32B SA85-33B EB071009-SO	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 8304612

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304612

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson**

LDC #: 21494L17	VALIDATION COMPLETENESS WORKSHEET	Date: 9/11/09
SDG #: 8304612	Stage 2B	Page: lof )
Laboratory: <u>Test America</u>		Reviewer: NC
METHOD: GC Organophosph	orus Pesticides (EPA SW 846 Method 8141A)	2nd Reviewer:

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 7 /oq - 10 /oq
ila.	Initial calibration	A	2 RSD =207 r2
IIb.	Calibration verification/ICV	sW	COV/1W = 20 2
111.	Blanks	A	
IVa.	Surrogate recovery	L A	
IVb.	Matrix spike/Matrix spike duplicates	A	D9 C020173-061D
IVc.	Laboratory control samples	SW	D9 C020173-061D LCS 15
V.	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	N	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	ND	D = 4,5
<u>x.</u>	Field blanks	ND	EB = 7 FB = FB072109-SO (from 8304616)

Note:

A = Acceptable

N = Not provided/applicable SW = See worksheet

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

D = Duplicate

TB = Trip blank EB = Equipment blank

Validated Samples: Soil + Water

		V) T	VICIE			
11	RSAM2-10B		11	9203338-MB	21	31
2 7	RSAM2-35B		12 7		22	32
3 >	SA35-10B		13 3	919 8202 MB	23	33
4 2	SA35-32B	Þ	14		24	34
- γ 5	SA35009-32B	b	15		25	35
6 7	SA85-33B		16		26	36
7	EB071009-SO	W	17		27	37
8			18		28	38
9			19		29	39
10			20		30	40

Notes:	

# VALIDATION FINDINGS WORKSHEET

METHOD: / GC HPLC

0700					
01.00	8330	8151	(8141	8141(Con't)	8021B
A. Acenaphthene	A. HMX	A. 2.4-D	Note:		
B. Acenaphthylene	>00 B		A Diction of	V. Fensulfothion	V. Benzene
A. A. C.	, C. C.	B. 2,4-DB	B. Mevinphos	W. Bolstar	CC. Toluene
V. Alluitacene	C. 1,3,5-Trinitrobenzene	C. 2,4,5-T	C. Demeton-O	X. EPN	
D. Benzo(a)anthracene	D. 1,3-Dinitrobenzene	D. 2,4,5-TP	D. Demeton-S	Y Azimhos-methul	- 1
E. Benzo(a)pyrene	E. Tetry(	E. Dinoseb	111111111111111111111111111111111111111		ooo. U-Xylene
F. Benzo(b)fluoranthene	F. Nitrobenzene	F Dichioman		4. Coumaphos	RRR. MP-Xylene
( 1 m) ( 1 m) ( 1 m)		doddod	F. Naled	AA. Parathion	GG. Total Xylene
o. Den zole, i., i) peryiene	G. 2.4.6-Trinitrotoluene	G. Dicemba	G. Sulfotep	BB. Trichloronate	
H. Benzo(k)fluoranthene	H. 4-Amino-2,6-dinitrotoluene	H. Dalapon	H. Phorate	CC. Trichlorinate	
1. Chrysene	l. 2-Amino-4,6-dinitrotoluene	I. MCPP	1. Dimethoats	DD. Trifluratio	
J. Dibenz(a,h)anthracene	J. 2,4-Dinitrotolune	J. MCPA	J. Diazingn	EF Det	
K. Fluoranthene	K. 2,6-Dinitrotoluene	K. Pentachiorophenol	K Disulfaton		
L. Fluorene			N. Claumorou	FF. Prowl	
	L. Z-Nitrotoluene	L 2,4,5-TP (silvex)	L. Parathion-methyl	GG. Ethlon	
M. Indeno(1,2,3-cd)pyrene	M. 3-Nitrotoluene	M. Silvex	M. Ronnel	HH. Tetrachiorvinphos	
N. Naphthalene	N. 4-Nitrotoluene		N. Malathion	II. Sulprofes	
O. Phenanthrene	О.		O. Chlorpyrifos		
P. Pyrene	a.		P Fenthlon	1	
Ö	ď			AR, FNOSmet	
0			d. Parathion-ethyl	11. 0.00-Triethylphosphorothiogie	Sephorothiogate
			R. Trichloronate	MM. Famphur	
S.			S. Merphos		
			T. Stirofos		00
			1 Tobushian	00. Carbophenothon	on - methy
			C. Longillon		

Notes:

LDC #: 2494 17 SDG #: 5ce Gv-7

METHOD: \_\_GC\_\_ HPLC

# **VALIDATION FINDINGS WORKSHEET** Continuing Calibration

Reviewer: 316

Page: of

2nd Reviewer:

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? — "AD or \_\_\_ RPD

Were continuing calibration standards analyzed at the required frequencies?

NAMA

Did the continuing calibration standards meet the "AD / RPD validation criteria of \$460%?

V N N/A V N N/A Level IV Only Y N (N/A

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

Ţ			$\overline{}$			_	T	1	T		1								T		Ī	_			$\overline{}$
	Qualifications	(2) streets (c)	3-145 1	11/8/10	5-/45/6		やりとっち	1	3-/NJ/p		J-145/A	THAUSA	1			J-/W/₩									
	Associated Samples	11 + BIKS						_			1. 9203338 MR					2-6 91944>1-MB	4								
	(Limit < 18:0) RT (limit)	( )	( )	( )	( )	( )	( )	( )	( )	(	( )	( )	( )	(	(	( )	( )	)	( )	( )	( )	( )	( )	( )	
	%D / RPD (Limit < 18:0)	8-28	(10)			315.0	47.0	88.7	78.8		9,05	32.6	34.7			28.	,								
	Compound	(4)	F (-)	<del>( ) (</del>	k (-)	d to	F (-)	130	(-) N		(-) A	‡ (4)	† (4)			F (-)									
	Detector/ Column	7.3			1	CH.2			<b>†</b>		Cad.	->	4.2			4.12									
	Standard ID	016 = 1001	(Da)								003 F0301	( CEN )	У			019 11901	(60,)								
	Date	1/20/00	,								7/24/69					7/16/09									
	**																								

LDC # 21494 L17 SDG#:

# VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

2nd Reviewer: Page: 10f 1 Reviewer

METHOD: GC HPLC

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

Were a laboratory control samples (LCS) and laboratory control sample duplicate (LCSD) analyzed for each matrix in this SDG? Were the LCS percent recoveries (%R) and relative percent differences (RPD) within the QC limits?

Level 1440 Only
Y. N. N/A.) Was an LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed?

			(1889)		T					T	T				-	T	T	1			T	T	7	1	Ī		
		Qualification	DA) Tons of																								
		Associated Samples	1 920238-11B																								
	RPD (1 Imite)		82 (140 )	(				)	)	( )	1				~	(	( )										
	LCSD %R (Limits)			)	( )	)			)			(			-	~		_				(		)			, ,
	LCS %R (Limits)	1			· ·				-		<del></del>									(	( )	( )	( )	( )	-	(	
	Compound	צ								-																	
	CS/CSD ID	9203328-165																									
-	1		_	+	+	$\dashv$	-		$\vdash$	-	-	+	+	_		$\vdash$	+	+	-	+	+	+	+	+	-	-	_

## Laboratory Data Consultants, Inc. Data Validation Report

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

Henderson, Nevada

Collection Date: July 13, 2009

LDC Report Date: October 22, 2009

Matrix: Soil

Parameters: Organophosphorus Pesticides

Validation Level: Stage 2B

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 8304613

Sample Identification

SA176-10B SA176009-37B SA176-37B RSAM3-30B

#### 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 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.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- 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.
- Partial Posta 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.

#### \*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
7/25/09	003F0301	1	Dichlorvos Ethoprop Tokuthion	23.2 20.4 21.1	All samples in SDG 8304613	J- (all detects) UJ (all non-detects)	А
7/25/09	003F0301	2	Naled	63.4	All samples in SDG 8304613	J- (all detects) UJ (all non-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:

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 8304613	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	Р

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
6/26/09	010F1001	2	Naled Malathion	47.6	All samples in SDG 8304613	J- (all detects) UJ (all non-detects)	Р
			Maiathion	23.2		J- (all detects) UJ (all non-detects)	

<sup>\*</sup>Added Malathion to table above for 010F1001 Column 2 on 6/26/09

#### III. Blanks

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

Sample FB072109-SO (from SDG 8304616) was identified as a field 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

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

Raw data were not reviewed for this SDG.

#### VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304613	All compounds reported below the PQL.	J (all detects)	Α

Raw data were not reviewed for this SDG.

#### VII. System Performance

Raw data were not reviewed for this SDG.

#### VIII. Overall Assessment of Data

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

#### IX. Field Duplicates

Samples SA176009-37B and SA176-37B were identified as field duplicates. No organophosphorus pesticides were detected in any of the samples.

#### \*Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Data Qualification Summary - SDG 8304613

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304613	SA176-10B SA176009-37B SA176-37B RSAM3-30B	Dichlorvos Ethoprop Tokuthion Naled	J- (all detects) UJ (all non-detects)	А	Continuing calibration (%D) (c)
*8304613	SA176-10B SA176009-37B SA176-37B RSAM3-30B	Naled Disulfoton Malathion	J- (all detects) UJ (all non-detects)	Р	Continuing calibration (ICV %D) (c)
8304613	SA176-10B SA176009-37B SA176-37B RSAM3-30B	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 8304613

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304613

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson**

LDC #: 21494M17	VALIDATION COMPLETENESS WORKSHEET	Date:9/11/09
SDG #: 8304613	Stage 2B	Page: \of \
Laboratory: Test America	_	Reviewer: JVC
METHOD: GC Organophosph	orus Pesticides (EPA SW 846 Method 8141A)	2nd Reviewer:

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

	Validation Area		Comments
1.	Technical holding times		Sampling dates: 7/13/6 G
lla.	Initial calibration	A	% RSD ≤ 20 CEN/ICN ≤ 20 Z
IIb.	Calibration verification/ICV	SW	CON/101 = 20 Z
111.	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	A	b9 C0 20 173-066
IVc.	Laboratory control samples	A	us
V.	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	N	
VIII.	Overall assessment of data	Α	
IX.	Field duplicates	ND	D = 23
X.	Field blanks	MD	FB = TB072109-50 (from 8304616)

Note:

A = Acceptable

N = Not provided/applicable SW = See worksheet

ND = No compounds detected

R = Rinsate FB = Field blank D = Duplicate

TB = Trip blank
EB = Equipment blank

Validated Samples:

Soil

_	3011			
1	SA176-10B	11	21	31
2	SA176009-37B D	12	22	32
3	SA176-37B	13	23	33
4	RSAM3-30B	14	24	34
5	92 04174 - MB	15	25	35
6		16	26	36
7_		17	27	37
8		18	28	38
9		19	29	39
10		20	30	40

otes:

# VALIDATION FINDINGS WORKSHEET

H	2
29/	
METHOD:	

0340					
0.00	8330	8151	(8141	814410	27000
A. Acenaphthene	A. HMX	A. 2.4-D		() Inchia	90218
B. Acenaphthylene	B. RDX		A. Ulchiorvos	V. Fensulfothion	V. Benzene
C. Anthracene	C. 13 S. Trinitrohonness.	B. 2,4-DB	B. Mevinphos	W. Boistar	CC. Toluene
D. Benzo(a)anthracene	1 1 3 Dielecchemicane	C. 2,4,5-T	C. Demeton-O	X. EPN	EE. Ethyl Benzene
E. Benzo(a)ovrene		D. 2,4,5-TP	D. Demeton-S	Y. Azinphos-methyl	SSS. O-Xylene
F. Renzo(h/Giosant	E. Ieuyi	E. Olnoseb	E. Ethoprop	Z. Coumaphos	RRR. MP-Xvlene
	F. Nitrobenzene	F. Dichlorprop	F. Nated	AA. Parathion	GG Total Video
G. Benzo(g,h,i)perylene	G. 2.4.6-Trinitrotoluene	G. Dicamba	G. Suffotep	BR Trickloweste	CC. 10tel Aylene
H. Benzo(k)fluoranthene	H. 4-Amino-2,6-dinitrotoluene	H. Dalapon	H. Phorate		
1. Chrysene	l. 2-Amino-4,6-dinitrotoluene	I. MCPP	1. Dimethoste	cc. Inchiormate	
J. Dibenz(a,h)anthracene	J. 2,4-Dinitrotolune	J. MCPA		Un. Influration	
K. Fluoranthene	K. 2,6-Dinitrotoluene	7	J. Diazinon	EE. Def	
L. Fluorene		n. rentachidrophenoi	K. Disulfoton	FF. Prowi	
N Indept of the Park	L. ATHILOGORAN	L 2,4,5-TP (silvex)	L. Parathion-methyl	GG. Ethion	
alle Jacob Common Commo	M. 3-Nitrotoluene	M. Silvex	M. Ronnel	HH. Tetrachlorvinphos	
N. Naphthalene	N. 4-Nitrotoluene		N. Melathion	II. Sulprofos	
O. Phenanthrene	О.		O. Chlorpyrifos		
P. Pyrene	a.		P Canthon		
Ö	O			KK, Phosmet	
£.			d. Parathion-ethyl	LL. O.O. Triethylphosphorothiogte	sphoro thio ate
Ġ			R. Trichloronate	MM. Famphur	
			S. Merphos	NN. Carbo pheno thion	S
			T. Stirofos		mod le.
			U. Tokuthlon		מו - ייין דמע

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Notes:

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SDG #: Sre Gury LDC # 2494 17

METHOD: \_\_GC \_\_ HPLC

# VALIDATION FINDINGS WORKSHEET Continuing Calibration

Page: Lof / Reviewer:

2nd Reviewer:

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

V N NIA V N NIA Level IV Only X N N/A

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

	Qualifications	C) Prest		184	S-/MT/P	****	7 /45 6	T. 40 (b)	5-747 7-		T- /127/4			7							
	Associated Samples	14. A11 + BIL																			
WD/RPD	erul = 20 c) KT (ilmit)			( )	( )				( )	( )	· ·	( )	( )	(			( )				
-	Compound (Limit 5.3)	6-(1) 187.8		D (-) 82.1	l		F C-) 47.6	A Corp	N (-) 23.7		A (C) 23.7	E (-) 20.4	U (-) 21. 1	FC) 63.4							
Detector	#	2 2				£ 1.2		1			201			Cod. Y							
- ted	$\downarrow$	6/26/09 010 = 100	( M)								7/25/04 003 7 070	(82)	`								
	╁	7																-			_

#### LDC Report# 21494N17

## 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: October 22, 2009

Matrix: Water

Parameters: Organophosphorus Pesticides

Validation Level: Stage 2B

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): 8304614

Sample Identification

TR-8B

#### 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.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. Calibration

#### a. Initial Calibration

Initial calibration of compounds was performed 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.

#### \*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)	Р
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)	Р

<sup>\*</sup>Added Malathion to table above for 010F1001 Column 2 on 6/26/09

#### 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

Raw data were not reviewed for this SDG.

#### VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

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

Raw data were not reviewed for this SDG.

#### VII. System Performance

Raw data were not reviewed for this SDG.

#### VIII. Overall Assessment of Data

Data flags 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)	Р	Continuing calibration (ICV %D) (c)
8304614	TR-8B	All compounds reported below the PQL.	J (all detects)	А	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

### **Tronox Northgate Henderson**

_DC #:21494N17	VALIDATION COMPLETENESS WORKSHEET	Date: 9/11/69
SDG #: 8304614	Stage 2B	Page: <u>  of )</u>
_aboratory: <u>Test America</u>	——————————————————————————————————————	Reviewer: NG
METHOD: GC Organophospho	rus Pesticides (EPA SW 846 Method 8141A)	2nd Reviewer:
S F F		/

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

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 7/14/09
Ila.	Initial calibration	A	2 RSD = 20 2 "
lib.	Calibration verification/ICV	SW	cov/10) 620 }
. 111.	Blanks	Á	
IVa.	Surrogate recovery	A	
iVb.	Matrix spike/Matrix spike duplicates	N	client spec (insufficient sample)
IVc.	Laboratory control samples	A	Client spec (insufficient sample) 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	
Χ.	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:

1	TR-8B	11	21		
2	9198202-MB	12	22	31	
3		13	23	32	
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	

lotes:

# VALIDATION FINDINGS WORKSHEET

H	2
29/	
METHOD:	f

8310 A. Acenaphthene	8330 A HMX	8151	8141	8141(con't)	8021B
B. Acenaphthylene	S BDX	A. 2,4-D	A. Dichlorvos	V. Fensulfothlon	V. Benzena
C. Anthracene	C. 1.3.5-Trinitrobenzene	B. 2,4-0B	B. Mevinphos	W. Bolstar	CC. Toluene
D. Benzo(a)anthracene	D. 1.3-Dinitrobenzene	C. 4,4,5-1	C. Demeton-O	X. EPN	EE. Ethyl Benzene
E. Benzo(a)pyrene	F. Tetoví	U. 2,4,0-1 P	D. Demeton-S	Y. Azinphos-methyl	SSS. O-Xylene
F. Benzo(b)fluoranthena	A March	E. Dinoseb	E. Ethoprop	Z. Coumaphos	RRR. MP-Xylene
	r. wirobenzene	F. Dichlorprop	F. Nafed	AA. Parathion	GG. Total Xylene
o. Denzolg,n, Jperylene	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	
	l. 2-Amino-4,6-dinitrotoluene	I. MCPP	1. Dimethoate	DD. Triffuratio	
J. Dibenz(a,h)anthracene	J. 2,4-Dinitrotolune	J. MCPA	J. Diazinon	5-6	
K. Fluoranthene	K. 2,6-Dinitrotoluene	K. Pentachiorophenol	K. Disulfoton		
	L. 2-Nitrotoluene	L 2,4,5-TP (silvex)	Derathics materia	rr. riowi	
M. Indeno(1,2,3-cd)pyrene	M. 3-Nitrotoluene	13 11	L. rainingii-membi	GG. Ethlon	
	T. A. Miller	III. OIIVEX	M. Ronnel	HH. Tetrachiorvinphos	
	n. 4-Nitrotoluene		N. Malathion	il. Sulprofes	
O. Fnenanthrene	o'.		O. Chlorpyrifos	7. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1.	
	· ai		P. Fenthion	1	
	g		Donathing of the	- 1	
			d. raramion-emyi	LL. 0.00-Triethylphosphorothiogte	sphoro thio ate
			R. Trichloronate	MM. Famphur	
			S. Merphos	NN. Carbophenothion	40
			T. Stirofos	OO. Carbo chen 41	The 1th 1
			U. Tokuthion		181

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Notes:

SDG #: See Gary LDC #: 2494 17

# **VALIDATION FINDINGS WORKSHEET** Continuing Calibration

2nd Reviewer:

Reviewer: 3/2 Page: of

METHOD: \_\_GC\_\_ HPLC

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

X N (N/A)

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

	Č	9	_			1	F		1																
	Qualifications	September 1	J-111 1	tide?	g/ IM/-S	TATAL TATAL	やケブーク	4	3-/45/																
Accordated Country	Associated Samples	A11 + B1K																							
S) RT (limits)	NI (milli)	(	( )	( )	( )	(		•	( )	( )	(		)	)			( )	(	(	(	(	(	)		)
%D/RPD (15.07) RT (Ilmin)	207 1 1 200 ( Table )	778	40.1	83.+	20.6	9/2 lz	47.0	458	79.7																
Compound	ninodiiio	( <del>*</del> )	下(-)	D-t-	k (-)	643	(-) I	<del>(34</del>	(-) N																
Detector/ Column		3			_	Catio		•																	
Standard ID		016 F 1001	(10)								- 0 18+180 h	( <del>-ccv</del> -)													
# Date	ŀ	1,0/77/3	,								7/9/69	,													
<u></u>	ال	L		$\perp$			1		$\perp$						_1	1	1	1	[		<u> </u>	[		- 1	- {

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 21, 2009

LDC Report Date:

September 19, 2009

Matrix:

Water

Parameters:

Organophosphorus Pesticides

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304615

Sample Identification

M-97B

#### 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.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. Calibration

### a. Initial Calibration

Initial calibration of compounds was performed 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 with the following exceptions:

Date	Column	Compound	r²	Associated Samples	Flag	A or P
8/6/09	2	Trichloronate	0.98937	All samples in SDG 8304615	J (all detects) UJ (all non-detects)	А

### 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.

### 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

Raw data were not reviewed for this SDG.

### VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304615	All compounds reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

### VII. System Performance

Raw data were not reviewed for this SDG.

### VIII. Overall Assessment of Data

Data flags 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 8304615

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304615	M-97B	Trichloronate	J (all detects) UJ (all non-detects)	А	Initial calibration (r²) (c)
8304615	M-97B	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (PQL) (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Laboratory Blank Data Qualification Summary - SDG 8304615

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304615

No Sample Data Qualified in this SDG

	21494O17 8304615	<b>VA</b>			_	ESS	derson WORKSI	HEET		Date: <u>1/1/6</u> Page: <u>\</u> of <u>/</u>
	tory: Test America				-					Reviewer: 3V
VIETH	OD: GC Organophosph	orus F	Pesticides (I	EPA SW 8	346 Metho	od 81	41A)		2nd F	Reviewer:
The sa	amples listed below were	revie	ewed for eac	ch of the fo	ollowing v	alida	tion areas. V	alidation fine	dings are	noted in attached
	ion findings worksheets.				J., J., J.	•				
	Validation	Δrea						Comments		
I.	Technical holding times	<i></i>		A	Sampling of	lates:	7/21/09			
IIa.	Initial calibration			W2			£ 20 Z			
IIb.	Calibration verification/ICV			SWA			CN = 50			
III.	Blanks			A						
IVa.	Surrogate recovery			A						
IVb.	Matrix spike/Matrix spike du	plicates	S	N	clie	nt	Spec	Cinsup	pierent	sample)
IVc.	Laboratory control samples			A	L	s/t	> '		<b>V</b>	. /
V.	Target compound identificat	ion		N						
VI.	Compound Quantitation and	I CRQL	.s	N						
VII.	System Performance			N						
VIII.	Overall assessment of data			A						
IX.	Field duplicates			N						
<u> X.</u>	Field blanks			l N						
Note:	A = Acceptable N = Not provided/applicable SW = See worksheet	<del>)</del>	R = Rin	o compound sate eld blank	s detected		D = Duplicat TB = Trip bla EB = Equipr	ank		
Validate	ed Samples:									
1	M-97B	11			21	ļ		31		
2	1205274 MB	12			22	ļ		32		
3		13			23			33_		
4		14			24	-		34_		
5		15			25	-		35		
6		16			26	ļ		36		
7		17			27			37		<u> </u>
8		18			28			38		
9		19			29			39		
10		20			30			40		

Notes:\_

# VALIDATION FINDINGS WORKSHEET

METHOD: /GC HPLC

8310	8330	8151	8141	8141(con't)	8021B
A. Acenaphthene	А. НМХ	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-methyi	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. Nated	AA. Parathion	GG. Total Xylene
G. Benzo(g,h,l)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. Tetrachiorvinphos	
N. Naphthalene	N. 4-Nitrotoluene		N. Maiathion	II. Sulprofos	
O. Phenanthrene	·o		O. Chlorpyrifos	JJ. Thionazin	
P. Pyrene	a:		P. Fenthion	kk, Phosmet	
Ö	a		Q. Parathion-ethyl	LL. 0.00-Triethylphosphorothiogte	osphoro thio ate
.Y.			R. Trichtoronate	MM. Famphur	
ú			S. Merphos	NN. Carbophenothion	00
			T. Stirofos	00. Carbophenothion - methy	on - methy/
			U. Tokuthion		

Notes:

cmpd\_list.wpd

SDG#: Sec large LDC # 21494 017

## VALIDATION FINDINGS WORKSHEET **Initial Calibration**

2nd Reviewer: Reviewer:

Page: of

METHOD: CGC HPLC

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

Was a 5 point calibration curve performed?

N N/A

A N N

Was a linear fit used for evaluation? If yes, the acceptance criteria for each compound is %RSD less than or equal to 20.0%. Was a curve fit used for evaluation? If yes, what was the acceptance criteria used for evaluation? エンスタ, 41

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? N/N/N Y'A N'A

Was initial calibration performed at the required frequency?

Level-IV Only Y N N/A

Were the retention time windows properly established for all compounds?

Were compounds run at the required concentrations in the initial calibrations?

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

Project/Site Name:

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 21 through July 24, 2009

LDC Report Date:

September 23, 2009

Matrix:

Soil/Water

Parameters:

Organophosphorus Pesticides

Validation Level:

Stage 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304616

### Sample Identification

EB072109-SO

FB072109-SO

SA166-10B

SA166-31B

EB072209-SO

SA182-10B

SA182-38B

RSAH3-0.5B

RSAH3009-0.5B

RSAH3-32B

EB072309-SO

SA131-0.5B

SA131009-0.5B

SA131-10B

SA131-27B

EB072409-SO

RSAH3-0.5BMS

RSAH3-0.5BMSD

### Introduction

This data review covers 13 soil samples and 5 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.

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 (r²) was greater than or equal to 0.990 with the following exceptions:

Date	Column	Compound	r²	Associated Samples	Flag	A or P
8/6/09	2	Trichloronate	0.98937	EB072109-SO FB072109-SO 9205274MB	J (all detects) UJ (all non-detects)	A

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% with the following exceptions:

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
8/10/09	003F0301	1	Dichlorvos Mevinphos	56.2 36.1	SA166-10B SA166-31B 9216459MB	J+ (all detects) J+ (all detects)	А
8/10/09	003F0301	1	Naled	44.1	SA166-10B SA166-31B 9216459MB	J- (all detects) UJ (all non-detects)	А

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
8/10/09	003F0301	2	Dichlorvos Mevinphos	81.4 25.9	SA166-10B SA166-31B 9216459MB	J+ (all detects) J+ (all detects)	А
8/10/09	003F0301	2	Naled Parathion-ethyl Bolstar	38.1 42.8 25.4	SA166-10B SA166-31B 9216459MB	J- (all detects) UJ (all non-detects)	А
8/8/09	066F6601	1	Mevinphos	23.9	SA131-0.5B SA131009-0.5B SA131-10B SA131-27B	J+ (all detects)	А
8/8/09	066F6601	2	Dichlorvos	21.5	SA131-0.5B SA131009-0.5B SA131-10B SA131-27B	J+ (all detects)	А

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
8/6/09	010F1001 (19:10)	1	Mevinphos	22.2	SA166-10B SA166-31B SA182-10B SA182-38B RSAH3-0.5B RSAH3-0.5B RSAH3-32B SA131-0.5B SA131-0.5B SA131-10B SA131-27B RSAH3-0.5BMS RSAH3-0.5BMS RSAH3-0.5BMSD 9216459MB	J- (all detects) UJ (all non-detects)	Р

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
8/6/09	010F1001 (19:10)	2	Mevinphos	21.1	SA166-10B SA166-31B SA182-10B SA182-38B RSAH3-0.5B RSAH3-09-0.5B RSAH3-32B SA131-0.5B SA131-0.5B SA131-10B SA131-27B RSAH3-0.5BMS RSAH3-0.5BMS PSAH3-0.5BMSD 9216459MB	J- (all detects) UJ (all non-detects)	Р
8/6/09	010F1001 (19:10)	1	Mevinphos	23.8	EB072209-SO EB072309-SO EB072409-SO 9206112MB	J- (all detects) UJ (all non-detects)	Р
8/6/09	010F1001 (19:10)	2	Mevinphos	21.4	EB072209-SO EB072309-SO EB072409-SO 9206112MB	J- (all detects) UJ (all non-detects)	Р

Retention times (RT) of all compounds in the calibration standards were within QC limits.

### III. Blanks

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

Samples EB072109-SO, EB072209-SO, EB072309-SO, and EB072409-SO were identified as equipment banks. No organophosphorus pesticide contaminants were found in these blanks.

Sample FB072109-SO was identified as a field 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

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.

### VI. Project Quantitation Limit

All project quantitation limits PQLs were within validation criteria.

The sample results for detected compounds from the two columns were within 40% relative percent difference (RPD) with the following exceptions:

Sample	Compound	RPD	Flag	A or P
SA131-0.5B	Coumaphos	58	J (all detects)	А

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304616	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

Samples RSAH3-0.5B and RSAH3009-0.5B and samples SA131-0.5B and SA131009-0.5B were identified as field duplicates. No organophosphorus pesticides were detected in any of the samples with the following exceptions:

	Concentrat	tion (ug/Kg)	DDD.	Difference			
Compound	SA131-0.5B	SA131009-0.5B	RPD (Limits)	Difference (Limits)	Flags	A or P	
Azinphos-methyl	20	14U	-	6 (≤14)	-	•	
Coumaphos	6.1	14U	-	7.9 (≤14)	-	-	

### Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Data Qualification Summary - SDG 8304616

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304616	EB072109-SO FB072109-SO	Trichloronate	J (all detects) UJ (all non-detects)	А	Initial calibration (r²) (c)
8304616	SA166-10B SA166-31B	Dichlorvos Mevinphos	J+ (all detects) J+ (all detects)	А	Continuing calibration (%D) (c)
8304616	SA166-10B SA166-31B	Naled Parathion-ethyl Bolstar	J- (all detects) UJ (all non-detects)	Α	Continuing calibration (%D) (c)
8304616	SA131-0.5B SA131009-0.5B SA131-10B SA131-27B	Mevinphos	J+ (all detects)	А	Continuing calibration (%D) (c)
8304616	SA131-0.5B SA131009-0.5B SA131-10B SA131-27B	Dichlorvos	J+ (all detects)	A	Continuing calibration (%D) (c)
8304616	SA166-10B SA166-31B EB072209-SO SA182-10B SA182-38B RSAH3-0.5B RSAH3-0.5B RSAH3-32B EB072309-SO SA131-0.5B SA131-0.5B SA131-10B SA131-27B EB072409-SO	Mevinphos	J- (all detects) UJ (all non-detects)	P	Continuing calibration (ICV %D) (c)
8304616	SA131-0.5B	Coumaphos	J (all detects)	A	Project Quantitation Limit (PQL) (dc)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304616	EB072109-SO FB072109-SO SA166-10B SA166-31B EB072209-SO SA182-10B SA182-38B RSAH3-0.5B RSAH3-0.5B RSAH3-32B EB072309-SO SA131-0.5B SA131-0.5B SA131-10B SA131-27B EB072409-SO	All compounds reported below the PQL.	J (all detects)	Α	Project Quantitation Limit (PQL) (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Laboratory Blank Data Qualification Summary - SDG 8304616

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304616

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson** T

LDC #: 21494P17	VALIDATION COMPLETENESS WORKSHEE
SDG #: 8304616	Stage 28 4
Laboratory: Test America	

Date:	9/14/09
Page:_	of '
Reviewer:	546
2nd Reviewer:	<u> </u>

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
l.	Technical holding times	A	Sampling dates: 7/21 - 24 /09
lla.	Initial calibration	sΨ	% RSD 6202 rx
llb.	Calibration verification/ICV	SM)	CW/10 620 Z
111.	Blanks	Å	
IVa.	Surrogate recovery	Ą	
IVb.	Matrix spike/Matrix spike duplicates	A	
IVc.	Laboratory control samples	A	las/b
V.	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	SM	
VII.	System Performance	N	
VIII.	Overall assessment of data	A	V
IX.	Field duplicates	SW	$4D_1 = 8, 9$ $D_2 = 12, 13$
X.	Field blanks	ND	FB = 1, 5, 11, 16 $FB = 2$

Note:

A = Acceptable

N = Not provided/applicable SW = See worksheet

★ ND = No compounds detected R = Rinsate

D = Duplicate TB = Trip blank

FB = Field blank EB = Equipment blank

Validated Samples:

		Soil	+	Water					
1 1	EB072109-SO	W	ー 11 多	EB072309-SO	W	21 !	9205274 -MB	31	
<u>-</u> 1	FB072109-SO		12 4	SA131-0.5B	025	22 2	- 9216459- MB	32	
<u>3</u> 7	SA166-10B	5	— 13 <b>4</b>	SA131009-0.5B	D 4	<b>3</b>	9206112- MB	33	
4 >	SA166-31B		14 <b>Y</b>	SA131-10B		24 4	9210468- MB	34	
- 5 3	EB072209-SO	W	15 <b>4</b>	SA131-27B	<u> </u>	25		35	
6	SA182-10B	\$	16 3	EB072409-SO	W	26		36	
- 7 <b>4</b>	SA182-38B		17 <b>4</b>	RSAH3-0.5BMS	5	27		37	
- 8 4	RSAH3-0.5B	pı	18 Y	RSAH3-0.5BMSD		28		38	
9 4	RSAH3009-0.5B	ь,	19		•	29		39	
10 4	RSAH3-32B	$\overline{}$	/ 20			30		40	

Notes:		

### **VALIDATION FINDINGS CHECKLIST**

Page: 1 of 2
Reviewer: 776
2nd Reviewer: 4

Method: GC \_\_\_\_HPLC

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.		Series nas		
II. Initial calibrations				
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?				
W. Continuing calibration				
Was a continuing calibration analyzed daily?				
Were all percent differences (%D) ≤ 15%.0 or percent recoveries 85-115%?	/			
Were all the retention times within the acceptance windows?				
V/Blanks				
Was a method blank associated with every sample in this SDG?				
Was a method blank analyzed for each matrix and concentration?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		_		
Mi-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?	-0.000 T-0.00 T-0.00		_	
WIE Matrik spike/Matrix spikelduplicates				Control of the state of the sta
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 a Paboratory control is amples in the same of the				
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)			1	
within the QC limits?  IX: Regional:Quality Assurance and Quality Control:		, ,		
Were performance evaluation (PE) samples performed?	<u> </u>		<u> </u>	
Were the performance evaluation (PE) samples within the acceptance limits?	<u> </u>		_	

### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 2
Reviewer: JV6
2nd Reviewer: \_\_\_\_\_\_

Validation Area	Yes	No	NA	Findings/Comments
X. (large), compound (destification				
Were the retention times of reported detects within the RT windows?				
XI. Compound quantitation/CRQLs		<b>A</b>		
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII System performances				rant the same of t
System performance was found to be acceptable.		-		
XIII. Överall assessment of data				And Andrew Control of the Control of
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 and A A A A A A A A A A A A A A A A A A A				
Field blanks were identified in this SDG.				
Target compounds were detected in the field blanks.				

# VALIDATION FINDINGS WORKSHEET

METHOD: /GC HPLC

	T	٦		Ī	T	T	T	T	T	$\overline{\top}$	T	T	T	T	T	T	T	T	T	T	T	T
8021B		V. Benzene	CC. Toluene	EE. Ethyl Benzene	SSS. O-Xviene		GG Total Xidana												osphoro Thio ate		no	on - methy/
8141(con't)	V Eanguistation		W. Bolstar	X. EPN	Y. Azinphos-methyl	Z. Coumaphos	AA. Parathion	BB. Trichloronate	CC. Trichlorinate	DD. Trifluralin	EE. Def	FF. Prowl	GG Ethion	HH. Tetrachiorvinnhoe	II. Sulprofos	11 T. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1.		1	기			00. Carbophenothen - methyl
8141	A. Dichlorvos		B. Mevinphos	C. Demeton-O	D. Demeton-S	E. Ethoprop	F. Nated	G. Sulfotep	H. Phorate	1. Dimethoate	J. Diazinon	K. Disulfoton	L. Parathion-methyl	M. Ronnel	N. Maiathion	O. Chlorpyrifos	P. Fenthion	Q. Parathion-ethyl	R. Trichloronate	S. Merohos	T Shrofoe	Tokuthon
8151	A. 2,4-D	00770	D. 4,4-UO	C. 2,4,5-T	D. 2,4,5-TP	E. Dinoseb	F. Dichlorprop	G. Dicamba	H. Dalapon	I. MCPP	J. MCPA	K. Pentachlorophenol	L 2,4,5-TP (silvex)	M. Silvex								
8330	A. HMX	B. RDX		C. 1,3,5-Trinitrobenzene	D. 1,3-Dinitrobenzene	E. Tetryí	F. Nitrobenzene	G. 2.4.6-Trinitrotoluene	H. 4-Amino-2,6-dinitrotoluene	l. 2-Amino-4,6-dinitrotoluene	J. 2,4-Dinitrotolune	K. 2,6-Dinitrotoluene	L. 2-Nitrotoluene	M. 3-Nitrotoluene	N. 4-Nitrotoluene	0,	<b>.</b>	σ				
8310	A. Acenaphthene	B. Acenaphthylene	C Anthracene	0. 21.	D. Benzo(a)anthracene	E. Benzo(a)pyrene	F. Benzo(b)fluoranthene	G. Benzo(g,h,i)perylene	H. Benzo(k)fluoranthene	I. Chrysene	J. Dibenz(a,h)anthracene	K. Fluoranthene	L. Fluorene	M. Indeno(1,2,3-cd)pyrene	N. Naphthalene	O. Phenanthrene	P. Pyrene	Ö.	Α.	·ŝ.		

LDC # 21494 P17

## VALIDATION FINDINGS WORKSHEET Initial Calibration

Page: 1 of 2nd Reviewer: Reviewer:

METHOD: CGC HPLC

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

Was a 5 point calibration curve performed?

Was a linear fit used for evaluation? If yes, the acceptance criteria for each compound is %RSD less than or equal to 20.0%. Was a curve fit used for evaluation? If yes, what was the acceptance criteria used for evaluation?

Was initial calibration performed at the required frequency? 

Did the initial calibration meet the acceptance criteria?

**Level IV Only** Y)N N/A

N/N N/A

Were the retention time windows properly established for all compounds?

Were compounds run at the required concentrations in the initial calibrations?

				(					
	#	Date	Standard ID	Column Detector	Compound	RSD Limit <20%	Associated Samples	Qualifications	
	~	8/00/69	1CAL	2.7	R	0.98937 (2)	(26,99) 12 920S	*	7
	$\dashv$	12:38-15:92	idy			1	1	2/ M2/ (c)	A
	$\dashv$								T
1	+								T
									T
	+								T
	╣								T
	$\dashv$								7
	$\dashv$								Τ
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LDC#: 21494 \$17 SDG #: Su (upry

## VALIDATION FINDINGS WORKSHEET Continuing Calibration

2nd Reviewer:

Page: / of / Reviewer: 3VC

> Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". GC HPLC METHOD:

ntinuing calibration calculation was performed? \_\_\_%D or \_\_\_RPD Were continuing calibration standards analyzed at the required frequencies? What type of continuing calibration calculation was performed? \_

Did the continuing calibration standards meet the %D / RPD validation criteria of <a 3.70%?

Y N N/A Y N N/A 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 <-45.0)	$\%D/RPD$ (Limit $45.0$ ) $(4.0 \lambda)$ RT (limit)		Associated Samples	Qualifications
<b>⊗</b>	8/10/09	003 F0301	3.	(+) Y	56.2	)		3,4,9216459 MB	J+115/4 C
	17:55	(cer)			36.1	)	(	1	
					44.	)	)		J-/NJ/A
			50.2	(+) A	81.4	)	(		3+ drts 1A
				( <del>)</del> \(\frac{1}{2}\)	25.9	)	(		
					38.1	)	(		J-745/A
					42,8	)	)		
$\vdash \downarrow$				(-) W	25.4	)	_		->
			<b>\</b>	,		)			
						)	_		
						)	(		
8,	40/80/8	066 F6681	C. 1	(±) &	23.9	Ú	^	12-,15	J+ 4cts /4
	•	$(\omega \omega)$	CM. 2	(+) ¥	21.5	)	)		
						)	)		
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		And the second of the second o					^	100 000 000 000 000 000 000 000 000 000	
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						)	)		
						)	(		

LDC# 21494 P17 SDG# 84 Green

# VALIDATION FINDINGS WORKSHEET Continuing Calibration

Reviewer: 006

2nd Reviewer:

METHOD: \_\_GC\_\_ HPLC

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

Did the continuing calibration standards meet the %D / RPD validation criteria of <15.9%?

Y N N/A Y N N/A Lexel IV Only

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

CA    C (4)   230.0   1,2,926274 - MB	Date Sta	Sta	Standard ID	Detector/ Column	Compound	%D / RPD (Limit < 15:0)	$\%D/RPD$ (Limit $\le 36.0$ ) RT (limit)	Associated Samples	Qualifications
CALL B (C) 23.8 (C) 1.0 (C) 1.	8/66/69 Q10 F1001	~100 F 100 1		[ ca. 1 _	(t) 9-	230.0		1,2 9205274-MB	3+ dets/ ? (c
C.A. 1787 (1) 1787 (1) 1 2 4 9216 459 MB (2.1) 2 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	_	XQ,		7	(H)	88.8			J-1R 18-
CA.1 B (-) 75.6 ( ) 3.4 9216.459 MB (-) 22.2 ( ) 3.4 9216.459 MB (-) 24.2 ( ) 6-10 12-15,17,18, 18, 18, 18, 18, 18, 18, 18, 18, 18,				City		1787			3+ dees 19
2 B (-) 22.2 ( ) 3,4 9216,459 MB 2 C (+) 24176- ( ) 6-10 12-15,17,18 2 B (-) 23.8 ( ) 5,11,16,9266112 MB 2 C (+) 2012- ( ) 5,11,16,9266112 MB 2 C (+) 2012- ( ) 5,11,16,9266112 MB 2 C (+) 2012- ( ) 5,11,16,9266112 MB 2 C (+) 2012- ( ) 5,11,16,9266112 MB 2 C (+) 2012- ( ) 5,11,16,9266112 MB 2 C (+) 2012- ( ) 5,11,16,9266112 MB 3 C (+) 21.2- ( ) 5,11,16,9266112 MB 4 C (+) 21.4- ( ) 6				7	)	12.6	( )	->	J-/R/P
2							( )		
2 B (-) 22.2 ( ) 3,4 9216,459 MB 2 B (-) 24.2 ( ) 6-10 12-15,17,18, 2 B (-) 21.1 ( ) 92.10 468-MB 2 B (-) 23.8 ( ) 5,11,16,926(12 MB 2 C (1) 201.2 ( ) ) 2 C (1) 25.8 ( ) ) 2 C (2) 43.4 ( ) ) 2 C (3) 221.2 ( ) ) 3 4 92.10 ( ) ) 4 92.10 ( ) ) 4 92.10 ( ) ) 5 11,16,926(12 MB 6 ( ) ) 7 11.2 ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) (							(		
CA. 2 B C 2 24.9 ( ) 6-10 12-15, 17, 18, 18, 19, 19, 19, 19, 19, 19, 19, 19, 19, 19	8/06/69 010 F 100)	001 F 100	_	CA.1		22,2	( )	3.4 9216459 MB	J-145/P
2 B C) 21.4 ( ) 92 10 468-MB ( ) 92 10 4		( M)				211.6-	(	6-10 12-15, 17, 18	The test (2)
2 b (-) 21.1 ( ) )		ر ر		^	( <u>+</u> ) 4	-84.9	(	9210468-MB	2-78-4P-
2		-		٠ ا	( <u>-</u> ) 4	7	(		J-/45/P
					o (F)	7.8.7	( )		2 radop xp.
Ced.   B (-) 23.8					( <del>)</del> 4	93.1	( )	,	**************************************
Col.   B (5) 23.8						-	( )		
Col.   B (-) 23.8 ( ) 5,11,16,926/12 MB ( ) 5,11,16,926/12 MB ( )   5,11,16,92									
2	8/00/04 610 F100	610 F 100		Cal.		23.8	( )	11 16	J-/NJ/P
2	19:10 (100)	(M)				201.3	( )		Frank 18
2					( <del>)</del>	85.0.			J-/R/16
B (-) 21.4 ( ) 3-/m5/					(b) -0	221.3-	( )		J+4645/P
(-) 21.4 ( ) J-/MJ ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) (					163-69	43.1	( )		7/18/8
						4.12	(		J-/WJ/P
								A district of the second of th	
							)		
							)		

LDC# 21 494 P17 SDG# Suckny

GC HPLC

METHOD:

# Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

Page: 1 of

2nd Reviewer: Reviewer:

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

Were CRQLs adjusted for sample dilutions, dry weight factors, etc.?

N N N X

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

Did the percent difference of detected compounds between two columns /detectors < 40%?

If no, please see findings bellow.

	TTT
Qualifications  J drth / A	
SS Limit (< 40%)	
Sample ID	
Compound Name	
	<del>                                     </del>

Comments: See sample calculation verification worksheet for recalculations

LDC#: 21494 P17 SDG #: SCC Corey

METHOD:

# VALIDATION FINDINGS WORKSHEET

Page: Lof \_

Reviewer.\_\_\_\_

Field Duplicates

GC HPLC
Were field duplicate pairs identified in this SDG?
Were target compounds detected in the field duplicate pairs? Y N N/A

Commonne	Concentration ( Mg/kg)	( 49/kg)	%RPD	Qualification
	. [2	(3		Parent only All Samples
χ .	20	N 41	(4.14.Dit)	
2	(2.)			
-				
	Concentration (	(	%RPD	Qualification
Compound			Limit	Parent only / All Samples
	-			
			_	

LDC # 21494 P17 SDG #: See Cover

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

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

> 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

(814/A-1) See (814/A-1) See (814/A-1) O.86756 (814/A-1) O.86756 (814/A-2) O.86014 (814/A-2) O.86014	L				10000	Doogland	700000	Dotelinie	petrode	December
# Standard ID Calibration Compound (3.10 std)  1 1 CAL 8 66 69 Dichlorors (8141A-1)  2 1 CAL 8 66 69 Dichlorors (8141A-1)  3 1 CAL 8 66 69 Dichlorors (8141A-1)  4 Malathian (8141A-1) 0.8675C 6  1.08977  Dichlorors (8141A-1) 0.86014  Andathian (8141A-2) 0.86014  Dichlorors (8141A-2) 0.86014  Dichlorors (8141A-2) 0.86014					Kepodeo	Ferallinated	REDOUGED	NEI AN INGLES		
1 1 CAL 8 6 6 6 9 Dichlarors (814A-2) SEE  2 Malatrian See See See 15:57 Malatrian See 16:00 6 9 Dichlarors (8141A-1) CRESCO 6 17:550-18:24 Phrate (8141A-1) C. 86750 6. 86014  4 Malatrian (8141A-1) C. 86014  Dichlarors (8141A-1) C. 86014  Dichlarors (8141A-2) C. 86014  Dichlarors (8141A-2) C. 86014  Dichlarors (8141A-2) C. 86014  Dichlarors (8141A-2) C. 86014	#		Calibration Date	Compound	CF (2,30 std)	CF (2,00std)	Average CF (initial)	Average CF (initial)	%RSD	%RSD
2 2 3 (CAL 8 (0,69) DICALLUADO (81414-1) C. 84084 3 (CAL 8 (0,69) DICALLUADO (81414-1) C. 86 756 0.86 4 (7:56-18:74) Phorate (81414-1) C. 86014 O. 8 4 (1:56-18:74) Phorate (81414-2) C. 86014 O. 8 4 (1:56-18:74) Phorate (81414-2) C. 86014 O. 8 4 (1:56-18:74) Phorate (81414-2) C. 84014 O. 8 4 (1:56-18:74) Ph	<u> </u>	1221	8/01/69	Dichlaraz (8141A.	268	rr all				
2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	l		/ Ca	phorate	2.03409	2,03409	1-566-1	1. 99571	1:65160	30159 10.65165
2 Phorate (8141A-1) 0.84084  Malathian  14:56-18:34 Phorate (8141A-1) 0.86756 0  Phorate Dichlarops (8141A-1) 0.86014  Phorate Dichlarops (8141A-2) 0.86014  Phorate Dichlarops (8141A-2) 0.84084			4 - 10/-01	Malatina V	ζις	rr calc.				
9 1 CAL 8 (86 69 01 Chlwro (81414-1) 0.86756 0  14:56-18:34 Phorate 1.08977  108977  108977  108977  108977  108977  108977  108977  108977  108977  108977										
3 (AL 8/06/69 DICALLINES (8/4/4-1) 0.86756 0  14:56-18:34 Phorate 1.07/17  4 Pichlunes (8/4/4-2) 0.86014  Phorate 0.84084  Phorate 0.84084	<u> </u>				0.84084		10.548.0		softe El	
3 1 CAL 8 (06 69 DICHLANDS (81414-1) 0.86756 0  1+156-18:24 Phonate 1.07117  1.08977  4 Dichlands (81414-2) 0.86014  Dhorate (81414-2) 0.84084	Ш			Malathim		$\rightarrow$				
14:56-18:34 Phorate 1.07117  Malatione 1.08977  pichlung (81414-2) 0.86014  Phorate 0.84084		727	8/06/69			0.84756	0,84 168	6.84168	3.52069	3 5206
Malatiere J 1.08977  Dichlurs (81414-2) 0.86014  Dhorate 0.84084		14:	76:81-93		1.07117	1.07117	1.0210+	1.63104	8.29536	8 29546
Dichlunes (81414-2) 0.86014  Dhurate 0.84084				malations J	1.08977	1.08977	1.00124	1.00124	\$ 6180	8.6120
+8048.0				(8141	0,86014	0.86014	6,83367	6. 83357	4.86+12	4.84272
رده	<u></u>			Phyrate	0.84084	6. 84084	0.84507	0.84507	13.29300	13. 29294
· · ·	<u> </u>			malarian	200	r calc				

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.

2.0 doc. = 2,0 (phonate & Dichloros) 15 = Tributal phosphate ıl \* \*

INICLC.1SB

= 2.0 ( Malathim ) から 15

21494 217 LDC #: SDG#:

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: 2 of 8 Reviewer: JVC 2nd Reviewer:

> HPLC METHOD: GC\_

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

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

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 (2.ø std)	CF (2:0 std)	Average CF (initial)	Average CF (initial)	%RSD	%RSD
-	1641	50/10/8	Pich Lurvas		1,25265	1.21037	1.21637	STETE.E	3.27372
			Phorace	1.54663	1. 54663	1.48247	1. 48247	7.97042	7.9704x 7.970498
	(10ch)	40.01-05.4	Malathion	1.089.77	1.08977	1.00124	1.00124	8.618	8.618
2			Dichlur vos	1. 16 144	1. 16 144 1. 16144	1.11 64	1.1141	4.50356	1
			physic	1.13537	1.13537	1. 12665	1.12665	16.63095	10.039g
			Medathim	97	41 2	cale. (same	٧٧	other one)	
က									
4									
	-								

Comments: Refer to Initial Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated the other Same raw datas as for all cade. IS wed Tog = S T results.

LDC # 21 444 P17 SDG# In Cone

# Initial Calibration Calculation Verification **VALIDATION FINDINGS WORKSHEET**

Reviewer: NC 2nd Reviewer: \_\_\_\_

Page: 3 of Z

GC EPA SW 846 Method 8141A METHOD:

Dichlorvos Parameter:

Date	Column	Compound	Y Area ratio	X Conc ratio	XvZ
08/06/2009	(8141A-2)	Dichlorvos	0.16666	0.100	
12:58-15:42			0.14234	0.250	
			1.14300	0.500	
			1.86832	1.000	
			2.84139	1.500	
			3.91604	2.000	
			4.54365	2.500	

Regression Output:			Reported	and Association and the Company of t
Constant		-0.04459	<b>=</b> 0	0.00661
Std Err of Y Est		0.19659		
R Squared		0.9900	r2 =	0.99400
No. of Observations		7.00000		Market Control of the
Degrees of Freedom		5.00000		The state of the s
	AND THE RESIDENCE AND THE PROPERTY OF THE PROP			
X Coefficient(s)	1.9024	-0.002208	a	1.91742E+000
Std Err of Coef.	0.087748	0.00		

LDC # 21 494 P17 SDG#

Initial Calibration Calculation Verification **VALIDATION FINDINGS WORKSHEET** 

Page: 24 of 9 Reviewer: 3VC

Reviewer: 20

METHOD:

Malathion

Parameter:

GC EPA SW 846 Method 8141A

Xv2		Who was a second						
X Conc ratio	0.100	0.250	0.500	1.000	1.500	2.000	2.500	
Y Area ratio	0.08761	0.08110	0.66683	1.12291	1.66312	2.22001	2.69919	
Compound	Malathion							
Column	(8141A-2)							
Date	08/06/2009	12:58-15:42		-				

		MANAGAN TO THE THE THE THE THE THE THE THE THE THE	
Regression Output:		Reported	
Constant	-0.02887	100	0.04272
Std Err of Y Est	0.10316		0.0
R Squared	0.99153	r? =	0.00453
No. of Observations	00000 2	1 <b>L.</b>	0000
Degrees of Freedom	00000		
X Coefficient(s)	137 -0.002208	æ	1 126305+000
Std Err of Coef. 0.046046	- W. C.	3	12000 T

LDC # 21 494 P17 SDG#

# Initial Calibration Calculation Verification **VALIDATION FINDINGS WORKSHEET**

Page: \$ of \$

Reviewer: 3VC 2nd Reviewer:

METHOD:

GC EPA SW 846 Method 8141A

Parameter:

Dichlorvos

			<b>&gt;</b>	×	X^2
Date	Column	Compound	Area ratio	Conc ratio	
08/06/2009	(8141A-1)	Dichlorvos	0.17514	0.100	
12:58-15:41			0.44788	0.250	
			1.21081	0.500	
			2.03738	1.000	
			3.11399	1.500	
			4.02458	2.000	
			5.10973	2.500	

Regression Output:		Reported	75
Constant	0.02613	II O	0.01094
Std Err of Y Est	0.09498		
R Squared	0.99782	r2 =	0.99581
No. of Observations	7.00000		Andrews are an analysis of the state of the
Degrees of Freedom	5.00000		
X Coefficient(s)	2.0301 -0.002208	a	2.07952E+000
Std Err of Coef. 0.042397			

214 AG P17 LDC # SDG#

# Initial Calibration Calculation Verification **VALIDATION FINDINGS WORKSHEET**

Page: 6 of 8

Reviewer:  $3 \sqrt{\mathcal{E}}$  2nd Reviewer:

GC EPA SW 846 Method 8141A METHOD:

Parameter:

Malathion

Date	Column	Compound	Υ Area ratio	X Conc ratio	Xv2
08/06/2009	(8141A-1)	Malathion	0.10725	0.100	
12:5K-15:4V			0.27812	0.250	
•			0.73013	0.500	
			1.19616	1.000	
			1.74866	1.500	
			2.23552	2.000	
			2.89440	2.500	

Regression Output:			Reported	
Constant		0.04712	11 0	-0.00342
Std Err of Y Est		0.07124		
R Squared		0.99605	r2 =	0.99300
No. of Observations		7.00000		
Degrees of Freedom		5.00000		
X Coefficient(s)	1.1287	-0.002208	О	1.17261E+000
Std Err of Coef.	0.031800	00.00		

LDC # 21 454 P17 SDG# 54 Cm

VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: 7 of 3

Reviewer: TYZ 2nd Reviewer: \_\_\_

METHOD: GC EPA S

GC EPA SW 846 Method 8141A

Parameter:

Phorate

Date	Column	Compound	Υ Area ratio	X Conc ratio	Xv2
08/06/2009	(8141A-1)	Phorate	0.18690	0.100	
12:58-15:42		•	0.49767	0.250	
			1.18283	0.500	
		,	1.93715	1.000	
			2.80443	1.500	
			3.62669	2.000	
		•	4.57604	2.500	

Regression Output:			Reported	
Constant		0.11374	11 0	-0.05994
Std Err of Y Est		0.10106		
R Squared		0.99682	r2 =	0.99682
No. of Observations		7.00000		
Degrees of Freedom		5.00000		
X Coefficient(s)	1.7854	-0.002208	Ø	1.79112E+000
Std Err of Coef.	0.045109	00.00		

LDC # 21 494 P17 SDG# Ly Cm

VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: 7 of 2

Reviewer: 3

METHOD: GC EPA SW 846 Method 8141A

Parameter:

Malathion

Conc ratio	0.100	0.250	0.500	1.000	1.500	2.000	2.500	
Y Area ratio	0.06172	0.16064	0.35869	0.71697	1.02499	1.35430	1.61327	
Compound	Malathion				The state of the s			
Column	(8141A-2)							
Date	08/06/2009	16: 81-25:31						

Regression Output:			Reported	
Constant		0.02098	. = 0	0.01814
Std Err of Y Est		0.03472		
R Squared		0.99721	r2 =	0.99782
No. of Observations		7.00000		
Degrees of Freedom		5.00000		
X Coefficient(s)	0.6553	-0.002208	Ф	9.45490E-001
Std Err of Coef.	0.015497	00.00		

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

LDC # 21494 P17 See Corer SDG #

# Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

1 of 7 2nd Reviewer:\_ Page: 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	%D	%D
-			- analyzed	what open	1461			
			8,11,16	2				
2	003 F0301	8/10/04	Dichlarus (8141-2)	2.500	3. 9054	3. 90SY	2.52	7, 72
			phorace		2.1678	2.1678	13.3	13.3
			martin 1		2. 26 38	2, 26.38	9.4	9.4
9			DICH [W VUS (8141-1)		4.5358	4.5358	81.4	81,4
			physale		2,4046	2.4096	3.6	3.6
			Martin		2,0320	2.0320	18.7	18.7
4								
		-						

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.

CONCLC.1S

LDC # 21494 P17 SDG#: Le Core

# Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

メダ Reviewer: JV Page: 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

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

Where:

	Table 1				Reported	Recalculated	Reported	Recalculated
	ID Production of the control of the	Date	Compound	Average CF(Ical)/ CCV Conc.	CF/ Conc. CCV	CF/ Conc. CCV	<b>0%</b>	Q%
ر ۹۰	1 652 F 5201	8/57/8	Pichlaruzos (8141-1)	2.500	2,57653	2, 5653	2,6	2.6
		2	phorate		2.4186	2,4186	3,3	3,3
			Malathing 1		2,4924	2.4924	0,3	6.3
	2		Dichlarys (8141-2)		2.8464	2.8464	13.9	13.9
			Phorate		2.4649	2.4649	1.4	1.4
			Malashim t		7804-	2.4084	3.7	3.7
21-51	3 & CF6601	10/80/8	Dichluring (814-1)		2, 8136	2.8136	2,5	12,5
1		-	Phorate		2,5067	2.506.7	6.9	6.3
			Malathim		2.5 656	2.58 36	0.1	6.
	4		bardmang (8141-2)		3.0385	3.0385	2.15	21. 5
<u> 1</u>			phyrate		2. 4347	2.4347	2.6	グラ
			Medathim		2.4180	2.4180	w w	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.

4 P17	Core
2149	Ser
LDC #:	SDG#:

### VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page: l of Reviewer:\_\_\_\_\_

METHOD: \_\_ GC \_\_ 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 Sample ID: #

Surrogate	Columnipetector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
	)			Reported	Recalculated	
Triphenyl phosphat.	C4.7	1. 0	0. 86945	28	8 7	0
Mornita		∀	661250	25	7 12	

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

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

LDC #: 21494 P17 SDG #: See Const

## Matrix Spike/Matrix Spike Duplicates Results Verification VALIDATION FINDINGS WORKSHEET

Page: 1 of 1 Reviewer: 176

2nd Reviewer:

HPLC ပ္ပ METHOD:

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

the following calculation: %Recovery = 100 \* (SSC - SC)/SA

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

SC = Sample concentration

MSD = Matrix spike duplicate

1/8 MS/MSD samples:

RPD =(({SSCMS - SSCMSD} \* 2) / (SSCMS + SSCMSD))\*100

MS/MSD RPD Matrix Spike Duplicate Percent Recovery Percent Recovery Matrix spike Spike Sample Concentration <u>ક</u> Sample Conc. MS/E.) Spike Added Compound

Gasoline         (8015)           Diesel         (8015)           Benzene         (8021B)										
e e										
Methane (RSK-175)										
2,4-D (8151)										
Dinoseb (8151)										
Naphthalene (8310)										
Anthracene (8310)						,				
HMX (8330)										
2,4,6-Trinitrotoluene (8330)										
Dichlur vas (8141) 131	120	0	119	107	=	9	75%	87	0	=
malathin /			87.6	85	67	6.7	رد	وح	3.0	8
					,	,				

of the recalculated results.

LDC# 21464 P17 SDG #: See Gover

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

Reviewer: 376 Page: lof /

2nd Reviewer:

GC\_HPLC METHOD:

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

% Recovery = 100\* (SSC-SC)/SA RPD = 1 LCS - LCSD 1 \* 2/(LCS + LCSD)

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

LCSD = Laboratory control sample duplicate percent recovery

SC = Concentration

4205024 LCS/LCSD samples:

	S	pike	Spiked	Spiked Sample	Ţ,	SJT	Э П	LCSD	SOT	CS/LCSD
Compound	(3)	( 7/5 <sub>11</sub> )		intration i († )	Percent	Percent Recovery	Percent Recovery	Recovery		RPD
	SOT	LCSD	SOT	CSD	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)										
Dichluruss (8141)	4.00	4.00	3.42	3.29	98	86	۲8	8	4.0	4
Malathim /			3,09	3.01	77	77	75	2%	7 2	77

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.

21494 P17 SDG #: See Cover LDC #:

## VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Reviewer: 1/2 2nd Reviewer:

> 29 METHOD:

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

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

Example:

Area or height of the compound to be measured Final Volume of extract A= Area or height of i Fv= Final Volume of e 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

COUMAPhos - 0.08107 001) (IME Compound Name - 0.03646 0.08107) 0.89074 (258b/b) (2836) /( j Zž Concentration/= | Sample ID. find

Qualifications (0,88) Recalculated Results Concentrations Sparie Reported Concentrations Compound Sample ID #

Comments:

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

**Project/Site Name:** 

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 21 through July 22, 2009

LDC Report Date:

September 24, 2009

Matrix:

Soil

Parameters:

Organophosphorus Pesticides

Validation Level:

Stage 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304617

### Sample Identification

SA166-10BSPLP

SA166-10BSPLPDI

SA182-10BSPLP

SA182-10BSPLPDI

SA166-10BSPLPMS

SA166-10BSPLPMSD

SA166-10BSPLPDIMS

SA166-10BSPLPDIMSD

### Introduction

This data review covers 8 soil 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.

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. Calibration

### a. Initial Calibration

Initial calibration of compounds was performed 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.

Retention times (RT) of all compounds in the calibration standards were within QC limits..

### 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

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.

### VI. Project Quantitation Limit

All project quantitation limits PQLs were within validation criteria.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304617	All compounds reported below the PQL.	J (all detects)	А

### VII. System Performance

The system performance was acceptable.

### VIII. Overall Assessment of Data

Data flags 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 8304617

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304617	SA166-10BSPLP SA166-10BSPLPDI SA182-10BSPLP SA182-10BSPLPDI	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (PQL) (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Laboratory Blank Data Qualification Summary - SDG 8304617

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304617

No Sample Data Qualified in this SDG

SDG # Labora <b>METH</b> The sa		ON COMPI St (EPA SW 84	LETENI age 2B 46 Metho	d 8141A)		Date: 4/15/09 Page: 1 of / Reviewer: 5\7\7 2nd Reviewer: indings are noted in attached
	Validation Area				Commen	ts
I.	Technical holding times	A	Sampling d			
lla.	Initial calibration	A		2SD €20 %	22/09 r~	
IIb.	Calibration verification/ICV	NEWA		V/W = 20		
III.	Blanks	A				
IVa.	Surrogate recovery	A				
IVb.	Matrix spike/Matrix spike duplicates	A				
IVc.	Laboratory control samples	A	las			
V.	Target compound identification	N				
VI.	Compound Quantitation and CRQLs	N				
VII.	System Performance	N				
VIII.	Overall assessment of data	A HEVE				
IX.	Field duplicates	N				
X.	Field blanks	N				
Note: Validate	N = Not provided/applicable R = Ri	No compounds on sate Field blank	detected	D = Duplicate TB = Trip blan EB = Equipme		
- 1 s		42 MB	21		31	
	SA166-10BSPLPRE DI 12 7 9 2 /1		22		32	
		, -	<del>-   </del>			

7	901				
1 1	SA166-10BSPLP	11	9211412 MB	21	31
2 2	SA166-10BSPLPRE DI	12 )	92/1418 MB	22	32
3 1	SA182-10BSPLP	13		23	33
4 >	SA182-10BSPLPRE-DI	14		24	34
5	SA166-10BSPLPMS	15		25	35
6	SA166-10BSPLPMSD	16		26	36
7 <b>7</b>	SA166-10BSPLP <del>RE</del> MS	17		27	37
8 <b>7</b>	SA166-10BSPLP <del>RE</del> MSD	18		28	38
9		19		29	39
10		20		30	40

Notes:	

LDC#: 21494 & 17 SDG#: See Cover

### **VALIDATION FINDINGS CHECKLIST**

Page: L of 2
Reviewer: 576
2nd Reviewer: \_\_\_\_\_\_

Method: GC HPLC

Wethod: 2 GC HPLC	T		T	,
Validation Area	Yes	No	NA	Findings/Comments
Ik Technical holding times		-		
All technical holding times were met.	/_			
Cooler temperature criteria was met.			WEST TO	
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?			ļ	
Did the initial calibration meet the curve fit acceptance criteria of ≥ 0.990?			<u> </u>	
Were the RT windows properly established?				
IV Continuing/calibration/				
Was a continuing calibration analyzed daily?				
Were all percent differences (%D) ≤ 15%.0 or percent recoveries 85-115%?				
Were all the retention times within the acceptance windows?				
V:Blanks				
Was a method blank associated with every sample in this SDG?				
Was a method blank analyzed for each matrix and concentration?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/	\	
VI-Surrogate spikes:			<b>4</b>	
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?			/	
Mit Matrix spike/Matrix spike dtrplicates			,	
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?			ļ	
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?			4	

### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 2
Reviewer: 5%
2nd Reviewer: 4

Validation Area	Yes	No	NA	Findings/Comments
X 1 arget compound identification	<i>j</i>			T indings/confidents
Were the retention times of reported detects within the RT windows?				
XI Compound quantilation/GRQLs				
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 or data			•	
Overall assessment of data was found to be acceptable.				
XIV. Pield duplicates are to the state of th				
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.				

21444 Q17 See Cover LDC #: SDG #:

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

- 0 <del>1</del> Page: 2nd Reviewer: 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:

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

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 ( >.º std)	CF (کرک std)	Average CF (initial)	Average CF (initial)	%RSD	%RSD
+	1641	8/02/04	Dichlorvos (8141-1			1,82700	١٠ 8 ١٢٥٠	7.61929	7.61934
			Phorate	Sre	rr cale.				
			marthin J	1.36616	1.36616	1.2860)	1.28601	6.267,9	4.26.780
7			Dichlorys (8141-2		1.68620 1.68126	1.55884	1.55884	5.64768	X.6478Y
			Phurate /throprop	See or	suc				
			Madathian	1.16461	1.16461	22560-1	1.07563	5,2123	5.2/245
ю							-	,	
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

20 0/C) TOCP ij

LDC# 21494 Q17 Ste Corr \*SDC

## Initial Calibration Calculation Verification **VALIDATION FINDINGS WORKSHEET**

Page: 2 of 3

Reviewer: TV 2nd Reviewer:

METHOD:

GC EPA SW 846 Method 8141A

Parameter:

Phorate

Date	Column	Compound	Υ Area ratio	X Conc ratio	X^2
08/03/2009	(8141A-1)	Phorate	0.18145	0.100	
			0.53110	0.250	
			1.04001	0.500	
			2.13734	1.000	
		,	3.09373	1.500	T T T T T T T T T T T T T T T T T T T
			3.53449	2.000	0.000
			4.98370	2.500	

Regression Output:	:		Reported	
Constant		0.07126		-0.02464
Std Err of Y Est		0.20117		A.A.A.
R Squared		0.99000	r2 =	0.99500
No. of Observations		7.00000		
Degrees of Freedom		5.00000		
X Coefficient(s)	1.9112	-0.002208	ß	1.93230E+000
Std Err of Coef.	0.089791	0.00		

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

LDC # 21494 Q 17 SDG#

## Initial Calibration Calculation Verification **VALIDATION FINDINGS WORKSHEET**

Page: 3 of 3

Reviewer: JVC 2nd Reviewer:

GC EPA SW 846 Method 8141A

METHOD:

Phorate/Ethoprop

Parameter:

Z <b>v</b> Z								
X Conc ratio	0.200	0.500	1.000	2.000	3.000	4.000	5.000	
γ Area ratio	0.29174	0.73456	1.50219	3.33981	4.68224	5.72258	7.80615	
Compound	Phorate/Ethoprop							
Column	(8141A-2)							
Date	08/03/2009							

	AND AND AND AND AND AND AND AND AND AND	- The state of the	
Regression Output:		Reported	
Constant	0.02499	II O	0.01406
Std Err of Y Est	0.23214		
R Squared	0.99424	r2 =	0.99671
No. of Observations	7.00000		
Degrees of Freedom	5.00000		
		THE THE PERSON AND ADDRESS AND	
X Coefficient(s)	1.5226 -0.002208	r	1.55380E+000
Std Err of Coef. 0.05	0.051808 0.00		

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

LDC# 21494 Q 17 Sec Coner SDG:#:

## Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

) of 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  $^{\star}$  (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.	ge CF(Ical)/ CF/Conc.	CF/Conc. CCV	Q%	<b>0</b> %
+			V	ang in sect in section		CAL		
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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.

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# VALIDATION FINDINGS WORKSHEET

Surrogate Results Verification

/ of / Reviewer: Page:

METHOD: \_\_GC \_\_ 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

0 Recalculated Percent Recovery 22 Percent Recovery Reported 21% 58552 0. 78998 Surrogate Found Ö Surrogate Spiked 1.00 ColumnDetector 2 3 Tripheny phisphate Surrogate Chlormetos Sample ID: #

Sample ID:

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

Sample ID:

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

LDC #: 2149 4 Ø 17 SDG #: See Cover

## Matrix Spike/Matrix Spike Duplicates Results Verification VALIDATION FINDINGS WORKSHEET

Page: 1 of 1 Reviewer: 31/6

2nd Reviewer:\_\_\_

METHOD: \_\_\_ GC \_\_ HPLC
The percent recoveries (%R) and relative percent differences (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation: %Recovery = 100 \* (SSC - SC)/SA

Where

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

SC = Sample concentration

RPD =(((SSCMS - SSCMSD) \* 2) / (SSCMS + SSCMSD))\*100

MS/MSD samples:\_\_

MSD = Matrix spike duplicate

	Spike	ike	Sample	Spike (	Spike Sample	Matris	Matrix spike	Matrix Spik	Matrix Spike Duplicate	MS/MSD	(SD
Compound	7 48	17	1 1/5/1	, MS /	( MS/L )	Percent	Percent Recovery	Percent F	Percent Recovery	RPD	٥
	MS	MSD		MS	MSD	Reported	Recatc.	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)											
Dichler vos (8141)	4,00	4.0	Ö	3.07	3.36	77	77	<b>2</b>	4×	9.6	6
Malani m	$\rightarrow$		7	2.77	2,92	89	(%	2	73	7.3	-

Spike Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC # 21494 @17 SDG #: See Guer

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

Page: lof / Reviewer: 316 2nd Reviewer:

> GC HPLC METHOD:

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

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

SC = Concentration

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

LCSD = Laboratory control sample duplicate percent recovery

9211418 LCS/LCSD samples:

	S	Spike	Spiked	Spiked Sample	Ĩ	SOT	ΓC	rcsD	SOT	LCS/LCSD
Compound	) ( N/	( 1/ 5M)	/ SM )	ntration /L)	Percent	Percent Recovery	Percent I	Percent Recovery	æ	RPD
· · · · · · · · · · · · · · · · · · ·	LCS	CSD	rcs	GSOT	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)										
Dichlarvas (8141)	4.00	\$	3.27	NA	87	87	-			1
Malathin 1			5.77	>	69	59				

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.

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## VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page: \\_of\_ Reviewer: 2nd Reviewer:

> 00 METHOD:

NA	/ \ \ \ \	
	_	

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

Example:

Sample ID.

(A)(Fv)(Df)	(RF)(Vs or Ws)(%S/100)
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
Vs= Initial volume of the sample
Ws= Initial weight of the sample
Ws= Percent Solid

Concentration =

Compound Name \_

#±	Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications

SAMPCALew.wpd

Comments:

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

Project/Site Name:

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 29, 2009

LDC Report Date:

September 18, 2009

Matrix:

Soil/Water

Parameters:

Organophosphorus Pesticides

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304620

### Sample Identification

RSAU4-20

RSAU4-50

FB072909-SO

SA73-0.5B

SA73-30B

RSAU4-20MS

RSAU4-20MSD

### Introduction

This data review covers 6 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 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.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. Calibration

### a. Initial Calibration

Initial calibration of compounds was performed 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.

### 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
8/9/09	048F4801	1	Naled	24.3	RSAU4-20 RSAU4-50 SA73-0.5B SA73-30B RSAU4-20MS RSAU4-20MSD 9215329MB	J- (all detects) UJ (all non-detects)	А
8/9/09	048F4801	2	Dichlorvos	24.8	RSAU4-20 RSAU4-50 SA73-0.5B SA73-30B RSAU4-20MS RSAU4-20MSD 9215329MB	J+ (all detects)	А

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
8/6/09	010F1001	1	Mevinphos	22.2	All samples in SDG 8304620	J- (all detects) UJ (all non-detects)	Р
8/6/09	010F1001	2	Mevinphos	21.1	All samples in SDG 8304620	J- (all detects) UJ (all non-detects)	Р

### III. Blanks

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

Sample FB072909-SO was identified as a field 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

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

### c. Laboratory Control Samples

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

### V. Target Compound Identification

Raw data were not reviewed for this SDG.

### VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304620	All compounds reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

### **VII. System Performance**

Raw data were not reviewed for this SDG.

### VIII. Overall Assessment of Data

Data flags 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 8304620

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304620	RSAU4-20 RSAU4-50 SA73-0.5B SA73-30B	Naled	J- (all detects) UJ (all non-detects)	A	Continuing calibration (%D) (c)
8304620	RSAU4-20 RSAU4-50 SA73-0.5B SA73-30B	Dichlorvos	J+ (all detects)	А	Continuing calibration (%D) (c)
8304620	RSAU4-20 RSAU4-50 FB072909-SO SA73-0.5B SA73-30B	Mevinphos	J- (all detects) UJ (all non-detects)	Р	Continuing calibration (ICV %D) (c)
8304620	RSAU4-20 RSAU4-50 FB072909-SO SA73-0.5B SA73-30B	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 8304620

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304620

No Sample Data Qualified in this SDG

	imples listed below were ion findings worksheets.  Validation			EPA SW 84			areas. Valid	dation findi	ngs are noted in attacl
		1164		A :	Sampling	dates: 7			
<u>l.</u>	Technical holding times			7T 1			202	·~	
lla.	Initial calibration			SW			€20}	-	
llb.	Calibration verification/ICV			A		<i>y</i> , , , , ,	- 0		
<u>   .</u>	Blanks			A					
√a.	Surrogate recovery	liantas		A					
Vb Vc.	Matrix spike/Matrix spike dur Laboratory control samples	meates		A	us	(p			
vc. V.	Target compound identificati	on		N					
<u>v.</u> √I.	Compound Quantitation and			N					
/II.	System Performance	ONGLS		N				***************************************	
/III.	Overall assessment of data			A					
IX.	Field duplicates			1)					
X.	Field blanks			ND	₹₿	- 3			
te:	A = Acceptable N = Not provided/applicable SW = See worksheet ed Samples: 2 m 1 +	Wat	R = Rin FB = Fi	o compounds sate eld blank	detected	Т	= Duplicate B = Trip blank B = Equipmen		
-	RSAU4-20 S	11 /	9215	329 MB	21			31	
	RSAU4-20 S RSAU4-50	12	9215		3 22			32	
X	FB072909-SO W	13			23			33	
$\overline{}$	SA73-0.5B S	14			24			34	
	SA73-30B	15			25			35	
1	RSAU4-20MS	16			26			36	
	RSAU4-20MSD	17			27			37	
		18			28			38	
		19			29			39	
,		20			30			40	

# VALIDATION FINDINGS WORKSHEET

METHOD: GC HPLC

8310	8330	8151	(8141	8141(Con't)	8021B
A. Acenaphthene	А. НМХ	A. 2,4-D	A. Dichlorvos	V. Fensulfothion	V. Benzene
B. Acenaphthylene	B. RDX	B. 2,4-DB	B. Mevinphos	W. Bolstar	
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	l. 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	Ġ		P. Fenthlon	l	
Ö	8		Q. Parathion-ethyl	0	ocebaro this ate
R.			R. Trichloronate	1	
·s			S. Merphos		20
			T. Stirofos		en - methul
			U. Tokuthion		

omud liet

LDC # 21 49 4 717 SDG #: Ly Con HPLC

METHOD: \_\_GC\_

## **VALIDATION FINDINGS WORKSHEET** Continuing Calibration

Page: 1 of / Reviewer: 3/C

2nd Reviewer:

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

Y N/A

Did the continuing calibration standards meet the %D / RPD validation criteria of <15.9%?

Level JA-Qnly Y N (N/A

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

Qualifications	5-/W5/P (C)	Frankston 1	56/R/p	5- /W3 /P	1+ 405X	2/8/Z		J-145 /A	J+Arts A					-										
Associated Samples	AN + BIKS					<b>\</b>		12, 4-7, 9215329 MB				9 4215362 MB												
	)		)	) (	)	)	)	)   ',	)			٤ (	)	)	_	_	)	)	)	)	)	)	)	
RT (limit)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)	)
2 6 2																								
%D / RPD (Limit ≤45.8)°26 &	22.2	211.6	84.9	21.1	-218:1	93.1		34.2	24.8															
Compound	(-)q	(1) 3	4	(-) A	0	b es		(~) 7	A (4)															
Defector/ (Colump	1,25		->	3.				E. 1	4.12															
Standard ID	010 F1001	CMI		A Company of the Comp				048 F4801	(25)	\ 		014 1401	(484)											
Date	8/01/04							8/69/8	/			2/2/20	1///											
#																								

### LDC Report# 21494U17

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

**Project/Site Name:** 

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

July 31, 2009

LDC Report Date:

October 6, 2009

Matrix:

Soil

Parameters:

Organophosphorus Pesticides

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304621

### Sample Identification

RSAU4-20BSPLP

RSAU4-20BSPLPRE

RSAU4-20BSPLPDI

RSAU4-50BSPLP

RSAU4-50BSPLPDI

RSAU4-20BSPLPREMS

RSAU4-20BSPLPREMSD

### Introduction

This data review covers 7 soil 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.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

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

Sample	Analyte	Total Days From Sample Collection Until Analysis	Required Holding Time (in Days) From Sample Collection Until Analysis	Flag	A or P
RSAU4-20BSPLPRE RSAU4-20BSPLPREMS RSAU4-20BSPLPREMSD	All TCL compounds	21	14	J- (all detects) UJ (all non-detects)	A

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<sup>2</sup>) was greater than or equal to 0.990.

### 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
8/14/09	040F4001	1	Naled	42.5	RSAU4-20BSPLP RSAU4-20BSPLPDI RSAU4-50BSPLP RSAU4-50BSPLPDI 9224150MB 9221012MB	J- (all detects) UJ (all non-detects)	А

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
8/14/09	040F4001	2	Naled	42.7	RSAU4-20BSPLP RSAU4-20BSPLPDI RSAU4-50BSPLP RSAU4-50BSPLPDI 9224150MB 9221012MB	J- (all detects) UJ (all non-detects)	А
8/25/09	003F0301	1.	Dichlorvos Mevinphos	22.7 25.5	RSAU4-20BSPLPRE RSAU4-20BSPLPREMS RSAU4-20BSPLPREMSD 9232166MB	J+ (all detects) J+ (all detects)	А
8/25/09	003F0301	1	Naled	21.8	RSAU4-20BSPLPRE RSAU4-20BSPLPREMS RSAU4-20BSPLPREMSD 9232166MB	J- (all detects) UJ (all non-detects)	А
8/25/09	003F0301	2	Dichlorvos Mevinphos	31.4 29.3	RSAU4-20BSPLPRE RSAU4-20BSPLPREMS RSAU4-20BSPLPREMSD 9232166MB	J+ (all detects) J+ (all detects)	А

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
8/6/09	010F1001	1	Mevinphos	22.2	RSAU4-20BSPLPRE RSAU4-20BSPLPREMS RSAU4-20BSPLPREMSD 9232166MB	J- (all detects) UJ (all non-detects)	Р
8/6/09	010F1001	2	Mevinphos	21.1	RSAU4-20BSPLPRE RSAU4-20BSPLPREMS RSAU4-20BSPLPREMSD 9232166MB	J- (all detects) UJ (all non-detects)	Р

### 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 with the following exceptions:

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
RSAU4-20BSP	LP Not specified	Triphenylphosphate Chlormefos	38 (60-154) 31 (49-171)	All TCL compounds	J- (all detects) UJ (all non-detects)	А

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

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

#### c. Laboratory Control Samples

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

#### V. Target Compound Identification

Raw data were not reviewed for this SDG.

#### VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304621	All compounds reported below the PQL.	J (all detects)	À

Raw data were not reviewed for this SDG.

#### VII. System Performance

Raw data were not reviewed for this SDG.

#### \*VIII. Overall Assessment of Data

The overall assessment of data was acceptable. In the case where more than one result was reported for an individual sample, the least technically acceptable results were rejected as follows:

Sample	Compound	*Flag	A or P
RSAU4-20BSPLPRE	All TCL compounds	Х	А

<sup>\*</sup>Corrected Flag in above table from "R" to "X".

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 8304621

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304621	RSAU4-20BSPLPRE	All TCL compounds	J- (all detects) UJ (all non-detects)	А	Technical holding times (h)
8304621	RSAU4-20BSPLP RSAU4-20BSPLPDI RSAU4-50BSPLP RSAU4-50BSPLPDI RSAU4-20BSPLPRE	Naled	J- (all detects) UJ (all non-detects)	A	Continuing calibration (%D) (c)
8304621	RSAU4-20BSPLPRE	Dichlorvos Mevinphos	J+ (all detects) J+ (all detects)	А	Continuing calibration (%D) (c)
8304621	RSAU4-20BSPLPRE	Mevinphos	J- (all detects) UJ (all non-detects)	Р	Continuing calibration (ICV %D) (c)
8304621	RSAU4-20BSPLP	All TCL compounds	J- (all detects) UJ (all non-detects)	А	Surrogate recovery (%R) (s)
8304621	RSAU4-20BSPLP RSAU4-20BSPLPRE RSAU4-20BSPLPDI RSAU4-50BSPLP RSAU4-50BSPLPDI	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (PQL) (sp)
*8304621	RSAU4-20BSPLPRE	All TCL compounds	*X	А	Overall assessment of data (0)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Laboratory Blank Data Qualification Summary - SDG 8304621

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304621

No Sample Data Qualified in this SDG

DC #:	21494U17	VΔ		nox non N COMP			RKSHEE	Г	Date: 9/14
	8304621	<b>V</b> /\(\)			tage 2B		11110111111	•	Page: 1 of 1
	tory: Test America			Ŭ	tugo 20				Reviewer: 316
	2D- 00 0	F	Dontinidas (	TDA CIA/ C	AG Motha	~ 04.41.A.\			2nd Reviewer:
EIH	OD: GC Organophospho	orus F	esticides (	EPA SVV C	946 Metric	u 6141A)			1
		revie	ewed for ea	ch of the fo	ollowing v	alidation a	reas. Validat	ion findiı	ngs are noted in attache
lidati	on findings worksheets.								
	Validation	A = 0.0		·			Comi	ments	
		ALEA		Civi	Camaniina	7	31/09	ТСПП	
<u>l,                                     </u>	Technical holding times			SW	Sampling of		07 r2	···	
IIa.	Initial calibration				i				
llb.	Calibration verification/ICV			SW		WIN	2201		
III.	Blanks			A					
IVa.	Surrogate recovery			2M			,		
IVb.	Matrix spike/Matrix spike du	olicates	<u> </u>	<u> </u>		- /-			
IVc.	Laboratory control samples			A	LC.	s/p			
V.	Target compound identificat	ion		N	ļ				
VI.	Compound Quantitation and	CRQL	.s	N					
VII.	System Performance			N		·			
VIII.	Overall assessment of data		· · · · · · · · · · · · · · · · · · ·	SW					
IX.	Field duplicates			N.					
Х	Field blanks			N	<u> </u>				
ote:	A = Acceptable N = Not provided/applicable SW = See worksheet		R = Rir	lo compound: nsate ield blank		TB =	Duplicate - Trip blank - Equipment bla	ank	1978 to the second seco
lidate	d Samples:	l							
- 1 F	RSAU4-20BSPLP	17 1	92241	50 MB	21			31	
_	RSAU4-20BSPLPRE1	12 7		66 MB	22			32	
	RSAU4-20BSPLP <del>RE2</del> DI	13 3		120 MB				33	
- 1	RSAU4-50BSPLP	14			24			34	
	RSAU4-50BSPLPRE DI	15			25			35	
	RSAU4 - 20 BSPLPRE M				26			36	
P	l '	017			27			37	······································
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SDG #: 124 12-1 LDC#: 24494 117

## **VALIDATION FINDINGS WORKSHEET Technical Holding Times**

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

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

Sample ID Matrix Preserved					
Soi)	ed Sampling Date	Extraction date	Analysis date	Total # of Days	Qualifier
	7/21/69	8/21/09	8/25/09	2	5-/45/A Ch)
			, ,		
	•				
		12 VST 10 12 J 11 12 J			
			-		
	-				
			-		

# **TECHNICAL HOLDING TIME CRITERIA**

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

Both within 14 days of sample collection.

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

Soils:

Water: Soil: EXTRACTABLES:

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

HTNew.wpd

LDC # 21 494 117 SDG #: METHOD: GC HPLC

## VALIDATION FINDINGS WORKSHEET Continuing Calibration

Page: 76f Reviewer:

2nd Reviewer:

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

What type of continuing calibration standards analyzed at the required frequencies?

Not applicable questions are identified as "N/A".

Not applicable questions are identified as "N/A".

Do a substitution standards analyzed at the solidation criteria of 46.0%?

Level IV Only
Y N (V/A)

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

I																		T	耳	T		Ţ	7	Ŋ	$\neg$	
	Qualifications	-3+ dets/p (c	\$ 600 F	5+ det 19	J-1R/P			J-105/2	5+ Ath 6	J=19/10	J-145/P	2+AUB-F	J. 18/10			J-14JA	>			J+445A		J-757 /A	0+A02/A	>		
	Associated Samples	1, 3-5 9224150 MB	922 1042 MB					2, 6,7, 9232166 MB								1, 3-5, 9224 150MB	9221012MB			2,6 7 9332166 MB						
ŀ		_	_	7	^	_	_	`	(	_	_	_	_	_	_	^	(	$\hat{}$	<u> </u>		^		)			
	%D/RPD (Limit ≤ 45:0] (4-20 ユン RT (limit)	)		_	\ \ \	)		)	)	_			)	Ú	·	)	)	)	)	)	)	)	)	)	)	
	%D / RPD (Limit < 45:0)	197 0	74.9	238.0	di.s		100 A	22.2	211.6	84.0	21,1	1.812	92.			42,5	42.7	,		22.7	20,52	21.8	¥.14	29.3		
	Compound	(÷)	1		D (-)			B (-)	600	4	B 5-7	140	4			3	F (-)			(+) ¥	B (+)	£	i			
	Detector/ Column	3	1	2,75				3			23.					7,7	?			- :5		>	2.7			
	Standard ID	\mu\mu\mu\mu\mu\mu\mu\mu\mu\mu\mu\mu\mu\	Sign					010 7 1001	CMD							040 F 400 I	(ca)			003 F0301	( B)					
	Date	1	_					8/06/69								8/14/09				8 65/09						
	*																					L				

## **VALIDATION FINDINDS WORKSHEET** Surrogate Recovery

Page: 1 of

Reviewer:\_\_\_\_\_

LDC#: 21 444 417 SDG#: 24 (W)~

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

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

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

#	Sample ID		Detector/ Column	Surrogate Compound		%R (Limits)				Quali	Qualifications	
		Nat	Sheer	×		) 86	451-09	24 )	- り	グリノーク	/A	(٤)
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						)		(				
						)		)				
	Surrogate Compound	Į.	Surre	Surrogate Compound		Surrogate Compound		Surrogate Compound	punodu			
<	Chlorobenzene (CBZ)		0	Octacosane	Σ	Benzo(e)Pyrene	S	1-Chloro-3-Nitrobenzene	euezuego.	<b>&gt;</b>	Tetrachloro-m- xylene	900
В	4-Bromofluorobenzene (BFB)	FB)	н	Ortho-Terphenyl	z	Terphenyl-D14	⊦	3,4-Dinitrotoluene	luene	N	Chlormates	9
O	a,a,a-Trifluorotoluene		t Flu	Fluorobenzene (FBZ)	٥	Decachlorobiphenyl (DCB)	ם	Tripentyltin	ţį	1		
۵	Bromochiorobenene		· 4	n-Triacontane	۵	1-methylnaphthalene	7	Tri-n-propyttin	vitin	+		
ш	1,4-Dichlorobutane		×	Hexacosane	o	Dichlorophenyl Acetic Acid (DCAA)	3	Tributyl Phosphate	sphate			
F	1.4-Difluorobenzene (DFB)	(B)	_	Bromobenzene	R	4-Nitrophenol	×	Triphenyl Phosphate	sphate			

LDC #: 21 464 M17 SDG #: 54 Cm

# VALIDATION FINDINGS WORKSHEET Overall Assessment of Data

Page: lof L Reviewer: We

METHOD: \_\_GC \_\_HPLC

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

All available information pertaining to the data were reviewed using professional judgement to compliment the determination of the overall quality of the data.

Y N/A Was the overall quality and usability of the data acceptable?

	Sample #		Acconisted Samples	Qualifications
#	Compound name—	TH T. Z. Z.+W		X K/A (0)
		Est property of		
			4	
Comments:	nents:			

OVRNew.wpd

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

Project/Site Name:

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

August 5, 2009

LDC Report Date:

September 24, 2009

Matrix:

Water

Parameters:

Organophosphorus Pesticides

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304622

Sample Identification

FB080409-GW

#### 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.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. Calibration

#### a. Initial Calibration

Initial calibration of compounds was performed 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.

#### 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
8/9/09	031F3101	1	Naled Merphos	21.9 23.8	All samples in SDG 8304622	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	А
8/9/09	031F3101	2	Dichlorvos	21.4	All samples in SDG 8304622	J+ (all detects)	А

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
8/6/09	010F1001	1	Mevinphos	22.2	All samples in SDG 8304622	J- (all detects) UJ (all non-detects)	Р

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
8/6/09	010F1001	2	Mevinphos	21.1	All samples in SDG 8304622	J- (all detects) UJ (all non-detects)	Р

#### III. Blanks

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

Sample FB080409-GW was identified as a field 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 were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

#### c. Laboratory Control Samples

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

#### V. Target Compound Identification

Raw data were not reviewed for this SDG.

#### VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304622	All compounds reported below the PQL.	J (all detects)	А

Raw data were not reviewed for this SDG.

#### VII. System Performance

Raw data were not reviewed for this SDG.

#### VIII. Overall Assessment of Data

Data flags 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 8304622

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304622	FB080409-GW	Naled Merphos	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	A	Continuing calibration (%D) (c)
8304622	FB080409-GW	Dichlorvos	J+ (all detects)	А	Continuing calibration (%D) (c)
8304622	FB080409-GW	Mevinphos	J- (all detects) UJ (all non-detects)	Р	Continuing calibration (ICV %D) (c)
8304622	FB080409-GW	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (PQL) (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Laboratory Blank Data Qualification Summary - SDG 8304622

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304622

No Sample Data Qualified in this SDG

### Tropov Northasta Handarson

_DC #:	21494V17			LETENESS	WORKSHEET		Date: 9/v/
SDG#	: 8304622	<del>-</del> -		Stage 2B			Date: 9/11/ Page:of Reviewer:V d Reviewer:
_abora	tory: <u>Test America</u>			_			Reviewer: 3V
VIETH:	<b>OD:</b> GC Organophosph	norus Pesticides (	EPA SW 8	346 Method 81	41A)	200	a Reviewer:
						<i>c</i> : ::	/
ne sa /alidati	imples listed below were ion findings worksheets	e reviewed for ea	ch of the f	ollowing valida	tion areas. Validation	on findings a	e noted in attache
	<b>J</b>						
	Validation	Area			Comm	ents	
<b>I</b> ,	Technical holding times		A	Sampling dates:	8/05/09 rr		
lla.	Initial calibration		A	7, RSI	o rr		
IIb.	Calibration verification/ICV		SW)	4	W = 20 Z		
III.	Blanks		A				
IVa.	Surrogate recovery		À				
IVb.	Matrix spike/Matrix spike du	ıplicates	N	Clien	t sker		
IVc.	Laboratory control samples		A	4CS /	t spec		
V.	Target compound identifica		N				
VI.	Compound Quantitation and		N				
VII.	System Performance		N				
VIII.	Overall assessment of data	ı	A				
IX.	Field duplicates		l N				
Χ.	Field blanks		ND	FB =	1		
Note:	A = Acceptable N = Not provided/applicable SW = See worksheet	e R = Rin	o compound:	s detected	D = Duplicate TB = Trip blank EB = Equipment blan	k	
/alidated	d Samples: Nater						
1 F	- B080409GW	11		21		31	
2	9217511 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	
lotes:							

# VALIDATION FINDINGS WORKSHEET

METHOD: /GC HPLC

8310	8330	8151	8141	8141(Con't)	8021B
A. Acenaphthene	А. НМХ	A. 2,4-D	A. Dichlorvos	V. Fensulfothion	V. Benzene
B. Acenaphthylene	B. RDX	B. 2,4-DB	B. Mevinphos	W Boleter	1:
C. Anthracene	C. 1,3,5-Trinitrobenzene	C. 2,4,5-T	C. Demeton-O	X. EPN	
D. Benzo(a)anthracene	D. 1,3-Dinitrobenzene	D. 2,4,5-TP	D. Demeton-S	Y. Azinphos-methyi	SSS. O-Xylene
E. Benzo(a)pyrene	E. Tetryl	E. Dinoseb	E. Ethoprop	Z. Coumaphos	
F. Benzo(b)fluoranthene	F. Nitrobenzene	F. Dichlorprop	F. Naled	AA. Parathion	GG. Total Xylene
G. Benzo(g,h,l)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	
1. Chrysene	l. 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. Tetrachiorvinphos	
N. Naphthalene	N. 4-Nitrotoluene		N. Malathion	II. Sulprofos	
O. Phenanthrene	o.		O. Chlorpyrlfos	JJ. Thionazin	
P. Pyrene	a:		P. Fenthion		
Ġ	ď		Q. Parathion-ethyi	0	sephera this ate
Ж.			R. Trichloronate	1	
S.			S. Merphos		20
			T. Stirofos		ion - methul
			U. Tokuthion		

LDC #: 21 494 V 17 See Cons SDG #:

## VALIDATION FINDINGS WORKSHEET Continuing Calibration

2nd Reviewer:

Page: \of \ Reviewer: 3V6

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

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

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

Were the retention times for all calibrated compounds within their respective acceptance windows? Level W-Only Y N/N/A

Ouslifications	J 4 - 17	49	18/18	3+ 4th/p	18/10	-/WJ/P		5-65/4	-	Jacts/4														
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Associated Samples	왕																							
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RT (limit)				)	)	)	)	)	)	)	)	~	_	)	)	)		)	)	)	)	)	)	,
%D / RPD (Limit < 15.0)	22.22	31.6	2	218.2	42.1	21.1		21.9	23.8	21.4														
<b> </b>	15	$  \  $		Y																				
Compound	11	ا د د	() d	J	Y d	( <del>-</del> ) 8		F (-)	( <del>-</del> ) S	A (+)	•													
Detector/ Column	3			とって		<b>~</b>		Cot		2 2														
								3																
Standard ID	010#1001	(%)						051F3161	(101)	,														
Date	8/06/09	<u></u>						8/69/69	-															
*																								

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

**Project/Site Name:** 

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

August 3 through August 5, 2009

LDC Report Date:

September 23, 2009

Matrix:

Soil/Water

Parameters:

Organophosphorus Pesticides

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304623

Sample Identification

FB080309-SO RSAU5-0.5B RSAU5-50B RSAU5-0.5BMS RSAU5-0.5BMSD

#### 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 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.

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- 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 (r²) was greater than or equal to 0.990.

#### 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
8/9/09	031F3101	1	Naled Merphos	21.9 23.8	FB080309-SO 9217511MB	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	А
8/9/09	031F3101	2	Dichlorvos	21.4	FB080309-SO 9217511MB	J+ (all detects)	А
8/14/09	055F5501	1	Naled EPN	43.9 27.1	RSAU5-0.5B RSAU5-50B RSAU5-0.5BMS RSAU5-0.5BMSD 9223449MB	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	A
8/14/09	055F5501	2	Naled Dimethoate Parathion-ethyl EPN	63.9 20.6 20.3 28.7	RSAU5-0.5B RSAU5-50B RSAU5-0.5BMS RSAU5-0.5BMSD 9223449MB	J- (all detects) UJ (all non-detects)	А

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
8/6/09	010F1001	1	Mevinphos	22.2	FB080309-SO 9217511MB	J- (all detects) UJ (all non-detects)	Р
8/6/09	010F1001	2	Mevinphos	21.1	FB080309-SO 9217511MB	J- (all detects) UJ (all non-detects)	Р

#### III. Blanks

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

Sample FB080309-SO was identified as a field 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

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

#### c. Laboratory Control Samples

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

#### V. Target Compound Identification

Raw data were not reviewed for this SDG.

#### VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304623	All compounds reported below the PQL.	J (all detects)	Α

Raw data were not reviewed for this SDG.

#### **VII. System Performance**

Raw data were not reviewed for this SDG.

#### VIII. Overall Assessment of Data

Data flags 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 8304623

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304623	FB080309-SO	Naled Merphos	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	A	Continuing calibration (%D) (c)
8304623	FB080309-SO	Dichlorvos	J+ (all detects)	А	Continuing calibration (%D) (c)
8304623	RSAU5-0.5B RSAU5-50B	Naled Dimethoate Parathion-ethyl EPN	J- (all detects) UJ (all non-detects)	А	Continuing calibration (%D) (c)
8304623	FB080309-SO	Mevinphos	J- (all detects) UJ (all non-detects)	Р	Continuing calibration (ICV %D) (c)
8304623	FB080309-SO RSAU5-0.5B RSAU5-50B	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (PQL) (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Laboratory Blank Data Qualification Summary - SDG 8304623

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304623

No Sample Data Qualified in this SDG

SDG #	: 21494W17 t: 8304623 atory: Test America	Tro	N COMP	thgate Hend LETENESS tage 2B		≣ET	Date: 9/14/o Page: 1 of 1 Reviewer: 3/6 2nd Reviewer: 1
METH	OD: GC Organophospho	orus Pesticides (	EPA SW 8	346 Method 814	I1A)		]
	amples listed below were tion findings worksheets.		ch of the fo	ollowing validati	on areas. Val	idation findi	ngs are noted in attached
	Validation	Area			C	omments	
I.	Technical holding times		A	Sampling dates:	8/63 -		
lla.	Initial calibration		A	% RED	€ 20 %	rγ	
IIb.	Calibration verification/ICV		SW	ICV/COV =	20 %		
111.	Blanks		A				
IVa.	Surrogate recovery		A				
IVb.	Matrix spike/Matrix spike du	olicates	A				
IVc.	Laboratory control samples		Ą	45/0			
V.	Target compound identificat	ion	N				
VI.	Compound Quantitation and	CRQLs	N				
VII.	System Performance		N				
VIII.	Overall assessment of data		A				
IX.	Field duplicates		N N				
X.	Field blanks		ND	FB =	,		
Note: /alidate	A = Acceptable N = Not provided/applicable SW = See worksheet ed Samples:	R = Rin	o compounds sate eld blank	s detected	D = Duplicate TB = Trip blank EB = Equipmen		
	FB080309-SO V	11		21		31	
	RSAU5-0.5B Ş	12		22		32	
_	RSAU5-50B	13		23		33	
	RSAU5-0.5BMS	14		24		34	
1	RSAU5-0.5BMSD	15		25		35	
6	9217511 MB	16		26		36	
7 7	9223449 MB	17		27		37	
8	-	18		28		38	
9		19		29		39	
10		20		30		40	

Notes:\_

# VALIDATION FINDINGS WORKSHEET

METHOD: /GC HPLC

8310	8330	8151	(8141	8141(Con't)	8021B
A. Acenaphthene	А. НМХ	A. 2,4-D	A. Dichiorvos	V. Fensulfothion	V. Benzene
B. Acenaphthylene	B. RDX	B. 2,4-DB	B. Mevinphos	W. Bolstar	
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-methyi	1
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,l)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	1. 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. Tetrachiorvinphos	
N. Naphthalene	N. 4-Nitrotoluene		N. Maiathion	II. Sulprofos	
O. Phenanthrene	0.		O. Chlorpyrlfos	JJ. Thionazin	
P. Pyrene	من		P. Fenthion	1	
Ö	ď		Q. Parathion-ethyl	LL. 000-Triethulphocophorothicate	ocahara this ate
R.			R. Trichloronate	1 .	
S.			S. Merphos		20
			T. Stirofos	00. Carbophenothing - methu	on - methul
			U. Tokuthion		

cmpd\_list.wpd

LDC #: 21494 WI7 St. Car SDG #: GC HPLC

METHOD:

**VALIDATION FINDINGS WORKSHEET** 

Continuing Calibration

Reviewer: JV6

Page: of

2nd Reviewer:

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

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

	<del></del>	`	_						 	_=			-	 		<del></del>	<del>-</del> 7		-		_			\
Qualifications	∥ ~	7 7 7 7 7 7	ST PROPER	1/4/2	J-105/P	Freeze	**************************************		J+ 10t5/R	J-/45/10	I + deters	1-/8/6	,	J-/101 A		J+046/4			J-/NJ/A					
Associated Samples	1071CB								2-5 9223449 MB			<i></i>		1, 9217511 MB					2-5 9223449 MB					
%D / RPD (Limit < 15.0 € 20 ∑) RT (limit)			( )	( )	( )			( )							(	( )	( )	(						
%D/RPD	, cc	7.7	211.6	84.9	٧.)	218, 1	93.1		8.791	74.9	2 38.8	96.3		21,9	23.8	21.4			43.9		63.9	20,6	20,3	7.8.7
Compound	Sumodino 6	(-) A		d	(-) B		444		C (+)	7		1 ^		(J) H	5	$\sim$			( <del>)</del> =	(-) X	(A 7	ĬÓ	(F) Ø	(-) X
Detector/		3	-	->	7 3				3		475	:ـــا		3	ì	۲. ح			7.75		W. 7			>
Standard ID	Stalldaid ID	010 F 100/	(3)						1001 F 1001					1914-160					055 F5501	(col >				
	# Date	8/06/09		7					8/2/60	2001				0/00/0					8/4 69					
'	Ш	- 1																						

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

Project/Site Name:

Tronox LLC Facility, 2009 Phase B Investigation,

Henderson, Nevada

**Collection Date:** 

August 3 through August 5, 2009

LDC Report Date:

September 19, 2009

Matrix:

Soil

Parameters:

Organophosphorus Pesticides

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): 8304624

#### Sample Identification

RSAJ3-10BSPLP

RSAJ3-10BSPLPDI

RSAJ3-29BSPLP

RSAJ3-29BSPLPDI

RSAU5-0.5BSPLP

RSAU5-0.5BSPLPDI

RSAU5-0.5BSPLPMS

RSAU5-0.5BSPLPMSD

#### Introduction

This data review covers 8 soil 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.

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

#### II. Calibration

#### a. Initial Calibration

Initial calibration of compounds was performed 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.

#### 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
8/14/09	040F4001	1	Naled	42.5	RSAJ3-10BSPLP RSAJ3-10BSPLPDI RSAJ3-29BSPLP RSAJ3-29BSPLPDI 9224150MB 9221012MB	J- (all detects) UJ (all non-detects)	A
8/14/09	040F4001	2	Naled	42.7	RSAJ3-10BSPLP RSAJ3-10BSPLPDI RSAJ3-29BSPLP RSAJ3-29BSPLPDI 9224150MB 9221012MB	J- (all detects) UJ (all non-detects)	А
8/14/09	055F5501	1	Naled EPN	43.9 27.1	RSAU5-0.5BSPLP RSAU5-0.5BSPLPDI RSAU5-0.5BSPLPMS RSAU5-0.5BSPLPMSD 9224512MB 9224510MB	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	А

Date	Standard	Column	Compound	%D	Associated Samples	Flag	A or P
8/14/09	055F5501	2	Naled	47.5	RSAU5-0.5BSPLP RSAU5-0.5BSPLPDI RSAU5-0.5BSPLPMS RSAU5-0.5BSPLPMSD 9224512MB 9224510MB	J- (all detects) UJ (all non-detects)	А

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

#### III. Blanks

Method blanks were reviewed for each matrix as applicable. No 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

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

#### c. Laboratory Control Samples

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

#### V. Target Compound Identification

Raw data were not reviewed for this SDG.

#### VI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 8304624	All compounds reported below the PQL.	J (all detects)	A

Raw data were not reviewed for this SDG.

#### VII. System Performance

Raw data were not reviewed for this SDG.

#### VIII. Overall Assessment of Data

Data flags 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 8304624

SDG	Sample	Compound	Flag	A or P	Reason (Code)
8304624	RSAJ3-10BSPLP RSAJ3-10BSPLPDI RSAJ3-29BSPLP RSAJ3-29BSPLPDI	Naled	J- (all detects) UJ (all non-detects)	А	Continuing calibration (%D) (c)
8304624	RSAU5-0.5BSPLP RSAU5-0.5BSPLPDI	Naled EPN	J- (all detects) UJ (all non-detects) J- (all detects) UJ (all non-detects)	А	Continuing calibration (%D) (c)
8304624	RSAJ3-10BSPLP RSAJ3-10BSPLPDI RSAJ3-29BSPLP RSAJ3-29BSPLPDI RSAU5-0.5BSPLP RSAU5-0.5BSPLPDI	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (PQL) (sp)

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Laboratory Blank Data Qualification Summary - SDG 8304624

No Sample Data Qualified in this SDG

Tronox LLC Facility, 2009 Phase B Investigation, Henderson, Nevada Organophosphorus Pesticides - Field Blank Data Qualification Summary - SDG 8304624

No Sample Data Qualified in this SDG

#### **Tronox Northgate Henderson** RKSHEET

LDC #: 21494X17	_ VALIDATION COMPLETENESS WO
SDG #: 8304624	Stage 2B
Laboratory: Test America	
METHOD: GC Organophosph	norus Pesticides (EPA SW 846 Method 8141A)

Date:	9/15/09
Page:_	of
Reviewer:	JVG
2nd Reviewer:	

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: 8/63 - 05 / 09
lla.	Initial calibration	A	Sampling dates: $8/03 - 05/09$ $7_2$ RJD $\leq 20$ $2$ $CCV/16V \leq 20$ $2$
IIb.	Calibration verification/ICV	W2	CCV/1W = 20 Z
111.	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	A	
lVc.	Laboratory control samples	A	ucs /b
V.	Target compound identification	N	
VI.	Compound Quantitation and CRQLs	N	
VII.	System Performance	N	
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N	
X.	Field blanks	V\$	#B = FB 072+89 SD frm 83046+6

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

TB = Trip blank
EB = Equipment blank

Validated Samples:

Soil

	341						
1	RSAJ3-10B SPLP	111	9224150 MB	21		31	
2 >	RSAJ3-10BRE SPLP DI	12 7	9221012 MB	22		32	
3 <b>l</b>	RSAJ3-29B SPLP	13 >	9224517 MB	23		33	
4 >	RSAJ3-29BRE SPLP DI	14 4	922 d 5 10 MB	24		34	
5 <b>3</b>	RSAU5-0.5BSPLP	15		25		35	
6 <b>4</b>	RSAU5-0.5BSPLPRE DI	16		26		36	
7 3	RSAU5-0.5BSPLPMS	17		27	·	37	
8 <b>3</b>	RSAU5-0.5BSPLPMSD	18		28		38	
9		19		29		39	
10		20		30		40	

Notes:			

# 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. Fenerifothion	
B. Acenaphthylene	B. RDX	B. 2.4.DB	O Marie Land		
C. Anthracege			D. Mevinphos	W. Bolstar	CC. Toluene
	C. 1,3,3-1 finitrobenzene	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-Xviene
F. Benzo(b)fluoranthene	F. Mitrobenzene	F. Dichlorprop	F. Naled	AA. Parathion	GG. Total Xviene
G. Benzo(g,h,i)perylene	G. 2.4.6-Trinitrotoluene	G. Dicemba	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	1. 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 (slivex)	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	J.T. This age in	
P. Pyrene	ď		P. Fenthion	1	
Ö	g		Q. Parathion-ethyl	1 6	1
R.			R. Trichloronate		25 prioro into att
S.			S. Merphos	1	200
			T. Stirofos	l	m+4/
			U. Tokuthlon		160.11

cmpd

Notes:\_\_

LDC #: 21 494 X17 SDG #: 54 Care

# VALIDATION FINDINGS WORKSHEET Continuing Calibration

Page: 1 of 1 Reviewer: **3/6** 

2nd Reviewer:

METHOD: \_\_GC\_\_ HPLC

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

Did the continuing calibration standards meet the %D / RPD validation criteria of <45.0%?

Y N N/A Y N N/A Y N N/A

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

	ন													Z	r				-					
Qualifications	J. 12400 (C)	J-/MJ/P	J+dens/P	J-1R1P	-		J-147/4	-				J-/MA		<b>→</b>										
Associated Samples	A11 + BIKC						1-4 922450 MB	9 221012 1015				5-8 923 4512 MB	GUYACIO MB		•									
	(	)	)	<u> </u>	(	(	_		(	)	)	)	)	)	^	_		)	)	(	)	)	)	
20 D RT (limit)	)			)	)	)	)	)	)	)	)	)	)	)	)	J	·	)	)	)	)	)	)	)
%D / RPD (Limit ≤ 45:0)	197.8	74.9	288,0	96.3			42.5	42.7	<u> </u>			43.9	27,	274										
Compound	(+) 0	(-) 9	_	Jan A			(-) ヨ	(-) <del>d</del>	1			(-) ≠	$\mathcal{L} \mathcal{L}$	(-) =										
Detector/ Column	7.5	/	ノイ	7			Cal. 1	270				- 3	1	7.3										
Standard ID	010 + 1001	\x\3\-					040 F400 1	(cos)	\			055 1450	(CCN)											
Date	8/13/00	Z					8/4/09					8/4/09												
#	8											(2)												