



## LABORATORY DATA CONSULTANTS, INC.

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GSI Pacific, Inc.  
181 S. Kukui Street  
Honolulu, HI 96813  
ATTN: Ms. Sonia Shjegstad

June 18, 2014

SUBJECT: Makua Military Reservation, Oahu, HI, Data Validation

Dear Ms. Shjegstad

Enclosed are the final validation reports for the fractions listed below. These SDGs were received on May 21, 2014. Attachment 1 is a summary of the samples that were reviewed for each analysis.

### LDC Project #31852:

#### SDG #

006611, 006612, 006615,  
006616/006617, 10265141,  
320-6209-1, 320-6210-1

#### Fraction

Volatiles, Chlorinated Pesticides, Dioxins/Dibenzofurans,  
Perchlorate

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

- Final Supplemental Marine Resources Study Sampling and Analysis Plan at Makua Military Reservation, Oahu, Hawaii, August 2013
- Final Draft Version of the U.S. Department of Defense, and Department of Energy, Consolidated Quality Systems Manual, for Environmental Laboratories, Version 5.0, March 2013
- USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, June 2008
- 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,

Andrew Kong  
Project Manager/Senior Chemist



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

**Project/Site Name:** Makua Military Reservation  
**Collection Date:** January 13, 2014  
**LDC Report Date:** June 3, 2014  
**Matrix:** Tissue  
**Parameters:** Volatiles  
**Validation Level:** EPA Level III  
**Laboratory:** ARDL, Inc.  
**Sample Delivery Group (SDG):** 006611

**Sample Identification**

MAK101O  
MAK102O  
MAK103O  
MAK104O  
MAK105O  
MAK106O  
MAK107O  
MAK108O  
MAK104OMS  
MAK104OMSD

## Introduction

This data review covers 10 tissue samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8260B for Volatiles.

This review follows the Final Supplemental Marine Resources Study Sampling and Analysis Plan at Makua Military Reservation, Oahu, Hawaii (August 2013), the Final Draft Version of the U.S. Department of Defense (DoD) and Department of Energy (DoE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0 (March 2013), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008).

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

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

The following are definitions of the data qualifiers:

- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

## **I. Technical Holding Times**

All technical holding time requirements were met.

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

## **II. GC/MS Instrument Performance Check**

Instrument performance was checked at 12 hour intervals. All ion abundance requirements were met.

## **III. Initial Calibration**

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 15.0% for all compounds.

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

## **IV. Continuing Calibration**

Continuing calibration was performed at the required frequencies.

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

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

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

## **V. Blanks**

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

No field blanks were identified in this SDG.

## **VI. Surrogate Spikes**

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

**VII. Matrix Spike/Matrix Spike Duplicates**

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

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

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

**IX. Regional Quality Assurance and Quality Control**

Not applicable.

**X. Internal Standards**

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

**XI. Target Compound Identifications**

Raw data were not reviewed for this SDG.

**XII. Compound Quantitation**

Raw data were not reviewed for this SDG.

**XIII. Tentatively Identified Compounds (TICs)**

Raw data were not reviewed for this SDG.

**XIV. System Performance**

Raw data were not reviewed for this SDG.

**XV. Overall Assessment of Data**

The laboratory indicated sample Trip Blank was received frozen and all vials shattered, therefore no results were provided.

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

**XVI. Field Duplicates**

No field duplicates were identified in this SDG.

**Makua Military Reservation  
Volatiles - Data Qualification Summary - SDG 006611**

No Sample Data Qualified in this SDG

**Makua Military Reservation  
Volatiles - Laboratory Blank Data Qualification Summary - SDG 006611**

No Sample Data Qualified in this SDG

**Makua Military Reservation  
Volatiles - Field Blank Data Qualification Summary - SDG 006611**

No Sample Data Qualified in this SDG

LDC #: 31852A1

## VALIDATION COMPLETENESS WORKSHEET

Date: 6/13/14

SDG #: 006611

Level III

Page: 1 of 1

Laboratory: ARDL, Inc.

Reviewer: JVG

2nd Reviewer: [Signature]

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

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: 1/13/14
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	A	% RSD $\leq$ 15%
IV.	Continuing calibration/ICV	A	CV/ICV $\leq$ 20%
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	LCS
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	N	
XII.	Compound quantitation/RL/LOQ/LODs	N	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	N	
XV.	Overall assessment of data	A	Laboratory indicated TB samples received frozen and shattered, therefore no results provided
XVI.	Field duplicates	N	
XVII.	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:

Tissue

1	MAK101O	11	006611-04B1	21		31	
2	MAK102O	12		22		32	
3	MAK103O	13		23		33	
4	MAK104O	14		24		34	
5	MAK105O	15		25		35	
6	MAK106O	16		26		36	
7	MAK107O	17		27		37	
8	MAK108O	18		28		38	
9	MAK104OMS	19		29		39	
10	MAK104OMSD	20		30		40	

BTEX, Styrene, 1,2,4-TMB

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

**Project/Site Name:** Makua Military Reservation  
**Collection Date:** January 13 through January 14, 2014  
**LDC Report Date:** June 3, 2014  
**Matrix:** Tissue  
**Parameters:** Volatiles  
**Validation Level:** EPA Level III  
**Laboratory:** ARDL, Inc.  
**Sample Delivery Group (SDG):** 006612

**Sample Identification**

MAK102C  
MAK103C  
MAK104C  
MAK105C  
MAK106C  
MAK107C  
MAK108C  
MAK110C  
MAK110CMS  
MAK110CMSD

## Introduction

This data review covers 10 tissue samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8260B for Volatiles.

This review follows the Final Supplemental Marine Resources Study Sampling and Analysis Plan at Makua Military Reservation, Oahu, Hawaii (August 2013), the Final Draft Version of the U.S. Department of Defense (DoD) and Department of Energy (DoE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0 (March 2013), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008).

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

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

The following are definitions of the data qualifiers:

- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

## **I. Technical Holding Times**

All technical holding time requirements were met.

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

## **II. GC/MS Instrument Performance Check**

Instrument performance was checked at 12 hour intervals. All ion abundance requirements were met.

## **III. Initial Calibration**

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 15.0% for all compounds.

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

## **IV. Continuing Calibration**

Continuing calibration was performed at the required frequencies.

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

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

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

## **V. Blanks**

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

No field blanks were identified in this SDG.

## **VI. Surrogate Spikes**

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

## **VII. Matrix Spike/Matrix Spike Duplicates**

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

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

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

## **IX. Regional Quality Assurance and Quality Control**

Not applicable.

## **X. Internal Standards**

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

## **XI. Target Compound Identifications**

Raw data were not reviewed for this SDG.

## **XII. Compound Quantitation**

Raw data were not reviewed for this SDG.

## **XIII. Tentatively Identified Compounds (TICs)**

Raw data were not reviewed for this SDG.

## **XIV. System Performance**

Raw data were not reviewed for this SDG.

## **XV. Overall Assessment of Data**

The laboratory indicated sample Trip Blank was received frozen and all vials shattered, therefore no results were provided.

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

## **XVI. Field Duplicates**

No field duplicates were identified in this SDG.

**Makua Military Reservation  
Volatiles - Data Qualification Summary - SDG 006612**

No Sample Data Qualified in this SDG

**Makua Military Reservation  
Volatiles - Laboratory Blank Data Qualification Summary - SDG 006612**

No Sample Data Qualified in this SDG

**Makua Military Reservation  
Volatiles - Field Blank Data Qualification Summary - SDG 006612**

No Sample Data Qualified in this SDG

LDC #: 31852B1

## VALIDATION COMPLETENESS WORKSHEET

Date: 6/02/14

SDG #: 006612

Level III

Page: 1 of 1

Laboratory: ARDL, Inc.

Reviewer: JVG

2nd Reviewer:

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

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 - 14 / 14
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	A	2 RSD $\leq$ 15%
IV.	Continuing calibration/ICV	A	CCV/ICV $\leq$ 20%
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	VCS
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	N	
XII.	Compound quantitation/RL/LOQ/LODs	N	
XIII.	Tentatively identified compounds (TICs)	N	
XIV.	System performance	N	
XV.	Overall assessment of data	A	Laboratory indicated TB samples received frozen and shattered, therefore no results provided.
XVI.	Field duplicates	N	
XVII.	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:

TISSUE

1	MAK102C	11	006612-0832	21		31	
2	MAK103C	12		22		32	
3	MAK104C	13		23		33	
4	MAK105C	14		24		34	
5	MAK106C	15		25		35	
6	MAK107C	16		26		36	
7	MAK108C	17		27		37	
8	MAK110C	18		28		38	
9	MAK110CMS	19		29		39	
10	MAK110CMSD	20		30		40	

BTEX, Styrene, 1,2,4-TMB

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** Makua Military Reservation  
**Collection Date:** January 31 through February 11, 2014  
**LDC Report Date:** June 4, 2014  
**Matrix:** Tissue  
**Parameters:** Chlorinated Pesticides  
**Validation Level:** EPA Level III  
**Laboratory:** ARDL, Inc.  
**Sample Delivery Group (SDG):** 006615

### Sample Identification

MAK111C  
MAK113C  
MAK114C  
MAK115C  
MAK116C  
MAK117C  
MAK118C  
MAK120C  
MAK116CMS  
MAK116CMSD

## Introduction

This data review covers 10 tissue samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8081B for Chlorinated Pesticides.

This review follows the Final Supplemental Marine Resources Study Sampling and Analysis Plan at Makua Military Reservation, Oahu, Hawaii (August 2013), the Final Draft Version of the U.S. Department of Defense (DoD) and Department of Energy (DoE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0 (March 2013), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008).

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

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

The following are definitions of the data qualifiers:

- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

## **I. Technical Holding Times**

All technical holding time requirements were met.

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

## **II. GC Instrument Performance Check**

Instrument performance was checked at 12 hour intervals.

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

## **III. Initial Calibration**

Initial calibration was performed as required by the method.

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

In the case where the laboratory used a calibration curve to evaluate the compounds, all coefficients of determination ( $r^2$ ) were greater than or equal to 0.990 .

## **IV. Continuing Calibration**

Continuing calibration was performed at required frequencies.

The percent differences (%D) were less than or equal to 20.0% for all compounds.

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

## **V. Blanks**

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

No field blanks were identified in this SDG.

## **VI. Surrogate Spikes**

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

## **VII. Matrix Spike/Matrix Spike Duplicates**

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

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

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

## **IX. Regional Quality Assurance and Quality Control**

Not applicable.

## **X. Florisil Cartridge Check**

Florisil cleanup was not reviewed in this SDG.

## **XI. GPC Calibration**

GPC cleanup was not reviewed in this SDG.

## **XII. Target Compound Identification**

Raw data were not reviewed for this SDG.

## **XIII. Compound Quantitation**

Raw data were not reviewed for this SDG.

## **XIV. Overall Assessment of Data**

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

## **XV. Field Duplicates**

No field duplicates were identified in this SDG.

**Makua Military Reservation  
Chlorinated Pesticides - Data Qualification Summary - SDG 006615**

No Sample Data Qualified in this SDG

**Makua Military Reservation  
Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG  
006615**

No Sample Data Qualified in this SDG

**Makua Military Reservation  
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG 006615**

No Sample Data Qualified in this SDG

LDC #: 31852C3a  
 SDG #: 6615  
 Laboratory: ARDL, Inc.

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III

Date: 6-2-14  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

**METHOD:** GC Chlorinated Pesticides (EPA SW 846 Method 8081B)

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: 1/31/14 → 2/11/14
II.	GC Instrument Performance Check	A	
III.	Initial calibration	A	≤ 20 / Y <sub>2</sub>
IV.	Continuing calibration/ICV	A	CCV/ICV ≤ 20
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	LCS
IX.	Regional quality assurance and quality control	N	
X.	Florisil cartridge check	N	
XI.	GPC Calibration	N	
XII.	Target compound identification	N	
XIII.	Compound quantitation/RL/LOQ/LODs	N	
XIV.	Overall assessment of data	A	
XV.	Field duplicates	N	
XVI.	Field blanks	N	

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

Validated Samples: tissue

1	MAK111C	11		21		31	
2	MAK113C	12		22		32	
3	MAK114C	13		23		33	
4	MAK115C	14		24		34	
5	MAK116C	15		25		35	
6	MAK117C	16		26		36	
7	MAK118C	17		27		37	
8	MAK120C	18		28		38	
9	MAK116CMS	19	B10175	29		39	
10	MAK116CMSD	20		30		40	

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** Makua Military Reservation  
**Collection Date:** January 31 through February 11, 2014  
**LDC Report Date:** June 5, 2014  
**Matrix:** Tissue  
**Parameters:** Chlorinated Pesticides  
**Validation Level:** EPA Level III  
**Laboratory:** ARDL, Inc.  
**Sample Delivery Group (SDG):** 006616/006617

### Sample Identification

MAK120L  
MAK121L  
MAK122L  
MAK123L  
MAK124L  
MAK126L  
MAK127L  
MAK128L  
MAK116L  
MAK117L  
MAK118L  
MAK123LMS  
MAK123LMSD

## Introduction

This data review covers 13 tissue samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8081B for Chlorinated Pesticides.

This review follows the Final Supplemental Marine Resources Study Sampling and Analysis Plan at Makua Military Reservation, Oahu, Hawaii (August 2013), the Final Draft Version of the U.S. Department of Defense (DoD) and Department of Energy (DoE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0 (March 2013), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008).

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

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

The following are definitions of the data qualifiers:

- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

## **I. Technical Holding Times**

All technical holding time requirements were met.

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

## **II. GC Instrument Performance Check**

Instrument performance was checked at 12 hour intervals.

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

## **III. Initial Calibration**

Initial calibration was performed as required by the method.

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

In the case where the laboratory used a calibration curve to evaluate the compounds, all coefficients of determination ( $r^2$ ) were greater than or equal to 0.990 .

## **IV. Continuing Calibration**

Continuing calibration was performed at required frequencies.

The percent differences (%D) were less than or equal to 20.0% for all compounds.

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

## **V. Blanks**

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

No field blanks were identified in this SDG.

## **VI. Surrogate Spikes**

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

Sample	Column	Surrogate	%R (Limits)	Compound	Flag	A or P
MAK120L	STX-CLP	Tetrachloro-m-xylene	8 (25-125)	All TCL compounds	J (all detects) R (all non-detects)	P
MAK121L	STX-CLP	Tetrachloro-m-xylene	22 (25-125)	All TCL compounds	J (all detects) UJ (all non-detects)	P
MAK124L	STX-CLP	Tetrachloro-m-xylene	19 (25-125)	All TCL compounds	J (all detects) UJ (all non-detects)	P

### VII. Matrix Spike/Matrix Spike Duplicates

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

Spike ID (Associated Samples)	Compound	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
MAK123LMS/MSD (MAK123L)	alpha-BHC	124.8 (45-113)	-	-	J (all detects)	A
	Heptachlor	128 (52-114)	-	-	J (all detects)	
	Heptachlor epoxide	123.7 (53-110)	-	-	J (all detects)	
	beta-BHC	-	121.9 (50-111)	-	J (all detects)	

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Florisil Cartridge Check

Florisil cleanup was not reviewed in this SDG.

### XI. GPC Calibration

GPC cleanup was not reviewed in this SDG.

### XII. Target Compound Identification

Raw data were not reviewed for this SDG.

### **XIII. Compound Quantitation**

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

<b>Sample</b>	<b>Compound</b>	<b>RPD</b>	<b>Flag</b>	<b>A or P</b>
MAK117L	alpha-BHC	87	J (all detects)	A

Raw data were not reviewed for this SDG.

### **XIV. Overall Assessment of Data**

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

### **XV. Field Duplicates**

No field duplicates were identified in this SDG.

**Makua Military Reservation  
Chlorinated Pesticides - Data Qualification Summary - SDG 006616/006617**

SDG	Sample	Compound	Flag	A or P	Reason
006616/006617	MAK121L MAK124L	All TCL compounds	J (all detects) UJ (all non-detects)	P	Surrogate spikes (%R)
006616/006617	MAK120L	All TCL compounds	J (all detects) R (all non-detects)	P	Surrogate spikes (%R)
006616/006617	MAK123L	alpha-BHC Heptachlor Heptachlor epoxide beta-BHC	J (all detects) J (all detects) J (all detects) J (all detects)	A	Matrix spike/Matrix spike duplicate (%R)
006616/006617	MAK117L	alpha-BHC	J (all detects)	A	Compound quantitation (RPD)

**Makua Military Reservation  
Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG 006616/006617**

No Sample Data Qualified in this SDG

**Makua Military Reservation  
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG 006616/006617**

No Sample Data Qualified in this SDG

LDC #: 31852D3a

**VALIDATION COMPLETENESS WORKSHEET**

Date: 6-2-14

SDG #: 6616/6617

Level III

Page: 1 of 1

Laboratory: ARDL, Inc.

Reviewer: [Signature]

2nd Reviewer: [Signature]

**METHOD:** GC Chlorinated Pesticides (EPA SW 846 Method 8081B)

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: 1/31/14 → 2/11/14
II.	GC Instrument Performance Check	A	
III.	Initial calibration	A	≤ 20/r <sub>r</sub>
IV.	Continuing calibration/ICV	A	CCV/ICV ≤ 20
V.	Blanks	A	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	A	LCS
IX.	Regional quality assurance and quality control	N	
X.	Florisol cartridge check	N	
XI.	GPC Calibration	N	
XII.	Target compound identification	N	
XIII.	Compound quantitation/RL/LOQ/LODs	SW	
XIV.	Overall assessment of data	A	
XV.	Field duplicates	N	
XVI.	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: *Plant tissue*

1	MAK120L	11	MAK118L	21		31	
2	MAK121L	12	MAK123LMS	22		32	
3	MAK122L	13	MAK123LMSD	23		33	
4	MAK123L	14		24		34	
5	MAK124L	15		25		35	
6	MAK126L	16		26		36	
7	MAK127L	17		27		37	
8	MAK128L	18		28		38	
9	MAK116L	19	<i>B10178</i>	29		39	
10	MAK117L	20		30		40	

## VALIDATION FINDINGS WORKSHEET

**METHOD:** Pesticide/PCBs (EPA SW 846 Method 8081/8082)

A. alpha-BHC	I. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	GG. Chlordane
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH. Chlordane (Technical)
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	II. oxy-Chlordane
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	JJ. Mirex
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. 2,4'-DDD	KK.
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. 2,4'-DDE	LL.
G. Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	EE. 2,4'-DDT	MM.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	FF. Hexachlorobenzene	NN.

Notes: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_







## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** Makua Military Reservation  
**Collection Date:** January 31 through March 28, 2014  
**LDC Report Date:** June 17, 2014  
**Matrix:** Tissue  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** EPA Level III  
**Laboratory:** Pace Analytical Services, Inc.  
**Sample Delivery Group (SDG):** 10265141

### Sample Identification

MAK109O	MAK138L
MAK110O	MAK121C
MAK111O	MAK122C
MAK112O	MAK123C
MAK113O	MAK124C
MAK114O	MAK125C
MAK115O	MAK126C
MAK116O	MAK127C
MAK117O	MAK128C
MAK118O	MAK109OMS
MAK119O	MAK109OMSD
MAK120O	MAK120OMS
MAK121O	MAK120OMSD
MAK122O	
MAK123O	
MAK124O	
MAK130L	
MAK132L	
MAK134L	
MAK135L	

## Introduction

This data review covers 33 tissue samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 1613B for Polychlorinated Dioxins/Dibenzofurans.

This review follows the Final Supplemental Marine Resources Study Sampling and Analysis Plan at Makua Military Reservation, Oahu, Hawaii (August 2013), the Final Draft Version of the U.S. Department of Defense (DoD) and Department of Energy (DoE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0 (March 2013), and the USEPA Contract Laboratory Program (CLP) National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Data Review (September 2011).

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.

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

The following are definitions of the data qualifiers:

- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

## **I. Technical Holding Times**

All technical holding time requirements were met.

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

## **II. HRGC/HRMS Instrument Performance Check**

Instrument performance was checked at the required daily frequency.

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

## **III. Initial Calibration**

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

Percent relative standard deviations (%RSD) were less than or equal to 20.0% for unlabeled compounds and less than or equal to 35.0% for labeled compounds.

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

## **IV. Continuing Calibration**

Continuing calibration was performed at the required frequencies.

All of the continuing calibration results were within the QC limits for unlabeled compounds and labeled compounds.

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

## **V. Blanks**

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
BLANK-40418	5/6/14	2,3,7,8-TCDF Total TCDF Total HpCDD	0.080 ng/Kg 0.064 ng/Kg 0.089 ng/Kg	MAK109O MAK110O MAK111O MAK112O MAK113O MAK114O MAK115O MAK116O MAK117O MAK118O MAK119O MAK120O MAK121O MAK122O MAK123O MAK124O MAK130L MAK132L
BLANK-40456	5/7/14	2,3,7,8-TCDF Total TCDF Total TCDD 1,2,3,4,6,7,8-HpCDD Total HpCDD OCDD	0.14 ng/Kg 0.150 ng/Kg 0.150 ng/Kg 0.035 ng/Kg 0.035 ng/Kg 0.12 ng/Kg	MAK134L MAK135L MAK138L MAK121C MAK122C MAK123C MAK124C MAK125C MAK126C MAK127C MAK128C

Sample concentrations were compared to concentrations detected in the method blanks. The sample concentrations were either not detected or were significantly greater (>5X blank contaminants) than the concentrations found in the associated method blanks with the following exceptions:

Sample	Compound	Reported Concentration	Modified Final Concentration
MAK109O	2,3,7,8-TCDF	0.12 ng/Kg	0.12U ng/Kg
MAK110O	2,3,7,8-TCDF Total TCDF	0.14 ng/Kg 0.14 ng/Kg	0.14U ng/Kg 0.14J ng/Kg
MAK111O	2,3,7,8-TCDF Total TCDF	0.12 ng/Kg 0.12 ng/Kg	0.12U ng/Kg 0.12J ng/Kg
MAK113O	2,3,7,8-TCDF Total TCDF	0.14 ng/Kg 0.14 ng/Kg	0.14U ng/Kg 0.14J ng/Kg
MAK114O	2,3,7,8-TCDF	0.140 ng/Kg	0.140U ng/Kg
MAK116O	2,3,7,8-TCDF Total TCDF	0.19 ng/Kg 0.19 ng/Kg	0.19U ng/Kg 0.19J ng/Kg

Sample	Compound	Reported Concentration	Modified Final Concentration
MAK117O	2,3,7,8-TCDF Total TCDF	0.97 ng/Kg 0.74 ng/Kg	0.97U ng/Kg 0.74J ng/Kg
MAK118O	2,3,7,8-TCDF Total TCDF	0.140 ng/Kg 0.11 ng/Kg	0.140U ng/Kg 0.11J ng/Kg
MAK119O	2,3,7,8-TCDF	0.19 ng/Kg	0.19U ng/Kg
MAK121O	2,3,7,8-TCDF	0.13 ng/Kg	0.13U ng/Kg
MAK122O	2,3,7,8-TCDF	0.11 ng/Kg	0.11U ng/Kg
MAK123O	2,3,7,8-TCDF	0.18 ng/Kg	0.18U ng/Kg
MAK124O	2,3,7,8-TCDF Total TCDF	0.11 ng/Kg 0.10 ng/Kg	0.11U ng/Kg 0.10J ng/Kg
MAK130L	2,3,7,8-TCDF	0.16 ng/Kg	0.16U ng/Kg
MAK134L	1,2,3,4,6,7,8-HpCDD OCDD	0.042 ng/Kg 0.350 ng/Kg	0.042U ng/Kg 0.350U ng/Kg
MAK135L	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDD Total HpCDD OCDD	0.15 ng/Kg 0.088 ng/Kg 0.088 ng/Kg 0.530 ng/Kg	0.15U ng/Kg 0.088U ng/Kg 0.088J ng/Kg 0.530U ng/Kg
MAK138L	2,3,7,8-TCDF Total TCDF OCDD	0.190 ng/Kg 0.310 ng/Kg 0.380 ng/Kg	0.190U ng/Kg 0.310J ng/Kg 0.380U ng/Kg
MAK121C	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDD OCDD	0.150 ng/Kg 0.064 ng/Kg 0.210 ng/Kg	0.150U ng/Kg 0.064U ng/Kg 0.210U ng/Kg
MAK122C	2,3,7,8-TCDF Total TCDF 1,2,3,4,6,7,8-HpCDD OCDD	0.150 ng/Kg 0.150 ng/Kg 0.12 ng/Kg 0.27 ng/Kg	0.150U ng/Kg 0.150J ng/Kg 0.12U ng/Kg 0.27U ng/Kg
MAK123C	2,3,7,8-TCDF Total TCDF OCDD	0.120 ng/Kg 0.11 ng/Kg 0.390 ng/Kg	0.120U ng/Kg 0.11J ng/Kg 0.390U ng/Kg
MAK124C	2,3,7,8-TCDF Total TCDF OCDD	0.14 ng/Kg 0.11 ng/Kg 0.29 ng/Kg	0.14U ng/Kg 0.11J ng/Kg 0.29U ng/Kg
MAK125C	OCDD	0.39 ng/Kg	0.39U ng/Kg

Sample	Compound	Reported Concentration	Modified Final Concentration
MAK126C	2,3,7,8-TCDF Total TCDF	0.13 ng/Kg 0.13 ng/Kg	0.13U ng/Kg 0.13J ng/Kg
MAK128C	2,3,7,8-TCDF Total TCDF 1,2,3,4,6,7,8-HpCDD Total HpCDD OCDD	0.15 ng/Kg 0.15 ng/Kg 0.10 ng/Kg 0.10 ng/Kg 0.46 ng/Kg	0.15U ng/Kg 0.15J ng/Kg 0.10U ng/Kg 0.10J ng/Kg 0.46U ng/Kg

No field blanks were identified in this SDG.

### VI. 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 the QC limits with the following exceptions:

Spike ID (Associated Samples)	Compound	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Affected Compounds	Flag	A or P
MAK1200MS/MSD (MAK1200)	1,2,3,6,7,8-HxCDD	-	134 (70-130)	-	1,2,3,6,7,8-HxCDD Total HxCDD	J (all detects)	A

### VII. Laboratory Control Samples (LCS)

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

### VIII. Regional Quality Assurance and Quality Control

Not applicable.

### IX. Internal Standards

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

### X. Target Compound Identifications

Raw data were not reviewed for this SDG.

### XI. Compound Quantitation

All compound quantitations were within validation criteria with the following exceptions:

Sample	Compound	Flag	A or P
MAK1190 MAK1230	All compounds flagged "P" due to DiPhenylEther interference	J (all detects)	P

The 2,3,7,8-TCDF confirmation was performed with the following exceptions:

Sample	Compound	Finding	Criteria
MAK1090 MAK1100 MAK1110 MAK1130 MAK1140 MAK1160 MAK1170 MAK1180 MAK1190 MAK1210 MAK1220 MAK1230 MAK1240 MAK130L MAK135L MAK138L MAK121C MAK122C MAK123C MAK124C MAK126C MAK128C	2,3,7,8-TCDF	2nd column confirmation was not performed for this compound.	2,3,7,8-TCDF must be confirmed on the 2nd column per the method.

Raw data were not reviewed for this SDG.

## XII. System Performance

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.

**Makua Military Reservation  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG 10265141**

SDG	Sample	Compound	Flag	A or P	Reason
10265141	MAK120O	1,2,3,6,7,8-HxCDD Total HxCDD	J (all detects)	A	Matrix spike/Matrix spike duplicate (%R)
10265141	MAK119O MAK123O	All compounds flagged "P" due to DiPhenylEther interference	J (all detects)	P	Compound quantitation

**Makua Military Reservation  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG  
10265141**

SDG	Sample	Compound	Modified Final Concentration	A or P
10265141	MAK109O	2,3,7,8-TCDF	0.12U ng/Kg	A
10265141	MAK110O	2,3,7,8-TCDF Total TCDF	0.14U ng/Kg 0.14J ng/Kg	A
10265141	MAK111O	2,3,7,8-TCDF Total TCDF	0.12U ng/Kg 0.12J ng/Kg	A
10265141	MAK113O	2,3,7,8-TCDF Total TCDF	0.14U ng/Kg 0.14J ng/Kg	A
10265141	MAK114O	2,3,7,8-TCDF	0.140U ng/Kg	A
10265141	MAK116O	2,3,7,8-TCDF Total TCDF	0.19U ng/Kg 0.19J ng/Kg	A
10265141	MAK117O	2,3,7,8-TCDF Total TCDF	0.97U ng/Kg 0.74J ng/Kg	A
10265141	MAK118O	2,3,7,8-TCDF Total TCDF	0.140U ng/Kg 0.11J ng/Kg	A
10265141	MAK119O	2,3,7,8-TCDF	0.19U ng/Kg	A
10265141	MAK121O	2,3,7,8-TCDF	0.13U ng/Kg	A
10265141	MAK122O	2,3,7,8-TCDF	0.11U ng/Kg	A
10265141	MAK123O	2,3,7,8-TCDF	0.18U ng/Kg	A

SDG	Sample	Compound	Modified Final Concentration	A or P
10265141	MAK124O	2,3,7,8-TCDF Total TCDF	0.11U ng/Kg 0.10J ng/Kg	A
10265141	MAK130L	2,3,7,8-TCDF	0.16U ng/Kg	A
10265141	MAK134L	1,2,3,4,6,7,8-HpCDD OCDD	0.042U ng/Kg 0.350U ng/Kg	A
10265141	MAK135L	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDD Total HpCDD OCDD	0.15U ng/Kg 0.088U ng/Kg 0.088J ng/Kg 0.530U ng/Kg	A
10265141	MAK138L	2,3,7,8-TCDF Total TCDF OCDD	0.190U ng/Kg 0.310J ng/Kg 0.380U ng/Kg	A
10265141	MAK121C	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDD OCDD	0.150U ng/Kg 0.064U ng/Kg 0.210U ng/Kg	A
10265141	MAK122C	2,3,7,8-TCDF Total TCDF 1,2,3,4,6,7,8-HpCDD OCDD	0.150U ng/Kg 0.150J ng/Kg 0.12U ng/Kg 0.27U ng/Kg	A
10265141	MAK123C	2,3,7,8-TCDF Total TCDF OCDD	0.120U ng/Kg 0.11J ng/Kg 0.390U ng/Kg	A
10265141	MAK124C	2,3,7,8-TCDF Total TCDF OCDD	0.14U ng/Kg 0.11J ng/Kg 0.29U ng/Kg	A
10265141	MAK125C	OCDD	0.39U ng/Kg	A
10265141	MAK126C	2,3,7,8-TCDF Total TCDF	0.13U ng/Kg 0.13J ng/Kg	A
10265141	MAK128C	2,3,7,8-TCDF Total TCDF 1,2,3,4,6,7,8-HpCDD Total HpCDD OCDD	0.15U ng/Kg 0.15J ng/Kg 0.10U ng/Kg 0.10J ng/Kg 0.46U ng/Kg	A

**Makua Military Reservation  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG 10265141**

No Sample Data Qualified in this SDG

LDC #: 31852E21

## VALIDATION COMPLETENESS WORKSHEET

Date: 6-2-14

SDG #: 10265141

Level III

Page: 1 of 1

Laboratory: Pace Analytical Services, Inc.

Reviewer: *TM*2nd Reviewer: *J*

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

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: 1/31/14 → 3/28/14
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	≈ 20/35
IV.	Continuing calibration/ <del>rev</del>	A	CCV QC limits
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	LCS/LCSD
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
X.	Target compound identifications	N	
XI.	Compound quantitation/RL/LOQ/LODs	SW	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	N	

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

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

D = Duplicate  
TB = Trip blank  
EB = Equipment blank

Validated Samples:

tissue / plant tissue

1	MAK1090	11	MAK1190	21	MAK138L	PT	31	MAK109OMSD
2	MAK1100	12	MAK1200	22	MAK121C		32	MAK120OMS
3	MAK1110	13	MAK1210	23	MAK122C		33	MAK120OMSD
4	MAK1120	14	MAK1220	24	MAK123C		34	
5	MAK1130	15	MAK1230	25	MAK124C		35	
6	MAK1140	16	MAK1240	26	MAK125C		36	
7	MAK1150	17	MAK130L	PT	27	MAK126C	37	
8	MAK1160	18	MAK132L	PT	28	MAK127C	38	BLANK-40418
9	MAK1170	19	MAK134L	PT	29	MAK128C	39	BLANK-40456
10	MAK1180	20	MAK135L	PT	30	MAK109OMS	40	

Notes:

## VALIDATION FINDINGS WORKSHEET

**METHOD:** HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

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

Notes: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

**VALIDATION FINDINGS WORKSHEET**  
**Blanks**

**METHOD:** HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

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

- N N/A Were all samples associated with a method blank?
- N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?
- N N/A Was the method blank contaminated?

Blank extraction date: 05/06/14 Blank analysis date: 05/12/14 Associated samples: 1-18 Qual U/Qual J  
Conc. units: ng/Kg

Compound	Blank ID	Sample Identification								
		5x	1	2	3	5	6	8	9	10
	BLANK-40418									
H	0.080*	0.400	0.12* U	0.14 U	0.12 U	0.14 U	0.140* U	0.19 U	0.97* U	0.140* U
V	0.064	--		0.14 <del>U</del> J	0.12 <del>U</del> J	0.14 <del>U</del> J		0.19 <del>U</del> J	0.74 J	0.11 J
U	0.089	--								

\*EMPC

Blank extraction date: 05/06/14 Blank analysis date: 05/12/14 Associated samples: 1-18  
Conc. units: ng/Kg

Compound	Blank ID	Sample Identification								
		5x	11	13	14	15	16	17		
	BLANK-40418									
H	0.080*	0.400	0.19* U	0.13* U	0.11* U	0.18* U	0.11* U	0.16* U		
V	0.064	--					0.10 J			
U	0.089	--								

\*EMPC

**VALIDATION FINDINGS WORKSHEET**  
**Blanks**

**METHOD:** HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

Blank extraction date: 05/07/14    Blank analysis date: 05/13/14    Associated samples: 19-29 *Qual U / Qual J*  
Conc. units: ng/Kg

Compound	Blank ID	Sample Identification								
		5x	19	20	21	22	23	24	25	26
	BLANK-40456									
H	0.14*	0.700		0.15* U	0.190 U	0.150* U	0.150 U	0.120* U	0.14* U	
V	0.150	--			0.310 J		0.150 <i>UJ</i>	0.11 J	0.11 J	
R	0.150	--								
F	0.035	0.175	0.042* U	0.088 U		0.064* U	0.12* U			
U	0.035	--		0.088 <i>UJ</i>						
G	0.12*	0.600	0.350 U	0.530 U	0.380 U	0.210 U	0.27* U	0.390* U	0.29 U	0.39* U

\*EMPC

Blank extraction date: 05/07/14    Blank analysis date: 05/13/14    Associated samples: 19-29  
Conc. units: ng/Kg

Compound	Blank ID	Sample Identification								
		5x	27	29						
	BLANK-40456									
H	0.14*	0.700	0.13 U	0.15 U						
V	0.150	--	0.13 <i>UJ</i>	0.15 <i>UJ</i>						
R	0.150	--								
F	0.035	0.175		0.10 U						
U	0.035	--		0.10 <i>UJ</i>						
G	0.12*	0.600		0.46 U						

\*EMPC

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



**VALIDATION FINDINGS WORKSHEET**  
**Compound Quantitation and Reported RLs**

**METHOD:** GC/MS Dioxins/Dibenzofurans (Method 1613B)

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

Y N N/A Were the correct internal standard (IS), quantitation ions and relative response factors (RRF) used to quantitate the compound?  
 Y N N/A Compound quantitation and RLs were adjusted to reflect all sample dilutions and dry weight factors (if necessary).

#	Date	Compound	Finding	Associated Samples	Qualifications
			no 2,3,7,8-TCDF confirmation	1-3, 5-6, 8-11, 13-17, 20-25, 27, 29	Text
		H	"P" flagged as DiPhenylEther interference	11, 15	Jdets/P

Comments: See sample calculation verification worksheet for recalculations

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## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** Makua Military Reservation  
**Collection Date:** January 13, 2014  
**LDC Report Date:** June 3, 2014  
**Matrix:** Tissue  
**Parameters:** Perchlorate  
**Validation Level:** EPA Level III  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** 320-6209-1

### Sample Identification

MAK101O  
MAK102O  
MAK103O  
MAK104O  
MAK105O  
MAK106O  
MAK107O  
MAK108O  
MAK104OMS  
MAK104OMSD

## Introduction

This data review covers 10 tissue samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6850 for Perchlorate.

This review follows the Final Supplemental Marine Resources Study Sampling and Analysis Plan at Makua Military Reservation, Oahu, Hawaii (August 2013), the Final Draft Version of the U.S. Department of Defense (DoD) and Department of Energy (DoE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0 (March 2013), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008).

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

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

The following are definitions of the data qualifiers:

- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- J Indicates an estimated value.
- R Quality control indicates the data is not usable.
- NJ Presumptive evidence of presence of the compound at an estimated quantity.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

## **I. Technical Holding Times**

All technical holding time requirements were met.

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

## **II. LC/MS Instrument Performance Check**

Instrument performance check is not required by the method.

## **III. Initial Calibration**

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 15.0% for all compounds.

## **IV. Continuing Calibration**

Continuing calibration was performed at required frequencies.

The percent differences (%D) were less than or equal to 15.0% for all compounds.

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

The percent differences (%D) of the limit of detection verification (LODV) standard were less than or equal to 30.0% for all compounds.

## **V. Blanks**

Method blanks were reviewed for each matrix as applicable. No perchlorate was found in the method blanks.

No field blanks were identified in this SDG.

## **VI. Surrogate Spikes**

Surrogates were not required by the method.

## **VII. Matrix Spike/Matrix Spike Duplicates**

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

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

Sample	Internal Standards	Area (Limits)	Compound	Flag	A or P
MAK101O	18-O Perchlorate	112126000 (243055801-972223204)	Perchlorate	J (all detects) UJ (all non-detects)	P
MAK102O	18-O Perchlorate	120946685 (243055801-972223204)	Perchlorate	J (all detects) UJ (all non-detects)	P
MAK103O	18-O Perchlorate	119108963 (243055801-972223204)	Perchlorate	J (all detects) UJ (all non-detects)	P
MAK104O	18-O Perchlorate	115685291 (243055801-972223204)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK105O	18-O Perchlorate	130329064 (243055801-972223204)	Perchlorate	J (all detects) UJ (all non-detects)	P
MAK106O	18-O Perchlorate	131607893 (243055801-972223204)	Perchlorate	J (all detects) UJ (all non-detects)	P
MAK107O	18-O Perchlorate	125172417 (243055801-972223204)	Perchlorate	J (all detects) UJ (all non-detects)	P
MAK108O	18-O Perchlorate	103548948 (239803517-959214066)	Perchlorate	J (all detects) UJ (all non-detects)	P

### XI. Target Compound Identifications

Raw data were not reviewed for this SDG.

### XII. Compound Quantitation

Raw data were not reviewed for this SDG.

### **XIII. System Performance**

Raw data were not reviewed for this SDG.

### **XIV. Overall Assessment**

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

### **XV. Field Duplicates**

No field duplicates were identified in this SDG.

**Makua Military Reservation  
Perchlorate - Data Qualification Summary - SDG 320-6209-1**

SDG	Sample	Compound	Flag	A or P	Reason
320-6209-1	MAK101O MAK102O MAK103O MAK105O MAK106O MAK107O MAK108O	Perchlorate	J (all detects) UJ (all non-detects)	P	Internal standards (area)
320-6209-1	MAK104O	Perchlorate	J (all detects) UJ (all non-detects)	A	Internal standards (area)

**Makua Military Reservation  
Perchlorate - Laboratory Blank Data Qualification Summary - SDG 320-6209-1**

No Sample Data Qualified in this SDG

**Makua Military Reservation  
Perchlorate - Field Blank Data Qualification Summary - SDG 320-6209-1**

No Sample Data Qualified in this SDG

LDC #: 31852F87

**VALIDATION COMPLETENESS WORKSHEET**

Date: 6/02/14

SDG #: 320-6209-1

Level III

Page: 1 of 1

Laboratory: Test America, Inc.

Reviewer: SVL

2nd Reviewer: A

**METHOD:** LC/MS Perchlorate (EPA SW846 Method 6850)

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: 1/13/14
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	A	% RSD $\leq$ 15
IV.	Continuing calibration/ICV	A	CV/AV $\leq$ 15% LODV $\leq$ 30%
V.	Blanks	A	
VI.	Surrogate spikes	N	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	LCs
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	SW	
XI.	Target compound identification	N	
XII.	Compound quantitation/RL/LOQ/LODs	N	
XIII.	System performance	N	
XIV.	Overall assessment of data	A	
XV.	Field duplicates	N	
XVI.	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:

Tissue

1	MAK101O	11	MB 320-38755/1-A21	31
2	MAK102O	12		32
3	MAK103O	13		33
4	MAK104O	14		34
5	MAK105O	15		35
6	MAK106O	16		36
7	MAK107O	17		37
8	MAK108O	18		38
9	MAK104OMS	19		39
10	MAK104OMSD	20		40

**VALIDATION FINDINGS WORKSHEET**  
**Internal Standards**

**METHOD:** LC/MS Perchlorate (EPA SW 846 Method 6850)

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

Y  N/A Were all internal standard area counts within -50 to +100% of the associated calibration standard?

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

#	Date	Sample ID	Internal Standard	Area (Limits)	RT (Limits)	Qualifications
237		1	18-O Perchlorate	112 126 000 (243 055 801-972 223 204)	<del>10.85 (11.08 12.08)</del>	J/UJ/P
		2		120 946 685		
		3		119 108 963		
		4		115 685 291		J/UJ/A
		5		130 329 064		J/UJ/P
		6		131 607 893		
		7		125 172 417		
		to 9		121 946 592		No qual (QC)
		10		125 637 331		
		8		103 548 948 (239 803 517-959 214 066)		J/UJ/P

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

**Project/Site Name:** Makua Military Reservation  
**Collection Date:** January 13 through January 14, 2014  
**LDC Report Date:** June 4, 2014  
**Matrix:** Tissue  
**Parameters:** Perchlorate  
**Validation Level:** EPA Level III  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** 320-6210-1

**Sample Identification**

MAK102C  
MAK103C  
MAK104C  
MAK105C  
MAK106C  
MAK107C  
MAK108C  
MAK110C  
MAK110CMS  
MAK110CMSD

## Introduction

This data review covers 10 tissue samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 6850 for Perchlorate.

This review follows the Final Supplemental Marine Resources Study Sampling and Analysis Plan at Makua Military Reservation, Oahu, Hawaii (August 2013), the Final Draft Version of the U.S. Department of Defense (DoD) and Department of Energy (DoE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0 (March 2013), and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008).

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

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

The following are definitions of the data qualifiers:

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- 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. LC/MS Instrument Performance Check**

Instrument performance check is not required by the method.

## **III. Initial Calibration**

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 15.0% for all compounds.

## **IV. Continuing Calibration**

Continuing calibration was performed at required frequencies.

The percent differences (%D) were less than or equal to 15.0% for all compounds.

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

The percent differences (%D) of the limit of detection verification (LODV) standard were less than or equal to 30.0% for all compounds.

## **V. Blanks**

Method blanks were reviewed for each matrix as applicable. No perchlorate was found in the method blanks.

No field blanks were identified in this SDG.

## **VI. Surrogate Spikes**

Surrogates were not required by the method.

## **VII. Matrix Spike/Matrix Spike Duplicates**

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

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

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

### **IX. Regional Quality Assurance and Quality Control**

Not applicable.

### **X. Internal Standards**

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

### **XI. Target Compound Identifications**

Raw data were not reviewed for this SDG.

### **XII. Compound Quantitation**

Raw data were not reviewed for this SDG.

### **XIII. System Performance**

Raw data were not reviewed for this SDG.

### **XIV. Overall Assessment**

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

### **XV. Field Duplicates**

No field duplicates were identified in this SDG.

**Makua Military Reservation  
Perchlorate - Data Qualification Summary - SDG 320-6210-1**

No Sample Data Qualified in this SDG

**Makua Military Reservation  
Perchlorate - Laboratory Blank Data Qualification Summary - SDG 320-6210-1**

No Sample Data Qualified in this SDG

**Makua Military Reservation  
Perchlorate - Field Blank Data Qualification Summary - SDG 320-6210-1**

No Sample Data Qualified in this SDG

LDC #: 31852G87

**VALIDATION COMPLETENESS WORKSHEET**

Date: 6/02/14

SDG #: 320-6210-1

Level III

Page: 1 of 1

Laboratory: Test America, Inc.

Reviewer: SVL

2nd Reviewer: A

**METHOD:** LC/MS Perchlorate (EPA SW846 Method 6850)

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: 1/13-14 / 14
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	A	% RSD ≤ 15%
IV.	Continuing calibration/ICV	A	CCV / ICV ≤ 15%
V.	Blanks	A	
VI.	Surrogate spikes	N	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	LCS
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	N	
XII.	Compound quantitation/RL/LOQ/LODs	N	
XIII.	System performance	N	
XIV.	Overall assessment of data	A	
XV.	Field duplicates	N	
XVI.	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:

Tissue

1	MAK102C	11	MB 320-38755/-A	21		31	
2	MAK103C	12		22		32	
3	MAK104C	13		23		33	
4	MAK105C	14		24		34	
5	MAK106C	15		25		35	
6	MAK107C	16		26		36	
7	MAK108C	17		27		37	
8	MAK110C	18		28		38	
9	MAK110CMS	19		29		39	
10	MAK110CMSD	20		30		40	