



# LABORATORY DATA CONSULTANTS, INC.

2701 Loker Ave. West, Suite 220, Carlsbad, CA 92010 Bus: 760-827-1100 Fax: 760-827-1099

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

July 11, 2014

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

Dear Ms. Shjegstad

Enclosed are the final validation reports for the fraction listed below. These SDGs were received on June 27, 2014. Attachment 1 is a summary of the samples that were reviewed for each analysis.

**LDC Project #32064:**

<b><u>SDG #</u></b>	<b><u>Fraction</u></b>
320-6211-1, 320-7224-1 320-7224-2, 320-7224-3	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 through January 14, 2014  
**LDC Report Date:** July 11, 2014  
**Matrix:** Tissue  
**Parameters:** Perchlorate  
**Validation Level:** EPA Level III  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** 320-6211-1

### Sample Identification

MAK101L  
MAK102L  
MAK105L  
MAK106L  
MAK108,109,110,113L  
MAK111L  
MAK112L  
MAK115L  
MAK101LMS  
MAK101LMSD

## 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.
- 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
MAK101L	18-O Perchlorate	215127698 (254430497-1017721986)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK102L	18-O Perchlorate	244923321 (254430497-1017721986)	Perchlorate	J (all detects) UJ (all non-detects)	P
MAK105L	18-O Perchlorate	220027786 (254430497-1017721986)	Perchlorate	J (all detects) UJ (all non-detects)	P
MAK106L	18-O Perchlorate	184582799 (254430497-1017721986)	Perchlorate	J (all detects) UJ (all non-detects)	P
MAK108,109,110,113L	18-O Perchlorate	212524822 (254430497-1017721986)	Perchlorate	J (all detects) UJ (all non-detects)	P
MAK111L	18-O Perchlorate	245015978 (254430497-1017721986)	Perchlorate	J (all detects) UJ (all non-detects)	P
MAK112L	18-O Perchlorate	208752610 (254430497-1017721986)	Perchlorate	J (all detects) UJ (all non-detects)	P
MAK115L	18-O Perchlorate	213204275 (254430497-1017721986)	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 320-6211-1**

SDG	Sample	Compound	Flag	A or P	Reason
320-6211-1	MAK101L	Perchlorate	J (all detects) UJ (all non-detects)	A	Internal standards (area)
320-6211-1	MAK102L MAK105L MAK106L MAK108,109,110,113L MAK111L MAK112L MAK115L	Perchlorate	J (all detects) UJ (all non-detects)	P	Internal standards (area)

**Makua Military Reservation  
Perchlorate - Laboratory Blank Data Qualification Summary - SDG 320-6211-1**

No Sample Data Qualified in this SDG

**Makua Military Reservation  
Perchlorate - Field Blank Data Qualification Summary - SDG 320-6211-1**

No Sample Data Qualified in this SDG

**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: <u>1/13-14/14</u>
II.	GC/MS Instrument performance check	N	
III.	Initial calibration	A	<u>7. RSD ≤ 15%</u>
IV.	Continuing calibration/ICV	A	<u>CCV/ICV ≤ 15% LODV ≤ 30%</u>
V.	Blanks	A	
VI.	Surrogate spikes	N	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	<u>LCS</u>
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	<u>SW</u>	
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 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

<u>1</u>	MAK101L	<u>11</u>	<u>MB 320-38877/A</u>	<u>21</u>		<u>31</u>	
<u>2</u>	MAK102L	<u>12</u>		<u>22</u>		<u>32</u>	
<u>3</u>	MAK105L	<u>13</u>		<u>23</u>		<u>33</u>	
<u>4</u>	MAK106L	<u>14</u>		<u>24</u>		<u>34</u>	
<u>5</u>	MAK108,109,110, <del>113</del> L	<u>15</u>		<u>25</u>		<u>35</u>	
<u>6</u>	<del>AK</del> M111L	<u>16</u>		<u>26</u>		<u>36</u>	
<u>7</u>	MAK112L	<u>17</u>		<u>27</u>		<u>37</u>	
<u>8</u>	MAK115L	<u>18</u>		<u>28</u>		<u>38</u>	
<u>9</u>	MAK101LMS	<u>19</u>		<u>29</u>		<u>39</u>	
<u>10</u>	MAK101LMSD	<u>20</u>		<u>30</u>		<u>40</u>	

LDC #: 32064A87

### VALIDATION FINDINGS WORKSHEET Internal Standards

Page: 1 of 1  
Reviewer: JVG  
2nd Reviewer: [Signature]

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

N N/A Are all internal standard recoveries were within the 50-150% criteria?  
Y N N/A Were the retention times of the internal standards within +/- 30 seconds of the retention times of the associated calibration standard?

#	Date	Lab ID/Reference	Internal Standard	Area (Limits)	Qualifications
		1	18-O Perchlorate	215 127 698 ( 254 930 497 - 1017 721 986)	J/UJ/A
		2		244 923 321 ( )	J/UJ/P
		3		220 027 786 ( )	
		4		184 582 799 ( )	
		5		212 524 822 ( )	
		6		245 015 978 ( )	
		7		208 752 610 ( )	
		8		213 204 275 ( )	↓
		9		192 806 976 ( )	No qual (Q)
		10		188 416 339 ( )	↓
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

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

**Project/Site Name:** Makua Military Reservation  
**Collection Date:** January 31 through March 28, 2014  
**LDC Report Date:** July 10, 2014  
**Matrix:** Tissue  
**Parameters:** Perchlorate  
**Validation Level:** EPA Level III  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** 320-7224-1

**Sample Identification**

MAK109O  
MAK110O  
MAK111O  
MAK112O  
MAK113O  
MAK114O  
MAK115O  
MAK116O  
MAK117O  
MAK118O  
MAK118ODL  
MAK119O  
MAK119ODL  
MAK120O  
MAK121O  
MAK122O  
MAK123O  
MAK124O  
MAK109OMS  
MAK109OMSD

## Introduction

This data review covers 20 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.
- J Indicates an estimated value.
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- 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
MAK1090	18-O Perchlorate	1864142 (2132703-8530810)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK1100	18-O Perchlorate	1500888 (2132703-8530810)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK1110	18-O Perchlorate	1364552 (2132703-8530810)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK1120	18-O Perchlorate	1465211 (2132703-8530810)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK1130	18-O Perchlorate	1509460 (2132703-8530810)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK1140	18-O Perchlorate	1698762 (2226829-8907316)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK1150	18-O Perchlorate	1601802 (2226829-8907316)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK1160	18-O Perchlorate	1754138 (2226829-8907316)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK1170	18-O Perchlorate	1646493 (2226829-8907316)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK1180	18-O Perchlorate	1380828 (2226829-8907316)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK118ODL	18-O Perchlorate	281296 (2228734-8914934)	Perchlorate	J (all detects) R (all non-detects)	A

Sample	Internal Standards	Area (Limits)	Compound	Flag	A or P
MAK119O	18-O Perchlorate	1523483 (2226829-8907316)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK119ODL	18-O Perchlorate	415846 (2228734-8914934)	Perchlorate	J (all detects) R (all non-detects)	A
MAK120O	18-O Perchlorate	1546262 (2226829-8907316)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK121O	18-O Perchlorate	1365441 (2226829-8907316)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK122O	18-O Perchlorate	1354760 (2226829-8907316)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK123O	18-O Perchlorate	1563911 (2226829-8907316)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK124O	18-O Perchlorate	1552974 (2157351-8629404)	Perchlorate	J (all detects) UJ (all non-detects)	A

#### 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

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
MAK118ODL MAK119ODL	All TCL compounds	R R	A

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 320-7224-1**

SDG	Sample	Compound	Flag	A or P	Reason
320-7224-1	MAK109O MAK110O MAK111O MAK112O MAK113O MAK114O MAK115O MAK116O MAK117O MAK118O MAK119O MAK120O MAK121O MAK122O MAK123O MAK124O	Perchlorate	J (all detects) UJ (all non-detects)	A	Internal standards (area)
320-7224-1	MAK118ODL MAK119ODL	Perchlorate	J (all detects) R (all non-detects)	A	Internal standards (area)
320-7224-1	MAK118ODL MAK119ODL	All TCL compounds	R R	A	Overall assessment of data

**Makua Military Reservation  
Perchlorate - Laboratory Blank Data Qualification Summary - SDG 320-7224-1**

No Sample Data Qualified in this SDG

**Makua Military Reservation  
Perchlorate - Field Blank Data Qualification Summary - SDG 320-7224-1**

No Sample Data Qualified in this SDG

**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: 1/31 - 3/28/14
II.	GC/MS Instrument performance check	N	
III.	Initial calibration	A	% RSD ≤ 15%
IV.	Continuing calibration/ICV	A	CV/ICV ≤ 15%      LOBV ≤ 30%
V.	Blanks	A	
VI.	Surrogate spikes	N	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	ICS
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	SW	
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	MAK1090	11	MAK118ODL	21	MB 320-41549 / 1-A 31/21	
2	MAK1100	12	MAK1190	22		↓
3	MAK1110	13	MAK119ODL	23		33
4	MAK1120	14	MAK1200	24		34
5	MAK1130	15	MAK1210	25		35
6	MAK1140	16	MAK1220	26		36
7	MAK1150	17	MAK1230	27		37
8	MAK1160	18	MAK1240	28		38
9	MAK1170	19	MAK109OMS	29		39
10	MAK1180	20	MAK109OMSD	30		40

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

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

Y (N) N/A Are all internal standard recoveries were within the 50-150% criteria?

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

#	Date	Lab ID/Reference	Internal Standard	Area (Limits)	Qualifications
		1	18-O Perchlorate	186414 ✓ ( 2132703 - 8530810 )	J/WJ/A
		2		1500888 ( )	
		3		1264552 ( )	
		4		1465211 ( )	
		5		1509460 ( ) ✓	
		6		1698762 ( 2226829 - 8907316 )	
		7		1601802 ( )	
		8		1754138 ( )	
		9		1646493 ( )	
3.7		10		1380828 ( ) ✓	✓
6.2		11		281296 ( 2228734 - 8914934 )	J/R/A
		12		1523483 ( 2226829 - 8907316 )	J/WJ/A
9.1		13	✓	415846 ( 2228734 - 8914934 )	J/R/A





## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** Makua Military Reservation  
**Collection Date:** March 27 through March 28, 2014  
**LDC Report Date:** July 10, 2014  
**Matrix:** Tissue  
**Parameters:** Perchlorate  
**Validation Level:** EPA Level III  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** 320-7224-2

### Sample Identification

MAK130L  
MAK132L  
MAK134L  
MAK135L  
MAK138L  
MAK130LMS  
MAK130LMSD

## Introduction

This data review covers 7 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.
- 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
MAK132L	18-O Perchlorate	1855798 (2157351-8629404)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK134L	18-O Perchlorate	1675361 (2157351-8629404)	Perchlorate	J (all detects) UJ (all non-detects)	A
MAK135L	18-O Perchlorate	1624067 (2157351-8629404)	Perchlorate	J (all detects) UJ (all non-detects)	A

### 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 320-7224-2**

SDG	Sample	Compound	Flag	A or P	Reason
320-7224-2	MAK132L MAK134L MAK135L	Perchlorate	J (all detects) UJ (all non-detects)	A	Internal standards (area)

**Makua Military Reservation  
Perchlorate - Laboratory Blank Data Qualification Summary - SDG 320-7224-2**

No Sample Data Qualified in this SDG

**Makua Military Reservation  
Perchlorate - Field Blank Data Qualification Summary - SDG 320-7224-2**

No Sample Data Qualified in this SDG

**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: <u>3/27-28/14</u>
II.	GC/MS Instrument performance check	N	
III.	Initial calibration	A	<u>% RSD ≤ 15%</u>
IV.	Continuing calibration/ICV	A	<u>CCV/ICV ≤ 15%    LODV ≤ 30%</u>
V.	Blanks	A	
VI.	Surrogate spikes	N	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	<u>LCS</u>
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	<u>SW</u>	
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                      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	MAK130L	11	<u>MB 320-41554 / 1-A 6/21</u>	31
2	MAK132L	12	<u>↓ 6/22</u>	32
3	MAK134L	13		23
4	MAK135L	14		24
5	MAK138L	15		25
6	MAK130LMS	16		26
7	MAK130LMSD	17		27
8		18		28
9		19		29
10		20		30



## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** Makua Military Reservation  
**Collection Date:** March 28, 2014  
**LDC Report Date:** July 10, 2014  
**Matrix:** Tissue  
**Parameters:** Perchlorate  
**Validation Level:** EPA Level III  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** 320-7224-3

### Sample Identification

MAK121C  
MAK122C  
MAK123C  
MAK124C  
MAK125C  
MAK126C  
MAK127C  
MAK128C

## Introduction

This data review covers 8 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.
- 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**

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.

### **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 320-7224-3**

No Sample Data Qualified in this SDG

**Makua Military Reservation  
Perchlorate - Laboratory Blank Data Qualification Summary - SDG 320-7224-3**

No Sample Data Qualified in this SDG

**Makua Military Reservation  
Perchlorate - Field Blank Data Qualification Summary - SDG 320-7224-3**

No Sample Data Qualified in this SDG

**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: 3/28/14
II.	GC/MS Instrument performance check	N	
III.	Initial calibration	A	? RSD ≤ 15%
IV.	Continuing calibration/ICV	A	COV/ICV ≤ 15% LODV ≤ 30%
V.	Blanks	A	
VI.	Surrogate spikes	N	
VII.	Matrix spike/Matrix spike duplicates	N	CS
VIII.	Laboratory control samples	A	ICS
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	<del>N</del> 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 ND = No compounds detected D = Duplicate  
 N = Not provided/applicable R = Rinsate TB = Trip blank  
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Validated Samples:

Tissue

1	MAK121C	11	MB 320-41554/1-A	6/21	31
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6	MAK126C	16		26	36
7	MAK127C	17		27	37
8	MAK128C	18		28	38
9		19		29	39
10		20		30	40

(All 15 failed in original run, reran at all)