

January 25, 2011

Analytical Report for Service Request No: K1100460

[REDACTED]  
Martek Biosciences Corporation  
6480 Dobbin Road  
Columbia, MD 21045

**RE: Methyl Mercury Tests**

[REDACTED]

Enclosed are the results of the rush samples submitted to our laboratory on January 18, 2011. For your reference, these analyses have been assigned our service request number K1100460.

Analyses were performed according to our laboratory's NELAP-approved quality assurance program. The test results meet requirements of the current NELAP standards, where applicable, and except as noted in the laboratory case narrative provided. For a specific list of NELAP-accredited analytes, refer to the certifications section at [www.caslab.com](http://www.caslab.com). All results are intended to be considered in their entirety, and Columbia Analytical Services, Inc. (CAS) is not responsible for use of less than the complete report. Results apply only to the items submitted to the laboratory for analysis and individual items (samples) analyzed, as listed in the report.

[REDACTED]

Respectfully submitted,

Columbia Analytical Services, Inc.

[REDACTED]  
Client Services Manager

LH/lb

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## Acronyms

ASTM	American Society for Testing and Materials
A2LA	American Association for Laboratory Accreditation
CARB	California Air Resources Board
CAS Number	Chemical Abstract Service registry Number
CFC	Chlorofluorocarbon
CFU	Colony-Forming Unit
DEC	Department of Environmental Conservation
DEQ	Department of Environmental Quality
DHS	Department of Health Services
DOE	Department of Ecology
DOH	Department of Health
EPA	U. S. Environmental Protection Agency
ELAP	Environmental Laboratory Accreditation Program
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
LUFT	Leaking Underground Fuel Tank
M	Modified
MCL	Maximum Contaminant Level is the highest permissible concentration of a substance allowed in drinking water as established by the USEPA.
MDL	Method Detection Limit
MPN	Most Probable Number
MRL	Method Reporting Limit
NA	Not Applicable
NC	Not Calculated
NCASI	National Council of the Paper Industry for Air and Stream Improvement
ND	Not Detected
NIOSH	National Institute for Occupational Safety and Health
PQL	Practical Quantitation Limit
RCRA	Resource Conservation and Recovery Act
SIM	Selected Ion Monitoring
TPH	Total Petroleum Hydrocarbons
tr	Trace level is the concentration of an analyte that is less than the PQL but greater than or equal to the MDL.

### Inorganic Data Qualifiers

- \* The result is an outlier. See case narrative.
- # The control limit criteria is not applicable. See case narrative.
- B The analyte was found in the associated method blank at a level that is significant relative to the sample result as defined by the DOD or NELAC standards.
- E The result is an estimate amount because the value exceeded the instrument calibration range.
- J The result is an estimated value that was detected outside the quantitation range.
- U The analyte was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL.  
*DOD-QSM 4.1 definition*: Analyte was not detected and is reported as less than the LOD or as defined by the project. The detection limit is adjusted for dilution.
- i The MRL/MDL or LOQ/LOD is elevated due to a matrix interference.
- X See case narrative.
- Q See case narrative. One or more quality control criteria was outside the limits.

### Metals Data Qualifiers

- # The control limit criteria is not applicable. See case narrative.
- J The result is an estimated value that was detected outside the quantitation range.
- E The percent difference for the serial dilution was greater than 10%, indicating a possible matrix interference in the sample.
- M The duplicate injection precision was not met.
- N The Matrix Spike sample recovery is not within control limits. See case narrative.
- S The reported value was determined by the Method of Standard Additions (MSA).
- U The analyte was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL.  
*DOD-QSM 4.1 definition*: Analyte was not detected and is reported as less than the LOD or as defined by the project. The detection limit is adjusted for dilution.
- W The post-digestion spike for furnace AA analysis is out of control limits, while sample absorbance is less than 50% of spike absorbance.
- i The MRL/MDL or LOQ/LOD is elevated due to a matrix interference.
- X See case narrative.
- + The correlation coefficient for the MSA is less than 0.995.
- Q See case narrative. One or more quality control criteria was outside the limits.

### Organic Data Qualifiers

- \* The result is an outlier. See case narrative.
- # The control limit criteria is not applicable. See case narrative.
- A A tentatively identified compound, a suspected aldol-condensation product.
- B The analyte was found in the associated method blank at a level that is significant relative to the sample result as defined by the DOD or NELAC standards.
- C The analyte was qualitatively confirmed using GC/MS techniques, pattern recognition, or by comparing to historical data.
- D The reported result is from a dilution.
- E The result is an estimate amount because the value exceeded the instrument calibration range.
- J The result is an estimated value that was detected outside the quantitation range.
- N The result is presumptive. The analyte was tentatively identified, but a confirmation analysis was not performed.
- P The GC or HPLC confirmation criteria was exceeded. The relative percent difference is greater than 40% between the two analytical results.
- U The analyte was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL.  
*DOD-QSM 4.1 definition*: Analyte was not detected and is reported as less than the LOD or as defined by the project. The detection limit is adjusted for dilution.
- i The MRL/MDL or LOQ/LOD is elevated due to a chromatographic interference.
- X See case narrative.
- Q See case narrative. One or more quality control criteria was outside the limits.

### Additional Petroleum Hydrocarbon Specific Qualifiers

- F The chromatographic fingerprint of the sample matches the elution pattern of the calibration standard.
- L The chromatographic fingerprint of the sample resembles a petroleum product, but the elution pattern indicates the presence of a greater amount of lighter molecular weight constituents than the calibration standard.
- H The chromatographic fingerprint of the sample resembles a petroleum product, but the elution pattern indicates the presence of a greater amount of heavier molecular weight constituents than the calibration standard.
- O The chromatographic fingerprint of the sample resembles an oil, but does not match the calibration standard.
- Y The chromatographic fingerprint of the sample resembles a petroleum product eluting in approximately the correct carbon range, but the elution pattern does not match the calibration standard.
- Z The chromatographic fingerprint does not resemble a petroleum product.

**Columbia Analytical Services, Inc.**  
**Kelso, WA**  
**State Certifications, Accreditations, and Licenses**

<b>Program</b>	<b>Number</b>
Alaska DEC UST	UST-040
Arizona DHS	AZ0339
Arkansas - DEQ	88-0637
California DHS	2286
Florida DOH	E87412
Hawaii DOH	-
Idaho DHW	-
Indiana DOH	C-WA-01
Louisiana DEQ	3016
Louisiana DHH	LA050010
Maine DHS	WA0035
Michigan DEQ	9949
Minnesota DOH	053-999-368
Montana DPHHS	CERT0047
Nevada DEP	WA35
New Jersey DEP	WA005
New Mexico ED	-
North Carolina DWQ	605
Oklahoma DEQ	9801
Oregon - DHS	WA200001
South Carolina DHEC	61002
Washington DOE	C1203
Wisconsin DNR	998386840
Wyoming (EPA Region 8)	-

## **Case Narrative**

**COLUMBIA ANALYTICAL SERVICES, INC.**

**Client:** Martek Biosciences Corporation  
**Project:** Methyl Mercury Tests  
**Sample Matrix:** Misc. Liquid

**Service Request No.:** K1100460  
**Date Received:** 01/18/11

**CASE NARRATIVE**

All analyses were performed consistent with the quality assurance program of Columbia Analytical Services, Inc. (CAS). This report contains analytical results for samples designated for Tier IV validation deliverables including summary forms and all of the associated raw data for each of the analyses. When appropriate to the method, method blank results have been reported with each analytical test.

**Sample Receipt**

Five samples were received for analysis at Columbia Analytical Services on 01/18/11. The samples were received in good condition and consistent with the accompanying chain of custody form. The samples were stored in a refrigerator at 4°C upon receipt at the laboratory.

**Methyl Mercury by EPA Method 1630**

**Matrix Spike Recovery Exceptions:**

The matrix spike and matrix spike duplicate recoveries of Methyl Mercury for sample DHB 08-6530 were outside control criteria. Several attempts have been made to analyze this sample type with various modifications to CAS standard procedures, however low recoveries were produced with each attempt. The low recoveries equates to a potential low bias in the sample matrix. Formulation of a procedure that will adequately recover Methyl Mercury in this matrix would require a more extensive development initiative. No further corrective action was appropriate.

No other anomalies associated with the analysis of these samples were observed.

Approved by \_\_\_\_\_

Date \_\_\_\_\_

**INTENDED**

**TO BE**

**BLANK**

## **Chain of Custody**

SR# K100460 PAGE 1 OF 1

1317 South 13th, Kelso, WA 98626  
(360) 577-7222 FAX (360) 636-1068

Project Name: Methyl mercury tests Project Number: \_\_\_\_\_  
Project Manager: [REDACTED] Company: Martek Biosciences  
Company/Address: 6480 Dobbin Road Phone: 4435422368  
City, State, Zip: Columbia, MD FAX: \_\_\_\_\_  
Sampler's Signature: \_\_\_\_\_

Sample ID.	Date	Time	LAB ID	Matrix
DHB 08-6530	7-Dec-10		Algal oils, 30g	
DHB 08-6586	7-Dec-10		Algal oils, 30g	
DHB 08-6592	7-Dec-10		Algal oils, 30g	
DHB 48-6585			Algal oil, 30g	
DHB 48-6643			Algal oil, 30g	

URNAROUND REQUIREMENTS	REPORT REQUIREMENTS
24 hr <u>2</u> 48 hr <u>X</u> 5 day	I. Routine Report: Results, Method Blank, Surrogate, as required
Standard (21 days)	II. Report Dup., MS, MSD as required
Provide FAX Preliminary Results	III. Data Validation Report (includes raw data)
Requested Report Date: _____	IV. CLP Deliverable Report
Invoice Information	V. EDD
P.O. # <u>914484</u>	
Bill to: Martek Biosciences Corporation, 6480 Dobbin Road, Columbia, MD 21045	

	RECEIVED BY:	[Redacted]
RELINQUISHED BY:	Signature: _____	
	Printed Name: _____	
	Firm: _____	
Date/Time:	_____	_____

RECEIVED BY: A  
Date/Time: 11/8/11 1030

RELINQUISHED BY: \_\_\_\_\_  
Signature: \_\_\_\_\_  
Printed Name: \_\_\_\_\_  
Firm: \_\_\_\_\_  
Date/Time: \_\_\_\_\_

RECEIVED BY: \_\_\_\_\_  
Signature: \_\_\_\_\_  
Printed Name: \_\_\_\_\_  
Firm: \_\_\_\_\_  
Date/Time: \_\_\_\_\_

**Comments/Special Instructions:**

Please purge the bottles with Nitrogen after opening to store the rest of the sample.

**Columbia Analytical Services, Inc.**  
**Cooler Receipt and Preservation Form**

PC LH

Client / Project: Mar tek Bio Sciences Ice Request K11 0460  
 Received: 1/18/11 Opened: 1/18/11 By: [Redacted] Unloaded: 1/18/11 By: [Redacted]

1. Samples were received via? Mail Fed Ex UPS DHL PDX Courier Hand Delivered  
 2. Samples were received in: (circle) Cooler Box Envelope Other NA  
 3. Were custody seals on coolers? NA Y N If yes, how many and where? \_\_\_\_\_  
 If present, were custody seals intact? Y N If present, were they signed and dated? Y N

Cooler Temp °C	Temp Blank °C	Thermometer ID	Cooler/COC ID	NA	Tracking Number	NA	Filed
<u>frozen</u>					<u>9329 7293 1168</u>		

7. Packing material used. Inserts Baggies Bubble Wrap Gel Packs Wet Ice Sleeves Other Dry ice  
 8. Were custody papers properly filled out (ink, signed, etc.)? NA Y N  
 9. Did all bottles arrive in good condition (unbroken)? Indicate in the table below. NA Y N  
 10. Were all sample labels complete (i.e analysis, preservation, etc.)? NA Y N  
 11. Did all sample labels and tags agree with custody papers? Indicate major discrepancies in the table on page 2. NA Y N  
 12. Were appropriate bottles/containers and volumes received for the tests indicated? NA Y N  
 13. Were the pH-preserved bottles (see SMO GEN SOP) received at the appropriate pH? Indicate in the table below NA Y N  
 14. Were VOA vials received without headspace? Indicate in the table below. NA Y N  
 15. Was C12/Res negative? NA Y N

Sample ID on Bottle	Sample ID on COC	Identified by:

Sample ID	Bottle Count	Bottle Type	Out of Temp	Head-space	Broke	pH	Reagent	Volume added	Reagent Lot Number	Initials	Time

Notes, Discrepancies, & Resolutions:

CCC not signed by client

**RUSH**

## **Metals**

## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

Client: Martek Biosciences Corporation  
 Project: Methyl Mercury Tests  
 Sample Matrix: Liquid

Service Request: K1100460  
 Date Collected: 12/07/10  
 Date Received: 01/18/11

## Methyl Mercury

Prep Method: CAS SOP  
 Analysis Method: CAS SOP  
 Test Notes:

Units: ng/g  
 Basis: Dry

Sample Name	Lab Code	MRL	MDL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
DHB 08-6530	K1100460-001	10	4.0	1	01/20/11	01/21/11	ND	N
DHB 08-6586	K1100460-002	10	4.0	1	01/20/11	01/21/11	ND	N
DHB 08-6592	K1100460-003	10	4.0	1	01/20/11	01/21/11	ND	N
DHB 08-6585	K1100460-004	10	4.0	1	01/20/11	01/21/11	ND	N
DHB 08-6643	K1100460-005	10	4.0	1	01/20/11	01/21/11	ND	N
Method Blank 1	K1100460-MB1	10	4.0	1	01/20/11	01/21/11	7.4	J
Method Blank 2	K1100460-MB2	10	4.0	1	01/20/11	01/21/11	6.1	J
Method Blank 3	K1100460-MB3	10	4.0	1	01/20/11	01/21/11	6.0	J

## COLUMBIA ANALYTICAL SERVICES, INC.

## QA/QC Report

Client: Martek Biosciences Corporation  
 Project: Methyl Mercury Tests  
 Sample Matrix: Liquid

Service Request: K1100460  
 Date Collected: 12/07/10  
 Date Received: 01/18/11  
 Date Extracted: 01/20/11  
 Date Analyzed: 01/21/11

Matrix Spike/Duplicate Matrix Spike Summary  
 Metals

Sample Name: DHB 08-6530  
 Lab Code: K1100460-001S, K1100460-001SD  
 Test Notes:

Units: ng/g  
 Basis: Dry

Analyte	Prep Method	Analysis Method	MRL	Percent Recovery										Result Notes
				Spike Level		Sample Result	Spike Result		CAS Acceptance Limits		Relative Percent Difference			
				MS	DMS		MS	DMS	MS	DMS				
Methyl Mercury	CAS SOP	CAS SOP	10	1818	1931	ND	290	443	16	23	65-135	42	N	

**COLUMBIA ANALYTICAL SERVICES, INC.**

**QA/QC Report**

**Client:** Martek Biosciences Corporation  
**Project:** Methyl Mercury Tests  
**LCS Matrix:** Water

**Service Request:** K1100460  
**Date Collected:** NA  
**Date Received:** NA  
**Date Extracted:** 1/20/2011  
**Date Analyzed:** 1/21/2011

**Ongoing Precision and Recovery (OPR) Sample Summary**  
**Metals**

**Sample Name:** Ongoing Precision and Recovery (Initial)

**Units:** picograms (pg)  
**Basis:** NA

Analyte	Prep Method	Analysis Method	True Value	Result	Percent Recovery	CAS Percent Recovery Acceptance Limits	Result Notes
Methyl Mercury	CAS SOP	CAS SOP	100	100	100	67-133	

**COLUMBIA ANALYTICAL SERVICES, INC.**

**QA/QC Report**

**Client:** Martek Biosciences Corporation  
**Project:** Methyl Mercury Tests  
**LCS Matrix:** Water

**Service Request:** K1100460  
**Date Collected:** NA  
**Date Received:** NA  
**Date Extracted:** 1/20/2011  
**Date Analyzed:** 1/21/2011

**Ongoing Precision and Recovery (OPR) Sample Summary**  
**Metals**

**Sample Name:** Ongoing Precision and Recovery (Final)

**Units:** picograms (pg)  
**Basis:** NA

Analyte	Prep Method	Analysis Method	True Value	Result	Percent Recovery	CAS Percent Recovery Acceptance Limits	Result Notes
Methyl Mercury	CAS SOP	CAS SOP	100	73.7	74	67-133	

**COLUMBIA ANALYTICAL SERVICES, INC.**

**QA/QC Report**

**Client:** Martek Biosciences Corporation  
**Project:** Methyl Mercury Tests  
**LCS Matrix:** Tissue

**Service Request:** K1100460  
**Date Collected:** NA  
**Date Received:** NA  
**Date Extracted:** 1/20/2011  
**Date Analyzed:** 1/21/2011

**Quality Control Sample (QCS) Summary**  
**Total Metals**

**Sample Name:** Quality Control Sample

**Units:** ng/g  
**Basis:** Dry

**Source:** NRCC Tort-2

Analyte	Prep Method	Analysis Method	True Value	Result	Percent Recovery	CAS Percent Recovery	Result Notes
						Acceptance Limits	
Methyl Mercury	CAS SOP	CAS SOP	163	173	106	67-133	

Service Request #	K1100460		
MS/MSD with #	K1100460-001		
Star Lims Prep #	127580		
Star Lims Run #	233421		
OPR Parent Std	AF1-57-A	Exp.	8/27/2011
OPR Intermediate Std	AF1-60-B	Exp.	3/4/2011
QCS Parent Std	NA	Exp.	NA
QCS Intermediate Std	NA	Exp.	NA

## 1630M Tissue Data Review Form

	Yes	No	N/A
1 20 samples (or less) in batch	X		
2 MS/MSD every 20 samples	X		
3 Mean of Ethylation Blanks less than 2 pg	X		
4 3 Method Blanks Run	X		
5 Method blank below MRL	X		
6 Current Calibration factor used	X		
7 Calibration data included	X		
8 OPR, QCS in control (67-133%)	X		
9 MS/MSD recovery (65 -135%)		X	
10 MS/MSD RPD within 35%		X	
11 All samples within the linear range	X		
12 All corresponding charts included	X		
13 Dilution factors calculated	X		
14 Bench sheet signed	X		

### Comments

Samples were oils so MS/MSD recovered only ~20%, flag and report as instructed.  
 K1100460-001 & -003 did not have plastic zip seal when checked out by analyst.

Primary Reviewed by



Date 1/21/2011

Secondary Reviewed by

Date 1/25/11

## Batch Information Report

Batch Number: StarLIMS #233421

Method Number: 1630M

Project Number(s): MeHg in Tissues

Instrument ID: K-AFS-04

Date Analyzed: 1/21/11

Analyst Name: XXXXXXXXXX

Run Duration:	7.0	Method Blank Type:	Concentration
Heating Time:	1.00	Integration Mode:	Methyl Hg
Retention Start Time:	2.5	Integration Type:	Peak Height
Retention Stop Time:	3.5	Result Units:	µg/Kg
Calibration File:	010511calsoil&tissue.brd		

### Reagents

Name	Lot Number
1% NaBEt4	RE2-33-I
2M KOAc	RE2-33-J
25% KOH	RE2-33-P
MeOH	RE2-31-L

### Standards

Name	Concentration	Lot Number
MeHgCl 1000pg	1000 pg/mL	AF1-60-A
MeHgCl 100pg	100 pg/mL	AF1-60-B
QCS	100 pg/mL	AF1-60-C
QCS Intermediate	1000 pg/mL	AF1-60-D
MeHgCl 10pg	10 pg/mL	AF1-60-E

### Analyst Comments:

Noise: 54  
PMT: 741  
Offset: 50,484

OPR1.00 mL(100 pg/mL) = 100 pg  
Matrix Spike0.50 mL(1000ng/mL) = 2.0 mg/Kg

Freeze Dried:No

TORT Solids:94.7%

# Run Report

Batch Number: StarLIMS #233421

Method Number: 1630M

Project Number(s): MeHg in Tissues

Instrument ID: K-AFS-04

Date Analyzed: 1/21/11

Analyst Name: [REDACTED]

Run	Run Type	Name/ID	Method Blank	Peak	Peak Height	Analyzed Result (pg)	Final Result (ug/Kg)	QA Results	Criteria	Notes
1	OPR	OPR		6	46,745	100.		100.	67-133	accept
2	QCS	TORT	MBA	2	16,637	35.6	173	106	67-133	accept
3	MBA	MBLK 1		6	860	1.84	7.36	7.36	< 10	accept
4	MBA	MBLK 2		8	709	1.52	6.07	6.07	< 10	accept
5	MBA	MBLK 3		3	701	1.50	6.00	6.00	< 10	accept
6	S	K1100460-001	MBA	3	619	1.32	-1.36		< HS	accept
7	MS	K1100460-001	MBA	3	38,044	81.4	290.	16.0	65-135	reject
8	MSD	K1100460-001	MBA	2	54,316	116	442	23.0	65-135	reject
9	S	K1100460-002	MBA	3	683	1.46	-1.08		< HS	accept
10	S	K1100460-003	MBA	3	772	1.65	0.0788		< HS	accept
11	S	K1100460-004	MBA	4	683	1.46	-1.06		< HS	accept
12	S	K1100460-005	MBA	4	754	1.61	-0.365		< HS	accept
13	OPR	OPR		2	34,453	73.7		73.7	67-133	accept

## Analyst Comments:

Noise: 54

PMT: 741

Offset: 50,484

OPR1.00 mL(100 pg/mL) = 100 pg

Matrix Spike0.50 mL(1000ng/mL) = 2.0 mg/Kg

Freeze Dried:No

TORT Solids:94.7%

## Peak Report

Batch Number: StarLIMS #233421

Method Number: 1630M

Project Number(s): MeHg in Tissues

Date Analyzed: 1/21/11

Instrument ID: K-AFS-04

Analyst Name: [REDACTED]

Bias and Precision										
Run Type	Name/ID	Final Result	Units	Spike Level	Source Result	% REC	% REC Limit	RPD	RPD Limit	Notes
MS	K1100460-001	290.	µg/Kg	1818	-1.36	16.0	65-135			reject
MSD	K1100460-001	442	µg/Kg	1931	-1.36	23.0	65-135			reject
OPR	OPR	100.	pg	100		100.	67-133			accept
	OPR	73.7	pg	100		73.7	67-133			accept
QCS	TORT	173	µg/Kg	163		106	67-133			accept

Calibration									
QA Sample Type	Name/ID	Analyzed Result	Units	Spike Level	% REC	% REC Limit	RSD	RSD Limit	Notes
Calibration	STD 2	1.67	pg	2	83.5	75-125			accept
	STD 20	18.3	pg	20	91.5	75-125			accept
	STD 50	53.0	pg	50	106	75-125			accept
	STD 100	111	pg	100	111	75-125			accept
	STD 1000	1,080	pg	1000	108	75-125			accept
	STD 2000	2,120	pg	2000	106	75-125			accept
Calibration Factor		0.00214	pg/PH				7.09	< 15	accept
Calibration Date		1/5/11							

## Peak Report

Batch Number: StarLIMS #233421

Method Number: 1630M

Project Number(s): MeHg in Tissues

Instrument ID: K-AFS-04

Date Analyzed: 1/21/11

Analyst Name: [REDACTED]

Blank Summary							
QA Sample Type	Name/ID	Analyzed Result	Units	Criteria	StDev	StDev Limit	Notes
MBA	MBLK 1	7.36	µg/Kg	< 10			accept
	MBLK 2	6.07	µg/Kg	< 10			accept
	MBLK 3	6.00	µg/Kg	< 10			accept
Average		6.48	µg/Kg		0.766		

QA Comments:

## QA Summary Report

Batch Number: StarLIMS #233421

Method Number: 1630M

Project Number(s): MeHg in Tissues

Instrument ID: K-AFS-04

Date Analyzed: 1/21/11

Analyst Name: [REDACTED]

Run	Name/ID	Final Result (µg/Kg)	Notes
6	K1100460-001	-1.36	accept
9	K1100460-002	-1.08	accept
10	K1100460-003	0.0788	accept
11	K1100460-004	-1.06	accept
12	K1100460-005	-0.365	accept

## Run Information Report

Batch Number: StarLIMS #233421

Method Number: 1630M

Project Number(s): MeHg in Tissues

Instrument ID: K-AFS-04

Date Analyzed: 1/21/11

Analyst Name: [REDACTED]

Run	Run Type	Name/ID	Method Blank	Sample Vol/Wt	Dilution Vol (ml)	Analyzed Vol (ml)	Expected Value	Notes
1	OPR	OPR					100	
2	QCS	TORT	MBA	198	50	0.050	163	mg/Kg
3	MBA	MBLK 1		250	50	0.050		
4	MBA	MBLK 2		250	50	0.050		
5	MBA	MBLK 3		250	50	0.050		
6	S	K1100460-001	MBA	259	50	0.050		
7	MS	K1100460-001	MBA	275	50	0.050	1818	
8	MSD	K1100460-001	MBA	259	50	0.050	1931	
9	S	K1100460-002	MBA	271	50	0.050		
10	S	K1100460-003	MBA	252	50	0.050		
11	S	K1100460-004	MBA	270	50	0.050		
12	S	K1100460-005	MBA	264	50	0.050		
13	OPR	OPR					100	

**Columbia Analytical Services, Inc.**

Sample Number(s): As Listed	Service Request Number(s): K1100460
Analysis for: MeHg in Tissues	Method: 1630m

## DATA

**StarLIMS #127580**

[illegible]

Comments: Spike Standard: 0.5ml of 1000ng/mL AF1-57-A  
25% KOH; RE2-33-P

Ar		Date: 1/20/2011
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**Columbia Analytical Services, Inc.**

Service Request Number(s):

K1100460

prep # 127580

## DATA

[illegible]

Comments: 1+3 no seq.

Date: 1/20/2011

# Sample Results Summary Report

Batch Number: StarLIMS #233421

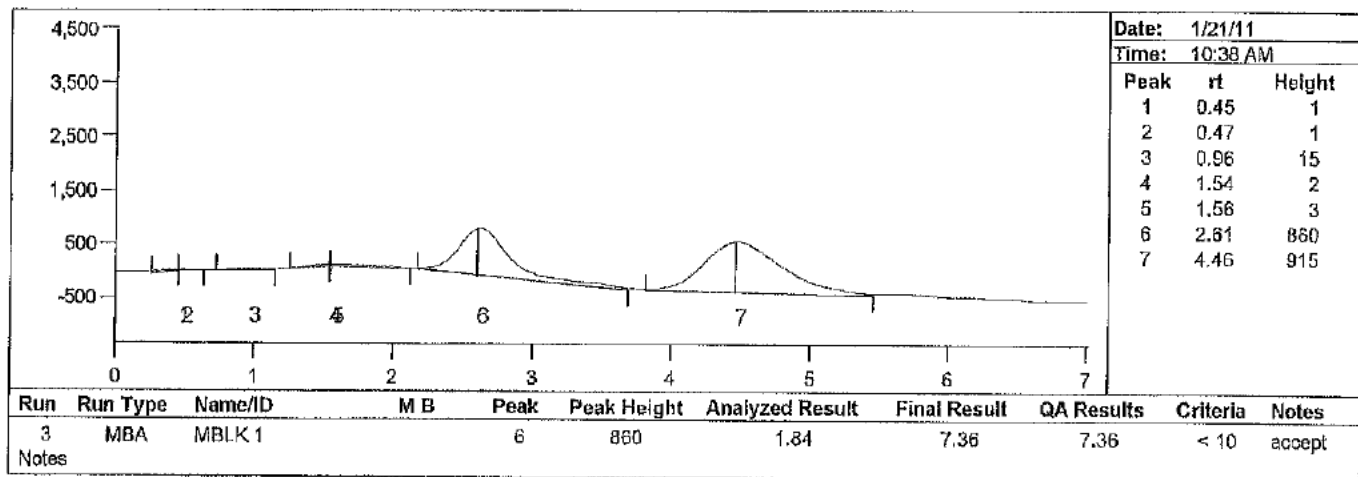
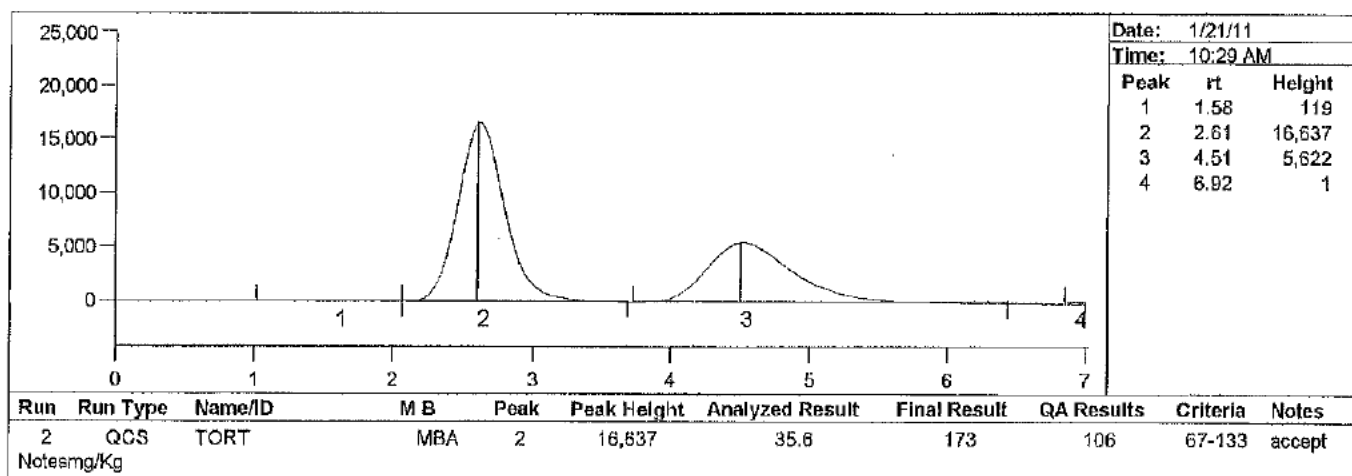
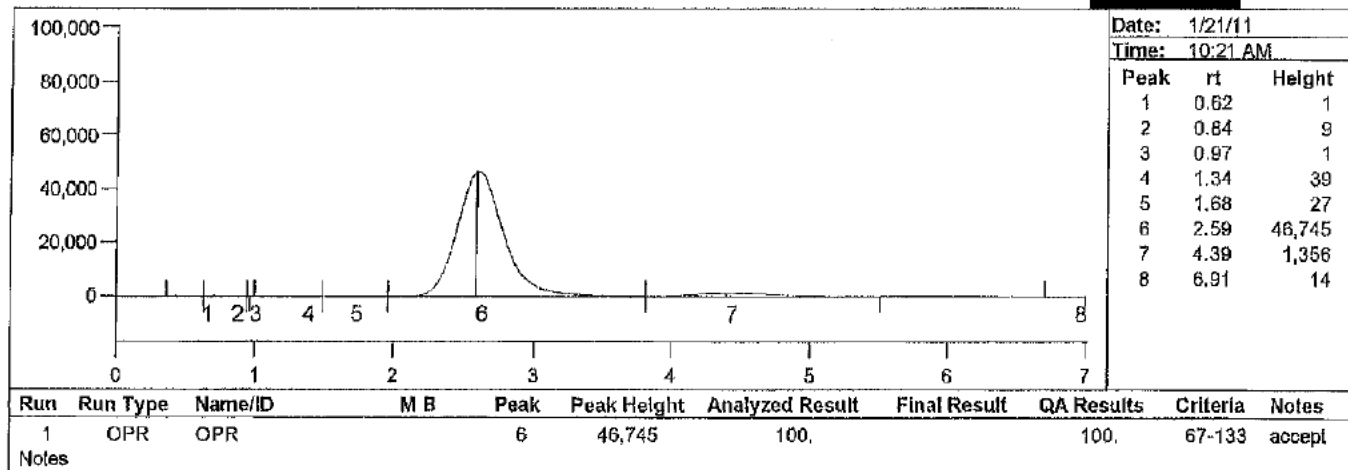
Method Number: 1630M

Project Number(s): MeHg in Tissues

Instrument ID: K-AFS-04

Date Analyzed: 1/21/11

Analyst Name: [REDACTED]



# Sample Results Summary Report

Batch Number: StarLIMS #233421

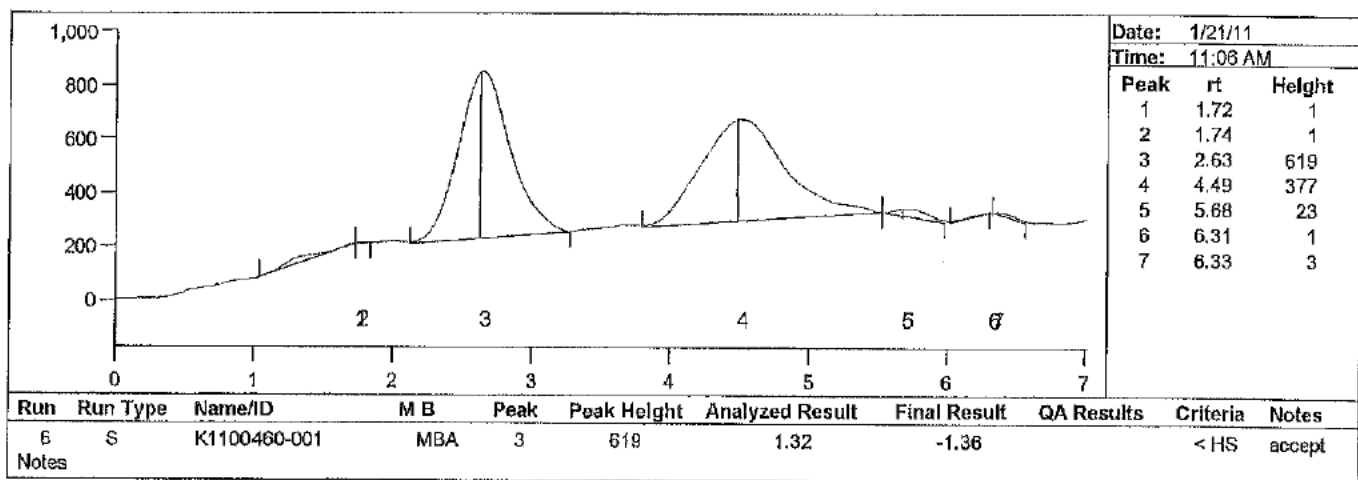
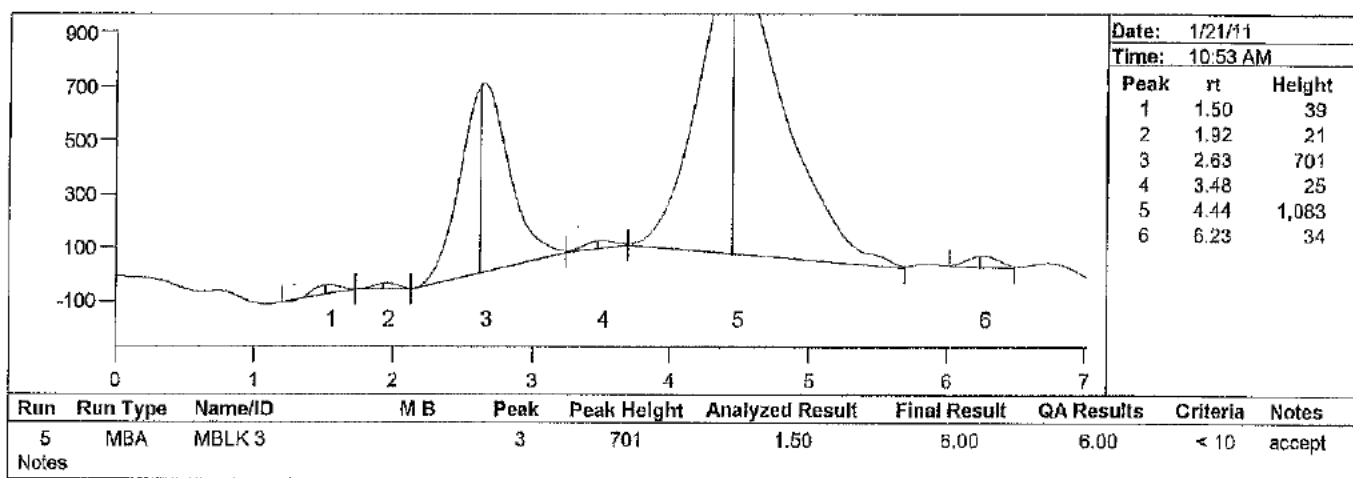
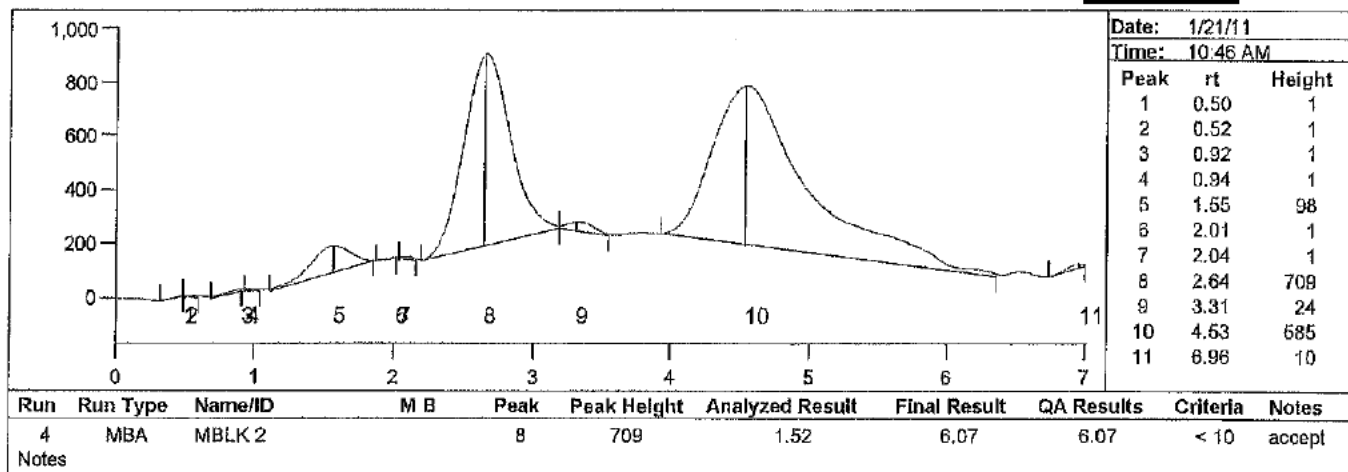
Method Number: 1630M

Project Number(s): MeHg in Tissues

Instrument ID: K-AFS-04

Date Analyzed: 1/21/11

Analyst Name: [REDACTED]



# Sample Results Summary Report

Batch Number: StarLIMS #233421

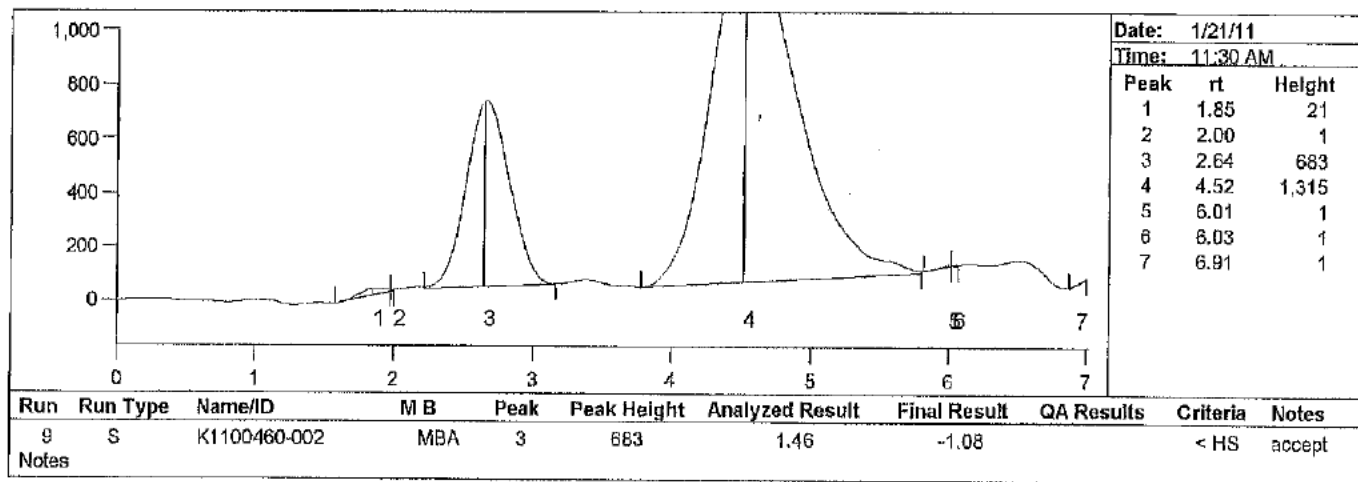
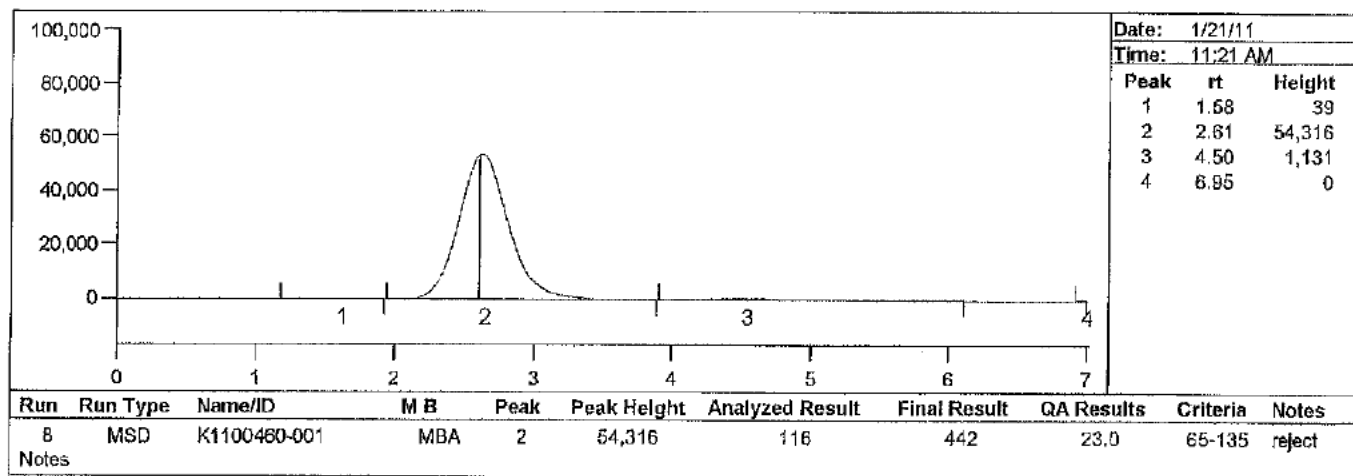
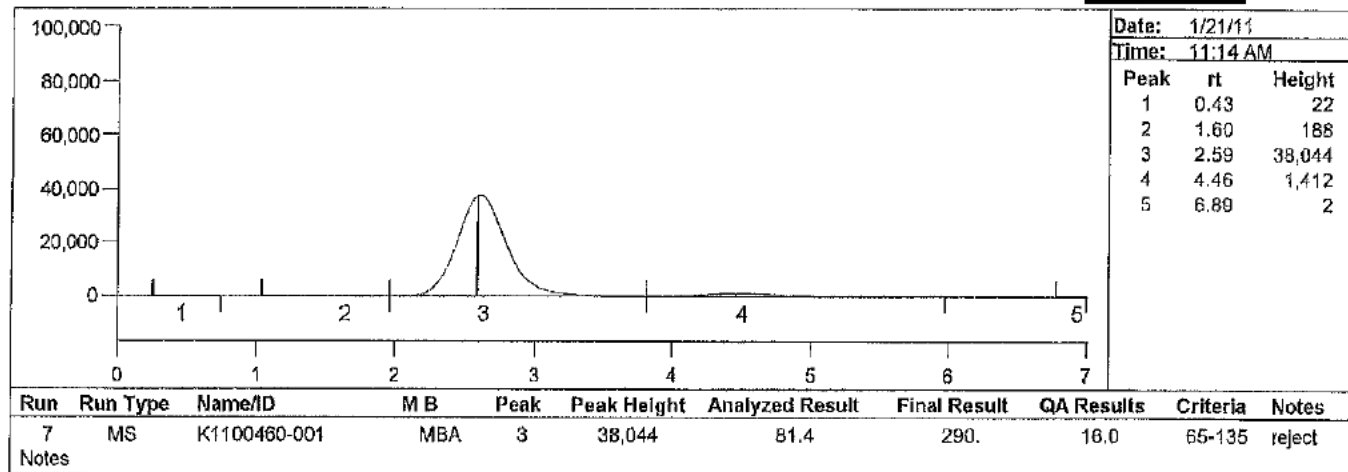
Method Number: 1630M

Project Number(s): MeHg in Tissues

Instrument ID: K-AFS-04

Date Analyzed: 1/21/11

Analyst Name: [REDACTED]



# Sample Results Summary Report

Batch Number: StarLIMS #233421

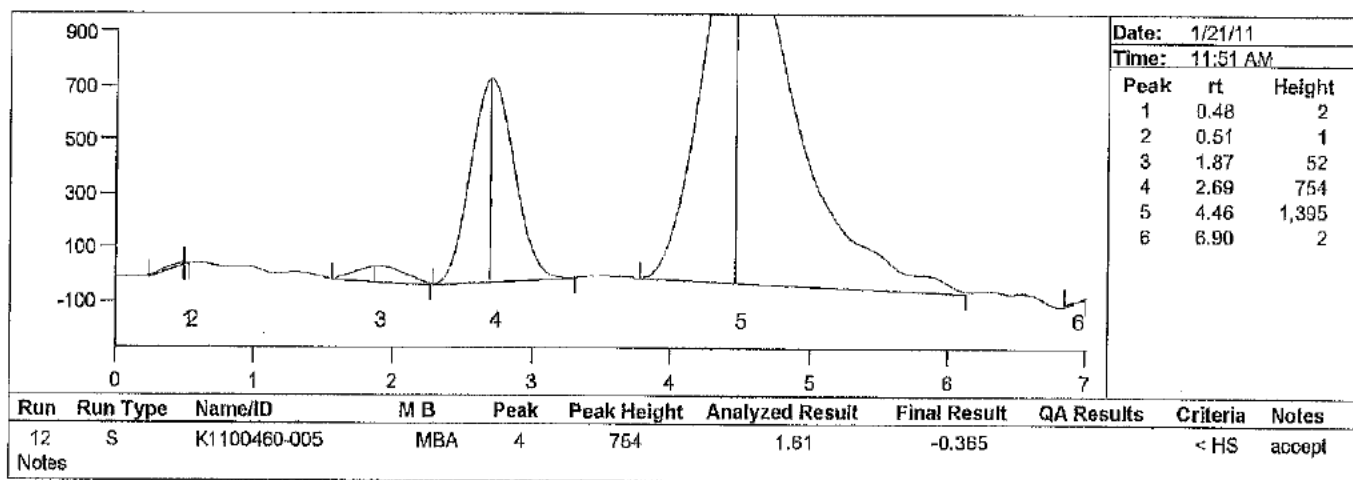
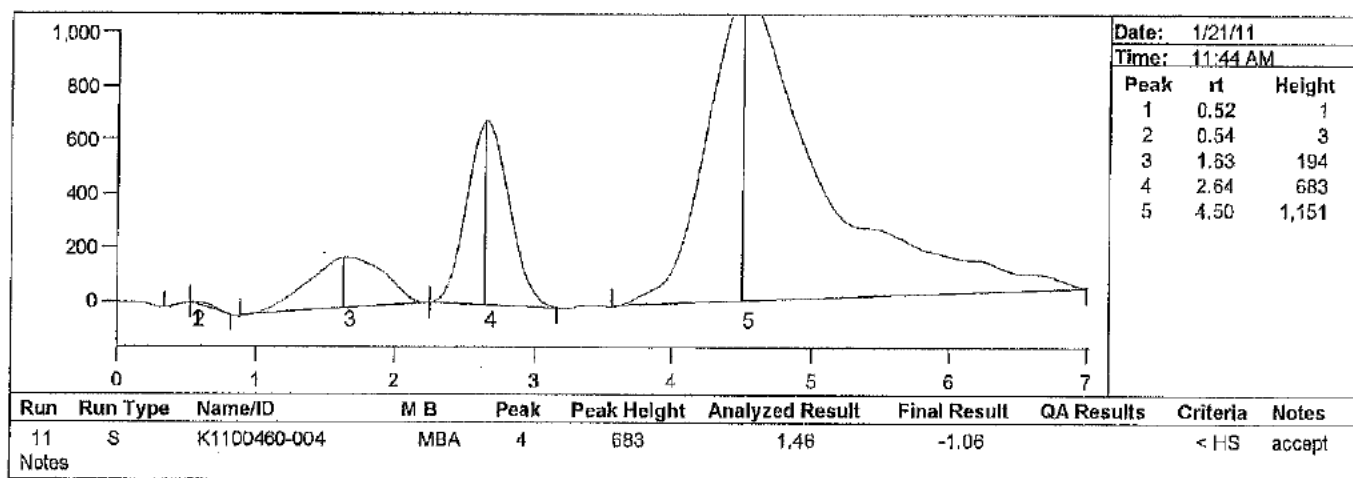
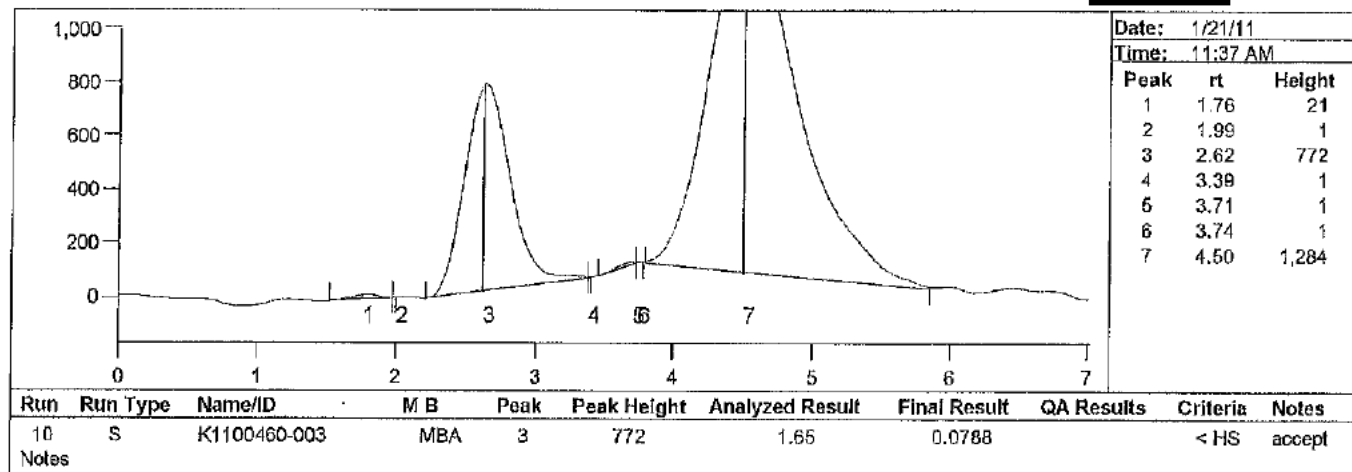
Method Number: 1630M

Project Number(s): MeHg in Tissues

Instrument ID: K-AFS-04

Date Analyzed: 1/21/11

Analyst Name: [REDACTED]



# Sample Results Summary Report

Batch Number: StarLIMS #233421

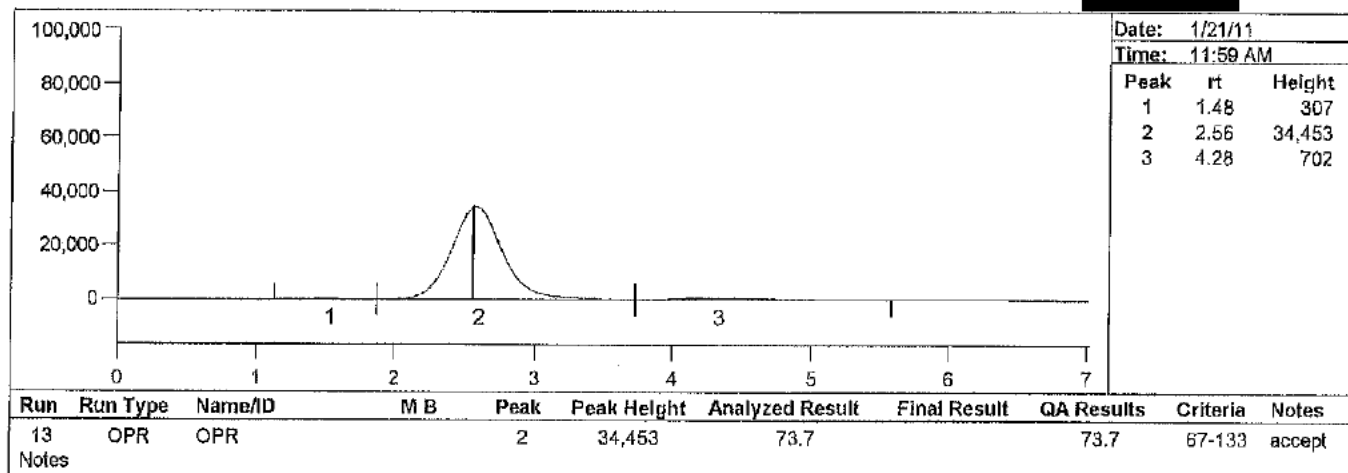
Method Number: 1630M

Project Number(s): MeHg in Tissues

Instrument ID: K-AFS-04

Date Analyzed: 1/21/11

Analyst Name





# CERTIFICATE OF ACCREDITATION

**ANSI-ASQ National Accreditation Board/ACCLASS**  
500 Montgomery Street, Suite 625, Alexandria, VA 22314, 877-344-3044

This is to certify that

**Columbia Analytical Services, Inc.**  
**1317 South 13<sup>th</sup> Ave.**  
**Kelso, WA 98626**

has been assessed by ACLASS  
and meets the requirements of international standard

**ISO/IEC 17025:2005**

while demonstrating technical competence in the field(s) of

**TESTING**

Refer to the accompanying Scope(s) of Accreditation for information regarding the types of tests to which this accreditation applies.

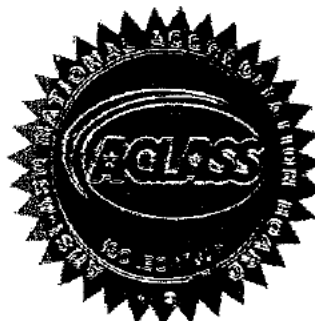
AT-1412

Certificate Number



ACCLASS Approval

Certificate Valid: 11/06/2009-11/06/2011  
Version No. 001



This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2005. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated January 2009).



## ANSI-ASQ National Accreditation Board/ACCLASS

### SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

#### Columbia Analytical Services, Inc.

1317 South 13<sup>th</sup> Ave. Kelso, WA. 98626

Phone: 360-577-7222

#### TESTING

Valid to: November 6, 2011

Certificate Number: AT - 1412

#### I. Environmental and Chemical

ITEMS, MATERIALS OR PRODUCTS TESTED	SPECIFIC TESTS OR PROPERTIES MEASURED	SPECIFICATION, STANDARD METHOD OR TECHNIQUE USED	*DETECTION LIMIT/ RANGE/ EQUIPMENT
Water / Solid	Analysis of Volatile compounds	EPA 8260B / 8260C	GC-MS
Water / Solid	Analysis of Semivolatile Organic Compounds	EPA 8270C / 8270D	GC-MS
Water / Solid	Polychlorinated Biphenyls (PCBs)	EPA 8082 / 8082B	GC-ECD
Water / Solid	Organochlorine Pesticides	EPA 8081A / 8081B	GC-ECD
Water / Solid	Gasoline Range Organics/Diesel Range Organics	EPA 8015B	GC-FID
Water / Solid	Analysis of Explosives	EPA 8330 EPA 8330B	HPLC
Water / Solid	Aromatic and Halogenated Volatiles	EPA 8021B / 8021D	GC-FID
Water / Solid	Chlorinated Herbicides using Methylation	EPA 8151A	GC-ECD
Water / Solid	Metals	EPA 6010B / 6010C	ICP



ITEMS, MATERIALS OR PRODUCTS TESTED	SPECIFIC TESTS OR PROPERTIES MEASURED	SPECIFICATION, STANDARD METHOD OR TECHNIQUE USED	*DETECTION LIMIT/ RANGE/ EQUIPMENT
Water / Solid	Metals	EPA 6020 / 6020A	ICP-MS
Water / Solid	Anions	EPA 9056 / 9056A EPA 5050 & 1312	Ion Chromatograph
Water / Solid	Total and Amenable Cyanides	EPA 9010C / 9012B	Midi-Distillation Unit
Water / Solid	Hexavalent Chromium	EPA 7196A EPA 3060	Spectrophotometer
Water / Solid	Mercury in Liquid Waste	EPA 7470A	Cold vapor AA
Water / Solid	Mercury in Solid or Semi-solid	EPA 7471A / 7471B	Cold vapor AA
Water / Solid	Total Organic Halides (TOX)	EPA 9020B	Microcoulometric-titration detector
Water / Solid	Acid-soluble and Acid- insoluble Sulfides	EPA 9030B	Distillation Unit
Water / Solid	pH	EPA 9045D EPA 9040C	pH Meter
Water / Solid	Phenolics	EPA 9065	Spectrophotometer
Water / Solid	Total Organic Carbon	EPA 9060A	TOC Meter
Water / Solid	n-Hexane Extractable Material (HEM)	EPA 9071A EPA 1664	Gravimetric
Solids	Phthalates	CPSC-CH-C1001-09.1 Modified 8270	GC



ITEMS, MATERIALS OR PRODUCTS TESTED	SPECIFIC TESTS OR PROPERTIES MEASURED	SPECIFICATION, STANDARD METHOD OR TECHNIQUE USED	*DETECTION LIMIT/ RANGE/ EQUIPMENT
Soil	Acid Digestion for Sediments, Sludges and Soils	EPA 3050B	Hot plate
Waste Water	Acid Digestion of Aqueous Samples and Extracts for Total Metals by ICP	EPA 3010A	Hot plate
Waste Water	Acid Digestion of Aqueous Samples and Extracts for Total Metals by ICP-MS	EPA 3020A	Hotplate
Waste Water	Continuous Liquid- Liquid Extraction	EPA 3520C	
Soil	Automated Soxhlet Extraction	EPA 3541	
Waste Water / Soil	Gel-Permeation Clean- up	EPA 3640A	
Waste Water	Solid Phase Extraction	EPA 3535 / 3535A	
Lead in Paint by ICP	Lead	16 CFR 1303 (using EPA 3051M/6010)	ICP
Total Lead in Children's Metal Jewelry	Lead	CPSC-CH-E1001-08 (using EPA 3051M/6010)	ICP

**Notes:**

1. \* = As Applicable
2. This scope is part of and must be included with the Certificate of Accreditation No. AT- 1412



Vice President



01/25/11

## Certificate of Analysis

<b>Sponsor:</b>  An-Ni Chang Analytical Sciences Martek Biosciences Corporation 6480 Dobbin Road Columbia, MD 21045  <b>Client #:</b> MK499	<div style="background-color: black; width: 100px; height: 20px; margin-bottom: 5px;"></div> <b>Sample ID#:</b> 11010011  <b>Product Description:</b> Algal Oil Lot: DHB08-6530  <b>Date Received:</b> 01/13/11
<b>Quotation #:</b> CyQ101207M-2	
<b>Report #:</b> R110125A <span style="float: right;"><b>Version:</b> 0</span>	

Test Description	Method Reference	Results (Test Date) (Reference)
Freezing Point	European Pharmacopoeia 7.0, 2.2.18 Freezing Point 01/2008:20218	-9.7°C (01/21/11) (NB: 225 ; P: 11 )

No Quality Assurance review or approval was performed on this report.

Prepared By:

Cyanta Analytical Services  
Maryland Heights, MO

01/25/11  
Date

Reviewed By:

Laboratory Manager  
Cyanta Analytical Services  
Maryland Heights, MO

01/25/11  
Date

01/25/11

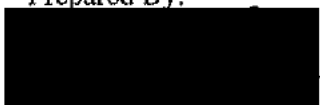
## Certificate of Analysis

<b>Sponsor:</b>  An-Ni Chang Analytical Sciences Martek Biosciences Corporation 6480 Dobbin Road Columbia, MD 21045  Client #: MK499	<div style="background-color: black; width: 100px; height: 15px; margin-bottom: 5px;"></div> <b>Sample ID#:</b> 11010012  <b>Product Description:</b> Algal Oil Lot: DHB08-6586  <b>Date Received:</b> 01/13/11
<b>Quotation #:</b> CyQ101207M-2	
<b>Report #:</b> R110125B	<b>Version:</b> 0

Test Description	Method Reference	Results (Test Date) (Reference)
Freezing Point	European Pharmacopoeia 7.0, 2.2.18 Freezing Point 01/2008:20218	-10.3°C (01/21/11) (NB: 225 ; P: 11 )

No Quality Assurance review or approval was performed on this report.

Prepared By:



Cyanta Analytical Services  
Maryland Heights, MO

01/25/11  
Date

Reviewed By:



Laboratory Manager  
Cyanta Analytical Services  
Maryland Heights, MO

01/25/11  
Date

01/25/11

## Certificate of Analysis

<b>Sponsor:</b> <div style="background-color: black; width: 100px; height: 15px; margin: 5px 0;"></div> Analytical Sciences Martek Biosciences Corporation 6480 Dobbin Road Columbia, MD 21045  <b>Client #:</b> MK499	<b>CONFIDENTIAL</b>  <b>Sample ID#:</b> 11010013  <b>Product Description:</b> Algal Oil Lot: DHB08-6592  <b>Date Received:</b> 01/13/11
<b>Quotation #:</b>	<b>CyQ101207M-2</b>
<b>Report #:</b>	<b>R110125C</b> <span style="float: right;"><b>Version:</b> 0</span>

Test Description	Method Reference	Results (Test Date) (Reference)
Freezing Point	European Pharmacopoeia 7.0, 2.2.18 Freezing Point 01/2008:20218	-8.6°C (01/21/11) (NB: 225 ; P: 11 )

No Quality Assurance review or approval was performed on this report.

Prepared By:

Cyanta Analytical Services  
Maryland Heights, MO

01/25/11  
Date

Reviewed By:

Laboratory Manager  
Cyanta Analytical Services  
Maryland Heights, MO

01/25/11  
Date

01/25/11

## Certificate of Analysis

<b>Sponsor:</b> <div style="background-color: black; width: 100px; height: 20px; margin: 5px 0;"></div> Analytical Sciences Martek Biosciences Corporation 6480 Dobbin Road Columbia, MD 21045  <b>Client #:</b> MK499	<div style="background-color: black; width: 100px; height: 20px; margin: 5px 0;"></div> <b>Sample ID#:</b> 11010014  <b>Product Description:</b> Algal Oil Lot: DHB08-6585  <b>Date Received:</b> 01/13/11
<b>Quotation #:</b> CyQ101207M-2	
<b>Report #:</b> R110125D	<b>Version:</b> 0

Test Description	Method Reference	Results (Test Date) (Reference)
Freezing Point	European Pharmacopoeia 7.0, 2.2.18 Freezing Point 01/2008:20218	-8.7°C (01/21/11) (NB: 225 ; P: 11 )

No Quality Assurance review or approval was performed on this report.

Prepared By:

Cyanta Analytical Services  
Maryland Heights, MO

01/25/11  
Date

Reviewed By:

Cyanta Analytical Services  
Maryland Heights, MO

Laboratory Manager

01/25/11  
Date

01/25/11

## Certificate of Analysis

<b>Sponsor:</b> <div style="background-color: black; width: 100px; height: 15px; margin: 5px 0;"></div> Analytical Sciences Martek Biosciences Corporation 6480 Dobbin Road Columbia, MD 21045  <b>Client #:</b> MK499	<div style="background-color: black; width: 100px; height: 15px; margin: 5px 0;"></div> <b>Sample ID#:</b> 11010015  <b>Product Description:</b> Algal Oil Lot: DHB08-6643  <b>Date Received:</b> 01/13/11
<b>Quotation #:</b> CyQ101207M-2	
<b>Report #:</b> R110125E	<b>Version:</b> 0

Test Description	Method Reference	Results (Test Date) (Reference)
Freezing Point	European Pharmacopoeia 7.0, 2.2.18 Freezing Point 01/2008:20218	-8.4°C (01/21/11) (NB: 225 ; P: 11 )

No Quality Assurance review or approval was performed on this report.

Prepared By:

Cyanta Analytical Services  
Maryland Heights, MO

01/25/11  
Date

Reviewed By:

Laboratory Manager  
Cyanta Analytical Services  
Maryland Heights, MO

01/25/11  
Date