

Battelle Marine Sciences Laboratory

MDL - Total Hg in Water

MDL	ANALYSIS DATE	PEAK SEQ#	DIGESTED VOLUME	TOTAL VOLUME	SAMPLE VOL ANALYZED ml	AREA	BLK CORR [Hg] ng/l	QA DATA
MDL 1	12/29/1999	38101	98.557	99.307	71.4	40854	0.585	
MDL 2	12/29/1999	38102	103.353	104.103	60.48	25089	0.382	Mean = 0.450
MDL 3	12/29/1999	38103	102.445	103.195	48.66	25129	0.492	S.D. = 0.0702
MDL 4	12/29/1999	38114	97.049	97.799	52.45	25656	0.464	RSD = 15.6%
MDL 5	12/29/1999	38105	96.202	96.952	57.27	29543	0.503	
MDL 6	12/29/1999	38106	98.206	98.956	70.92	30730	0.414	
MDL 7	12/29/1999	38108	107.797	108.547	50.31	20513	0.361	MDL = 0.203
MDL 8	12/29/1999	38109	95.614	96.364	63.16	26915	0.399	

All units are in ng/liter

Samples were prepared with tap water spiked at 0.5 ng/liter

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MDL - Methyl Hg in Water

	ANALYSIS DATE	DISTILLED VOLUME	SAMPLE VOL ANALYZED ml	AREA	DISTILLATION CORRECTION	BLK CORR [Hg] ng/l	QA DATA
MDL 1	12/29/1999	49.999	49.999	473	0.770	0.0713	
MDL 2	12/29/1999	51.369	51.369	439	0.770	0.0582	Mean=0.0867
MDL 3	12/29/1999	50.274	50.274	439	0.770	0.0595	StdDev=0.0291
MDL 4	12/29/1999	50.911	50.911	480	0.770	0.0723	MDL=0.0873
MDL 5	12/29/1999	51.215	51.215	646	0.770	0.1263	
MDL 6	12/29/1999	49.803	49.803	541	0.770	0.0945	
MDL 7	12/29/1999	49.750	49.75	631	0.770	0.1250	

All units are in ng/liter

Samples were prepared with tap water spiked at 0.1 ng/liter

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MDL - Total mercury in sediment

	ANALYSIS DATE	SAMPLE MASS.g	TOTAL DIGESTED VOLUME	PEAK AREA	BLK CORR [Hg], μ g/g	QA DATA
MDL 1	12/20/1999	0.208	24.45	262378	0.0465	
MDL 2	12/20/1999	0.202	24.35	290292	0.0543	Mean = 0.0477
MDL 3	12/20/1999	0.192	24.32	260412	0.0509	StdDev = 0.00316
MDL 4	12/20/1999	0.201	24.36	260518	0.0480	MDL = 0.00915
MDL 5	12/20/1999	0.192	24.36	239309	0.0461	
MDL 6	12/20/1999	0.203	24.42	248626	0.0449	
MDL 7	12/20/1999	0.21	24.38	254440	0.0440	
MDL 8	12/20/1999	0.202	24.35	257910	0.0471	

All units are in μ g/gram

Samples prepared with quartz sand spiked at 0.05 ng/g

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MDL - Methylmercury in sediment

	ANALYSIS DATE	SAMPLE MASS.g	TOTAL EXTRACTED VOLUME	SAMPLE VOL ANALYZED ml	PEAK AREA	BLK CORR [Hg] ng/g	QA DATA
MDL r1	12/22/1999	0.202	10.18	2.09	917	0.153	
MDL r2	12/22/1999	0.203	10.16	1.98	858	0.141	Mean=0.137
MDL r3	12/22/1999	0.200	10.20	2.02	768	0.111	Std Dev=0.0200
MDL r4	12/22/1999	0.196	10.16	2.09	929	0.162	MDL=0.0578
MDL r5	12/22/1999	0.198	10.17	2.12	891	0.146	
MDL r6	12/22/1999	0.204	10.18	2.02	859	0.138	
MDL r7	12/22/1999	0.201	10.18	2.03	875	0.145	
MDL r8	12/22/1999	0.199	10.09	2.02	752	0.104	

All units are in ng/gram

Samples prepared with quartz sand spiked at 0.1 ng/g

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MDL - Total mercury in tissue

	ANALYSIS DATE	SAMPLE MASS, g	TOTAL DIGESTED VOLUME	PEAK AREA	BLK CORR [Hg] μ g/g	QA DATA
MDL 1	12/29/1999	0.480	22.35	607347	0.0587	
MDL 2	12/29/1999	0.464	22.34	589479	0.0591	Mean = 0.0558
MDL 3	12/29/1999	0.518	22.39	630000	0.0560	StdDev = 0.00441
MDL 4	12/29/1999	0.485	22.11	612708	0.0578	MDL = 0.0128
MDL 5	12/29/1999	0.493	21.06	546607	0.0471	
MDL 6	12/29/1999	0.486	22.51	640166	0.0618	
MDL 7	12/29/1999	0.482	22.46	561571	0.0539	
MDL 8	12/29/1999	0.504	22.27	572842	0.0517	

All units are in μ g/gram

Samples prepared with egg yolkspiked at 0.05 ng/g

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MDL - Methylmercury in tissue

	ANALYSIS DATE	SAMPLE MASS g	TOTAL SAMPLE VOLUME	SAMPLE VOL ANALYZED ml	PEAK AREA	BLK CORR [Hg] μ g/g	QA DATA
MDL r1	12/16/1999	0.207	20	0.025	494	0.0133	
MDL r2	12/16/1999	0.196	20	0.025	523	0.0157	Mean = 0.0125
MDL r3	12/16/1999	0.206	20	0.025	461	0.0115	Std Dev = 0.00167
MDL r4	12/16/1999	0.205	20	0.025	444	0.0106	MDL= 0.00482
MDL r5	12/16/1999	0.201	20	0.025	452	0.0113	
MDL r6	12/16/1999	0.199	20	0.025	490	0.0136	
MDL r7	12/16/1999	0.207	20	0.025	486	0.0128	
MDL r8	12/16/1999	0.205	20	0.025	459	0.0114	

All units are in μ g/gram

Samples prepared with egg yolk spiked at 0.01 μ g/g

Beverly van Buuren

From: hetaylor@usgs.gov
Sent: Thursday, February 24, 2000 12:27 PM
To: BeverlyvB@frontier.wa.com
Cc: cnalpers@maildcasr.wr.usgs.gov; joed@usgs.gov
Subject: MDL information for determination of Hg in sediment



Paintbrush

Beverly,

We have completed the spike addition calculations for the determination of Hg in sediments. The general approach was as follows:

- 1) Reagent/glassware matrix blanks were measured on 7 replicates.
- 2) The mean and standard deviation were calculated on a mass equivalent basis as 0.13 ng/g and 0.17 ng/g, respectively. This results in a matrix blank detection limit of 0.5 ng/g.
- 3) Seven samples of Ottawa sand were digested and preserved with nitric acid and potassium dichromate.
- 4) The mean and standard deviation were calculated to be 0.20 ng/g with a standard deviation of 0.23.
- 5) Seven individual Ottawa sand samples were each spiked and digested (1 hour) with the equivalent of 2 ng/g Hg.
- 6) Spiked samples were processed as in step 3.
- 7) The mean of the spiked samples was 1.97 ng/g with a standard deviation of 0.25. A detection limit (MDL) calculated from this data was determined to be 0.8 ng/g.

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Hopefully, this information will be satisfactory for the approval process.
Thanks for your prompt attention to this matter.

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Mercury in Sediment MDL Study Data

Matrix Blank	net [Hg], ng/g	comment
Flsk1 blank	0.39	
Flsk2 blank	0.35	
Flsk4 blank	0.03	
Flsk6 blank	0.00	
Flsk8 blank	0.00	
Flsk13 blank	0.00	
Flsk14 blank	0.12	
mean	0.13	
stddev (s)	0.17	note: the MDL calculated from the matrix blank = 0.5 ng/g
Ottawa Sand		
Flsk2+OS	0.57	
Flsk4+OS	0.00	
Flsk6+OS	0.12	
Flsk8+OS	0.12	
Flsk13+OS	0.12	
Flsk14+OS	0.48	
Flsk1+OS	0.00	
mean	0.20	
stddev (s)	0.23	
Ottawa Sand Plus 2 ng/g spike		
F2+52.4OS+Sp	2.18	net 1.98 = 98.9 % rec
F4+52.0OS+Sp	2.04	net 1.84 = 92.2 % rec
F6+51.2OS+Sp	2.22	net 2.02 = 101.1 % rec
F8+55.4OS+Sp	2.04	net 1.84 = 92.2 % rec
F13+55.0OS+Sp	1.60	net 1.40 = 69.8 % rec
F14+52.0OS+Sp	2.09	net 1.89 = 94.4 % rec
F1+52.0OS+Sp	1.64	net 1.44 = 72.0 % rec
mean	1.97	mean net 1.74 = 89 % rec
stddev (s)	0.25	13 % RSD

Using 40 CFR 136, the MDL was calculated using the standard deviation of the spiked samples, with n = 6 degrees of freedom. In this case, the t value of 3.143 was used in the following equation, where s is the standard deviation of the results obtained on samples spiked at a level near the MDL:

$$\text{MDL} = t*s.$$

The MDL calculated from these data is $(3.143)*(0.25)$, or **0.8 ng/g**.

Spike recovery data indicate that good recovery is found in the concentration range normally encountered in environmental samples. Duplicate samples were collected and processed, one was preserved with the normal preservative while the other received the same preservative with a known concentration of Hg at approximately 0.0040 µg/L depending on the exact original sample volume. The recoveries ranged from 89.7 % to 116.3 % and had a mean of 101.5 % with a relative standard deviation of 8.2 % from a statistical population of 16 observations.

Method Performance

MDL Estimate Based upon Variability of the Method Blanks of Individual Analysis Sets

Recent results for the method detection limit for total dissolved Hg in water are presented in Table 2. The average MDL calculated using the method of Skogerboe and Grant, 1970, of these analysis sets is 0.3 ng/L.

Demonstrated Method Accuracy for Low Level Water Samples.

Results for spike recoveries of a 4.0 ng/L spike addition on natural water samples are presented in Table 3. The estimated accuracy of the method is 101.5 ± 8.2 % (n=16).

Demonstrated Precision for Low Level Water Samples.

Results for replicate analyses on SRMs are presented in Table 4. Percent relative standard deviation is presented as a measure of precision. The estimated precision of the method at various concentrations is given in Table 4.

Demonstrated Accuracy.

Recent results for recoveries of total Hg on standard reference material are presented in Table 5. Percent recoveries for several SRMs are listed.

Table 2 Recent Method Detection Limits-Dissolved

Date	Analyst	Detection Limit (ng/L)	Analysis Set Type
11/10/99	DBP/DAR	0.4	Dissolved
10/06/99	DBP/DAR	0.4	Dissolved
10/05/99	DBP/DAR	0.4	Dissolved
10/01/99	DBP/DAR	0.4	Dissolved
09/30/99	DBP/DAR	0.3	Dissolved
09/24/99	DBP/DAR	0.2	Dissolved
09/21/99	DBP/DAR	0.3	Dissolved
09/17/99	DBP/DAR	0.2	Dissolved
09/16/99	DBP/DAR	0.3	Dissolved
09/13/99	DBP/DAR	0.3	Dissolved
09/10/99	DBP/DAR	0.3	Dissolved
09/09/99	DBP/DAR	0.2	Dissolved
09/03/99	DBP/DAR	0.3	Dissolved
MEAN Detection Limit (ng/L)		0.30	
SD		0.07	
N		13	
ESTIMATED MDL		0.3	

Table 3. Accuracy data (results for recovery of 4.0 ng/L matrix spikes, ~ 2-4 x ambient concentration) for total mercury in low level water samples.

Date	% Recovery	
9/96	95	
9/96	96.6	
9/96	101.4	
9/96	98.1	
9/96	98.9	
9/96	105.3	
9/96	92	
9/96	93.4	
9/96	100.3	
1/97	89.7	
1/97	110.3	
1/97	116.3	
1/97	101.5	
1/97	111.7	
1/97	115.7	
1/97	97.2	
MEAN Matrix Accuracy (%)		101.5
SD		8.2
N		16

Table 4 Precision data for total dissolved mercury in low level water samples: Values for low level total dissolved Hg in water determinations

SRM	Dilution	[Hg] ng/L		N	Relative Percent Deviation
		Mean	Std Dev		
Hg7	1:100	2.4	0.2	102	8.3
Hg12	1:100	14.6	0.4	84	2.7
Hg14	1:100	6.8	0.2	30	2.9
Hg15	1:100	4.0	0.3	30	7.5

Table 5 Accuracy data for total dissolved mercury.

SRM	Dilution	[Hg] ng/L				Percent recovery
		Mean	Std Dev	Reported Value	Reported Std Dev	
Hg7	1:100	2.5	0.2	2.2	0.8	114
Hg12	1:100	14.6	0.3	14.4	2.5	101
Hg14	1:100	6.8	0.4	7.0	2.9	97
Hg15	1:100	4.0	0.3	4.1	2.0	98
NIST 1641b	1:40000	38.2	0.8	38.0	1.0	101

Reference

Corns, W.T., Ebdon, L., Hill, S.J. and Stockwell, P.B., *Analyst*, 1992, 117, 717.

Friedman, L.C. and Fishman, M.J., 1989, U. S. Geological Survey Water-Supply Paper 2293, 126 p.

Hatch, W.R. and Ott, W.L., 1968, *Analytical Chemistry*, 40, 2085

Kuehner, E.C., R. Alvarez, P.J. Paulsen, and T.J. Murphy, 1972, Production and analysis of special high-purity acids purified by sub-boiling distillation. *Anal. Chem.* 44, 2050-2056.

Roth, D.A., 1994, "Ultratrace Analysis of Mercury and its Distribution in Some Natural Waters in the United States", Ph.D. Dissertation, Colorado State University, Fort Collins, Colorado.

Roth, D.A. and Taylor, H.E., 1997, *39th Annual Rocky Mountain Conference on Analytical Chemistry, Professor R.K. Skogerboe Honorary Symposium on Atomic and Inorganic-Mass Spectrometry*, August 3-7.

Total Hg Water MDL Study

Sample	[Hg], pg/mL	Comment
blank #1	0.22	
blank #2	0.12	
blank #3	0.05	
blank #4	0.12	
mean	0.13	
SD	0.07	
Spike 1 (+.5007pg/mL)	0.55	net 0.42=83.9% recovery
Spike 2 (+.5009pg/mL)	0.77	net 0.64=127.8% recovery
Spike 3 (+.5009pg/mL)	0.49	net 0.36=71.9% recovery
Spike 4 (+.5009pg/mL)	0.54	net 0.41=81.9% recovery
Spike 5 (+.5007pg/mL)	0.59	net 0.46=91.9% recovery
Spike 6 (+.5007pg/mL)	0.59	net 0.46=91.9% recovery
Spike 7 (+.5008pg/mL)	0.59	net 0.46=91.9% recovery
Spike 8 (+.5009pg/mL)	0.60	net 0.47=93.8% recovery
Spike 9 (+.5007pg/mL)	0.58	net 0.45=89.9% recovery
mean	0.59	mean net .46=91.9% recovery
SD	0.076	
Dorm-2 (pg/mL)	4129600.0	89.0% recovery
certified value	4640000 +/- 260000	

MDL Calculated $(2.896) * (.076) = .22 \text{ pg/mL}$

Matrix Specific MDL Study: Methyl Mercury in Sediments and Soils (CH₂Cl₂ Extraction)

February 23, 2000

Department of Fish and Game & Moss Landing Marine Labs
P.O. Box 747
Moss Landing, Ca 95039

Objective. Determine the method detection limit (MDL) for methyl mercury in soils and sediments, using EPA Method 1630 and 40 CFR 136, after distillation extraction. The MDL found for this matrix is **0.019 ng/g Hg.**

Analytical Method. One gram aliquots of clean reagent grade river sand were mixed with 5.0 mL of 18 % KBr + 5 % H₂SO₄ solution, 1.0 mL 1 M CuSO₄ solution, and 10.0 mL of CH₂Cl₂ in 35 mL Teflon centrifuge tubes. Samples were shaken for 8 hours to extract the MMHg, and then centrifuged to separate the layers. Two milliliters of the CH₂Cl₂ was then placed into 40 mL of DI water, and purged out of solution, leaving the MMHg in aqueous solution. The study set consisted of 4 samples of pure sand, 4 method blanks, 9 samples of sand spiked with 0.056 ng/g MMHg, 4 samples spiked with 1.121 ng/g MMHg, and 1 reference soil (FGS 1944). A calibration, covering most of the reading range of the instrument (0-0.160 ng Hg) was performed using Frontier Geosciences Methods FGS-045 & FGS-045.2. Briefly, this method incorporates aqueous phase ethylation, collection on Carbotrap, isothermal GC separation, and cold vapor atomic fluorescence spectrometry (CVAFS). The calibration was linear over the entire range. Recoveries on the CRM (DORM-1, certified value of 0.731 mg/Kg) were 92.5-109.1 %. The results of these measurements are found in the table on the next page. Results are **corrected** for the method blanks.

MDL Calculation. Using 40 CFR 136, the MDL was calculated using the standard deviation of the spiked samples, with n = 8 degrees of freedom. In this case, the t value of 2.896 was used in the following equation, where σ is the standard deviation of the results obtained on samples spiked at a level near the MDL: **MDL = t * σ .**

The MDL calculated from these data is (2.896)*(0.0064), or **0.019 ng/g.**

Sediments and Soils Methyl Hg (by CH₂Cl₂ Extraction) MDL Data
February 23, 2000

sample	net [MMHg], ng/g as Hg	comment
Blank # 1	0.0012	
Blank # 2	0.0010	
Blank # 3	0.0018	
Blank # 4	0.0025	
Mean	0.0016	
SD	0.0006	
Sand + 1.121 ng/g #1	1.0680	101.6 % rec
Sand + 1.121 ng/g #2	1.0305	98.9 % rec
Sand + 1.121 ng/g #3	1.1768	111.8 % rec
Sand + 1.121 ng/g #4	0.8735	81.3 % rec
Mean	1.0374	98.4 % rec
SD	0.1256	12.9 % RSD
Sand #1	0.0011	< MDL
Sand #2	0.0003	< MDL
Sand #3	-0.0017	< MDL
Sand #4	0.0012	< MDL
Mean	0.0002	
SD	0.0013	
Sand + 0.056 ng/g #1	0.0566	102.5 % rec
Sand + 0.056 ng/g #2	0.0556	108.1 % rec
Sand + 0.056 ng/g #3	0.0620	110.7 % rec
Sand + 0.056 ng/g #4	0.0557	104.6 % rec
Sand + 0.056 ng/g #5	0.0469	87.2 % rec
Sand + 0.056 ng/g #6	0.0566	107.8 % rec
Sand + 0.056 ng/g #7	0.0474	87.2 % rec
Sand + 0.056 ng/g #8	0.0428	78.0 % rec
Sand + 0.056 ng/g #9	0.0592	109.3 % rec
Mean	0.0536	99.5 % rec
SD	0.0064	12.06 % RSD
FGS 1944*	4.110	

Note: FGS 1944 was included in the MDL study for future reference as currently there is no certified value for the material.

Total Hg Tissue MDL Study Data

sample	[Hg], ng	Comment
blank #1	0.00	
blank #2	0.00	
blank #3	0.00	
blank #4	0.00	
mean	0.00	
SD	0.00	
chicken #1	4.00	
chicken #2	2.83	
chicken #3	3.19	
chicken #4	2.17	
mean	3.05	
SD	0.76	
chicken + 120 ng/g #1	99.1	net 96.1=80.0% rec
chicken + 120 ng/g #2	112	net 109.0=90.8% rec
chicken + 120 ng/g #3	117.7	net 114.7=95.5% rec
chicken + 120 ng/g #4	101.8	net 98.75=82.3% rec
chicken + 120 ng/g #5	120.8	net 117.8=98.1% rec
chicken + 120 ng/g #6	116.3	net 113.3=94.4% rec
chicken + 120 ng/g #7	122.3	net 119.3=99.4% rec
chicken + 120 ng/g #8	102.8	net 99.8=83.1% rec
chicken + 120 ng/g #9	115	net 112=93.3% rec
mean	111.98	mean net 109=90.8% rec
SD	8.650	
DORM-2 (ng/g)	4,304	92.75 % recovery
certified value	4640 +/- 260	

MDL Calculated

$$(2.896) * (8.65) = 25.1 \text{ ng/g}$$

California Department of Fish and Game/Moss Landing Marine Labs
Tissue MMHg (by 25% KOH/Methanol digestion) MDL Data
 March 17, 2000

Sample	[MMHg] ng/g	% recovery
blank #1	0.000282	
blank #2	-0.000793	
blank #3	0.000233	
blank #4	0.0000741	
mean	-0.0000510	
SD	0.000503	
chicken #1	0.760	
chicken #2	0.879	
chicken #3	0.924	
chicken #4	0.961	
mean	0.881	
SD	0.0874	
chicken + 2 ng #1	17.43	81.9%
chicken + 2 ng #2	15.23	76.7%
chicken + 2 ng #3	15.50	81.0%
chicken + 2 ng #4	16.40	82.2%
chicken + 2 ng #5	14.20	77.5%
chicken + 2 ng #6	16.20	77.5%
chicken + 2 ng #7	15.00	72.7%
chicken + 2 ng #8	14.50	75.1%
chicken + 2 ng #9	14.20	76.7%
mean	15.41	77.9%
SD	1.10	3.21%
chicken + 4 ng #1	27.3	75.2%
chicken + 4 ng #2	29.3	80.9%
chicken + 4 ng #3	28.4	78.3%
chicken + 4 ng #4	27.5	73.3%
mean	28.1	76.9%
SD	0.9	3.4%
2976	22.100	79.3%
certified value	27.8 ± 1.1	

MDL calculation

n = 8 degrees of freedom

t value = 2.896

MDL = (2.896)*(1.10)

MDL = 3.19 ng/g

Method Detection Limit Determination For Total Hg Fresh Water

1/19/2000

Replicate runs of Lake Conroe Dam water yielded the following:

	Peak Area
1	0.1033
2	0.1081
3	0.1063
4	0.1027
5	0.1046
6	0.1041
7	0.1044
8	0.1052
9	0.1039
10	0.1097
STDEV	0.0022
3X STDEV	0.0066

Standard Curve				
Std Conc.(ng/L)	Ave. Area	Peak Area		
		Rep #1	Rep #2	Rep#3
0	0.02506	0.02471	0.02544	0.02502
0.5	0.12393	0.1242	0.1229	0.1247
1	0.23060	0.2295	0.2294	0.2329
3	0.65397	0.6485	0.6545	0.6589

SUMMARY OUTPUT

<i>Regression Statistics</i>	
Multiple R	0.999948988
R Square	0.999897979
Adjusted R Square	0.999846968
Standard Error	0.003423598
Observations	4

<i>Coefficients</i>	
Intercept	0.0216
X Variable 1	0.2105

$$[\text{Hg}]_{\text{MDL}} = 3 * \text{STDEV}_{\text{sample}} / \text{Slope} = 0.0066 / 0.2105 = \mathbf{0.031 \text{ ng/L}}$$

Method Detection Limit Determination For Total Hg Salt Water

1/19/2000

Replicate runs of Offats Bayou water yielded the following:

	Peak Area
1	0.1031
2	0.1020
3	0.1031
4	0.1040
5	0.1015
6	0.1036
7	0.1034
8	0.1044
9	0.1025
10	0.1018
STDEV	0.0010
3X STDEV	0.0029

Standard Curve				
Std Conc.(ng/L)	Ave. Area	Peak Area		
		Rep #1	Rep #2	Rep#3
0	0.02506	0.02471	0.02544	0.02502
0.5	0.12393	0.1242	0.1229	0.1247
1	0.23060	0.2295	0.2294	0.2329
3	0.65397	0.6485	0.6545	0.6589

SUMMARY OUTPUT

<i>Regression Statistics</i>	
Multiple R	0.999948988
R Square	0.999897979
Adjusted R Square	0.999846968
Standard Error	0.003423598
Observations	4

<i>Coefficients</i>	
Intercept	0.0216
X Variable 1	0.2105

$$[\text{Hg}]_{\text{MDL}} = 3 \cdot \text{STDEV}_{\text{sample}} / \text{Slope} = 0.0029 / 0.2105 = \mathbf{0.014 \text{ ng/L}}$$

Methylmercury in Estuarine Water (by distillation)
MDL Study Data

Texas A&M University
February, 2000

Sample	net MMHg (ng Hg/L)	Recovery
blank #1	0.002	
blank #2	0.003	
blank #3	0.004	
blank #4	0.003	
mean	0.003	
St. Dev.	0.001	
Filtered HSC water + 0.184 ng/L #1	0.186	90.2%
Filtered HSC water + 0.184 ng/L #2	0.165	78.8%
Filtered HSC water + 0.184 ng/L #3	0.183	88.5%
Filtered HSC water + 0.184 ng/L #4	0.192	93.4%
mean	0.182	87.7%
St. Dev.	0.012	6.3%
Filtered HSC water #1	0.018	
Filtered HSC water #2	0.019	
Filtered HSC water #3	0.025	
Filtered HSC water #4	0.018	
Filtered HSC water #5	0.019	
Filtered HSC water #6	0.020	
Filtered HSC water #7	0.019	
Filtered HSC water #8	0.023	
Filtered HSC water #9	0.017	
Filtered HSC water #10	0.018	
Filtered HSC water #11	0.025	
mean	0.020	
St. Dev.	0.003	
MDL	0.009	= st. dev. * 3

Sample collected in Houston Ship Channel; salinity ~17 ppt
Volume of sample distilled = 150 mL

Methylmercury in Galveston beach sand (Acidic KBr extraction)
MDL study data

Texas A&M University
 February, 2000

Sample	net MMHg (ng Hg/g)	Recovery
blank #1	0.004	
blank #2	0.006	
blank #3	0.006	
blank #4	0.005	
mean	0.005	
St. Dev.	0.001	
Galveston beach sand + 0.987 ng/g #1	0.846	84.9%
Galveston beach sand + 0.987 ng/g #2	0.871	87.5%
Galveston beach sand + 0.987 ng/g #3	0.950	95.5%
Galveston beach sand + 0.987 ng/g #4	0.871	87.5%
mean	0.858	86.2%
St. Dev.	0.018	
Galveston beach sand #1	0.005	
Galveston beach sand #2	0.010	
Galveston beach sand #3	0.002	
Galveston beach sand #4	0.011	
mean	0.007	
St. Dev.	0.004	
Galveston beach sand + 0.0592 ng/g #1	0.078	118.7%
Galveston beach sand + 0.0592 ng/g #2	0.074	113.1%
Galveston beach sand + 0.0592 ng/g #3	0.074	112.3%
Galveston beach sand + 0.0592 ng/g #4	0.057	83.3%
Galveston beach sand + 0.0592 ng/g #5	0.056	82.6%
Galveston beach sand + 0.0592 ng/g #6	0.050	72.6%
Galveston beach sand + 0.0592 ng/g #7	0.076	115.4%
Galveston beach sand + 0.0592 ng/g #8	0.062	91.6%
Galveston beach sand + 0.0592 ng/g #9	0.064	95.0%
Galveston beach sand + 0.0592 ng/g #10	0.055	79.5%
mean	0.066	98.7%
St. Dev.	0.011	
MDL	0.032	= st. dev. * 3

The average weight of sand used ~ 0.5 g

UC DAVIS SOP #1:

ANALYSIS OF TOTAL MERCURY IN BIOLOGICAL TISSUES USING THE PERKIN-ELMER FLOW INJECTION MERCURY SYSTEM (FIMS)

Last revised by Darell Slotton
December 18, 1999

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6. **SCOPE AND APPLICATION-** This SOP outlines sample digestion and subsequent analysis of samples for determination of total mercury in biological tissues. The analysis is performed by the Perkin-Elmer Flow Injection Mercury System, Model 100 (better known as the FIMS 100). The analysis method is based on EPA Method 245.6, as well as methodology developed within the UC Davis Mercury Group (refs).
7. **METHOD DETECTION LIMITS-** MDL= 0.01 ug/g Hg (both wet and dry samples, 9/9/99 data)
8. **WORKING RANGE-** 0.01-4.0 ug/g (dry samples), 0.01-1.0 ug/g (wet samples)- (These ranges are based on a 1-ml sample loop. These ranges can be extended by use of smaller sample loops, allowing for analysis of high-level mercury samples).
9. **SUMMARY OF METHOD-** In order to be analyzed by the cold vapor atomic absorption technique, total sample mercury needs to be in the +2 oxidation state. For this to occur, tissue samples of various types are oxidized in reaction tubes by digestion with a nitric acid/sulfuric acid mixture, followed by further oxidation with a potassium permanganate/potassium persulfate solution. Both oxidations are carried out in a 95-degree water bath. After reduction of any remaining permanganate by hydroxylamine hydrochloride, the samples are brought to volume in the reaction tubes with deionized water. The prepared samples are allowed to flow into the FIMS, along with an acidic carrier solution. The mercury in the sample digests is reduced to elemental mercury by reaction with stannous chloride in a reaction manifold. The gaseous elemental mercury is stripped from the sample solution by argon gas in a gas/liquid separator. The mercury is swept by the gas into a long glass cell that is positioned along the light path of a high-intensity mercury lamp. The sample mercury in the cell absorbs some of the light emitted by the mercury lamp. This