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A Laboratory Intercomparison of Mercury Analyses

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by
Pete Rawlik

Resource Assessment Division
Water Resources Evaluation Department
South Florida Water Management District
3301 Gun Club Road
West Palm Beach, FL 33406

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Executive Summary

The South Florida Mercury Science Program is a consortium of Federal, State and Local agencies and private entities involved with mercury research in South Florida. Many of these organizations are operating or contracting with laboratories that are analyzing environmental samples for mercury content. A laboratory intercomparison was conducted to ensure that data produced by distinct laboratories are comparable and that any data discrepancies are identified and resolved.

In January 1998, homogenized samples of water and preyfish were given to laboratories for analysis. Results were mixed with a total mercury in water ranging from 19% to 193% of the grand mean of 1.5 ng/L. Results for methylmercury in water ranged from 69% to 132% of the grand mean of 0.15 ng/L. Results for fish from WCA-2B were also variable, with laboratory total mercury averages for ranging from 57% to 129% of the grand mean of 5.19 ng/g. Results for WCA-3A were more acceptable with laboratory total mercury averages ranging from 84% to 117% of the grand mean of 165.01 ng/g.

In addition to the high variability between laboratories, the analysis also revealed areas of concern within the various laboratories. Several laboratories failed to notice that they had violated their own internal quality assurance protocols. Other laboratories provided results below their reported detection limit. Additionally, when duplicate samples were submitted without the laboratory's knowledge, the analytical results were not always comparable. Finally, several laboratories reported values as below the method detection limit. All of these questionable results cast some doubt on whether or not the laboratories are capable of providing valid and comparable data on mercury levels in South Florida.

Through the course of analyzing the data, it became apparent that the existence of a laboratory quality assurance plan provided no guarantee for the quality of data produced. Furthermore, it is highly likely, despite claims to the contrary, that the analytical methods used by the various laboratories are not equal or comparable. As a result, it is necessary for the standard method recently promulgated by the U.S. Environmental Protection Agency to be applied to laboratories uniformly. Finally, it is suggested that results from laboratories with method detection limits higher than the concentrations in environmental samples be used with caution. Continuing use of quality assurance programs like this one, will provide data on the analytical uncertainty of laboratories assessing the status of mercury in the Florida Everglades.

A Laboratory Intercomparison of Mercury Analyses

Introduction

The South Florida Mercury Science Program (SFMSP) is a consortium of Federal, State, and Local agencies and private entities involved in mercury research in South Florida. This research is currently being conducted by or for the U. S. Environmental Protection Agency (USEPA), the Florida Department of Environmental Protection (FDEP), the Florida Game and Freshwater Fish Commission (FGFWFC), the South Florida Water Management District (SFWMD), the Wisconsin Department of Natural Resources (WiDNR) and the United States Geological Survey (USGS). Only the District and the USGS have a formal Memorandum of Understanding governing study coordination, mutual in-kind support and data sharing for Everglades mercury research, however.

As the regional steward of the Everglades, the District is a primary consumer of the monitoring and research data being generated by the SFMSP agencies to guide restoration decision-making, because it has neither the fiscal resources nor administrative authority to conduct all of the required studies unilaterally. Consequently, there is an inherent need to combine data sets from different agencies and laboratories to support its core mission. However, this presents a potential problem, because, while each laboratory possesses an approved Comprehensive Quality Assurance Project Plan governing field sample collection and laboratory analysis for ultra-trace mercury species, not all agencies are using the same methods and procedures for field sample collection and laboratory analysis. The consequences of these differences needed to be characterized and quantified if informed use was to be made of these combined data sets in guiding restoration decision-making or in evaluating permit compliance.

The ANS, USGS, WiDNR, SFWMD and FGS have formed a loose association of reseachers known as the Aquatic Cycling of Mercury in the Everglades (ACME) Group. ACME had already instituted an inter-laboratory study of analytical data comparability among the ACME laboratories beginning in 1995. At the request of the District, in 1997 members of ACME agreed to participate in a study of analytical data comparability with the District. To ensure that data produced by individual laboratories are comparable, a split sample intercomparison program was initiated. The goal of this effort was to ensure that analytical data generated by the various laboratories of the ACME were both valid and comparable with Frontier Geosciences of Seattle, WA, an ultra-trace mercury analytical laboratory being used by the District for mercury research and monitoring projects mandated by Florida/Federal operation/discharge permits. Additionally, the intercomparison program was intended to provide a mechanism for identifying and resolving discrepancies in both past and future results. At the request of USEPA Region 4 and FDEP, the intercomparison program was expanded to include Florida International University of Miami, FL, a contract laboratory to USEPA Region 4, FDEP's Mercury Clean Laboratory in Tallahassee, and USEPA Region 4's Environmental Services Division laboratory in Athens, Georgia.

Methods and Procedures

Water

In January 1998, six liters of filtered surface water were collected using clean-hands technique from Water Conservation Area 2B (WCA-2B) with a peristaltic pump and an in-line 0.45 micron Meissner filter. The water was stored in an acid-washed, 10-L polycarbonate carboy suitable for ultra-trace mercury sampling. The carboy was vigorously swirled to homogenize the sample. Water was then decanted into twelve acid washed 500-ml Teflon bottles. During the process of filling the bottles, the carboy was continuously swirled to maintain sample homogeneity. Prior to filling the carboy, a bottle was filled with deionized (DI) water using the peristaltic pump and sampling train with Nitex pre-screen to act as an equipment blank. The true split samples were randomly distributed among the seven laboratories, with two laboratories receiving duplicate bottles. The remaining three bottles were archived at 4°C. The SFWMD standard operating procedure for surface water collection is attached.

Fish

In January 1998, several thousand small fish, primarily mosquitofish (*Gambusia* sp.) were collected in WCA-2B and Water Conservation Area 3A (WCA-3A). Samples were placed in plastic bags and placed on ice in coolers for transport. For each site, preyfish were homogenized using a food processor and divided into 10 gram aliquots, which were placed into pre-cleaned, polycarbonate sample containers (100 ml). All seven laboratories received samples of the fish from WCA-2B. However, due to a limited amount of sample, only six labs received samples from WCA-3A.

Shipping

Samples were stored at 4°C prior to shipping. Both water and fish samples were shipped overnight on dry ice. Despite this, many of the samples arrived at the laboratories either completely or partially thawed. Additionally, because the homogenization process destroys the structure of the fish, many of these samples showed evidence of separation of water from the solid portion of the sample. The participating laboratories were advised to rehomogenize the samples prior to subsampling and analysis.

Laboratory Analysis

Samples are assumed to have been analyzed in accordance with the Comprehensive Quality Assurance Project Plans (CompQAPP) maintained by each laboratory. Copies of these CompQAPPs and the bench sheets were requested from each participating laboratory to add in interpreting the analytical results. Quality assurance targets and method detection limits (MDL) were determined from these CompQAPPs.

Data Analysis

Sample results were supplied to the staffof the SFWMD and analyzed for both precision and accuracy. For laboratories that provided two results per bottle, in-bottle precision was calculated using relative standard deviation (RSD). RSD was used rather than relative percent deviation (RPD) or coefficient of variation (CV) because RSD is a routine component of the quality

assurance plan and is therefore directly applicable to generated data. RSD was calculated using the in-bottle mean of the individual laboratory data.

Additionally, data from laboratories that supplied more than two results per bottle was analyzed from a pair-wise perspective. This process entails taking all possible pair combinations and calculating the RSD for each pair. This is a more rigorous test than comparing the individual results to the overall mean. However, routine quality assurance protocols require the comparison of pairs for the determination of RSD, rather than the mean of triplicate or quadruplicate samples.

In cases where laboratories received multiple bottles of the same sample, RSD was also calculated for between bottle results. For purposes of quality assurance this split sample was treated as equivalent to a replicate.

Accuracy was determined by taking the average of all laboratory means and generating grand means with standard deviations. However, because of the small sample size, a single point can assert leverage and skew the grand mean substantially. To compensate for this a censored grand mean may be used to eliminate outliers when necessary.

Results

Mercury in Water

Results for total mercury (THg) in filtered water samples are presented in Table 1. Laboratory averages ranged from a low of 0.29 ng/L to a high of 2.90 ng/L. The majority of laboratories reported mean results from replicate subsamples, with Laboratories 02 and 05 reporting only single results. Laboratory 05 reported a value as less than their MDL of 1.00 ng/L. Laboratory 01 reported results for two distinct bottles. Laboratory 04 reported individual results and means for two distinct bottles.

Methylmercury in Water

Results for methylmercury (MeHg) in water samples are presented in Table 2. Laboratory averages ranged from a low of 0.104 ng/L to a high of 0.198 ng/L. The majority of laboratories reported mean results from replicate subsamples with only Laboratory 02 reporting a single result. Laboratory 05 did not report a result. Laboratory 01 reported results for two distinct bottles. Laboratory 04 reported individual results and means for two distinct bottles.

Mercury and Methylmercury in Fish

Table 3 presents analysis results for fish tissue composites from WCA-2B. Laboratory 07 reported the lowest THg value, while Laboratory 03 reported the highest MeHg value of 6.94 ng/g. Laboratory 05 and 07 did not report results, while Laboratory 04 withheld its MeHg data as a result of QA issues.

Table 4 presents analysis results for fish tissue composites from WCA-3A. Laboratory 04 reported the lowest value at 138.29 ng/g THg, while Laboratory 03 reported the highest value of 204.18 ng/g for MeHg. Laboratory 04 withheld its MeHg data do to QA issues.

Data Analysis for Precision and Accuracy

Precision for Mercury in Water

Table 1 includes the analytical precision data for the individual laboratory THg results. Since Laboratory 02 and Laboratory 05 failed to submit multiple results, it was not possible to calculate a measure of precision for these labs. For labs 03, 06 and 07 multiple analyses were carried out on the same bottle, which allowed for calculation of pair-wise RSDs, all of which were below 5%. Laboratory 01 ran single analyses on each of two split samples from distinct bottles. This allowed for the calculation of a between bottle RSD of 1%. Laboratory 04 ran replicate analyses on each of two split samples from distinct bottles. This allowed for the calculation of within-bottle, between bottle and pair-wise RSDs. The within-bottle RSDs were 0% for the first bottle and 19% for the second bottle. The RSD of 19% violates this laboratory's data quality objective of 10% RSD. The between bottle RSD was 11%, which again violates this laboratory's data quality objective of 10% RSD. Finally, the pair-wise analysis shows that the 0.18 ng/L had a RSD of 26% compared to the overall mean of all four results.

Precision for Methylmercury in Water

Table 1 includes the analytical precision data for the individual laboratory MeHg results. Since Laboratory 02 failed to submit multiple results, and Laboratory 05 did not submit any results, it was not possible to calculate a measure of precision for these labs. For labs 03, 06 and 07 multiple analyses were carried out on the same bottle, which allowed for calculation of pair-wise RSDs, all of which were below 15%. Laboratory 01 ran single analyses on each of two split samples in distinct bottles. This allowed for the calculation of a between bottle RSD of 0%. Laboratory 04 ran replicate analyses on each of two split samples from distinct bottles. This allows for the calculation of a within-bottle, between-bottle and pair-wise RSDs. The within-bottle RSD was 6% for the first bottle and 0% for the second bottle. The between-bottle RSD was 63%, which violates the data quality objective of 10% RSD, as specified in this laboratory's CompQAPP. Finally, the pair-wise analysis shows that all the values had RSDs in excess of 50% when compared to the overall mean of all four results.

Accuracy for Mercury in Water

Figure 1 shows the minimum detection limits, sample results, lab means and standard deviations for THg in split water samples. Also shown is the grand mean and standard deviation using all labs except Laboratory 05 which reported results as simply below the detection limit. Also included is a censored grand mean that does not include Laboratory 04, the lab with the lowest average results, and Laboratory 07, the lab with the highest average results. The censored grand mean was generated because it appeared that these two labs were asserting undue leverage on the grand mean. The majority of results fell between 1.0 ng/L and 2.0 ng/L, with only Laboratory 04 and Laboratory 07 reporting differently. Results from Laboratory 07 were approximately twice that of the grand mean, while Laboratory 04 results were only one-fifth that of the grand mean. The influence of these two diverging labs on the grand mean can be seen in the difference between the grand mean and the censored grand mean. Removal of Laboratory 04 and Laboratory 07 from the calculation of the grand mean had little effect on the mean, but a dramatic effect on the variability, reducing the standard deviations by more than half.

Accuracy for Methylmercury in Water

Figure 2 shows the minimum detection limits, sample results, lab means and standard deviations for MeHg in split water samples. Also shown are the grand mean and standard deviation using all labs except Laboratory 05, which did not report results. The most obvious point of concern is Laboratory 04, which reported a mean value of 0.178 ng/L, but individual results that ranged from 0.335 ng/L to below the method detection limit of 0.020 ng/L. This provided a laboratory standard deviation that exceeded the standard deviation of the grand mean.

Precision for Mercury and Methylmercury in Fish Tissue Composites

Tables 3 and 4 include the individual laboratory precision data for THg and MeHg in fish tissue composites from both WCA-2B and WCA-3A. All RSDs were less than 25%. Furthermore, nearly all laboratories were able to meet internal data quality objectives. The exception to this was Laboratory 04, which exceeded their internal data quality objective of 15% for THg by generating results from WCA-2B with an RSD of 24%. MeHg data from Laboratory 04 were withheld by that laboratory's Quality Assurance Officer. Additionally, Laboratory 02 had an RSD of 20% for WCA-3A. Pair-wise RSD analysis of the Laboratory 2 data revealed that the data point of 130 ng/g generates a maximum RSD of 27% when compared to other results from the same sample.

Accuracy for Mercury and Methylmercury in Fish Tissue Composites[1]

Figures 3 and 4 present the mean THg and MeHg results and standard deviations from individual laboratories and the calculated grand means and standard deviations for the results of analysis of fish tissue. For THg, the mean for Laboratory 04 was outside the standard deviation of the grand mean in both the WCA 2B and WCA-3A. Similarly, the mean THg for Laboratory 07 was outside the standard deviation of the WCA-2B grand mean, and the mean THg for Laboratory 03 was outside the standard deviation of the WCA-3A grand mean. Additionally, the Laboratory 03 mean for MeHg was outside the standard deviation of the grand mean for WCA-2B.

Discussion

Laboratory 01

This laboratory presented data that met their own internal quality assurance criteria for between-sample precision. The means of all the data fell within the standard deviation of the grand means.

Laboratory 02

This laboratory did not submit sufficient results in order to evaluate precision in surface water analysis. The means of all the data fell within the standard deviation of the grand means. Precision analysis for fish tissue composites showed that samples from WCA-3A had a mean RSD of 20%. A detailed analysis indicates that of the four replicate analyses of this sample, the

[1] While the THg grand mean for the WCA-2B samples was 5.19 ng/g, wet, other reports have shown differing values. Cleckner et al. (1998) reported results for WCA-2B ranging from 4 -6 ng/g, wet for December, 1995 and 5-15 ng/g, wet for December, 1996. In contrast Stober et al. (1996) reported results for WCA-2B ranging from 50 – 100 ng/g in April and September, 1995. Similarly, while the grand mean for the WCA-3A samples was 165.01 ng/g, wet, other reports have shown differing values. . Cleckner et al. (1998) reported results for WCA-3A ranging as 17 ng/g, wet for December, 1995 and 15 45 ng/g, wet for December, 1996. In contrast Stober et al. (1996) reported results for WCA-2B ranging from >450 ng/g, wet in April and September, 1995.

result of 130 ng/g had a RSD in excess of the 25% when compared to the other result of 190 ng/g. This violated the replicate analysis precision requirement for this laboratory.

Laboratory 03

This laboratory presented data that met their own internal quality assurance criteria for between sample precision. Mean results for MeHg in surface waters, MeHg in WCA-2B fish tissue composites, and THg in WCA-3A fish tissue composites were outside the standard deviation of the grand means.

Laboratory 04

A result of 0.24 ng/L for THg in water was reported. This value was actually the mean of two results, one of which was 0.18 ng/L, which is below the method detection limit of 0.30 ng/L for this laboratory. It may have been inappropriate to use a result below the MDL to generate a mean result, which was also below the MDL.

Additionally, by reporting values below the method detection limit, Laboratory 04 seriously compromised precision. For the THg analysis, Laboratory 04 analyzed two split samples. For the first split sample, the replicate analysis results were identical resulting in a RSD of 0%. For the second split sample, the replicate analyses were 0.31 ng/L and 0.18 ng/L, which are near or below the MDL. These divergent results generated a RSD of 19%, which exceeds the RSD precision of 10% required by Laboratory 04's quality assurance plan. It is important to note here that Laboratory 04 did report the value of this result as 0.24 ng/L, even though this is below their method detection limit and the replicate analysis failed precision requirements. As a result of reporting this value, the precision between bottles was 11% RSD, which again exceeded the precision requirement of 10% RSD. Laboratory 04 was not aware that the two bottles were split samples, and therefore, could not have been expected to recognize the need for between-bottle precision analysis and reject the results. Regardless, both split samples and replicate analyses are standard quality assurance criteria, and Laboratory 04 did not perform in a manner consistent with quality assurance criteria for THg in water.

Similarly, for MeHg in water, the first mean bottle result was 0.335 ng/L, while the second mean bottle result was identified as below the MDL of 0.020 ng/L. This vast difference results in a between-bottle RSD of 63%. Again, Laboratory 04 was not aware that the two bottles were split samples and therefore could not have been expected to recognize the need for between-bottle precision analysis and reject the results. Regardless, split samples are standard quality assurance criteria and Laboratory 04 did not perform in a manner consistent with quality assurance criteria for MeHg in water.

Furthermore, the mean THg in water result was outside the standard deviation associated with the grand mean. Additionally, while the mean MeHg in water result was within the standard deviation associated with the grand mean, neither the first bottle result of 0.335 ng/L, nor the second bottle result of <0.020 are within the standard deviation of the grand mean. Furthermore, while this analysis has used the MDL of 0.020 to estimate the split sample mean, other analyses could use half the MDL, 0, or simply reject the value altogether. Regardless, Laboratory 04 did not produce results for either THg or MeHg in water that could be accepted as accurate.

Laboratory 04 had similar problems analyzing fish tissue composites. For the WCA-2B sample, five replicate analyses were reported, and of these four were values below the MDL. Additionally, the mean THg result was below the MDL. Precision analysis using these raw values generated a RSD of 24%. Reanalysis of this data set by replacing the stated results with the MDL of 3.20 ng/g produced a RSD of 16%. Both of these RSDs violated the lab precision objective of 15%. The mean results for both the WCA-2B and WCA-3A fish tissue composites were both outside the standard deviation of the associated grand means. Finally, the laboratory quality assurance officer withheld results for MeHg in fish tissue.

Laboratory 05

This laboratory did not submit sufficient results in order to evaluate THg precision in surface water. THg in water results were submitted as below the method detection limit and therefore cannot be properly evaluated for accuracy. No MeHg in water results were submitted. Analytical results for THg in fish tissue met precision requirements. No MeHg in fish tissue results were submitted. THg in fish tissue composites fell within the standard deviation of the grand means.

Laboratory 06

This laboratory presented data that met their own internal quality assurance criteria for between sample precision. The means of all the data fell within the standard deviation of the grand means.

Laboratory 07

This laboratory presented data that met their own internal quality assurance criteria for between sample precision. The mean THg in water fell outside the standard deviation of the grand mean. The mean THg in fish tissue composites from WCA-2B fell outside the standard deviation of the grand mean.

Conclusions and Recommendations

1. Laboratories that report values for environmental samples as below the detection limit (BDL) create particular difficulties for the analysis of data. Even assuming that these laboratories are generating accurate data, a BDL cannot be used to adequately quantify environmental conditions. Data from laboratories that have practical quantitation limits (PQLs) greater than the 95th % lower bound concentration of the analyte of interest under routinely encountered environmental conditions should be flagged to guide appropriate use.

2. Despite the existence of internal quality assurance plans, some laboratories reported results that did not meet precision criteria for replicate or split-sample analysis. In part, this is because the precision criteria may be too strict. Given the ultra-trace levels at which these analyses are operating, the analytical method may be inherently variable and subject to a variety of uncontrollable factors. Based on SFWMD experience, it is suggested that a RSD of 40% be used to evaluate intra-laboratory precision. Data from laboratories that violate this criterion should be flagged to guide appropriate use.

3. The ability to reproduce the grand mean varied between laboratories, most likely because of differences in analytical methods among the laboratories. However, all participating laboratories are using approved methods that have been validated by their respective QA programs. At this time, the USEPA is in the process of finalizing a standard method for ultra-trace total mercury analysis and has already finalized a standard method for ultra-trace methylmercury analysis. It is suggested that these methods, with appropriate modifications for Everglades applications, be adopted and used by all laboratories in the SFMSP. Until such time as the alternate methods are validated to be strictly equivalent to the USEPA methods, data from laboratories that continue to use alternative methods should be flagged to guide appropriate use.

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Citations

Cleckner, L. B., P. J. Garrison, J. P. Hurley, M. L. Olson and D. P. Krabbenhoft (1998) Trophic transfer of methyl mercury in the northern Florida Everglades. Biogeochemistry 40 (2-3): 347-361.

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Table 1. Analytical results and quality control data for THg (ng/L) in filtered surface water split samples.

Lab ID	MDL	rep 1	rep 2	rep 3	rep 4	Lab mean	RSD DQO	RSD rep 1	RSD rep 2	RSD rep 3	RSD rep 4
Lab01	0.15	**1.69**		**1.72**		1.71	25%	1%		1%	
Lab02	0.50	1.20				1.20	20%				
Lab03	0.05	1.37	1.29	1.34	1.29	1.32	20%	3%	2%	1%	2%
		0.33		***0.24***		0.29	10%	11%		11%	
Lab04	0.30	0.33	0.33	*0.18*	0.31	0.29	10%	10%	10%	26%	6%
within bottle RSD =		0%		*19%*							
Lab05	1.00	<u>1.00</u>				<u>1.00</u>					
Lab06	0.15	1.10	1.20	1.20	1.20	1.18	20%	5%	2%	2%	2%
Lab07	0.10	3.04	2.76			2.90	10%	3%	3%		

bold values indicate distinct bottles

underlined values indicate a result reported as below the detection limit

italic values indicate a result that is below the supplied detection limit

Table 2. Analytical results and quality control data for MeHg (ng/L) in filtered surface water split samples.

Lab ID	MDL	rep 1	rep 2	rep 3	rep 4	Lab mean	RSD DQO	RSD rep 1	RSD rep 2	RSD rep 3	RSD rep 4
Lab01	0.009	**0.149**		**0.149**		0.149	25%	0%		0%	
Lab02		0.198				0.198	20%				
Lab03	0.077	0.120	0.088			0.104	20%	11%	11%		
		0.335		**<u>0.020</u>**		0.178	10%	63%		63%	
Lab04	0.020	0.358	0.309	<u>0.020</u>	<u>0.020</u>	0.177	10%	73%	53%	63%	63%
within bottle RSD =		6%		0%							
Lab05	No data provided										
Lab06	0.075	0.150	0.150			0.150	10%	0%	0%		
Lab07	0.030	0.175	0.168			0.172	20%	1%	1%		

bold values indicate distinct bottles

underlined values indicate a result reported as below the detection limit

italic values indicate a result that is below the supplied detection limit

Figure 1. Results and standard deviations for total mercury in filtered, split water samples from WCA-2B.

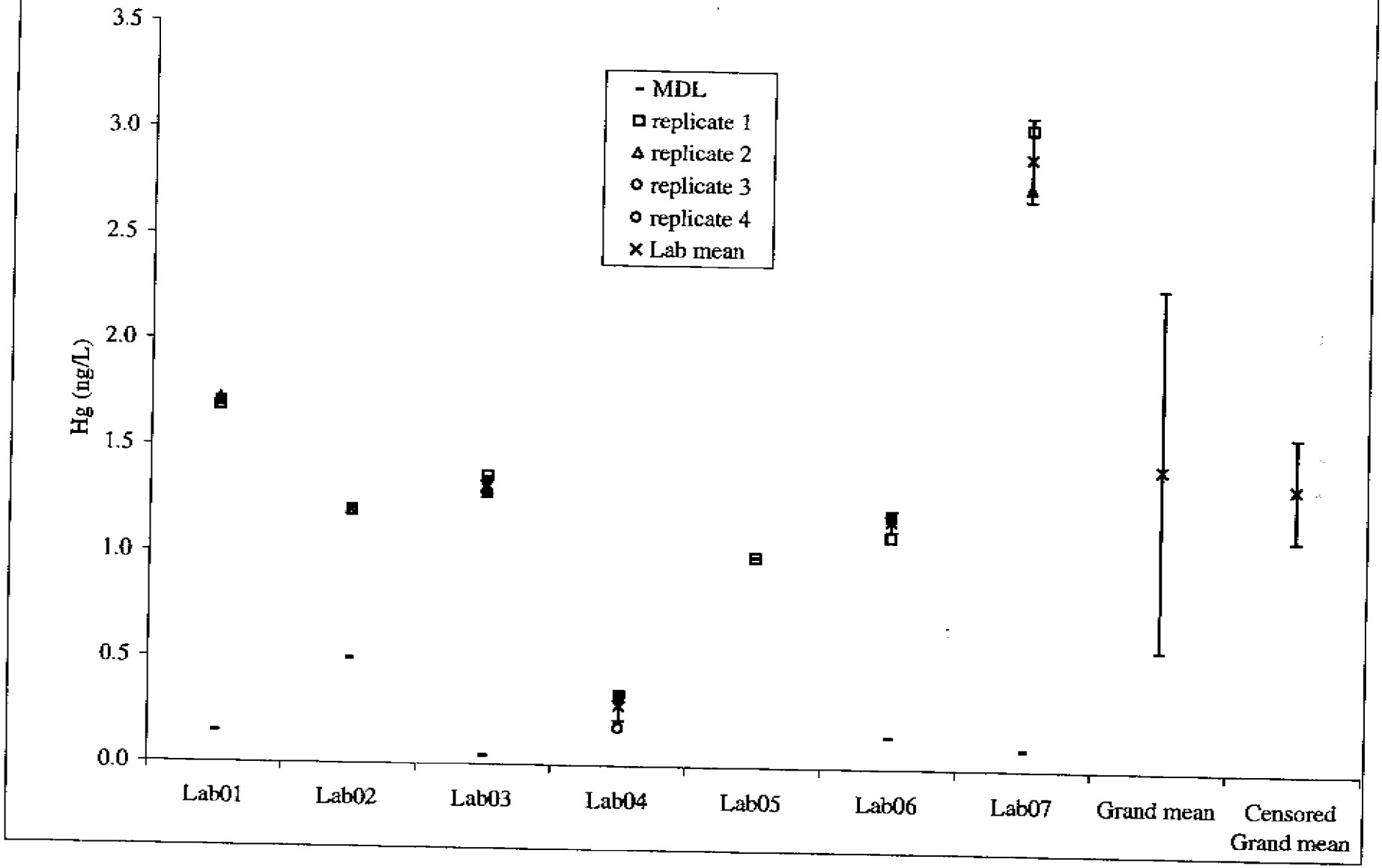

Figure 2. Results and standard deviations for methylmercury in filtered, split water samples fromWCA-2B.

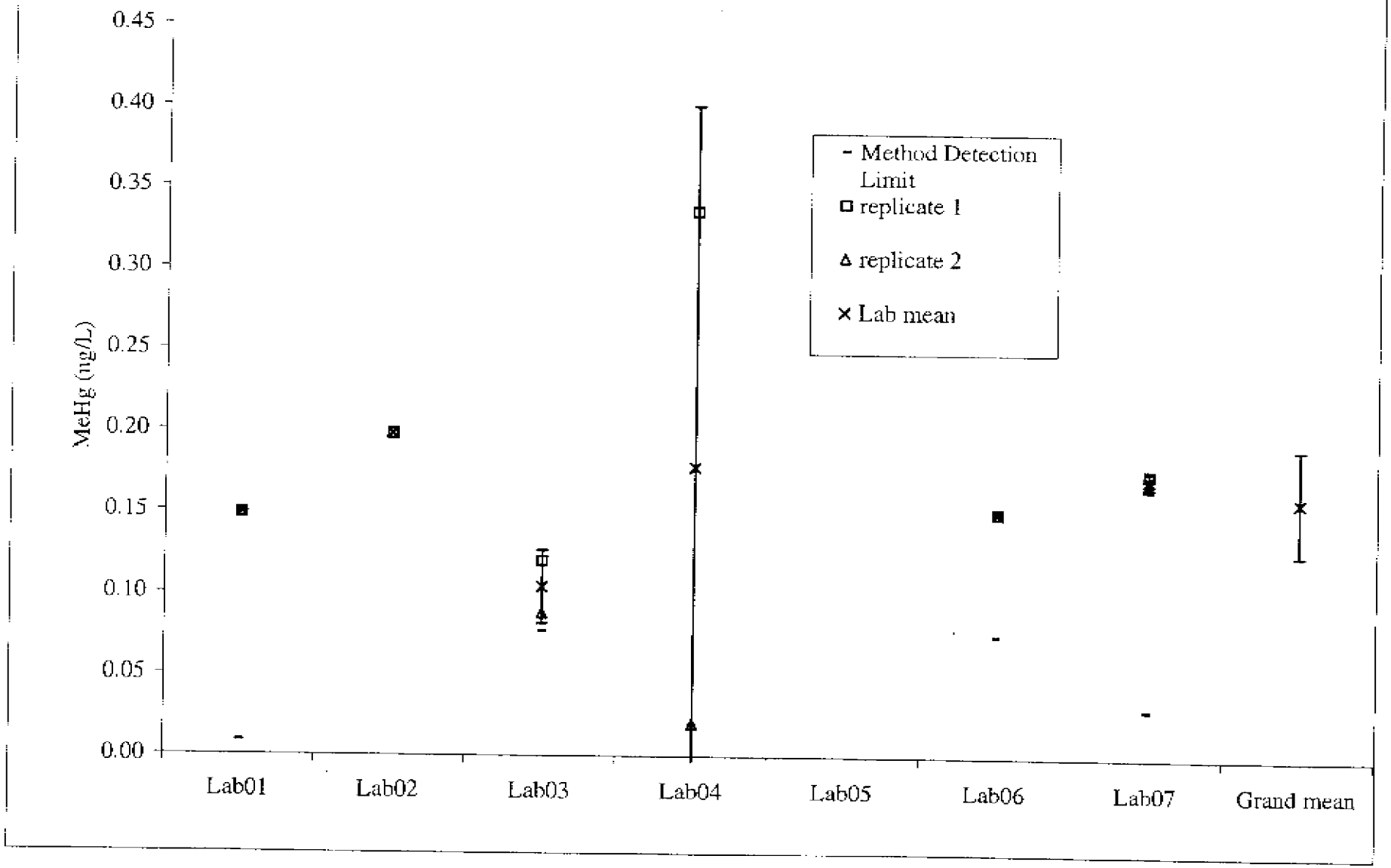

Figure 3. Mean mercury and standard deviations in composites of fish tissue from WCA-2B

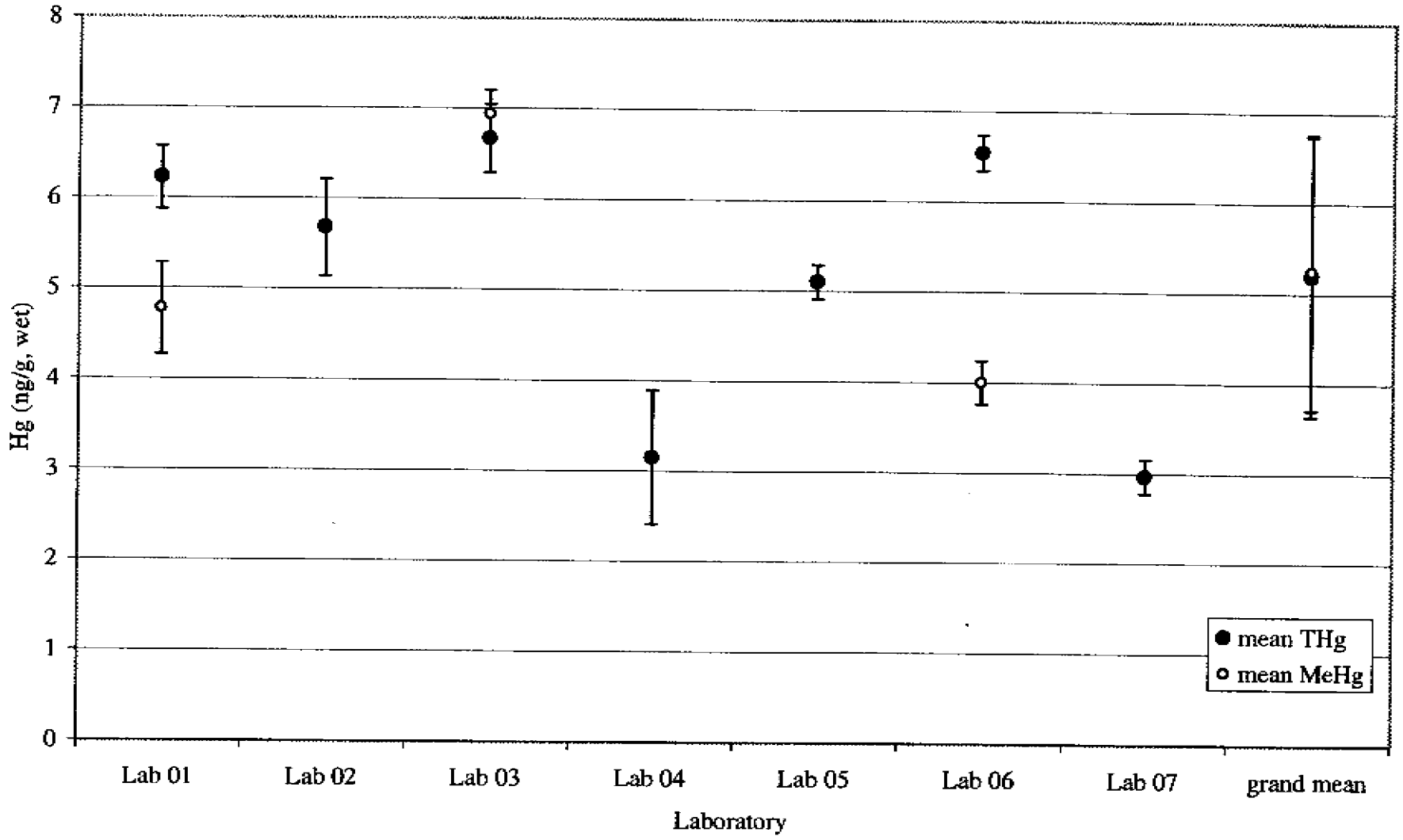

Figure 4. Mean Hg and standard deviations in composites of fish tissue from WCA-3A

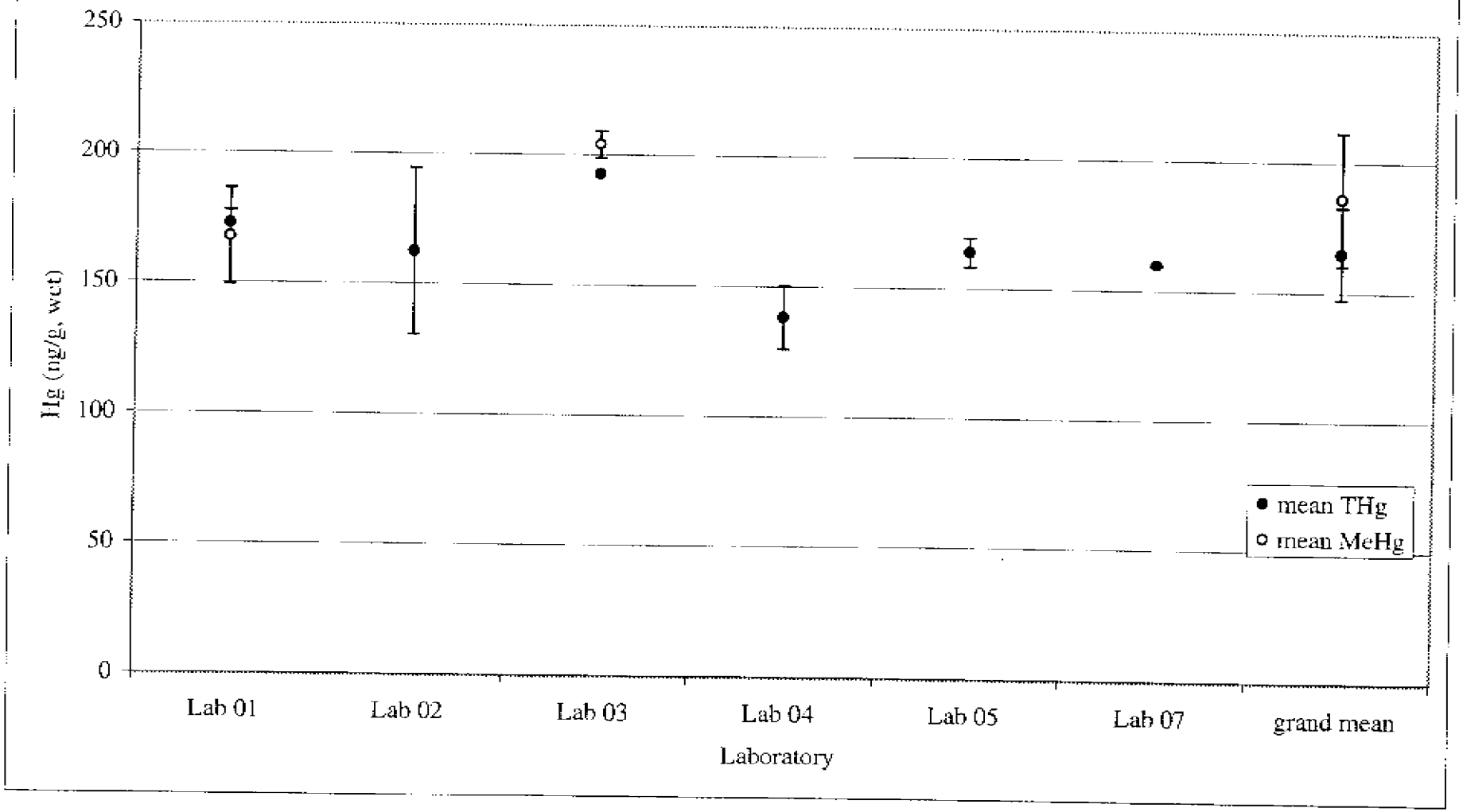

Table 3. THg and MeHg results for replicate composite fish tissue analysis from WCA-2B.

THg results (ng/g, wet)

lab	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	mean THg	stdev	RSD
Lab 01	5.80	6.08	6.34	6.74	6.17	6.23	0.35	6%
Lab 02	6.30	5.60	5.00	5.80		5.68	0.54	9%
Lab 03	7.10	6.40	6.50			6.67	0.38	6%
Lab 04	4.43	*3.09*	*2.55*	*2.83*	*2.85*	*3.15*	0.74	24%
Lab 05	5.1	5.4	5	5.1	4.9	5.10	0.19	4%
Lab 06	6.80	6.40	6.70	6.40	6.40	6.54	0.19	3%
Lab 07	2.83	3.1				2.97	0.19	6%
grand mean						5.19	1.55	

MeHg results (ng/g, wet)

lab	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	mean MeHg	stdev	RSD
Lab 01	5.13	4.42				4.78	0.50	11%
Lab 03	6.50	7.00	7.00	7.00	7.20	6.94	0.26	4%
Lab 04*	Data witheld by QA officer							
Lab 05								
Lab 06	4.30	4.20	3.90	3.70	3.90	4.00	0.24	6%
Lab 07								
grand mean						5.24	1.52	

italic values indicate a result that is below the supplied detection limit

Table 4. THg and MeHg results for replicate composite fish analysis from WCA-3A.

THg results (ng/g, wet)

lab	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	mean THg	stdev	RSD
Lab 01	165.74	176.43	175.34	173.63		172.79	4.84	3%
Lab 02	190.00	190.00	130.00	140.00		162.50	32.02	20%
Lab 03	192.60					192.60		
Lab 04	156.75	129.86	144.78	130.64	129.42	138.29	12.16	9%
Lab 05	160.00	170.00	160.00	170.00	160.00	164.00	5.48	3%
Lab 07	160.10	159.70				159.90	0.28	0%
grand mean						165.01	17.71	

MeHg results (ng/g, wet)

lab	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	mean MeHg	stdev	
Lab 01	179.36	177.64	146.56			167.85	18.46	11%
Lab 03	200.40	202.30	202.00	212.00		204.18	5.28	3%
Lab 04*	Data witheld by QA officer							
grand mean						186.01	25.68	