



Project Summary

Interlaboratory Evaluation of SW-846 Methods 7470 and 7471 for the Determination of Mercury in Environmental Samples

Werner F. Beckert, J.E. Gebhart, J. D. Messman, and G. F. Wallace

The EPA protocols for SW-846 Methods 7470 and 7471 are cold-vapor atomic absorption spectrometric (CV-AAS) methods for the determination of mercury in aqueous and solid environmental samples, respectively. In continuation of a previous single-laboratory study in which a more sensitive mercury CV-AAS method for environmental analyses was evaluated, the revised CV-AAS method has been subjected to an interlaboratory study. The revised CV-AAS system, operated in an open configuration, incorporates a dedicated gas sparging bottle for reduction-aeration and an on-line amalgamation/thermal desorption step. With these modifications, the CV-AAS method provides increased sensitivity and also alleviates non-specific background absorption interferences so that instrumental background correction is not required.

Silver-wool amalgamation cells, mercury stock standard and spiking solutions, a deionized water sample with spiking instructions, a coal fly ash reference material, and instructions for analysis by the amalgamation CV-AAS method were sent to 18 participating laboratories having prior experience with the current EPA protocols for Methods 7470 and 7471. Ten of the 18 laboratories were then invited to par-

ticipate in a more rigorous collaborative study. To evaluate the revised Method 7470, three aqueous sample types were analyzed: ground water, waste water, and dilute nitric acid. To evaluate the revised Method 7471, three solid sample types were analyzed: marine sediment, incinerator fly ash, and municipal sewage sludge. Some of the samples were designated for spiking with inorganic or organic mercury and also with copper, a potential interferent.

The analytical results reported by the collaborating laboratories were statistically examined in an attempt to characterize the overall accuracy, precision, and ruggedness of the amalgamation CV-AAS method. In general, the interlaboratory results indicated that the amalgamation CV-AAS method currently is not sufficiently rugged for routine use but, when properly implemented by proficient laboratory personnel, may serve as an alternative approach to the recirculating CV-AAS method described in the current EPA protocols. To fully realize the analytical benefits of the amalgamation CV-AAS method and to obtain accurate and precise data, a complete appreciation of the integral factors for successful trace analyses is essential, and a high level of sophisticated operation and operator skill is required.

This Project Summary was developed by EPA's Environmental Monitoring Systems Laboratory, Las Vegas, NV, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

Included in publication SW-846, entitled "Test Methods for Evaluating Solid Waste," by the Office of Solid Waste and Emergency Response of the U.S. Environmental Protection Agency are two analytical protocols, Methods 7470 and 7471, for the determination of mercury in aqueous and solid waste samples by cold-vapor atomic absorption spectrometry (CV-AAS). In a previous single-laboratory study, these EPA protocols were evaluated and revised to improve analytical performance. The protocols were evaluated using aqueous and solid environmental samples of homogeneous and known compositions in order to assess accuracies and precisions of the methods without introducing uncertainties due to sample heterogeneities.

The results of the single-laboratory study indicated, in general, that the digestion procedures were satisfactory for the samples analyzed; only minor revisions that would improve, clarify, or increase the flexibility of the digestion procedures were recommended. The recirculating CV-AAS method described in the current EPA protocols was found to be adequate only for mercury determinations in samples of relatively high mercury concentrations. Significant modifications of the cold-vapor apparatus were recommended and evaluated to overcome its inadequacy for mercury determinations in samples of low mercury levels and to minimize interferences caused by non-specific absorption of the primary mercury radiation by organic vapors. Instrument detectability was improved 10-fold by the use of a gas sparging bottle as a dedicated reduction-aeration vessel and silver-wool amalgamation in the CV-AAS system operated in an open configuration. The on-line amalgamation/thermal desorption process of the modified CV-AAS system also effectively eliminated interferences from water and organic matrix vapors so that

an instrument without dynamic background correction capabilities could be used. Good accuracy and precision were obtained with the amalgamation CV-AAS system for the analyses of four reference sediment materials.

The objective of this project was to conduct an interlaboratory evaluation of the revised EPA protocols for the determination of mercury in environmental samples. The interlaboratory study was conducted in three phases: Phase I - fabrication and testing of the silver-wool amalgamation cells and characterization of selected interlaboratory study samples by the lead laboratory; Phase II - preliminary evaluation of the technical capabilities of the participating laboratories; and Phase III - evaluation by the qualifying laboratories of the amalgamation CV-AAS method for the determination of mercury in a marine sediment reference material and in representative aqueous and solid waste samples. The test samples contained concentrations of endogenous mercury or were spiked with concentrations of inorganic or organic mercury that were in the optimal range of the amalgamation CV-AAS method.

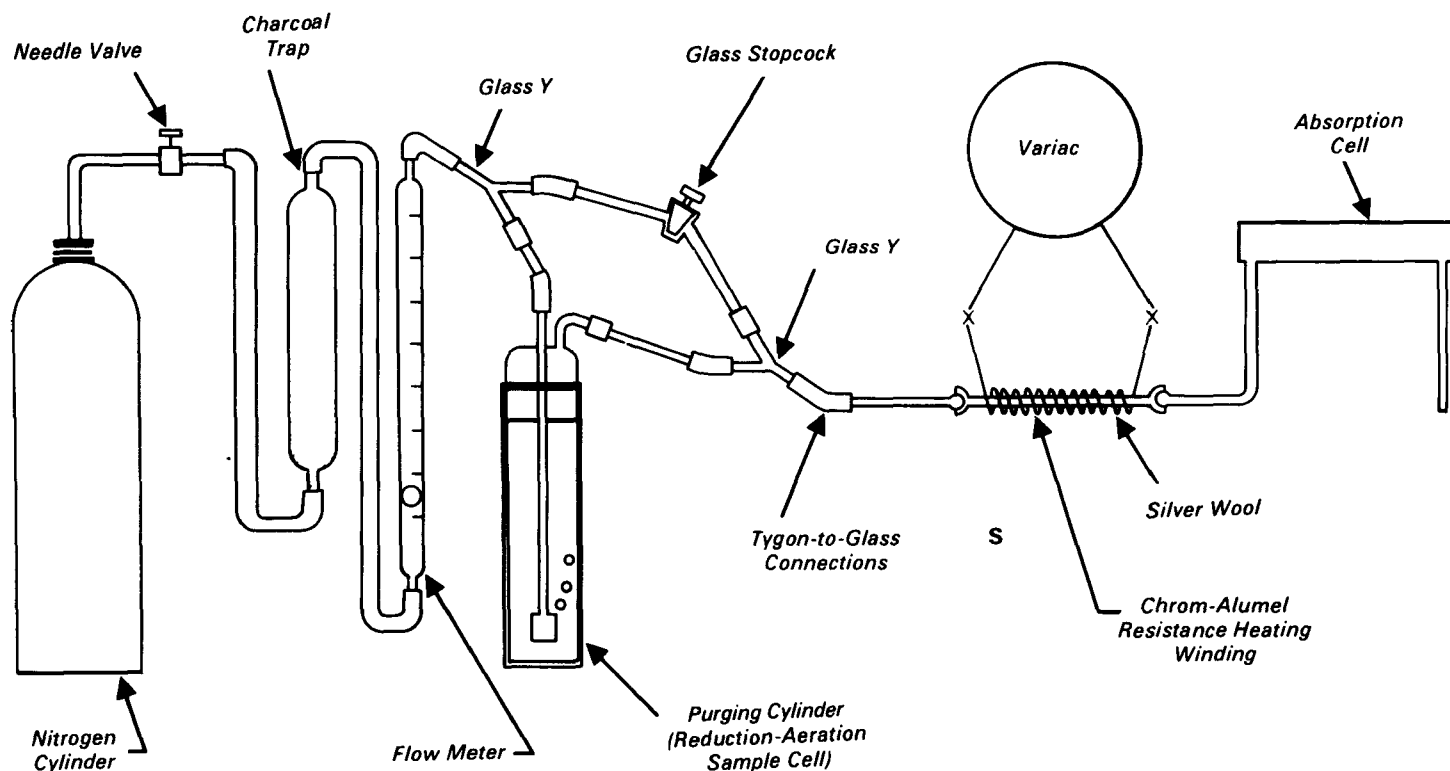


Figure 1. Schematic diagram of the amalgamation CV-AAS system.

statistical analysis of the mercury concentration data submitted to Battelle was conducted in an attempt to characterize the interlaboratory accuracy, precision and ruggedness of the amalgamation CV-AAS methods.

Experimental

Instrumentation

The schematic diagram of the amalgamation CV-AAS system is presented in Figure 1. Mercury vapor evolved from the reduction-aeration cell is trapped and concentrated by amalgamation on the silver-wool plug positioned in the center of the amalgamation cell. After the reduction-aeration and the on-line amalgamation steps are completed, mercury is desorbed from the silver wool by resistance heating to approximately 500°C. The mercury vapor is transported via a nitrogen-gas purge into the absorption cell, and the maximum peak absorbance is recorded. The appearance time of the mercury peak on the strip-chart recorder with a 1-second time constant is approximately 17 seconds; the maximum peak absorbance occurs between 25 and 30 seconds after heating of the silver-wool plug is initiated. The amalgamation/thermal desorption process eliminates interfering water and organic vapors prior to the mercury absorption measurement without compromising the instrument detection limit. Although continuum-source background correction should also compensate for such vapor interferences, the increased noise level in most of the older atomic absorption systems when operated in the background-correction mode degrades the detection limit. An additional benefit of amalgamation CV-AAS is that the method can be successfully implemented even by using an atomic absorption instrument that is not equipped with a dynamic background-corrector accessory.

Amalgamation Cells

To minimize experimental variability, all mercury amalgamation cells used in this study were manufactured, assembled, and tested by Battelle staff. The amalgamation cell, with an overall length of 105 mm, consists of ST 12/5 male and female ball joints annealed to opposite ends of Pyrex glass tubing (5 mm i.d.). A silver-wool plug (Fisher Scientific Company, Fair Lawn, NJ) of 0.7 g, with a mass uncertainty of 0.05 g, is inserted into the Pyrex tube and then compacted into a cylinder having approximate

dimensions of 5 mm diameter and 20 mm length.

A 92-cm length of 22-gauge Chromel A wire (Fisher Scientific Company, Fair Lawn, NJ) is wrapped around the Pyrex tubing, providing 30 windings and a resistance of 22 ohms/ft. The 0.7-g plug of silver wool quantitatively amalgamates mercury vapor at a nitrogen carrier-gas flow rate of 0.55 L/min with no apparent buildup of leak-inducing backpressure.

Reagents and Standards

All reagents and standards specified were reagent grade or better; the deionized water was specified as ASTM Type II water (ASTM D1193). All glassware for sample digestions, sample dilutions, and standard preparations must be prewashed sequentially with an aqueous detergent solution, mineral acids, and Type II water.

The following samples were used in this study:

- Coal fly ash (NBS-SRM 1633a) with a certified mercury concentration of $0.16 \pm 0.01 \mu\text{g/g}$ for a sample size of at least 250 mg.
- Marine sediment (MESS-1, National Research Council of Canada) with a reference mercury concentration of $0.171 \pm 0.014 \mu\text{g/g}$ for a sample size of at least 500 mg.
- Incinerator fly ash (crushed, sieved and homogenized) with no detectable mercury.
- Municipal sewage sludge (dried, sieved and homogenized) with a determined mercury concentration of approximately $1.4 \mu\text{g/g}$.
- Ground water (filtered and acidified with nitric acid) with no detectable mercury.
- Waste water with no detectable mercury.
- Deionized water (acidified with nitric acid) with no detectable mercury.

Sample Preparation

The samples were prepared for analysis by using the digestion procedures specified in the revised SW-846 Methods 7470 and 7471. Liquid samples were digested with sulfuric acid, nitric acid, permanganate and persulfate at 95°C; solid samples were digested either on a steam bath with aqua regia and permanganate, or in an autoclave with sulfuric acid, nitric acid and permanganate. All sample digests were diluted to calibrated volume in 100-mL volumetric flasks with deionized water following the addition of the sodium chloride-hydroxylamine hydro-

chloride reagent solution to reduce manganese dioxide and excess permanganate to the soluble divalent manganese form. This procedure permitted sampling of multiple aliquots and further dilutions as needed. The sample aliquot added to the reduction-aeration vessel was diluted to 100 mL with Type II water.

Results and Discussion

Phase I - Amalgamation Cell Testing and Sample Characterization

To minimize variability in the instrumental responses, the silver-wool amalgamation cells were fabricated and individually tested at Battelle. The parameter used to assess uniformity of performance among the amalgamation cells was the slope of their calibration curves. The slopes of four-point calibration curves for the first 8 cells ranged from 0.0048 to 0.0065 absorbance units/ng of mercury, with an average value of $0.0058 \pm 0.0006 \text{ abs/ng}$. The average recovery of a 50-ng mercury standard, using a calibration curve with a slope of 0.0052 abs/ng, for 15 additional cells was 98 ± 6 percent, with a recovery range of 89 to 108 percent. The overall testing results indicate uniformity in performance among the 23 amalgamation cells with respect to amalgamation efficiency and thermal-desorption characteristics. The differences in instrument responses for the different amalgamation cells are expected to be small compared to the overall measurement variabilities between the participating laboratories.

Battelle staff analyzed NBS-SRM 1633a, with a certified mercury value of $0.16 \pm 0.01 \mu\text{g/g}$; the results of duplicate 0.2-g portions were $0.145 \mu\text{g/g}$ and $0.160 \mu\text{g/g}$. The recoveries of inorganic mercury predigestion spikes added to the fly ash samples were 130 percent for duplicate 10-ng mercury spikes, and 112 percent and 104 percent for duplicate 50-ng mercury spikes. In triplicate 0.2-g samples of incinerator ash analyzed according to the revised Method 7471, no mercury was detected.

The average mercury concentration determined in 8 replicate 0.2-g portions of the sewage sludge sample was $1.4 \mu\text{g/g}$; the compiled results ranged from approximately $1.2 \mu\text{g/g}$ to $1.4 \mu\text{g/g}$ of mercury. These results indicate that the mercury content of the municipal sewage sludge is homogeneous to approximately $\pm 0.1 \text{ mg/kg}$.

The average recoveries of duplicate 90-ng and 180-ng Hg predigestion spikes (as methyl mercuric chloride) were 75 percent and 90 percent, respectively, and the average recoveries of duplicate 100-ng and 200-ng Hg predigestion spikes (as inorganic mercuric chloride) were 108 percent and 106 percent, respectively. The overall recoveries of organic and inorganic mercury added as predigestion spikes are within ± 25 percent of full recovery.

No mercury was found in four replicate 50-mL test aliquots of the ground-water sample. The average inorganic spike recoveries of four sample replicates were 106 percent for 50-ng mercury spikes and 105 percent for 100-ng mercury spikes.

Phase II - Preliminary Laboratory Evaluation

Method evaluation materials were sent to 19 laboratories participating in the preliminary laboratory evaluation phase. Each package contained: (1) a tested silver-wool amalgamation cell, (2) instructions for the installation and use of the amalgamation cell, (3) two samples and appropriate evaluation standard solutions, (4) instructions for the preparation and analysis of the samples, and (5) instructions for reporting the analytical results to Battelle. One laboratory withdrew at the beginning of Phase II after receiving the package, leaving a total of 18 participating laboratories. The two samples shipped to the participating laboratories consisted of deionized water for the evaluation of revised Method 7470, and NBS-SRM 1633a (Coal Fly Ash) for the evaluation of revised Method 7471. A spiking solution, containing mercury at a concentration of 1.5 mg/L, was included for spiking the deionized water sample. The participants were instructed to add 50 μ L of the 1.5-mg/L spiking solution (equivalent to 75 ng Hg) to a 20-mL aliquot of the water

sample as a predigestion spike. The laboratories were also provided a stock solution containing mercury at a concentration of 1000 mg/L to be used for preparing intermediate and final calibration standards.

For sample analyses, the participants were instructed to: (1) construct a 4-point calibration curve comprised of the absorbances for a reagent blank, 10-ng, 50-ng, and 100-ng Hg standards, (2) analyze the spiked water sample according to the revised Method 7470, (3) analyze the coal fly ash sample according to the revised Method 7471, and (4) prepare and analyze one reagent blank each for the revised Methods 7470 and 7471.

The slopes of the calibration curves generated by the 18 participating laboratories for the two revised methods ranged from 0.0001 to 0.0056 absorbance units/nanogram. Ten laboratories reported slopes between 0.003 and 0.006 absorbance units/nanogram for at least one of the revised methods. Battelle researchers have found that this is a typical range for the slope of the calibration curve obtained by using the amalgamation CV-AAS system according to the specific instructions described in revised Methods 7470 and 7471. Ten laboratories reported reagent blank values less than 0.03 absorbance unit. Battelle researchers have consistently measured absorbance values between 0.02 and 0.03 absorbance units for reagent blanks analyzed according to the revised Methods 7470 and 7471. Based on a typical instrument calibration slope of 0.005 absorbance units/nanogram, reagent blank absorbances in this range correspond to approximately 5 ng Hg. Reagent blanks for aqueous mercury calibration standards, consisting only of the stannous chloride reductant and deionized water, generally produce 0.001 absorbance unit; this is negligible relative to the absorbance values for the

reagent blanks carried through the digestions. Some of the apparent low reagent blank values reported by the laboratories may be suppressed in those cases in which the reported calibration slopes were lower than the typical range of values.

Two laboratories did not submit results for the spiked deionized water sample, and five laboratories did not report results for the coal fly ash sample. The results reported by one laboratory were excluded as obvious outliers. The other laboratories reported mercury concentrations for the spiked water sample ranging from 0.00076 to 0.0074 mg/L, and for the coal fly ash sample from 0.04 to 1.1 mg/kg. Thus, the calculated mercury recoveries for the water samples ranged from 20 percent (a 5-fold negative bias) to 200 percent (a 2-fold positive bias), and those for the coal fly ash samples ranged from 25 percent (a 4-fold negative bias) to 690 percent (a 7-fold positive bias).

A summary of the statistics across all laboratories (except the obvious outlier) for the mercury measurements on the spiked deionized water and coal fly ash samples is presented in Table 1. The means and standard deviations, reported as percent recoveries, were calculated for all laboratories and also for only those eventually selected for Phase III. After the outlier results had been removed, the remaining data were smoothly dispersed, and no additional outliers were obvious.

The data reported by some of the participating laboratories for these preliminary sample analyses are in appreciable error. Possible contributing factors to the wide range of results, especially for the coal fly ash sample, include high absorbance values for the reagent blanks resulting from sample digestions, a propagation of errors in the calculated data submitted to Battelle, and the general lack of operator experience with the technique of silver-wool amal-

Table 1. Statistical Summary of Mercury Measurements on Spiked Deionized Water and Coal Fly Ash

Number of Laboratories	Percent Recoveries	
	Spiked Deionized Water	Coal Fly Ash
All Labs ^a		
Mean	99	230
Standard Deviation	53	200
Phase III Labs		
Mean	110	230
Standard Deviation	57	210

^a Recoveries for the obvious outlier laboratory were not used in the calculations of the means and the standard deviations.

gamation. Some laboratories measured reagent blank values as high as 0.6 absorbance unit, which is equivalent to approximately 120 ng of mercury based on a calibration slope of 0.005 absorbance units/ng; such a reagent blank value exceeds the linear range of the amalgamation CV-AAS method. Battelle staff discussed the results with many of the laboratories; recommendations for improvements in controlling mercury contamination of the reagent blank and test sample, as well as suggestions for implementation of the amalgamation CV-AAS system, were provided. In general, the high dispersion of data between laboratories for both samples indicates a lack of ruggedness for the amalgamation CV-AAS method in this testing phase of the study.

Phase III- Interlaboratory Evaluation

The results of the preliminary sample analyses in Phase II were used as a qualitative guideline but not as absolute criteria for the selection of 10 laboratories for participation in Phase III.

Five laboratories obtained results which were in close agreement with the target mercury values for both samples. Five other laboratories obtained less satisfactory results but exhibited sufficient interest and enthusiasm in the silver-wool amalgamation technique; these 5 laboratories were judged to be capable participants in Phase III.

Two different sample sets, denoted as Option A and B and which comprised three solid and three aqueous samples, were shipped to the participants in Phase III, together with detailed instructions. Some of the samples required addition of a predigestion spike. Option A samples were supplied in duplicate and Option B samples in triplicate. The samples were shipped unspiked to avoid potential losses of trace mercury spikes added to samples with a reducing matrix. Predigestion spiking solutions containing organic and inorganic mercury were sent to the participants with spiking instructions. One solid sample in Option B was also to be spiked with copper, a potential interferent in CV-AAS analyses for mercury.

The slopes of the calibration curves reported by the 10 participating laboratories ranged from 0.0010 to 0.0065 absorbance units/nanogram of mercury. Six of the 10 laboratories reported slopes within the range experienced by Battelle staff (0.003 and 0.006 absorbance units/nanogram). The absorbance values for the reagent blanks ranged from -0.004

to 0.075 absorbance units. Eight of the 10 laboratories reported reagent blank values less than 0.03 absorbance unit. Based on a typical calibration slope of 0.005 absorbance unit/nanogram, a reagent blank value of 0.03 absorbance unit corresponds to a maximum concentration of 6 ng Hg in the reagent blank. In general, the 10 laboratories in Phase III reported a lower dispersion and a narrower range of results for calibration and reagent blank data than the 18 laboratories in Phase II. The 10 Phase-III laboratories represent a more consistent range of personnel skills and demonstrated competence in trace analysis. The lower dispersion in the data may also be a direct result of the acquired experience and familiarity with the silver-wool amalgamation technique by the 10 laboratories from their participation in the Phase II study.

The statistical summaries of the interlaboratory study results are presented in Tables 2 and 3. The means and standard deviations are reported in concentration units for those environmental samples originally having nondetectable endogenous mercury concentrations, and in percent recoveries for those samples having detectable target mercury concentrations. The percent recoveries are calculated as 100 times the measured concentration divided by the target concentration.

Outlier tests were performed on the laboratory means for each option within each sample type. One laboratory was identified as an outlier for most samples, and its results were excluded from statistical treatment for all sample types.

The average mercury concentration or percent recovery over all laboratories in a given option is reported in the third column of Tables 2 and 3. The fourth column contains the pooled within-laboratory standard deviations of the mercury concentrations or percent recoveries. The within-laboratory variances are simply calculated as the averages of the variances for the individual laboratories. The between-laboratory standard deviations of the mercury concentrations or percent recoveries are reported in the fifth column. This statistic characterizes the variabilities in the true average mercury concentration or percent recovery values across the laboratories. The between-laboratory variances are calculated according to the following formula:

$$S_B^2 = \frac{1}{n} (S_x^2 - S_w^2)$$

where

$$S_x^2$$

is the variance of the average percent recoveries for an individual laboratory, n is the number of sample results used to calculate the average percent recovery for each laboratory, and

$$S_w^2$$

is the within-laboratory variance. If

$$S_x^2$$

is less than

$$S_w^2,$$

then

$$S_B^2$$

is set to zero. The total standard deviations are reported in the last column. The total variance is simply the sum of the within-laboratory and between-laboratory variances.

The high dispersion of the interlaboratory data precludes any statistical treatment of the results for the revised Methods 7470 and 7471 that is more rigorous than the analyses conducted and summarized in Tables 2 and 3, respectively. As shown in Table 2 for the revised Method 7470, the total standard deviations for Options A and B are similar for each of the 3 test samples. However, as shown in Table 3 for the revised Method 7471, neither option demonstrates a consistently lower total standard deviation for all samples. For example, the total standard deviations of the results in Option A are 4-fold lower than those in Option B for the incinerator fly ash and the marine reference sediment samples. However, the total standard deviation in Option B is approximately 2-fold lower than that in Option A for the spiked incinerator fly ash sample. The total standard deviations of the results in both options are similar for the unspiked and the spiked municipal sewage sludge sample. The relative differences between the total standard deviations in Options A and B by the revised Method 7471 for the 5 samples appear to be random.

With the exception of the spiked waste-water sample in Table 2 and of the spiked incinerator fly-ash sample in Table 3, the grand means using the

Table 2. Statistical Summary of Mercury Measurements Using Revised Method 7470^a

Sample	Option	Mean	Pooled Standard Deviation	Between Standard Deviation	Total Standard Deviation
Samples Having Nondetectable Target Concentrations ^b					
Ground Water	A	4	1.8	1.4	2.3
Ground Water	B	6	1.5	2.1	2.6
Samples Having Detectable Target Concentrations ^c					
Waste Water, Spiked	A	140	17	45	48
Waste Water, Spiked	B	150	15	39	42
Dilute Nitric Acid, Spiked	A	82	27	27	38
Dilute Nitric Acid, Spiked	B	82	31	17	36

^a Results for an outlier laboratory in Option A were not used in the statistical analyses for any sample.

^b Results are in absolute concentration units (ng).

^c Results are in percent recovery.

Table 3. Statistical Summary of Mercury Measurements Using Revised Method 7471^a

Sample	Option	Mean	Pooled Standard Deviation	Between Standard Deviation	Total Standard Deviation
Samples Having Nondetectable Target Concentrations ^b					
Incinerator Fly Ash	A	0.021	0.0090	0.0076	0.012
Incinerator Fly Ash	B	0.074	0.021	0.041	0.046
Samples Having Detectable Target Concentrations ^c					
Marine Sediment MESS-1	A	110	6.5	10	12
Marine Sediment MESS-1	B	110	48	0	48
Incinerator Fly Ash, Spiked	A	74	8.6	32	34
Incinerator Fly Ash, Spiked	B	39	15	11	19
Municipal Sewage Sludge	A	90	14	18	22
Municipal Sewage Sludge	B	88	20	21	30
Municipal Sewage Sludge, Spiked	A	97	26	25	36
Municipal Sewage Sludge, Spiked	B	90	12	26	28

^a Results for an outlier laboratory in Option A were not used in the statistical analyses for any sample.

^b Results are in absolute concentration units (mg/kg).

^c Results are in percent recovery.

revised Methods 7470 and 7471, respectively, are generally within 25 percent of the target values. However, the total standard deviations for all sample types represent relative standard deviations as high as 50 percent. Because of such highly dispersed results, in addition to the contradictory results of the copper interference test, the revised Methods 7470 and 7471 presently lack the necessary ruggedness to be successfully used for routine testing by analytical laboratories without additional analyst training and demonstration of proficiency.

Conclusions

The results of this interlaboratory study indicate that only a few of the collaborating laboratories demonstrated proficiency in incorporating the silver-wool amalgamation apparatus into their

CV-AAS systems within a relatively short learning period. These laboratories reported calibration slopes in an acceptable range based on Battelle's experience with the amalgamation CV-AAS method. Only a few of the laboratories were able to perform accurate and precise measurements on both aqueous and solid environmental samples.

A statistical treatment of the interlaboratory results indicates that the amalgamation CV-AAS method currently lacks sufficient ruggedness to be successfully used by analytical laboratories for routine testing but, when properly implemented by proficient laboratory personnel, it may serve as an alternative to the recirculating CV-AAS method described in the current EPA protocols of Methods 7470 and 7471. However, to fully realize the analytical

benefits of the amalgamation CV-AAS method and to obtain accurate and precise data, a complete appreciation and awareness of the critical factors affecting successful trace analyses are essential, and a high level of sophisticated operation and operator skill is required. Moreover, because of the increased sensitivity of the amalgamation CV-AAS method, improved analytic techniques and the use of high-purity reagents are required to minimize mercury contamination that would not be a problem when using the recirculating CV-AAS method with samples of high mercury concentrations.

To achieve better results, many of the laboratories reported that they had modified the operating parameters of the silver-wool amalgamation apparatus from those specifically described in the revised protocols. This may have be

necessary when the silver-wool amalgamation apparatus was not assembled and configured properly according to the directions in the protocols, or when the amalgamation apparatus had to be reconfigured to fit a specific commercial or customized CV-AAS system. The different operating conditions used by some laboratories may have contributed to the high dispersion in the amalgamation CV-AAS data submitted to Battelle.

Most of the comments from the collaborating laboratories addressed the increased sensitivity and the longer analysis time of the amalgamation CV-AAS method. Many laboratories recognized the need for higher operator skill and the importance of the need for high-purity reagents to minimize mercury contamination. The benefit of an amalgamation CV-AAS method with increased sensitivity but at the expense of longer analysis time was challenged by some laboratories from a business economics viewpoint; the longer analysis time would increase sample analysis costs to the laboratory that would have to incur the additional costs or charge higher analysis fees to the customer. Although the faster recirculating CV-AAS method was considered adequate for the majority of their samples, other laboratories expressed special needs for the increased sensitivity of the amalgamation CV-AAS method and indicated that it would be valuable in research and for difficult analysis problems. Some laboratories expressed enthusiasm about the amalgamation CV-AAS method and indicated that they plan to incorporate this method into their laboratory operations for additional flexibility. The extent of time and cost constraints for adapting the amalgamation CV-AAS method to a specific sample workload may be influenced by whether the emphasis of the analytical laboratory is on commercial testing or on research, development and specialty problems in a support function of a larger organization.

In conclusion, it is recommended that the CV-AAS methods be used to characterize waste samples for mercury as described below:

(1) The current EPA protocols for the recirculating CV-AAS method should be used for analyses when the threshold concentration value for mercury is relatively high and when the samples do not contain volatile organic compounds that could cause nonspecific absorption interferences.

(2) The amalgamation CV-AAS method should be used for the analyses of samples containing low mercury concentrations which can not be easily determined by the recirculating CV-AAS method.

(3) The amalgamation CV-AAS method should also be used for the analyses of samples containing volatile organic compounds which will cause nonspecific absorption interferences and inaccurate results with the recirculating CV-AAS method.

Because of the greater sensitivity and the more sophisticated apparatus of the amalgamation CV-AAS system, it is emphasized that the laboratory analysts be properly trained and that they demonstrate proficiency with the amalgamation method before initiating regulatory analyses for mercury.

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The EPA author, **Werner F. Beckert**, (also the EPA Project Officer, see below) is with the Environmental Monitoring Systems Laboratory, Las Vegas, NV. Judy E. Gebhart, Jerry D. Messman, and Gordon F. Wallace are with the Battelle Columbus Division, Columbus, OH 43201-2693.

The complete report, entitled "Interlaboratory Evaluation of SW-846 Methods 7470 and 7471 for the Determination of Mercury in Environmental Samples," (Order No. PB 88-196 001/AS; Cost: \$14.95, subject to change) will be available only from:

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
Telephone: 703-487-4650

The EPA Project Officer can be contacted at:
Environmental Monitoring Systems Laboratory
U.S. Environmental Protection Agency
Las Vegas, NV 89193-3478

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