MERCURY IN SOLIDS AND SOLUTIONS BY THERMAL DECOMPOSITION, AMALGAMATION, AND ATOMIC ABSORPTION SPECTROPHOTOMETRY

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed standard operating procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 This method is for the determination of the following RCRA analyte in solids, aqueous samples, and digested solutions in both the laboratory and field environments:

Analyte	CAS No.[a]
Mercury total (organic and inorganic)	7439-97-6

[a]Chemical Abstract Service Registry Number

Integration of thermal decomposition sample preparation and atomic absorption detection reduces the total analysis time of most samples to less than 5 min in either the laboratory or field setting. Total mercury (organic and inorganic) in soils, sediments, bottom deposits, and sludge-type materials as well as in aqueous wastes and ground waters can be determined without sample chemical pretreatment using this method, except as noted. Alternatively, this method can be used for the detection of total mercury from total decomposition sample preparation methods, such as Method 3052, or for detection of extracted or leached mercury compounds or species from methods such as the 3000 series methods (as detailed in Chapter Three).

NOTE: For unique circumstances when mercury could be bound in silicates or other matrices that may not thermally decompose, validation of direct analysis of the solid should be confirmed with total decomposition with an appropriate method (such as Method 3052), followed by analysis with this method.

1.2 Prior to employing this method, analysts are advised to consult the manufacturer's instructions for additional information on quality control procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques

employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.3 Use of this method is restricted to use by, or under supervision of, properly experienced and trained personnel. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Controlled heating in an oxygenated decomposition furnace is used to liberate mercury from solid and aqueous samples in the instrument. The sample is dried and then thermally and chemically decomposed within the decomposition furnace. The decomposition products are carried by flowing oxygen to the catalytic section of the furnace. Oxidation is completed and halogens and nitrogen/sulfur oxides are trapped. The remaining decomposition products are then carried to an amalgamator that selectively traps mercury. After the system is flushed with oxygen to remove any remaining gases or decomposition products, the amalgamator is rapidly heated, releasing mercury vapor. Flowing oxygen carries the mercury vapor through absorbance cells positioned in the light path of a single wavelength atomic absorption spectrophotometer. Absorbance (peak height or peak area) is measured at 253.7 nm as a function of mercury concentration.

2.2 The typical working range for this method is 0.05 - 600 ng. The mercury vapor is first carried through a long pathlength absorbance cell and then a short pathlength absorbance cell. (The lengths of the first cell and the second cell are in a ratio of 10:1 or another appropriate ratio.) The same quantity of mercury is measured twice, using two different sensitivities (see Figure 1), resulting in a dynamic range that spans at least four orders of magnitude.

2.3 The instrument detection limit (IDL) for this method is 0.01 ng of total mercury.

3.0 DEFINITIONS

3.1 Thermal decomposition -- Partial or complete degradation of sample components using convection and conduction heating mechanisms resulting in the release of volatile components such as water, carbon dioxide, organic substances, elements in the form of oxides or complex compounds, and elemental gases.

3.2 Amalgamation -- The process by which mercury forms a metal alloy with gold.

3.3 Amalgamator -- A system composed of gold particles at a high surface area to volume ratio for the purpose of amalgamating mercury vapor.

3.4 Primary calibration -- A complete calibration of the instrument’s working range. This calibration is performed initially and when any significant instrumental parameters are changed. For example, in this method a primary calibration should be performed after the decomposition tube, amalgamator, or oxygen tank is replaced.

3.5 Daily calibration -- A calibration performed with minimal standards to ensure that the primary calibration is valid. For example, when two standards within the range of interest are analyzed and agree within 10% of their true value the primary calibration is assumed to be valid.

3.6 Memory effects -- Mercury vapor may remain in the decomposition tube, amalgamator, or absorbance cells and be released in a subsequent analysis resulting in a positive bias. For example, this may result when a low concentration sample is analyzed after a sample of high mercury content.

3.7 Sample boat -- The non-amalgamating thermally stable vessel used for containment and transport of the solid or liquid sample for thermal decomposition.

3.8 Also refer to Chapter One, Chapter Three, and the manufacturer's instructions for definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary. Refer to each method to be used for specific guidance on quality control procedures and to Chapter Three for general guidance on the cleaning of glassware. Also refer to Method 7000 for a discussion of interferences.

4.2 In areas where mercury contamination is an existing problem, the background signal may be significantly increased.

4.3 Memory effects between analyses may be encountered when analyzing a sample of high mercury concentration ($ 400 ng) prior to analyzing one of low concentration (# 25 ng). Typically, to minimize memory effects, analyze the samples in batches of low and high concentration, always analyzing those of low concentration first. If this batching process cannot be accomplished, a blank analysis with an extended decomposition time may be required following the analysis of a highly concentrated sample to limit memory effects.

4.4 Co-absorbing gases, such as free chlorine and certain organics (which are common interferants as indicated in Methods 7470 and 7471) should not interfere with mercury determination by this method due to the release of decomposition products by the decomposition furnace, removal of some decomposition products by the decomposition catalyst, and the selective entrapment of mercury vapor on the amalgamator.

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

5.2 Many mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Extreme care must be exercised in the handling of concentrated mercury

reagents. Concentrated mercury reagents should only be handled by analysts knowledgeable of their risks and of safe handling procedures.

5.3 Samples with a high organic content (e.g., oil saturated soil) should be analyzed with caution. The sample size should be scaled down as too large of a sample size may result in ignition of the sample within the decomposition tube.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

This section does not list common laboratory glassware (e.g., beakers and flasks).

6.1 The working scheme of the mercury analysis system is illustrated in Figure 2. The sample introduction device consists of a motorized support equipped with a metal or metal alloy sample boat that is appropriate for solids and liquids. An example of an appropriate boat would be one made of nickel with a liquid capacity of 0.5 - 1.0 mL. Once the sample is either manually or automatically dispensed into the sample boat, the boat is mechanically introduced automatically into a quartz decomposition tube. The decomposition tube is heated by two independently programmable furnaces -- the decomposition and catalyst furnaces -- and each furnace is capable of maintaining a temperature of at least 750 EC. The sample is dried and thermally decomposed in an oxygen environment, releasing mercury vapor. The mercury vapor is transported by oxygen over the amalgamator that traps the mercury. Once the sample is completely decomposed the trapped mercury is desorbed rapidly by heating the amalgamator with the mercury release furnace. The mercury vapor passes through two absorbance cuvettes, in series, that are separated by a collection flask outside the optical axis. The flow path through the spectrometer and cuvettes is maintained at approximately 120 EC, by a heating unit, to prevent condensation and minimize carry-over effects. A mercury vapor lamp is used as the light source. The detector is connected to a computer for data acquisition and analysis.

6.2 The DMA 80 automatic mercury analyzer (Milestone, Inc.) is the instrument used for the scheme outlined above. It has been tested for use with this method. Other instruments based on these principles may also be appropriate.

6.3 This method is not limited to mercury vapor generation by thermal decomposition. Alternatively, other mercury vapor introduction systems, such as mercury cold vapor generation, may be appropriate. Alternative sample introduction apparatus may be applied provided that the analyst can demonstrate and document performance appropriate for the data quality needs of the particular application.

6.4 This method is not limited to analyzing total mercury content. This detection scheme can be used for analysis of individual species of mercury that have been separated by an appropriate method or instrument system.

7.0 REAGENTS AND STANDARDS

7.1 Reagent-grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Reagent water -- Reagent water should be interference free. All references to water in this method refer to reagent water unless otherwise specified.

7.3 High purity oxygen gas -- High purity oxygen should be interference and mercury free. If the oxygen is possibly contaminated with mercury vapor, a gold mesh filter should be inserted between the gas cylinder and the mercury analysis instrument to prevent any mercury from entering the instrument.

7.4 Mercury stock solution -- Dissolve 0.1354 g of mercuric chloride in 75 mL of reagent water. Add 10 mL of concentrated nitric acid and adjust the volume to 100.0 mL (1.0 mL = 1.0 mg Hg). Alternatively, a mercury stock solution may be purchased from a reputable source with a concentration of 1.0 mg Hg/mL. Verify the quality of the standard by checking it against a second source standard (see second paragraph of Sec. 9.4).

7.5 Mercury working standards -- Make successive dilutions of the stock mercury solution to obtain standards containing 100 ppm and 10 ppm.

NOTE: The stability of the mercury standards is limited to 24 - 48 hours. Fresh mercury standards must be prepared daily.

7.6 Standard reference material -- In place of aqueous mercury standards, solid reference material with a certified value for mercury may by used for calibration.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 See the introductory material to Chapter Three, "Inorganic Analytes."

8.2 All sample containers must be prewashed with detergents, acids, and reagent water. Glass, plastic, and polytetraflouroethylene (PTFE) containers are suitable in most cases. Polymers are not suitable for samples containing metallic mercury.

8.3 Metallic mercury, some inorganic mercury compounds, and many organic mercury compounds are volatile and unstable. It is advantageous to analyze the samples as soon as possible to determine the total mercury in the sample but in no case exceed the 28-day limit as defined in Chapter Three of this manual. Non-aqueous samples must be analyzed as soon as possible. If solid samples are not analyzed immediately, refrigeration is necessary.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for additional guidance on quality assurance (QA) and quality control (QC) protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and those criteria given in Chapter One, and technique-specific QC criteria take precedence over the criteria in Chapter One. Any effort involving the collection of analytical data should include development

of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Initial demonstration of proficiency

Each laboratory must demonstrate initial proficiency by following the sample preparation and analytical procedures described in this method and generating data of acceptable accuracy and precision for the target analyte (mercury) in a clean matrix. The laboratory must also repeat the demonstration of proficiency whenever new staff members are trained or significant changes in instrumentation are made.

9.3 For each batch of samples processed, at least one method blank must be carried throughout the entire sample preparation and analytical process. A method blank is prepared by using a volume or weight of reagent water at the volume or weight specified in the preparation method and then carried through the appropriate steps of the analytical process. These steps may include but are not limited to digestion, dilution, filtering, and analysis. If the method blank does not contain the target analyte at a level that interferes with the project-specific data quality objectives then the method blank would be considered acceptable. In the absence of project-specific data quality objectives, if the blank is less than the lower level of quantitation or less than 10% of the lowest sample concentration for the analyte, whichever is greater, then the method blank would be considered acceptable. If the method blank cannot be considered acceptable, the method blank should be re-run once and if still unacceptable then all samples after the last acceptable method blank must be reprepped and reanalyzed along with the other appropriate batch QC samples. These blanks will be useful in determining if samples are being contaminated.

9.4 For each batch of samples processed, at least one laboratory control sample must be carried throughout the entire sample preparation and analytical process. The laboratory control samples should be spiked with each analyte of interest at the project-specific action level or, when lacking project-specific action levels, between the low and midlevel standards. Acceptance criteria should be set at a laboratory derived limit developed through the use of historical analyses. In the absence of historical data this limit should be set at ± 20% of the spiked value. After the determination of historical data, ± 20% should still be the limit of maximum deviation to express acceptability. If the laboratory control sample cannot be considered acceptable, the laboratory control sample should be re-run once and if still unacceptable then all samples after the last acceptable laboratory control sample must be reprepped and reanalyzed. Refer to Chapter One for more information.

If more than 10 samples per day are analyzed, the working standard curve must be verified by measuring satisfactorily a LCS or mid-range standard or reference standard after every 10 samples. This sample value should be within 20% of the true value, or the previous 10 samples must be reanalyzed.

9.5 Matrix spike/matrix spike duplicates (MS/MSDs) -- MS/MSDs are intralaboratory split samples spiked with identical concentrations of each analyte of interest. The spiking occurs prior to sample preparation and analysis. An MS/MSD is used to document the bias and precision of a method in a given sample matrix. Based on the analyst's discretion, a separate spike sample and a separate duplicate sample may be analyzed in lieu of the MS/MSD. For each batch of sample processed, at least one MS/MSD sample must be carried throughout the entire sample preparation and analytical process. MS/MSD samples should be spiked at the

same level as the corresponding laboratory control sample that is at the project-specific action level or, when lacking project-specific action levels, between the low and midlevel standards. Acceptance criteria should be set at a laboratory derived limit developed through the use of historical analyses. In the absence of historical data this limit should be set at ± 20% of the spiked value for precision and # 20 relative percent difference (RPD). After the determination of historical data, 20% should still be the limit of maximum deviation for both percent recovery and relative percent difference to express acceptability.

9.6 The method of standard additions can be used to verify linearity or if matrix interference is suspected. Refer to Method 7000 for standard addition procedures.

9.7 Refer to Method 7000 for additional QA and QC information that may be applicable.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Primary calibration -- Dose 100 µL (or other appropriate volume) of a working standard onto the sample boat. Choose analytical parameters for drying, decomposition, and wait times as recommended by the manufacturer for the analysis (Sec. 11.1). Analyze each standard solution twice. For the DMA 80, choose parameters of 70 sec for drying, 100 sec for decomposition, and 40 sec for wait times (abbreviated 70/100/40) for each standard analysis. Typical calibration curves obtained in laboratory conditions are illustrated in Figures 3a and b and a calibration curve obtained in field conditions is illustrated in Figure 4. Conduct the curve using the standards described in Sec. 7.5.

10.2 Daily calibration -- Analyze at least a high and low concentration standard for each working range using the analytical parameters as recommended by the manufacturer. The working calibration standards must be measured within 10% of their true value for the curve to be considered valid.

10.3 For calibration of the high range, standards of 0, 1, 2, 3, 4, 5, and 6 ppm are recommended. These are prepared by dilution of the 100 ppm standard. For calibration of the low range, standards of 0.00, 0.05, 0.1, 0.2, 0.3, 0.4, and 0.5 ppm are recommended. These are prepared by dilution of the 10 ppm standard. Alternatively, mercury standards of 10, 1.0, and 0.1 ppm (or other appropriate concentrations) may be used. The mercury standard dosed is then adjusted by changing the volume of the standard used. A blank calibration solution is also used for a zero calibration. Acidity of the working standards should be maintained at least 0.15% nitric acid, as also recommended in Methods 7470 and 7471.

NOTE: The concentrations listed above are only recommended concentrations. The concentration of the working standards may need adjustment according to specific instrumental working ranges and/or manufactures' recommendations.

10.4 An alternative calibration using standard reference materials (SRMs) may be used. In this method, an amount of the reference material is weighed (accurate to ± 0.001 g or better) onto a tared sample boat. The analytical parameters chosen are based on the weight, moisture content, and organic content of the soil and should be as similar to the matrix of interest as possible (refer to Sec. 11.1). This procedure is repeated with several different weights of the standard reference material containing mercury concentrations in the desired working range (see Figure 5).

CAUTION: Do not dry the standard reference material as indicated on the certificate of analysis unless the SRM was prepared and analyzed that way for mercury

certification. Drying may result in loss of mercury that is thermally unstable. Drying a separate sample at the time of analysis and correcting for moisture content is appropriate.

10.5 Construct a calibration curve by plotting the absorbances of the standards versus nanograms of mercury. Determine the peak height or peak area of the sample from the chart and calculate the mercury value from the standard curve.

11.0 PROCEDURE

11.1 General analytical parameters -- The analytical parameters depend on the sample size and matrix and are instrument specific. The following table shows the guidelines given for the DMA 80. Consult the operating manual for manufacturer's recommendations. Additional or alternative staged timings can be determined through validation experimentation using standard reference materials which are appropriate for the matrix of interest.

Analytical parameters as recommended by Milestone, Inc. for the DMA 80.

Sample Type	Maximum Capacity	Drying Time (s) [1]	Decomposition Time (s) [1]	Wait Time (s)
Aqueous	500 µL or 1000 µL[2]	= [0.7 s * vol. (µL)]	100	40
Solid (dry)	500 mg	10	= [0.4 s * wt. (mg) + 100 s]	40
Solid (moist)	500 mg	= [0.7 s * wt (mg) * % water content]	= [0.4 s * wt. (mg) + 100 s]	40
Solid (high organic content)	500 mg	= [0.7 s * wt (mg) * % water content]	100	40

[1] The variability of some matrices requires calculating the drying and decomposition times.
[2] Maximum sample size is dependent on the volume of the sample boat. Typical sample boat sizes are either 0.5 or 1.0 mL.

11.2 Sample analysis

For solids, weigh a homogenized amount of the sample (to ± 0.001 g or better) onto a tared sample boat. Insert the sample boat into the instrument with appropriate clean techniques. The analytical parameters chosen are based on the weight, moisture content, and organic content of the soil (refer to Sec. 11.1). For example, for 200 mg of sediment with a water content of 45%, the parameters for the DMA 80 would be: 63/180/40.

For aqueous samples and previous prepared samples (using appropriate 3000 series methods), dose a known volume of the sample onto the sample boat (see references listed in Secs. 13.3.2 and 13.3.3). The analytical parameters chosen are based on the volume of the sample dosed (refer to Sec. 11.1). For example, for 200 µL of prepared sample, the parameters for the DMA 80 would be: 140/100/40.

11.3 Field analysis -- With a stable power supply, this method can be transported to the field for direct sample analysis without acid digestions.

11.4 Duplicates, spiked samples, and check standards should be routinely analyzed as detailed in Sec. 9.0 of this method. Samples exceeding the calibration range should be diluted and reanalyzed. Refer to Sec. 10.0 for additional guidance on calibrating the instrument.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Results need to be reported in units commensurate with their intended use and all dilutions need to be taken into account when computing final results.

12.2 Calculate metal concentrations (1) by the method of standard additions, (2) from a calibration curve, or (3) directly from the instrument's concentration read-out. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 µg/g dry weight).

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance goals for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. <u>These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.</u>

13.2 This method was validated with both solid samples and digests of solid samples. National Institute of Standards and Technology (NIST) Solid Standard Reference Materials (SRMs) were selected because of their homogeneity and availability. The selected SRMs encompass various chemical forms of mercury, including biological forms, geological forms, and contaminated environmental forms. The SRMs were analyzed directly as the solid and as the digested sample as prepared by Method 3052. These results are summarized in Table 1. These data are provided for guidance purposes only.

Field capabilities of this instrumental method were tested. Direct analysis of various SRMs were performed in a field setting; a summary of the results is given in Table 2. In addition, randomly collected field soil samples were tested using this method to further validate its use in the field. Each soil sample was collected and homogenized in approximately 10 min and analyzed in triplicate in an additional 15 min. Field data of these randomly collected soil samples indicate that typical % RSD of less than 10% can be achieved, however this is dependent on many factors including concentration of mercury and homogeneity of the sample. These data are provided for guidance purposes only.

13.3 The following documents may provide additional guidance and insight on this method and technique:

13.3.1 N. Salvato and C. Pirola, "Analysis of Mercury Traces by Means of Solid Sample Atomic Absorption Spectrometry," *Mikrochimica Acta,* Vol. 123, 63 - 71, 1996.

13.3.2 P. J. Walter and H. M. Kingston, "The Fate of Mercury in Sample Preparation," The Pittsburgh Conference, Atlanta, GA, March 1997, paper #1223.

13.3.3 H. M. Kingston, P. J. Walter, S. Chalk, E. Lorentzen and D. Link, "Chapter 3: Environmental Microwave Sample Preparation: Fundamentals, Methods, and

Applications" in Microwave Enhanced Chemistry, H.M. Kingston and S. Haswell, Eds., American Chemical Society, Washington DC, 1997.

13.3.4 Milestone, Inc., DMA 80 Operating Manual, 160B Shelton Rd., Monroe, CN 06468.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, http://www.acs.org.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society.

16.0 REFERENCES

1. H. M. Boylan, P. J. Walter and H. M, Kingston, "Direct Mercury Analysis: Field and Laboratory Validation for EPA Method 7473."

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables and figures referenced by this method. A flow chart of the procedure follows the tables and figures.

TABLE 1

EXAMPLE LABORATORY ANALYSIS RESULTS (MEAN ± 95% CONFIDENCE INTERVAL) OF DIRECT AND DIGESTED (METHOD 3052) ANALYSES OF VARIOUS NIST SRMs USING THE DMA 80 (MILESTONE, INC.)

Standard Reference Material	Direct Analysis (ng/g)	Digested Sample Analysis (ng/g)	Certified Value (ng/g)
Apple Leaves NIST SRM 1515	48.3 ± 2.4	NA	44 ± 4
Citrus Leaves NIST SRM 1572	100 ± 12	97 ± 9	80 ± 20
Estuarine Sediment NIST SRM 1646		75.2 ± 4.9	65.7 ± 8.7
63 ± 12			
Oyster Tissue NIST SRM 1566a	67.1 ± 3.2	NA	64.2 ± 6.7
Coal Fly Ash NIST SRM 1633b	139 ± 6	132 ± 12	141 ± 19
Buffalo River SedimentNIST SRM 2704	1,450 ± 24	1,450 ± 26	1,440 ± 70
Montana Highly Contaminated Soil NIST SRM 2710	33,100 ± 310	33,400 ± 230	32,600 ± 1,800

NA: Not analyzed
n$3

Data taken from Ref. 1.

TABLE 2

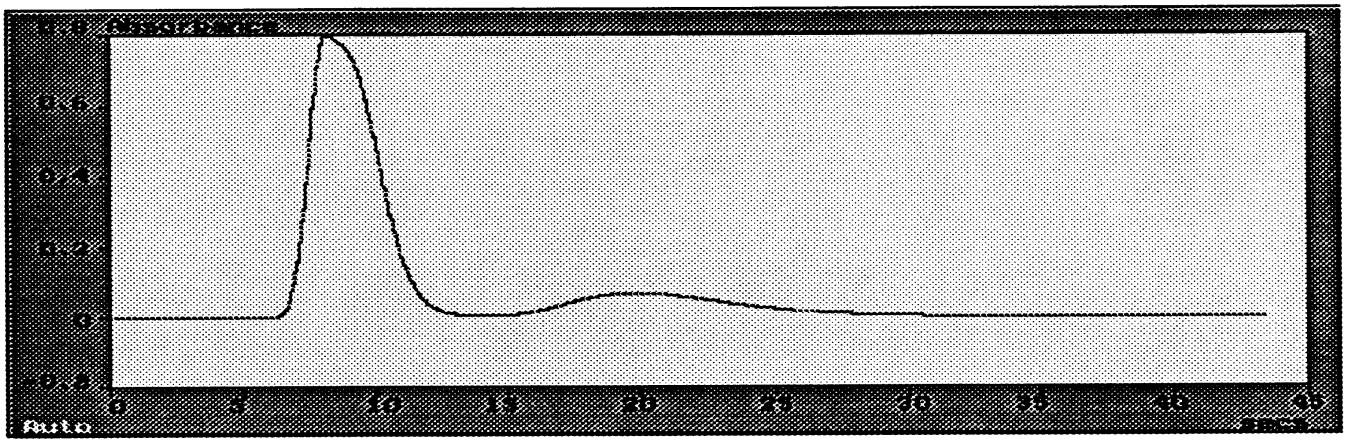
EXAMPLE FIELD ANALYSIS RESULTS (MEAN ± 95% CONFIDENCE INTERVAL) OF DIRECT ANALYSES OF VARIOUS NIST SRMs USING THE DMA 80 (MILESTONE, INC.)

Standard Reference Material	Direct Analysis (ng/g)	Certified Value (ng/g)
EstuarineSediment NIST SRM 1646	74.7 ± 2.4	63 ± 12
Oyster Tissue NIST SRM 1566a	68.0 ± 2.0	64.2 ± 6.7
Coal Fly Ash NIST SRM 1633b	139.2 ± 2.2	141 ± 19

Data taken from Ref. 1.

FIGURE 1

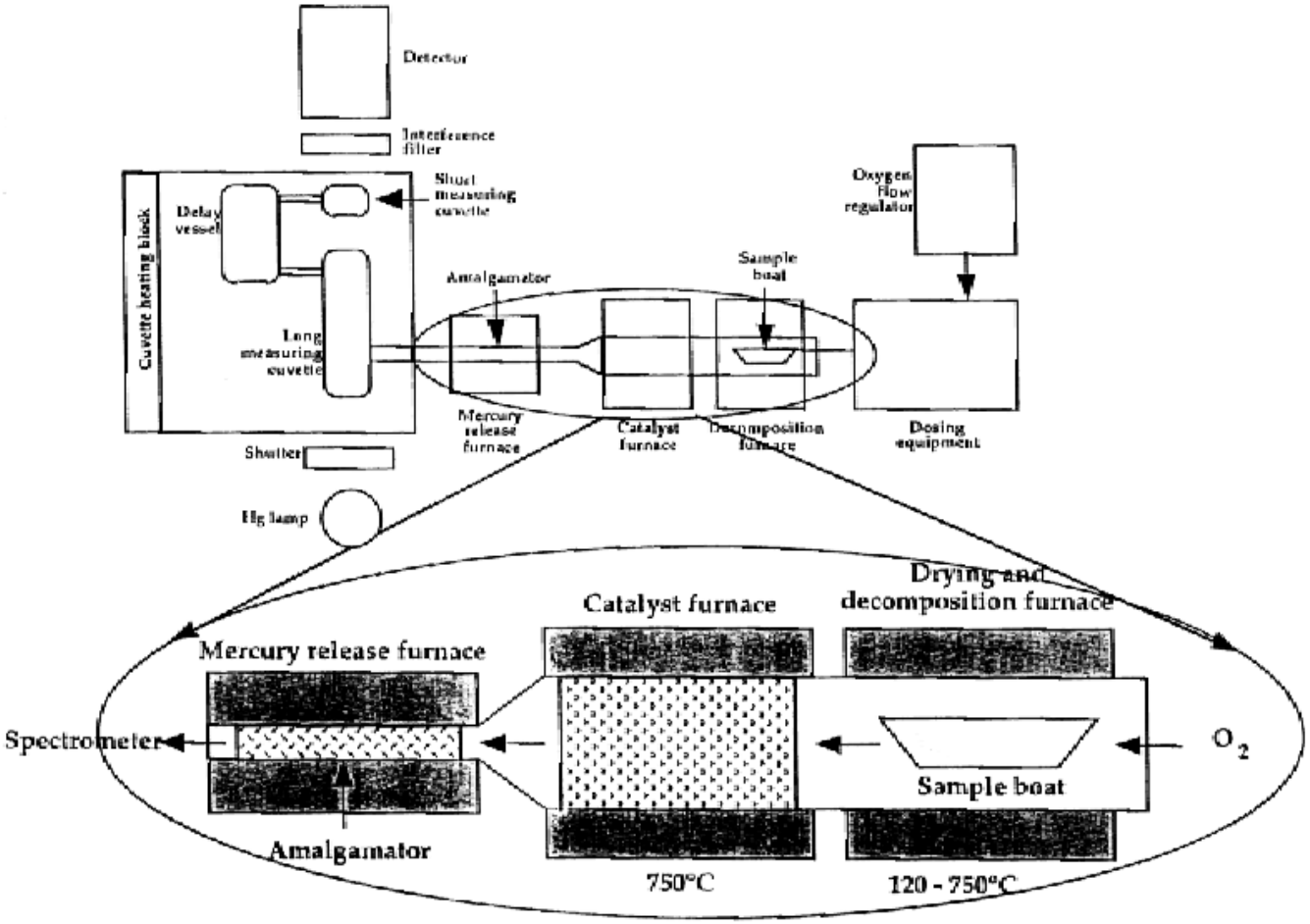
SPECTRAL OUTPUT OF DMA 80



The two individual peaks correspond to the two absorbance cells of different sensitivities. The maximum intensity of the long pathlength cuvette (low range cell) occurs at ~8 sec and the maximum intensity of the short pathlength cuvette (high range cell) occurs at ~20 sec.

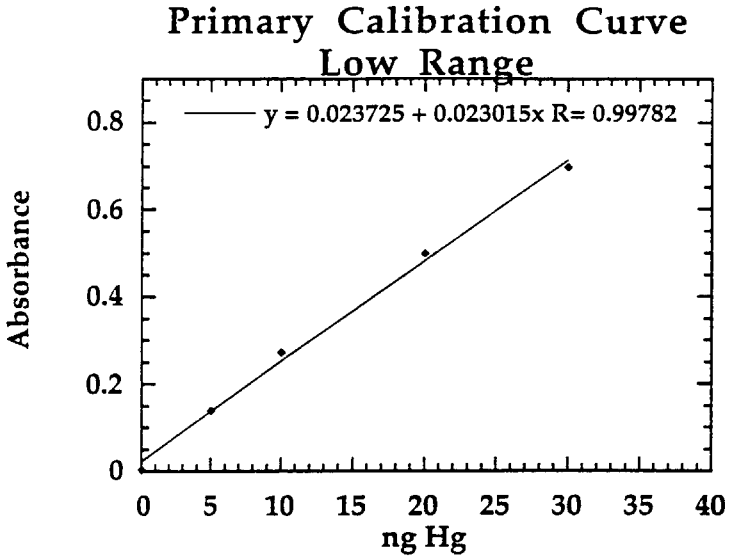
FIGURE 2

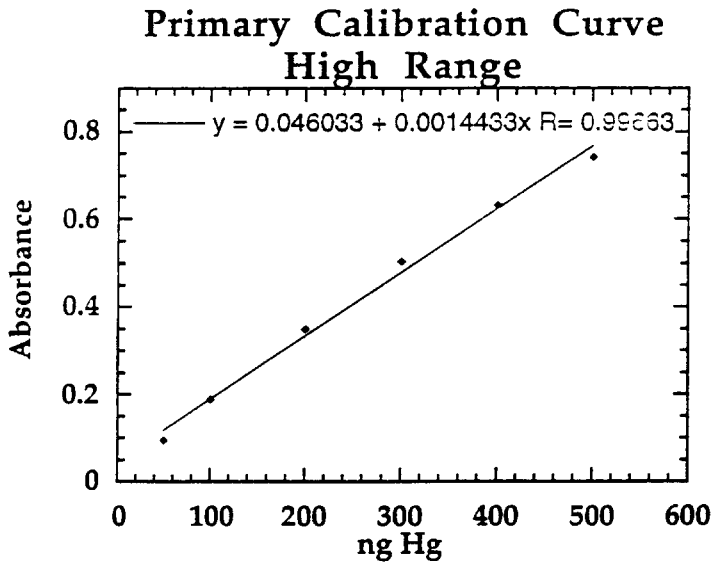
DIAGRAM OF THE MERCURY ANALYSIS SYSTEM



FIGURES 3a AND 3b

PRIMARY CALIBRATION CURVES USING THE DMA 80





The low range curve (3a) corresponds to the long pathlength cell. The high range curve (3b) corresponds to the short pathlength cell.

FIGURE 4

PRIMARY CALIBRATION CURVE USING THE DMA 80 IN FIELD ANALYSIS CONDITIONS

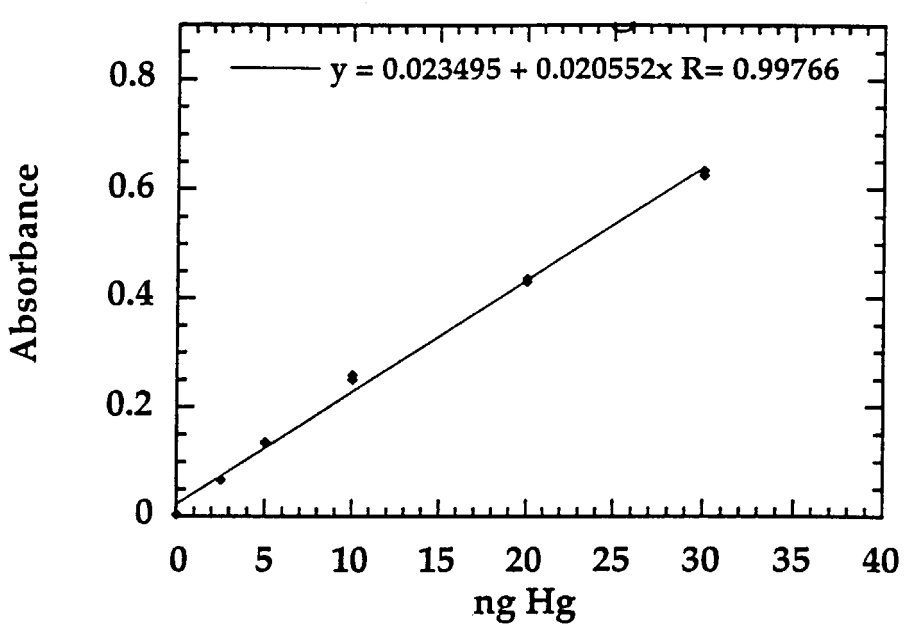
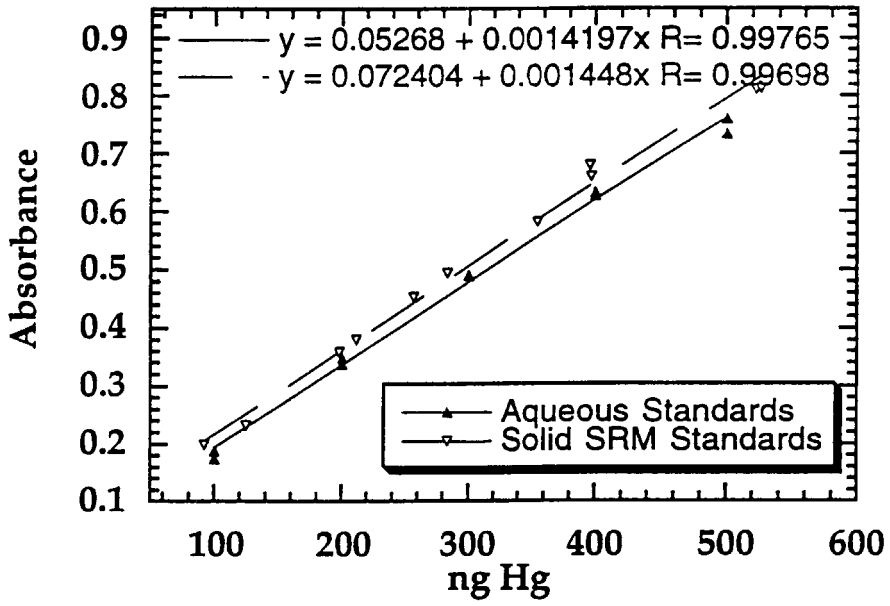
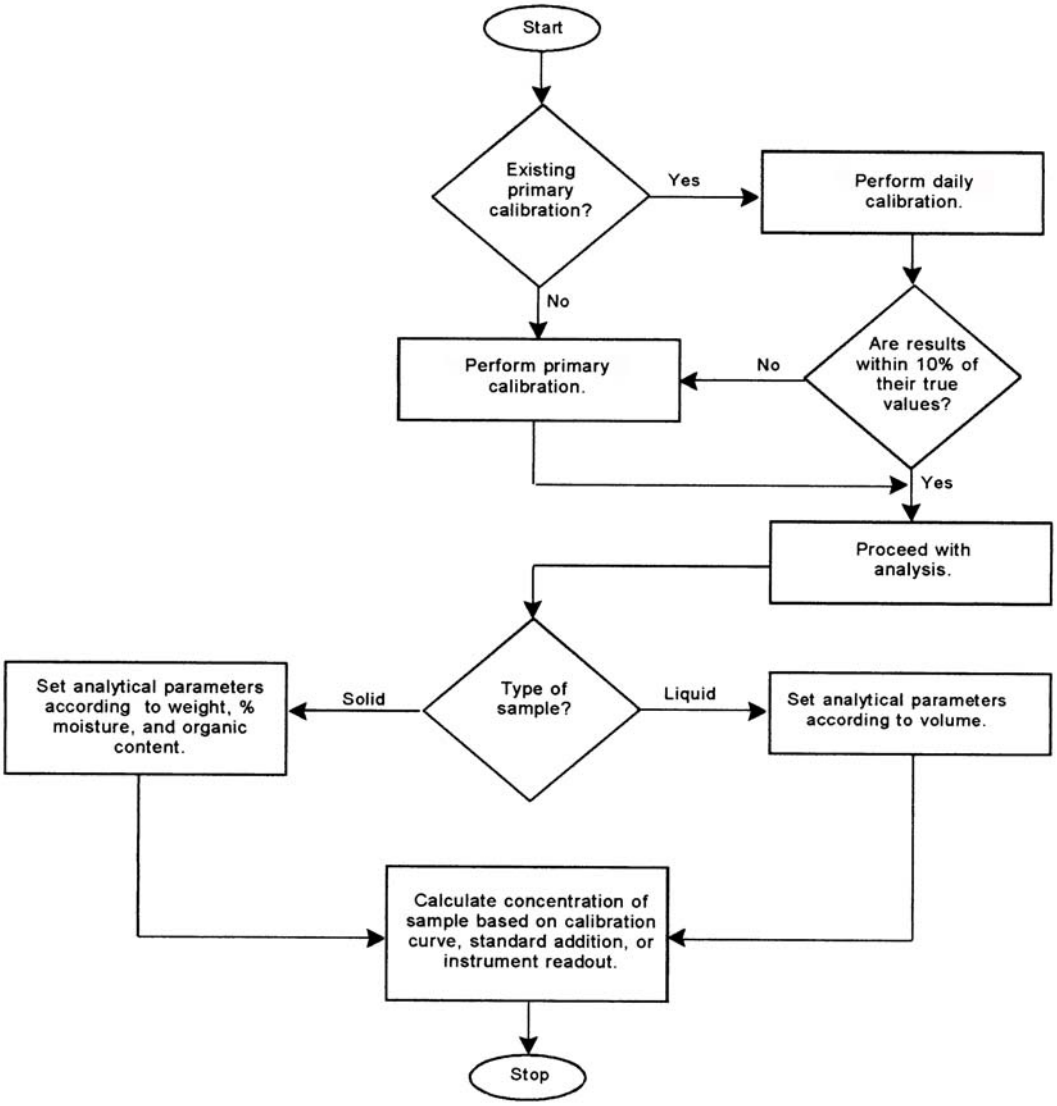


FIGURE 5

PRIMARY CALIBRATION CURVES USING THE DMA 80 -- COMPARISON OF THE CALIBRATION USING AQUEOUS STANDARD SOLUTIONS AND SOLID NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY STANDARD REFERENCE MATERIAL 2704 (BUFFALO RIVER SEDIMENT)




Start
Existing primary calibration?
Yes
Perform daily calibration.
No
Are results within 10% of their true values?
No
Perform primary calibration.
Yes
Proceed with analysis.
Type of sample?
Solid
Set analytical parameters according to weight, % moisture, and organic content.
Liquid
Set analytical parameters according to volume.
Calculate concentration of sample based on calibration curve, standard addition, or instrument readout.
Stop