PARTICULATE MERCURY IN WORKPLACE ATMOSPHERES

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Method Number:	ID-145	
Matrix:	Air*, Wipe, or Bulk	
OSHA Permissible Exposure Limits Aryl and Inorganic Compounds of Mercury (Final Rule Limit):	0.1 mg/m ³ as mercury (Ceiling) Also Skin Designation	
Mercury (Transitional Limit):	0.1 mg/m ³ as mercury (Time Weighted Average)	
Sampling (Air Samples) Collection Device:	A mixed-cellulose ester filter and a calibrated personal sampling pump are used.	
Recommended Sampling Rate:	2.0 L/min	
Recommended Air Volume:	10 L	
Analytical Procedure:	The collection medium is digested using nitric and sulfuric acids. Potassium permanganate and hydroxylamine hydrochloride are then added. The mercury in the sample is reduced using stannous chloride and analyzed using a cold vapor-atomic absorption spectrophotometer.	
Detection Limit:		
Qualitative:	0.001 mg/m ³ for a 10 L air sample	
Quantitative:	0.002 mg/m ³ for a 10 L air sample	
Precision and Accuracy (from samp	les prepared with phenyl mercuric acetate)	
CV ₁	0.086	
Bias	+0.033	
Method Classification:	Validated Method	
Date (Date Revised):	1987 (December, 1989)	
*If mercury vapor is also suspected to be present, OSHA Method No. ID-140 should also be consulted.		
Commercial manufacturers and products mentioned in this method are for descriptive use only and do not constitute endorsements by USDOL-OSHA. Similar products from other sources can be substituted.		

Division of Physical Measurements and Inorganic Analyses OSHA Technical Center Salt Lake City, Utah 1. Introduction

This method describes the collection of airborne particulate mercury on 0.8-µm mixed-cellulose ester membrane (MCE) filters and the subsequent analysis using a cold vapor-atomic absorption spectrophotometer (CV-AAS). Wipe and bulk samples can also be collected and analyzed for mercury content using this method.

1.1 History

This method is an adaptation of an analytical technique proposed by Hatch and Ott (8.1). Particulate mercury contained in air samples, as well as wipe and bulk samples have always been analyzed at the OSHA Salt Lake Technical Center (OSHA-SLTC) using CV-AAS. Elemental mercury vapor is sampled and analyzed using techniques described in Reference 8.2.

1.2 Principle

Airborne and non-airborne particulate mercury compounds and mercury-containing dust are collected, respectively, on 0.8-µm MCE and wipe filters. Bulk material is collected by grab sampling. Air filter, wipe, and bulk samples are initially dissolved using concentrated nitric and sulfuric acids. A potassium permanganate solution is added to help dissolve the sample matrix. A hydroxylamine hydrochloride solution is then added to reduce the excess potassium permanganate. Finally, stannous chloride is added to an aliquot of the sample to reduce the mercury to the vapor state. This vapor is then driven into an absorption cell of a flameless atomic absorption spectrophotometer for analysis.

- 1.3 Advantages and Disadvantages
 - 1.3.1 The air sampling device is small and portable.
 - 1.3.2 This method has adequate sensitivity for measuring workplace exposure to mercurycontaining dust and particulate mercury compounds.
 - 1.3.3 Sample preparation for analysis involves simple procedures.
 - 1.3.4 Particulate organo-mercury compounds may also be collected using 0.8 µm MCE filters.
 - 1.3.5 Elemental mercury vapor cannot be collected on the 0.8 µm MCE filters. If elemental mercury vapor is suspected to be present, a sample using Hydrar or hopcalite solid sorbent as the collection medium should be separately taken (8.2).
- 1.4 Workplace Exposure

Occupations with potential exposure to mercury and its compounds are listed (8.3):

embalmers silver extractors	amalgam makers bactericide makers barometer makers battery makers, mercury boiler makers calibration instrument makers cap loaders, percussion carbon brush makers caustic soda makers caustic soda makers ceramic workers chlorine makers dental amalgam makers dental amalgam makers dentists direct current meter workers disinfectors drug makers electric apparatus makers electroplaters embalmers	fur processors gold extractors histology technicians ink makers insecticide makers investment casting workers jewelers laboratory workers, chemical lampmakers, fluorescent manometer makers mercury workers miners, mercury neon light makers paper makers paper makers percussion cap makers pesticide workers photographers pressure gage makers refiners, mercury seed handlers silver extractors
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explosive makers farmers fingerprint detectors fireworks makers fungicide makers fur preservers switch makers, mercury tannery workers taxidermists textile printers thermometer makers wood preservative makers

1.5 Toxic Effects

Note: Information listed within this section is a synopsis of current knowledge of the physiological effects of mercury and is not intended to be used as a basis for OSHA policy.

Exposure to elemental mercury vapor can occur via the respiratory tract and skin. Possible symptoms from an acute exposure include severe nausea, vomiting, abdominal pain, bloody diarrhea, kidney damage, and death. These symptoms usually present themselves within 10 days of exposure. Potential symptoms from a chronic exposure include inflammation of the mouth and gums, excessive salivation, loosening of the teeth, kidney damage, muscle tremors, jerky gait, spasms of the extremities, personality changes, depression, irritability, and nervousness (8.3, 8.4).

1.6 Properties (8.3-8.5)

Elemental mercury (CAS No. 7439-97-6) is a silver-white, heavy, mobile, liquid metal at room temperature. Some physical properties and data for mercury are:

Atomic Number	80
Atomic Symbol	Hg
Atomic Weight	200.61
Freezing Point	-38.87 °C
Boiling Point	356.90 °C
Density	13.546 g/mL (20 °C)
Synonýms	Quicksilver, Hydrargyrum

Many different inorganic and aryl compounds of mercury exist. One of the more common aryl compounds is phenyl mercuric acetate ($C_6H_5HgOCOCH_3$) which is used as a fungicide:

CAS no.	62-38-4
Atomic Weight	336.75
Melting Point	149 °C
Volatility	slightly volatile at room temp.
Solubiliťy	soluble in alcohol, benzene, and glacial acetic acid, slightly soluble in water

2. Range

2.1 Detection Limits

The qualitative and quantitative detection limits for the analytical procedure are 0.01 μ g and 0.02 μ g mercury, respectively (8.6).

2.2 Working Range

The range of the analytical procedure has been determined to be 0.1 to 2 μ g mercury. Using the analytical conditions specified, a nonlinear response was noted above 2 μ g.

3. Method Performance

- 3.1 The average recovery of 88 quality control samples containing mercury spiked on MCE filters in the approximate range of 1 to 2 times the OSHA PEL (assuming a 20-L air volume) was 90.8% with a coefficient of variation (CV₁) of 0.149 (8.7). The variability and slight decrease in recovery for these samples, which were prepared from 1984 to 1986, was attributed to instability during storage. These samples had been spiked with dilute nitric acid solutions containing mercury and some samples were analyzed months after preparation.
- 3.2 An additional test to determine method performance was conducted using spikes of phenyl mercuric acetate (PMA) on 24 MCE filters (8.8). The filters were spiked with PMA solutions containing 50 to 200 µg total mercury. The recovery for 18 of the samples was 103.3% and a coefficient of variation of 0.086. The remaining six samples were subjected to a retention efficiency test where air was

drawn through the spiked filters for 3 h. The filters were then analyzed. Results indicated the filters sufficiently retained the PMA.

4. Interferences

Organic-free deionized water should be used during sample and standard preparation. Any compound with the same absorbance wavelength as mercury (253.7 nm) can be a positive interference. Some volatile organic compounds (i.e., benzene, toluene, acetone, carbon tetrachloride) absorb at this wavelength and are considered analytical interferences. They occur as contaminants in the reagents used during sample preparation. These compounds are not expected to be retained on an MCE filter during sample collection. Analytical interferences are rendered insignificant by using organic-free deionized water and at least reagent grade chemicals or by blank subtraction.

Increasing the concentration of nitric acid in the samples or standards appears to produce an elevated background signal. The nitric acid concentration in the samples and standards should not be greater than 10%.

5. Sampling - Particulate Mercury

For the sampling and analysis of mercury vapor, consult Reference 8.2.

- 5.1 Equipment Air Filters
 - 5.1.1 Filters: Mixed-cellulose ester (MCE) filters (0-8-µm pore size), cellulose backup pads, and cassettes, 37-mm diameter (part no. MAWP 037 AO, Millipore Corp., Bedford, MA).
 - 5.1.2 Gel bands (Omega Specialty Instrument Co., Chelmsford, MA) for sealing cassettes.
 - 5.1.3 Sampling pumps capable of sampling at 2 liters per minute (L/min).
 - 5.1.4 Assorted flexible tubing.
 - 5.1.5 Stopwatch and bubble tube or meter for pump calibration.
- 5.2 Equipment Wipe Samples

Smear tabs (part no. 225-24, SKC Inc., Eighty Four, PA), or wipe filters (Whatman no. 41 or no. 42 filters, Whatman LabSales Inc., Hillsboro, OR).

5.3 Equipment - Bulk Samples

Scintillation vials, 20-mL (part no. 74515 or 58515, Kimble, Div. of Owens-Illinois Inc., Toledo, OH) with polypropylene or Teflon cap liners. If possible, submit bulk or wipe samples in these vials. Tin or other metal cap liners should not be used since amalgamation can occur between the metal and mercury.

- 5.4 Sampling Procedure Air Samples
 - 5.4.1 Place a MCE filter and a cellulose backup pad in each two-or three-piece cassette. Seal each cassette with a gel band.
 - 5.4.2 Calibrate each personal sampling pump with a prepared cassette in-line to approximately 2 L/min.
 - 5.4.3 Attach prepared cassettes to calibrated sampling pumps (the backup pad should face the pump) and place in appropriate positions on the employee or workplace area.
 - 5.4.4 Collect the samples for at least 5 min.
 - 5.4.5 Place plastic end caps on each cassette after sampling. Attach an OSHA-21 seal around each cassette in such a way as to secure the end caps.

5.5 Sampling Procedure - Wipe Samples

A skin designation has been assigned to these mercury-containing compounds.

- 5.5.1 Wear clean, impervious, disposable gloves when taking each wipe sample.
- 5.5.2 Moisten the wipe filters with deionized water prior to use.
- 5.5.3 If possible, wipe a surface area covering 100 cm².
- 5.5.4 Fold the wipe sample with the exposed side in.
- 5.5.5 Transfer the wipe sample into a 20-mL scintillation vial and seal with vinyl or electrical tape. Securely wrap an OSHA-21 seal length-wise from vial top to bottom.
- 5.6 Sampling Procedure Bulk Samples

In order of laboratory preference, bulk samples may be one of the following:

- 1) a high-volume filter sample,
- 2) a representative settled dust (rafter) sample,
- 3) a sample of the bulk material in the workplace.
- 5.6.1 Transfer the bulk material into a 20-mL scintillation vial and seal with vinyl or electrical tape. Securely wrap an OSHA-21 seal length-wise from vial top to bottom.
- 5.6.2 The type of bulk sample should be stated on the OSHA 91 and cross-referenced to the appropriate air sample(s).
- 5.7 Shipment
 - 5.7.1 Submit at least one blank sample with each set of air or wipe samples. Blank filter samples should be handled in the same manner as other samples, except that an air or wipe sample is not taken.
 - 5.7.2 Send the samples to the laboratory as soon as possible with the OSHA 91A paperwork requesting particulate mercury analysis.
 - 5.7.3 Bulk samples should be shipped separately from air samples. They should be accompanied by Material Safety Data Sheets if available. Check current shipping restrictions and ship to the laboratory by the appropriate method.

6. Analysis

- 6.1 Safety Precautions
 - 6.1.1 Wear safety glasses, labcoat, and gloves at all times.
 - 6.1.2 Handle acid solutions with care. Avoid direct contact of acids with work area surfaces, eyes, skin, and clothes. Flush acid solutions which contact the skin or eyes with copious amounts of cold water.
 - 6.1.3 Prepare solutions containing hydrochloric acid in an exhaust hood and store in narrowmouthed bottles.
 - 6.1.4 Keep B.O.D. bottles containing stannous chloride/hydrochloric acid solutions capped when not in use to prevent inhalation of noxious vapors.
 - 6.1.5 Exercise care when using laboratory glassware. Do not use chipped pipets, volumetric flasks, beakers or any glassware with sharp edges exposed.
 - 6.1.6 Never pipet by mouth.
 - 6.1.7 Always purge the mercury from the CV-AAS into an exhaust vent.

- 6.1.8 Occasionally monitor the CV-AAS for mercury vapor leaks using an appropriate directreading instrument.
- 6.2 Equipment Cold Vapor Analysis
 - (Note: Specific equipment is listed for illustration only)
 - 6.2.1 Atomic absorption spectrophotometer (model 503, Perkin-Elmer, Norwalk, CT).
 - 6.2.2 Mercury hollow cathode lamp or electrodeless discharge lamp and power supply.
 - 6.2.3 Biological Oxygen Demand (B.O.D.) bottles, borosilicate glass, 300 mL.
 - 6.2.4 Peristaltic pump, 1.6 to 200 mL range, and controller, 1-100 rpm range (Masterflex model 7553-30 with model 7015 head, Cole-Parmer, Chicago, IL).
 - 6.2.5 Quartz absorption cell, 22 mm (7/8 in.) o.d. × 152 mm (6 in.) long (part no. 303-3101, Perkin-Elmer).
 - 6.2.6 Heating tape.
 - 6.2.7 Variable transformer 50-60 Hz, single phase, 10 A, 120 V input, 0-140 V output, 1.4 kW (Superior Electric, Bristol, CT).
 - 6.2.8 Tygon peristaltic pump tubing (part no. N06409-15, Cole-Parmer), and glass tubing.
 - 6.2.9 Aerator (part no. 0303-3102, Perkin-Elmer).
 - 6.2.10 Chart recorder.
 - 6.2.11 Desiccant (Drierite, W.A. Hammond Drierite Co., Xenia, OH).
 - 6.2.12 Volumetric flasks, volumetric pipets, beakers, and other laboratory glassware.
 - 6.2.13 Automatic pipets, adjustable, 0.1 to 5.0 mL range (models P-1000 and P-5000, Rainin Instruments Co., Woburn, MA).
 - 6.2.14 Exhaust vent.
 - 6.2.15 Automatic pipets, glass or Teflon, unlubricated (cat. no. 050-03-908-1, Brinkmann Dispensette, Brinkmann Instruments, Westbury, NY).
 - 6.2.16 Phillips beakers, 250 mL.
 - 6.2.17 Hot plate (used only for cleaning glassware, not for sample digestions).
 - 6.2.18 Analytical balance (0.01 mg).
- 6.3 Reagents All reagents should be at least reagent grade.

Potassium permanganate (KMnO₄) Hydroxylamine hydrochloride (NH₂OH•HC1) Stannous chloride (SnCl₂)

- 6.3.1 Deionized water (DI H₂O), organic-free.
- 6.3.2 Hydrochloric acid (HCI), concentrated (36.5 to 38%), with a mercury concentration less than 0.005 ppm.
- 6.3.3 Hydroxylamine hydrochloride (NH₂OH•HCl) solution, 20%: Dissolve 200 g NH₂OH•HCl in DI H₂O and dilute to 1 L. A glass automatic pipet is useful in dispensing this reagent. Rinse the automatic pipet dispenser with DI H₂O after the analysis is completed.
- 6.3.4 Mercury standard stock solution, 1,000 μg/mL: Use a commercially available certified standard or, alternatively, dissolve 1.0798 g of dry mercuric oxide (HgO) in 50 mL of 1:1

hydrochloric acid and then dilute to 1 L with DI H_2O . Store this reagent in a dark environment, preferably in an amber colored container.

- 6.3.5 Nitric acid (HNO₃) concentrated (69 to 71%), with a mercury concentration less than 0.005 ppm.
- 6.3.6 Nitric acid, 1:1: Carefully add equal portions of concentrated HNO₃ and DI H₂O.
- 6.3.7 Nitric acid, 10%: Carefully add 100 mL of concentrated HNO₃ to 900 mL of DI H₂O.
- 6.3.8 Potassium permanganate (KMnO₄) solution, 5%: Dissolve 50 g KMnO₄ in 1 L DI H₂O. This concentration is near saturation and the crystals will dissolve slowly. Stirring with a magnetic stirring bar/stirrer is recommended. An unlubricated glass automatic pipet is useful in dispensing the reagent during the analysis. After the analysis is completed, rinse and clean the automatic pipet dispenser to prevent seizing. Remove any dark deposits produced by permanganate reduction products with a 20% hydroxylamine hydrochloride solution.
- 6.3.9 Stannous chloride (SnCl₂) solution, 10%: Dissolve 20 g SnCl₂ in 100 mL concentrated HCl. Slowly and carefully pour this solution into 100 mL DI H₂O and then mix well. Transfer and store the final solution in a capped B.O.D. bottle to prevent oxidation. Prepare this solution before each new analysis.
- 6.3.10 Sulfuric acid (H_2SO_4), concentrated (95 to 98%), with a mercury concentration less than 0.005 ppm.
- 6.3.11 Sulfuric acid (H_2SO_4), 5 M: Cautiously add 278 mL concentrated H_2SO_4 to approximately 600 mL DI H_2O . Allow the solution to cool to room temperature and then dilute slowly to 1 L.
- 6.4 Glassware Preparation
 - 6.4.1 Clean the 250-mL Phillips beakers by refluxing with 1:1 HNO₃ on a hot plate in an exhaust hood. Thoroughly rinse with DI H_2O and allow to dry.
 - 6.4.2 Clean the B.O.D. bottles and stoppers with 1:1 HNO₃ and thoroughly rinse with DI H_2O prior to use.
 - 6.4.3 Rinse all other glassware with 10% HNO₃ and then with DI H₂O prior to use.
- 6.5 Standard Preparation
 - 6.5.1 Prepare a 1 μ g/mL mercury standard by making appropriate ten-fold serial dilutions of the 1,000 μ g/mL mercury standard stock solution with 10% HNO₃.
 - 6.5.2 Prepare working mercury standards (ranging from 0.1 to 2.0 μg) and reagent blanks immediately prior to use. A few standards at each concentration should be prepared. Add an appropriate aliquot of the 1 μg/mL standard to a clean B.O.D. bottle containing enough 10% HNO₃ to bring the total volume to 100 mL. A suggested dilution scheme is given:

Standard (µg)	Aliquot (mL)*	Final Volume (mL)
Reagent Blank	0	100
⁰ .1	0.1	100
0.2	0.2	100
0.5	0.5	100
1.0	1.0	100
1.5	1.5	100
2.0	2.0	100

* Aliquot taken from 1 µg/mL standard prepared in Section 6.5.1.

6.6 Sample Preparation

Sample digestion is performed at room temperature under oxidizing conditions to avoid loss of mercury.

- 6.6.1 Perform the transfer and digestion of samples in an exhaust hood. Transfer air and wipe samples, and previously weighed aliquots of bulk samples to separate labeled 250 mL Phillips beakers.
- 6.6.2 Add 5 mL conc. HNO₃ (per 100 mL final volume) to each Phillips beaker and allow to stand for a few min. Subsequently add 15 mL of 5 M H_2SO_4 (per 100 mL final volume) followed by the appropriate amount of 5% KMnO₄ to each sample to completely oxidize any additional organic material:

Bulk samples	40 mL of 5% KMnO₄ (per 100 mL final volume)
Large (> 4 cm diameter) wipe samples	40 mL of 5% KMnO $_{4}$ (per 100 mL final volume)
Air, small wipe, and smear tab samples	20 mL of 5% KMnO ₄ (per 50 mL final volume)

Swirl the samples frequently to break up the collection media and allow the samples to digest at least 1 h at room temperature.

6.6.3 Add the appropriate amount of 20% NH₂OH•HCl to each Phillips beaker:

Bulk samples	10 mL (per 100 mL final volume)
Large (> 4 cm diameter) wipe samples	10 mL (per 100 mL final volume)
Air, small wipe, and smear tab samples	5 mL (per 50 mL final volume)

The solution and suspended matter in each Phillips beaker should lose the dark-brown or purple color resulting from the $KMnO_4$ treatment and become clear. If it does not, add NH_2OH +HCI crystals directly to the beaker until clear.

- 6.6.4 Quantitatively transfer the sample solution from each Phillips beaker to an appropriate size volumetric flask and dilute to volume. This is a good place to stop if the analysis cannot be completed the same day.
- 6.7 Analysis Instrument Parameters
 - 6.7.1 Set up the CV-AAS as illustrated in Figure 1.
 - 6.7.2 Wrap the heating tape around the quartz cell and then turn on the variable transformer. The heat setting on the tape should be sufficient to prevent water vapor condensation in the absorption cell.
 - 6.7.3 Place the aerator in a B.O.D. bottle which contains approximately ½ to 1 inch of desiccant. Operate the peristaltic pump for approximately 30 min at full speed to remove any water vapor from the system.
 - 6.7.4 Operate the hollow cathode or electrodeless discharge mercury lamp at the manufacturer's recommended current or power rating.
 - 6.7.5 Use the following settings (Note: The mentioned instrument settings are for specific models used at the OSHA-SLTC. If instrumentation other than what is specified in Section 6.2 is used, please consult the instrument manufacturer's recommendations.):

Atomic Absorption Spectrophotometer: Slit 0.7 nm Signal Repeat Mode Function ABS Mode ABS UV Range 253.7 nm Wavelength Filter Out **EM Chopper** Off Phase Normal

Strip Chart Recorder:Chart Speed5 mm/minChart Range10 mV

6.7.6 Optimize the ENERGY meter reading at 253.7 nm.

- 6.7.7 Align the beam of the mercury lamp so it passes directly through the center of the quartz cell windows. This can be accomplished by adjusting the burner height, depth, and angle knobs to give a minimum ABSORBANCE reading.
- 6.7.8 Operate the peristaltic pump at full speed. Rinse the aerator with DI H_2O and insert it into a holder in the exhaust vent.
- 6.7.9 Perform the following steps to obtain a baseline signal near an absorbance of zero:
 - 1) start the chart recorder,
 - 2) set the spectrophotometer absorbance reading to zero,
 - 3) wait until the baseline stops drifting,
 - 4) set the reading to zero again.
- 6.8 Analytical Procedure
 - 6.8.1 Samples: <u>Immediately before analyzing</u>, transfer an appropriate aliquot (It is recommended to use 10 mL of each wipe and bulk and 25 to 50 mL of each air filter sample. Aliquot amounts from air filter sample solutions can be determined from the air volume taken. A general rule is to take an aliquot which will allow a detection limit of at least 0.1 times the PEL) of the sample solution to a clean B.O.D. bottle containing enough 10% HNO₃ solution to bring the total volume to 100 mL. The transfer must be done with a volumetric pipet.
 - 6.8.2 Standards: <u>Immediately before analyzing</u>, prepare standards according to instructions listed in Section 6.5.2.
 - 6.8.3 Deliver 5 mL of the 10% SnCl₂ solution with an automatic pipet to a B.O.D. bottle containing a standard, reagent blank, or sample to be analyzed. Immediately place the aerator into the solution with the peristaltic pump operating at full speed.
 - 6.8.4 Record the maximum absorbance reading and label the signal produced on the strip chart.
 - 6.8.5 Stop the pump, remove the B.O.D. bottle from the CV-AAS and stopper it. Rinse the aerator with DI H₂O and insert it into a holder in the exhaust vent. Turn the pump on at full speed until the CV-AAS system is purged of mercury and the baseline returns to zero.
 - 6.8.6 If the absorbance reading of a sample is greater than the highest standard <u>at any time</u> during analysis, <u>immediately</u> remove the B.O.D. bottle from the CV-AAS. Purge the system following the procedure listed in Section 6.8.5. Take a smaller aliquot or dilute the high concentration sample and re-analyze. Make any necessary sample dilutions with 10% HNO₃ and use the appropriate dilution factor when calculating results.
 - 6.8.7 Repeat Sections 6.8.3 through 6.8.5 for each prepared standard, reagent blank, or sample.
- 6.9 Analytical Recommendations
 - 6.9.1 It is recommended to analyze the reagent blank, lowest, and highest standard two or three times each to check for contamination, reproducibility, and sensitivity before starting the sample analysis. A 2.0-µg mercury standard should give a three-quarter to full-scale deflection on the chart recorder and an absorbance unit reading of about 0.850 when using the equipment and conditions specified. The lowest and highest standard should provide a linear response and the lowest standard should be at least two to three times the blank signal.
 - 6.9.2 It is also recommended to analyze an entire series of standards (including the reagent blank) at the beginning and end of the sample analysis to ensure standard readings are reproducible. As a general guideline, standard readings should be within ±10% throughout the analysis.
 - 6.9.3 A standard near the concentration range of the samples should be analyzed after every four to five samples.
 - 6.9.4 Quality control (QC) samples should be prepared and analyzed using the same matrix and analytical conditions as the samples. If possible, the QC samples should be generated from an independent source.

- 6.9.5 Approximately 10% of the samples should be reanalyzed.
- 7. Calculations
 - 7.1 Use a least squares regression program to plot a concentration-response curve of peak absorbance versus the amount (μ g) of mercury in each standard.
 - 7.2 Determine the amount (µg) of mercury, A, corresponding to the peak absorbance in each analyzed sample aliquot from this curve.
 - 7.3 Calculate the total amount (µg) of mercury, W, in each sample:

$$W = \frac{A \times \text{sample vol, } mL \times DF}{\text{aliquot, } mL}$$

Where:

DF = Dilution Factor (if none, DF = 1)

7.4 Calculate the total concentration of mercury in each sample using the appropriate equation:

Mercury mg/m³ =
$$\frac{W - Wb}{Air vol, L}$$
 (Air Samples)

Total Mercury = W – Wb (Wipe Samples)

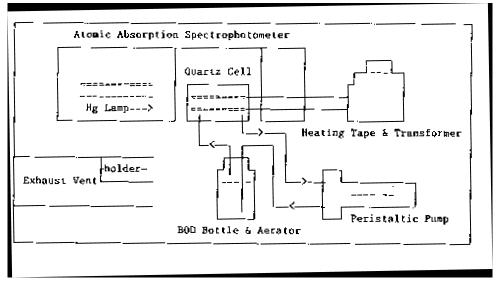
Mercury % (w / w) =
$$\frac{W \times 100\%}{\text{Sample wt, mg} \times 1000 \text{ mg/mg}}$$

Where:

Wb = total μ g of mercury in the blank sample. Sample wt = aliquot of bulk taken in Section 6.6.1

- 7.5 Reporting Results
 - 7.5.1 Air sample results are reported as mg/m^3 mercury.
 - 7.5.2 Wipe sample concentrations are reported as total micrograms or milligrams mercury.
 - 7.5.3 Bulk sample results are reported as approximate percent by weight mercury. Due to differences in sample matrices between bulks and standards, bulk results are approximate.
- 8. References
 - 8.1 Hatch, W.R. and W.L. Ott: Determination of Submicrogram Quantities of Mercury by Atomic Absorption Spectrophotometry. <u>Anal. Chem.</u>, 1968, <u>40</u>, 2085-87.
 - 8.2 Occupational Safety and Health Administration Technical Center: <u>Mercury Vapor in Workplace</u> <u>Atmospheres</u> (OSHA-SLTC Method No. ID-140). Salt Lake City, UT. Revised 1991.
 - 8.3 National Institute for Occupational Safety and Health: <u>Criteria for a Recommended Standard --</u> <u>Occupational Exposure to Inorganic Mercury</u> (DHEW/NIOSH Pub. No. HSM-73-11024). Cincinnati, OH: National Institute for Occupational Safety and Health, 1973.
 - 8.4 Windholz, M., ed.: The Merck Index. 10th ed. Rahway, NJ: Merck & Co. Inc., 1983.
 - 8.5 Sax, N.I. and R.J. Lewis Sr., ed.: <u>Hawley's Condensed Chemical Dictionary</u>, 11th ed.; New York: Van Nostrand Reinhold Co., 1987.
 - 8.6 Occupational Safety and Health Administration Analytical Laboratory: <u>Detection Limit Study for</u> <u>Mercury Cold Vapor Analysis</u> by C. Merrell. Salt Lake City, UT. 1987 (unpublished).

- 8.7 Occupational Safety and Health Administration Analytical Laboratory: <u>Quality Control Data -</u> <u>Mercury-Spiked Filter Cold Vapor Analysis</u> by B. Babcock. Salt Lake City, UT. 1987 (unpublished).
- 8.8 Occupational Safety and Health Administration Analytical Laboratory: <u>Phenyl Mercuric Acetate</u> (<u>PMA</u>) Procedure by S. Edwards. Salt Lake City, UT. 1981 (unpublished).



Cold Vapor-Atomic Absorption Spectrophotometer for Mercury Analysis

Figure 1