State of California Air Resources Board

Method 101

Determination of Particulate and Gaseous Mercury Emissions From Chlor-Alkali Plants – Air Streams

ADOPTED: March 28, 1986

1. Applicability and Principal

1.1 Applicability.

This method applies to the determination of particulate and gaseous mercury (Hg) emissions from chlor-alkali plants and other sources (as specified in the regulations), where the carrier-gas stream in the duct or stack is principally air.

1.2 Principle.

Particulate and gaseous Hg emissions are withdrawn isokinetically from the source and collected in acidic iodine monochloride ICI solution. The Hg collected (in the mercuric form) is reduced to elemental Hg, which is then aerated from the solution into an optical cell and measured by atomic absorption spectrophotometry.

2. Range and Sensitivity

2.1 Range.

After initial dilution, the range of this method is 0.5 to 120 ug Hg/ml. The upper limit can be extended by further dilution of the sample.

2.2 Sensitivity.

The sensitivity of this method depends on the recorder/spectrophotometer combination selected.

3. Interfering Agents

3.1 Sampling.

SO₂ reduces ICI and causes premature depletion of the ICI solution.

3.2 Analysis.

ICI concentrations greater than 10⁻⁴ molar inhibit the reduction of the Hg (II) ion in the aeration cell. Condensation of water vapor on the optical cell windows causes a positive interference.

4. Precision and Accuracy.

The following estimates are based on collaborative tests, wherein 13 laboratories performed duplicated analyses on two Hg-containing samples from a chlor-alkali

plant and on one laboratory prepared sample of known Hg concentration. The concentration ranged from 2 to 65 ug Hg/ml.

4.1 Precision.

The estimated within-laboratory and between-laboratory standard deviations are 1.6 and 1.8 ug Hg/ml, respectively.

4.2 Accuracy.

The participating laoboratories that analyzed a 64.3-ug Hg/ml (in 0.1 M ICl) standard obtained a mean of 63.7 Hg/ml.

5. Apparatus

5.1 Sampling Train.

A schematic of the sampling train is shown in Figure 101-1; it is similar to the Method 5 train (mention of Method 5 refers to Parts 60 of 40 CFR). The sampling train consists of the following components:

- 5.1.1 Probe Nozzle. Pitot Tube. Differential Pressure Gauge, Metering System, Barometer, and Gas Density Determination Equipment. Same as Method 5. Sections 2.1.1, 2.1.3, 2.1.4, 2.1.8, 2.1.9, and 2.1.10, respectively.
- 5.1.2 Probe Liner. Borosilicate or quartz glass tubing. The tester may use a heating system capable of maintaining a gas temperature of 120 _ 14°C (248 ± 25°F) at the probe exit during sampling to prevent water condensation.

Note. – Do not use metal probe liners.

- 5.1.3 Impingers. Four Greenburg-Smith impingers connected in series with leak-free ground glass fittings or any similar leak-free noncontaminating fittings. For the first, third, and fourth impingers, the tester may use impingers that are modified by replacing the tip with a 13-mm-ID (0.5-in.) glass tube extending to 13 mm (0.5 in.) from the bottom of the flask.
- 5.1.4 Acid Trap, Mine Safety Appliances Air Line Filter. Catalog number 81857, with acid absorbing cartridge and suitable connections, or equivalent.

5.2 Sample Recovery.

The following items are needed:

- 5.2.1 Glass Sample Bottles. Leakless, with Teflon-lined caps, 1000 and 100 ml.
- 5.2.2 Graduated Cylinder. 250 ml.
- 5.2.3 Funnel and Rubber Policeman. To aid in transfer of silica gel to container, not necessary if silica gel is weighed in the field.
- 5.2.4 Funnel Glass, to aid in sample recovery.
- 5.3 Sample Preparation and Analysis. The following equipment is needed:
 - 5.3.1 Atomic Absorption Spectrophotometer. Perkin-Elmer 303, or equivalent, containing a hollow-cathode mercury lamp and the optical cell described in Section 5.3.2.
 - 5.3.2 Optical Cell. Cylindrical shape with quartz end windows and having the dimensions shown in Figure 101-2. Wind the cell with approximately 2 meters of 24-gauge nichrome heating wire, and wrap with fiberglass insulation tape or equivalent. Do not let the wires touch each other.
 - 5.3.3 Aeration Cell. Constructed according to the specifications in Figure 101-3. Do not use a glass frit as a substitute for the blown glass bubbler tip shown in Figure 101-3.
 - 5.3.4 Recorder. Matched to output of the spectrophotometer described in Section 5.3.1.
 - 5.3.5 Variable Transformer. To vary the voltage on the optical cell from 0 to 40 volts.
 - 5.3.6 Hood. For venting optical cell exhaust.
 - 5.3.7 Flowmetering Valve.
 - 5.3.8 Flowmeter. Rotameter or equivalent, capable of measuring a gas flow of 1.5 liters/min.
 - 5.3.9 Aeration Gas Cylinder. Nitrogen or dry, Hg-free air, equipped with a single-stage regulator.

- 5.3.10 Connecting Tubing. Use glass tubing (ungreased ball and socket connections are recommended) for all tubing connections between the solution cell and the optical cell; do not use Tygon tubing, other types of flexible tubing, or metal tubing as substitutes. The tester may use Teflon, steel, or copper tubing between the nitrogen tank and flowmetering valve (5.3.7), and Tygon, gum, or rubber tubing between the flowmetering valve and the aeration cell.
- 5.3.11 Flow Rate Calibration Equipment. Bubble flowmeter or wet test meter for measuring a gas flow rate of 1.5 ± 0.1 liters/min.
- 5.3.12 Volumetric Flasks. Class A with penny head standard taper stoppers; 100-, 250-, 500-, and 1000-ml.
- 5.3.13 Volumetric Pipets. Class A; 1-, 2-, 3-, 4-, and 5-ml.
- 5.3.14 Graduated Cylinder. 50-ml.
- 5.3.15 Magnetic Stirrer. General-purpose laboratory type.
- 5.3.16 Magnetic Stirring Bar. Teflon-coated.
- 5.3.17 Balance. Capable of weighing to + 0.5 g.
- 5.4 Alternative Analytical Appratus.

Alternative systems are allowable as long as they meet the following criteria:

- 5.4.1 A linear calibration curve is generated and two consecutive samples of the same aliquot size and concentration agree within 3 percent of their average.
- 5.4.2 A minimum of 95 percent of the spike is recovered when an aliquot of a source sample is spiked with a known concentration of mercury (II) compound.
- 5.4.3 The reducing agent should be added after the aeration cell is closed.
- 5.4.4 The aeration bottle bubbler should not contain a frit.

- 5.4.5 Any Tygon used should be as short as possible and conditioned prior to use until blanks and standards yield linear and reproducible results.
- 5.4.6 If manual stirring is done before aeration, it should be done with the aeration cell closed.
- 5.4.7 A drying tube should not be used unless it is conditioned as the Tygon above.

6. Reagents.

Use ACS reagent-grade chemicals or equivalent, unless otherwise specified.

- 6.1 Sampling and Recovery. The reagents used in sampling and recovery are as follows:
 - 6.1.1 Water: Deionized distilled, meeting ASTM Specifications for Type I Reagent Water-ASTM Test Method D1193-77 (incorporated by reference-see § 61.18). If high concentrations of organic matter are not expected to be present, the analyst may eliminate the KMnO₄ test for oxidizable organic matter. Use this water in all dilutions and solution preparations.
 - 6.1.2 Nitric Acid (HNO₃), 50 percent (V/V). Mix equal volumes of concentrated HNO₃ and deionized distilled water, being careful to slowly add the acid to the water.
 - 6.1.3 Silica Gel. Indicating type, 6- to 16- mesh. If previously used, dry at 175°C (350°F) for 2 hours. The tester may use new silica gel as received.
 - 6.1.4 Potassium Iodide (KI, Solution, 25 Percent. Dissolve 250 g of KI in deionized distilled water and dilute to 1 liter.
 - 6.1.5 Iodine Maoochloride (ICI) Stock Solution 1.0 M. To 800 ml of 25 percent KI solution, add 800 ml of concentrated hydrochloric acid (HCI). Cool to room temperature. With vigorous stirring, slowly add 135 g of potassium iodate (KIO₃) and stir until all free iodine has dissolved. A clear orange-red solution occurs when all the KIO₃ has been added. Cool to room temperature and dilute to 1800 ml with deionized distilled water. Keep the solution in amber glass bottles to prevent degradation.

- 6.1.6 Absorbing Solution, 0.1 M ICI. Dilute 100 ml of the 1.0 M ICI stock solution to 1 liter with deionized distilled water. Keep the solution in amber glass bottles and in darkness to prevent degradation. This reagent is stable for at least two months.
- 6.2 Sample Preparation and Analysis.

The reagents needed are listed below:

- 6.2.1 Tin (II) Solution. Prepare fresh daily and keep sealed when not being used. Completely dissolve 20 g of tin (II) chloride (or 25 g of tin (II) sulfate) crystals (Baker Analyzed reagent grade or any other brand that will give a clear solution) in 25 ml of concentrated HCl. Dilute to 250 ml with deionized distilled water. Do not substitute HNO₃, H₂SO or other strong acids for the HCl.
- Mercury Stock Solution, 1 mg Hg/ml. Prepare and store all mercury standard solutions in borosilicate glass containers. Completely dissolve 0.1354 g of mercury (II) chloride in 75 ml of deionized distilled water in a 100 ml glass volumetric flask. Add 10 ml of concentrated HNO₂ and adjust the volume to exactly 100 ml with deionized distilled water. Mix thoroughly. This solution is stable for at least 1 month.
- 6.2.3 Sulfuric Acid. 5 Percent (V/V). Dilute 25 ml of concentrated H₂SO₄ to 500 ml with deionized distilled water.
- 6.2.4 Intermediate Mercury Standard Solution, 10 g Hg/ml. Prepare fresh weekly. Pipet 5.0 ml of the mercury stock solution (6.2.2) into a 500-ml glass volumetric flask and add 20 ml of the 5 percent H₂SO₄ solution. Dilute to exactly 500-ml with deionized distilled water. Thoroughly mix the solution.
- 6.2.5 Working Mercury Standard Solution, 200 ng Hg/ml. Prepare fresh daily. Pipet 5.0 ml from the "Intermediate Mercury Standard Solution" (6.2.4) into a 250-ml volumetric glass flask. Add 10 ml of the 5 percent H₂SO₄ and 2 ml of the 0.1 M ICl absorbing solution taken as a blank (7.2.3) and dilute to 250 ml with deionized distilled water. Mix thoroughly.

7. Procedure

7.1 Sampling.

Because of the complexity of this method, testers should be trained and experienced with the test procedures to assure reliable results. Since the amount of Hg that is collected generally is small, the method must be carefully applied to prevent contamination or loss of sample.

- 7.1.1 Pretest Preparation. Follow the general procedure given in Method 5, Section 4.1.1, except omit the directions on the filter.
- 7.1.2 Preliminary Determinations. Follow the general procedure given in Method 5, Section 4.1.2, except as follows: Select a nozzle size based on the range of velocity heads to assure that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates below 28 liters/min (1.0 cfm). Obtain samples over a period or periods that accurately determine the maximum emissions that occur in a 24-hour period. In the case of cyclic operations, run sufficient tests for the accurate determination of the emissions that occur over the duration of the cycle. A minimum sample time of 2 hours is recommended. In some instances, high Hg or high SO₂ concentrations make it impossible to sample for the desired minimum time. This is indicated by reddening (liberation of free iodine) in the first impinger. In these cases, the tester may divide the sample run into two or more subruns to insure that the absorbing solution is not depleted.
- 7.1.3 Preparation of Sampling Train. Clean all glassware (probe, impingers, and connectors) by rinsing with 50 percent HNO₂, tap water. 0.1 M ICI, tap water, and finally deionized distilled water. Place 100 ml of 0.1 M ICI in each of the first three impingers. Take care to prevent the absorbing solution from contacting any greased surfaces. Place approximately 200 g of preweighed silica gel in the fourth impinger. The tester may use more silica gel, but should be careful to ensure that it is not entrained and carried out from the impinger during sampling. Place the silica gel container in a clean place for later use in the sample recovery. Alternatively, determine and record the weight of the silica gel plus impinger to the nearest 0.5 g.

Install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260°C (500°F). Use a fiberglass string gasket if temperatues are higher. See APTD-0576 (Citation 9 in Section 10) for details. Other connecting

systems using either 316 stainless steel or Teflon ferrules may be used. Mark the probe with heat-resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point. Assemble the train as shown in Figure 101-1, using (if necessary) a very light coat of silicone grease on all ground glass joints. Grease only the outer portion (see APTD-0576) to avoid possibility of contamination by the silicone grease.

Note – An empty impinger may be inserted between the third impinger and the silica gel to remove excess moisture from the sample stream.

After the sampling train has been assembled, turn on and set probe, if applicable, at the desired operating temperature. Allow time for the temperatures to stabilize. Place crushed ice around the impingers.

- 7.1.4 Leak-Check Procedures. Follow the leak-check procedures outlined in Method 5, Sections 4.1.4.1 (Pretest Leak Check), 4.1.4.2 (Leak Checks During Sample Run), and 4.1.4.3 (Post-Test Leak Check).
- 7.1.5 Mercury Train Operation. Follow the general procedure given in Method 5, Section 4.1.5. For each run, record the data required on a data sheet such as the one shown in Figure 101-4.
- 7.1.6 Calculation of Percent Isokinetic. Same as Method 5, Section 4.1.6.

7.2 Sample Recovery.

Begin proper cleanup procedure as soon as the probe is removed from the stack at the end of the sampling period.

Allow the probe to cool. When it can be safely handled, wipe off any external particulate matter near the tip of the probe nozzle and place a cap over it. Do not cap off the probe tip tightly while the sampling train is cooling. Capping would create a vacuum and draw liquid out from the impingers.

Before moving the sampling train to the cleanup site, remove the probe from the train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe

off the silicone grease from the impinger. Use either ground-glass stoppers, plastic caps, or serum caps to close these openings.

Transfer the probe and impinger assembly to a cleanup area that is clean, protected from the wind, and free of Hg contamination. The ambient air in laboratories located in the immediate vicinity of Hg-using facilities is not normally free of Hg contamination.

Inspect the train before and during assembly, and note any abnormal conditions. Treat the sample as follows:

7.2.1 Container No. 1 (Impinger and Probe). Using a graduated cylinder, measure the liquid in the first three impingers to within ±1 ml. Record the volume of liquid present (e.g., see Figure 5-3 of Method 5). This information is needed to calculate the moisture content of the effluent gas. (Use only glass storage bottles and graduated cylinders that have been precleaned as in Section 7.1.3). Place the contents of the first three impingers into a 1000-ml glass sample bottle.

Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover the Hg (and any condensate) from the probe nozzle, probe fitting, and probe liner as follows: Rinse these components with two 50-ml portions of 0.1 M ICI. Next, rinse the probe nozzle, fitting and liner, and each piece of connecting glassware between the probe liner and the back half of the third impinger with a maximum of 400 ml of deionized distilled water. Add all washings to the 1000-ml glass sample bottle containing the liquid from the first three impingers.

After all washings have been collected in the sample container, tighten the lid on the container to prevent leakage during shipment to the laboratory. Mark the height of the liquid to determine later wheather leakage occurred during transport. Label the container to clearly identify its contents.

7.2.2 Container No. 2 (Silica Gel). Note the color of the indicating silica gel to determine whether it has been completely spent and make a notation of its condition. Transfer the silica gel from its impinger to its original container and seal. The tester may use as aids a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger. The small amount of particles that may adhere to the impinger wall need not be removed. Since the gain in weight

is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g. Record this weight.

7.2.3 Container No. 3 (Absorbing Solution Blank). For a blank, place 50 ml of the 0.1 M ICl absorbing solution in a 100-ml sample bottle. Seal the container. Use this blank to prepare the working mercury standard solution (6.2.5).

7.3 Sample Preparation.

Check the liquid level in each container to see whether liquid was lost during transport. If a noticeable amount of leakage occurred, either void the sample or use methods subject to the approval of the Control Agency's Authorized Representative to account for the losses. Then follow the procedures below:

- 7.3.1 Container No. 1 (Impinger and Probe). Carefully transfer the contents of Container No. 1 into a 1000-ml volumetric flask and adjust the volume to exactly 1000 ml with deionized distilled water.
- 7.3.2 Dilutions. Pipet a 2 ml aliquot from the diluted sample from 7.3.1 into a 250-ml volumetric flask. Add 10 ml of 5 percent H_2SO_4 and adjust the volume to exactly 250 ml with deionized distilled water. These solutions are stable for at least 72 hours.

Note – The dilution factor will be 250/2 for this solution.

7.4 Analysis.

Calibrate the spectrophotometer and recorder and prepare the calibration curve as described in Sectons 8.1 to 8.4.

7.4.1 Mercury Samples. Repeat the procedure used to establish the calibration curve with appropriately sized aliquots (1 to 5 ml) of each of the diluted samples (from Section 7.3.2) until two consecutive peak heights agree within ±3 percent of their average value. The peak maximum of an aliquot (except the 5-ml aliquot) must be greater than 10 percent of the recorder full scale. If the peak maximum of a 1.0-ml aliquot is off scale on the recorder, further dilute the original source sample to bring the Hg concentration into the calibration range of the spectrophotometer.

Run a blank and standard at least after every five samples to check the spectrophotometer calibration; recalibrate as necessary.

It is also recommended that at least one sample from each stack test be checked by the method of standard additions to confirm that matrix effects have not interfered in the analysis.

7.4.2 Container No. 2 (Silica Gel). Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. (This step may be conducted in the field.)

8. Calibration and Standards

Before use, clean all glassware, both new and used, as follows; brush with soap and water, liberally rinse with tap water, soak for 1 hour in 50 percent HNO₃ and then rinse with deionized distilled water.

8.1 Flow Calibration.

Assemble the aeration system as shown in Figure 101-5. Set the outlet pressure on the aeration gas cylinder regulator to a minimum pressure of 500 mm Hg (10 psi), and use the flowmetering valve and a bubble flowmeter or wet test meter to obtain a flow rate of 1.5 ± 0.1 liters/min through the aeration cell. After the flow calibration is complete, remove the bubble flowmeter from the system.

8.2 Optical Cell Heating System Calibration.

Using a 50-ml graduated cylinder, add 50 ml of deionized distilled water to the bottle section of the aeration cell and attach the bottle section to the bubbler section of the cell. Attach the aeration cell to the optical cell; and while aeratring at 1.5 liters/min determine the minimum variable transformer setting necessary to prevent condensation of moisture in the optical cell and in the connecting tubing. (This setting should not exceed 20 volts.)

8.3 Spectrophotometer and Recorder Calibration.

The mercury response may be measured by either peak height or peak area.

Note. – The temperature of the solution affects the rate at which elemental Hg is released from a solution and, consequently, it affects the shape of the absorption curve (area) and the point of maximum absorbance (peak

height). Therefore, to obtain reproducible results, bring all solutions to room temperature before use.

Set the spectrophotometer wavelength at 253.7 nm. and make certain the optical cell is at the minimum temperature that will prevent water condensation. Then set the recorder scale as follows: Using a 50-ml graduated cylinder, add 50 ml of deionized distilled water to the aeration cell bottle and pipet 5.0 ml of the working mercury standard solution into the aeration cell.

Note. – Always add the Hg-containing solution to the aeration cell after the 50 ml of deionized distilled water.

Place a Teflon-coated stirring bar in the bottle. Before attaching the bottle section to the bubbler section of the aeration cell, make certain that (1) the aeration cell exit arm stopcock (Figure 101-3) is closed (so that Hg will not prematurely enter the optical cell when the reducing agent is being added) and (2) there is no flow through the bubbler. If conditions (1) and (2) are met, attach the bottle section to the bubbler section of the aeration cell through the side arm of the cell and immediately stopper the side arm. Stir the solution for 15 seconds. Turn on the recorder, open the aeration cell exit arm stopcock, and then immediately initiate aeration with continued stirring. Determine the maximum absorbance of the standard and set this value to read 90 percent of the recorder full scale.

8.4 Calibration Curve.

After setting the recorder scale, repeat the procedure in Section 8.3 using 0.0-, 1.0-, 2.0-, 3.0-, 4.0-, and 5.0- ml aliquots of the working standard solution (final amount of Hg in the aeration cell is 0, 200, 400, 600, 800. and 1000 ng, respectively). Repeat this procedure on each aliquot size until two consecutive peaks agree within 3 percent of their average value. (Note: to prevent Hg carryover from one sample to another, do not close the aeration gas tank valve and do not disconnect the aeration cell from the optical cell until the recorder pen has returned to the baseline.) It should not be necessary to disconnect the aeration gas inlet line from the aeration cell when changing samples. After separating the bottle and bubbler sections of the aeration cell, place the bubbler section into a 600ml beaker containing approximately 400 ml of deionized distilled water. Rinse the bottle section of the aeration cell with a stream of deionized distilled water to remove all traces of the tin (II) reducing agent. Also, to prevent the loss of Hg before aeration, remove all traces of the reducing agent between samples by washing with deionized distilled water. It will be necessary, however, to wash the aeration cell parts with concentated HCl if any of the following conditions occur. (1) A white film appears on

any inside surface of the aeration cell, (2) the calibration curve changes suddenly, or (3) the replicate samples do not yield reproducible results.

Subtract the average peak height (or peak area) of the blank (0.0 ml aliquot)-which should be less than 2 percent of recorder full scale-from the averaged peak heights of the 1.0-, 2.0-, 3.0-, 4.0-, and 5.0-ml aliquot standards. If the blank absorbance is greater than 2 percent of full-scale, the probable cause is Hg contamination of a reagent or carry-over of Hg from a previous sample. Plot the corrected peak height of each standard solution versus the corresponding final total Hg weight in the aeration cell (in ng) and draw the best-fit straight line. This line should either pass through the origin or pass through a point no further from the origin than ±2 percent of the recorder full scale. If the line does not pass through or very near to the origin, check for nonlinearity of the curve and for incorrectly prepared standards.

8.5 Sampling Train Calibration.

Calibrate the sampling train components according to the procedures outlined in the following sections of Method 5: Section 5.1 (Probe Nozzle). Section 5.2 (Pitot Tube). Section 5.3 (Metering System). Section 5.4 (Probe Heater). Section 5.5 (Temperature Gauges). Section 5.7 (Barometer). Note that the leak-check described in Section 5.6 of Method 5 applies to this method.

Calculations

9.1 Dry Gas Volume.

Using the data from this test, calculate $V_{m(std)}$ the dry gas sample volume at standard conditions (corrected for leakage, if necessary) as outlined in Section 6.3 of Method 5.

9.2 Volume of Water Vapor and Moisture Content.

Using the data obtained from this test, calculate the volume of water vapor $V_{w(std)}$ and the moisture content B_{ws} of the stack gas. Use Equations 5-2 and 5-3 of Method 5.

9.3 Stack Gas Velocity.

Using the data from this test and Equation 2-9 of Method 2, calculate the average stack gas velocity $V_{\rm s}$.

9.4 Total Mercury.

For each source sample, correct the average maximum absorbance of the two consecutive samples whose peak heights agree within ±3 percent of their average for the contribution of the solution blank (see Section 8.4). Use the calibration curve and these corrected averages, to determine the final total weight of mercury in nanograms in the aeration cell for each source sample. Correct for any dilutions made to bring the sample in the working range of the spectrophotometer. Then calculate the Hg in ug (m Hg) in the original solution as follows:

$$m_{Hg} = C_{Hg}(AC)(DF)V_{f}10^{-3}$$
 Eq. 101-1

Where:

 $C_{Hg(AC)}$ = Total nanograms of mercury in aliquot analyzed (reagent blank subtracted).

D.F. = Dilution factor for the Hg-containing solution (before adding to the aeration cell; e.g., DF=250/2 if the source samples were diluted as described in Section 7.3.2).

V_f = Solution volume of original sample, 1000 ml for samples diluted as described in Section 7.2.1.

 10^{-3} = Conversion factor, ug/ng.

S = Aliquot volume added to aeration cell, ml.

9.5 Mercury Emission Rate.

Calculate the Hg emission rate R in g/day for continuous operations using Equation 101-2. For cyclic operations, use only the time per day each stack is in operation. The total Hg emission rate from a source will be the summation of results from all stacks.

$$R = K_{\underline{M_{Hg}}V_{\underline{s}}\underline{A_{\underline{s}}}} [86,400 \times 10^{-6}]$$
 Eq. 101-2
$$[V_{m(std)} \ V_{w(std)}] \ (T_{\underline{s}}/P_{\underline{s}})$$

Where:

 A_f = Stack cross-sectional area, m2 (ft²).

86,400= Conversion factor, sec/day.

 10^{-6} = Conversion factor, g/ug.

T_s = Absolute average stack gas temperature, °K (°R).

P_s = Absolute stack gas pressure, mm Hg (in. Hg).

K = 0.3858 °K/mm Hg for metric units.

= 17.85 °R/in. Hg for English units.

 $V_{m(std)} = See 9.1$

 $V_{w(std)} = See 9.2$

9.6 Isokinetic Variation and Acceptable Results.

Same as Method 5. Section 6.11 and 6.12 respectively.

9.7 Determination of Compliance.

Each performance test consists of three repetitions of the applicable test method. For the purpose of determining compliance with an applicable national emission standard, use the average of the results of all repetitions.

10. Bibliography

- Addendum to Specifications for Incinerator Testing at Federal Facilities. PHS, NCAPC. December 6, 1967.
- 2. Determining Dust Concentration in a Gas Stream. ASME Performance Test Code No. 27, New York, NY. 1957.
- 3. Devorkin, Howard, et al, Air Pollution Source Testing Manual. Air Pollution Control District, Los Angeles, CA. November 1963.
- 4. Hatch, W.R., and W.L. Ott. Determination of Sub-Microgram Quantities of Mercury by Atomic Absorption Spectrophotometry. Anal. Chem. 40:2085-87. 1968.
- 5. Mark, L. S. Mechanical Engineers' Handbook. McGraw-Hill Book Co., Inc. New York, NY. 1951.
- 6. Martin, Robert M. Construction Details of Isokinetic Source Sampling Equipment. U.S. Environmental Protection Agency. Research Triangle Park, NC. Publication No. APTD-0581, April 1971.

- 7. Western Precipitation Division of Joy Manufacturing Co. Methods for Determination of Velocity, Volume, Dust and Mist content of Gases. Bulletin WP-50. Los Angele4s, CA. 1966.
- 8. Perry J. H. Chemical Engineers' Handbook. McGraw-Hill Book Co., Inc. New York, NY. 1960.
- Rom, Jerome J. Maintenenace, Calibraton. and Operation of Isokinetic Source Sampling Equipment. U.S. Environmental Protection Agency. Research Triangle Park, NC. Publication No. APTD-0576. April 1972.
- 10. Shigehara, R. T., W. F. Todd, and W.S. Smith. Significance of Errors in Stack Sampling Measurements. Stack Sampling News. 1:(3):6-18, September 1973.
- 11. Smith, W.S., et al. Stack Gas Sampling Improved and Simplified with New Equipment. APCA Paper No. 67-119. 1967.
- 12. Smith, W.S., R.T. Shigehara, and W.F. Todd. A Method of Interpreting Stack Sampling Data. Stack Sampling News. 1(2):8-17. August 1973.
- 13. Specifications for Incinerator Testing at Federal Facilities, PHS, NCAPA. 1967.
- Standard Method for Sampling Stacks for Particulate Matter. In: 1971
 Annual Book of ASTM Standards, Part 23. ASTM Designation D-2928-71.
 Philadelphia, Pa. 1971.
- 15. Vennard, J.K. Elementary Fluid Mechanics, John Wiley and Sons, Inc. New York, 1947.
- Mitchell, W.J., and M. R. Midgett. Improved Procedure for Determining Mercury Emissions from Mercury Cell Chlor-Alkali Plants. J. APCA ______, July 1976.
- 17. Shigehara, R.T. Adjustments in the EPA Nomograph for Different Pitot Tube Coefficients and Dry Molecular Weights. Stack Sampling News. 2.4-11. October 1974.
- 18. Vollaro, R.F. Recommended Procedure for Sample traverses in Ducts Smaller than 12 inches in Diameter. U.S. Environmental Protection Agency, Emission Measurement Branch. Research Triangle Park, NC. November 1976.
- 19. Klein, R., and C. Hach Standard Additions: Uses and Limitation in Spectrophotometric Measurements. Amer. Lab. 9:21. 1977.



March 1986 CARB Method 101 Page 17

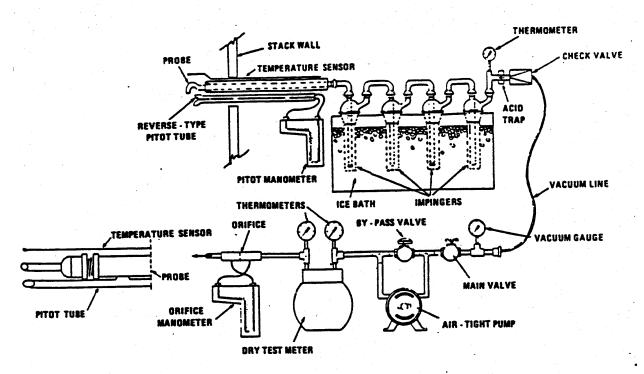


Figure 101-1 Mercury sampling train.

