VII. COMPATIBILITY WITH EMISSION STANDARDS

A national emission standard for mercury has been published by the Environmental Protection Agency (38 FR 8820). This standard is based upon specific operations and physical conditions, and is limited to emissions into the atmosphere. The standard specifies that emissions from stationary sources which process mercury ore to recover mercury and facilities which use mercury chlor-alkali cells to produce chlorine gas and alkali metal hydroxide shall not exceed 2,300 grams of mercury during a 24-hour period as measured in accordance with techniques set forth in the standard. This amount would limit the air concentration in the vicinity of emission sites to a daily level, averaged over 30 days, of 1 μg Hg/cu m. [10]

The standard is based upon information derived from many sources, including health effect levels, meteorology, technical analysis of control capability, and consideration of economic impact. The overriding considerations in developing the standard were health effects and the Environmental Protection Agency adopted the approach that mercury vapor and the more toxic methyl mercury are equal and additive.

A concentration in the air at or below 1 μg Hg/cu m is believed sufficient to protect the health of the public from illness due to inhalation of mercury with an ample margin of safety. [10]

There is no direct comparison possible between the proposed national emission standard for mercury and the recommended criteria for occupational exposure that the levels of exposure to the general public of varying health status and age on a 24-hour day, 7-day week, basis should be substantially lower than occupational standards based on an 8-hour day, 40-hour work week. However, the amount of mercury which an individual absorbs from the general atmosphere will be superimposed on that which he would receive from his occupational exposure. This additional amount is not expected to adversely affect workers when occupational levels are not above the 0.05 mg/cu m recommended in this document.

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IX. APPENDIX I

METHOD FOR SAMPLING OF MERCURY IN AIR

Elemental mercury vapor and mercury compounds are collected in an impinger and fritted bubbler in series, each containing acidic permanganate solution.

Equipment for Air Sampling

1. Stopwatch.
2. Constant rate vacuum pump with built-in rotameter.
3. Filtration adapter ₹ 24/40 joint. Two required.
4. 50-ml test tube and stopper ₹ 24/40 joint.
5. 150-ml ₹ 24/40 pear-shaped flasks. Two flasks are required for each air sample.
6. Solid borosilicate glass reagent bottle stopper, ₹24. One required for each 150-ml flask.
7. Fritted-glass bubbler tube, extra-coarse porosity.
8. Nonfritted bubbler tube.
9. Two No. 3 rubber stoppers bored to hold the tubes.

A complete set of glassware should be reserved solely for this sampling procedure and stored in a clean place when not in use. Only borosilicate glassware should be used. Clean all glassware initially by washing with brushes and a metal-free nonionic detergent, rinsing thoroughly with tap water until visibly clean. Then wash the complete inner surfaces with concentrated nitric acid. Rinse three to four

times with tap water, and then with deionized or distilled water. Once cleaned in this manner with concentrated nitric acid, glassware need only be rinsed three to four times with deionized water immediately after use and washed with 4 N nitric acid immediately before each subsequent use.

Reagents

All reagents should be prepared from reagent-grade materials.

1. 0.5 N potassium permanganate

Dissolve 7.90 g potassium permanganate crystals in water and dilute to 500 ml in a volumetric flask. Mix thoroughly and store in the volumetric flask protected from light. Discard when a precipitate of brown manganese dioxide develops.

2. 2.0 N sulfuric acid

Slowly add 56.2 ml of concentrated sulfuric acid to approximately 800 ml water in a 1-liter volumetric flask and mix. Because heat is evolved, sulfuric acid should be added to water with caution. Cool to 20 C, dilute to 1 liter, mix thoroughly, and store in the flask.

Sample Collection

The gas scrubbing devices recommended are shown in Figure XII-6. Similar devices may be used if they can be shown to have equivalent collection efficiencies for elemental mercury vapor, mercury compounds, and mercury-laden dust.

The absorbing solutions, 25 ml of 0.5 N potassium permanganate and 25 ml of 2.0 N sulfuric acid, are added to the pear-shaped flasks and stoppered with the glass stoppers. The time between addition of absorbing solution and the completion of sampling should not exceed 4 hours at room temperature. At higher ambient temperature, the bubbling solutions should be cooled by appropriate means. In the field, transfer the adapters and bubbling tubes to the flasks and stopper the test tube. Attach the bubblers to the sampling pump with tubing, forming a series arrangement with the fritted bubbler downstream from the nonfritted bubbler. Sample air at 2 liters per minute until 60 liters of air have been scrubbed. Measure the sampling time precisely. Remove the bubbler tubes and rinse the inside and outside of the tubes into the sample flasks with water from a polyethylene wash bottle. The same bubbler tubes are used for additional air samples.

The sampling pump must be checked for proper calibration prior to use.

The sampling routine will provide a 30-minute sample. Samples must be taken in a manner to allow the determination of a time-weighted average exposure in the workers breathing zone.

Samples should be returned to the laboratories for analysis as soon as possible.

X. APPENDIX II

METHOD FOR ANALYSIS OF MERCURY IN AIR

Equipment

1. Photometric analyzer.
2. Rapid response strip-chart recorder.
3. Automatic digital disc integrator, or planimeter.
4. Voltage regulator.
5. Rotameter.
6. Filtration adapter ℧ 24/40 joint.
7. 150-ml ℧ 24/40 pear-shaped flasks. One flask is required for each standard.
8. 3-way glass stopcock.
9. Fritted-glass bubbler tube, coarse porosity. One No. 3 rubber stopper bored to hold the tube.
10. Tygon, rubber, and borosilicate glass tubing.
11. Glass wool.
12. All-glass midget impinger.
13. Mercury vapor chemical cartridges.
14. Automatic dispensing bottle.
15. Pipettes, wash bottles, graduated cylinders, reagent bottles, glass-stoppered volumetric flasks, clamps, supports, rings, drying tubes, and other equipment and glassware as may be necessary.

Glassware is cleaned prior to use in a manner identical to that in Appendix I. Open vessels of reagents and sample solutions must be covered to protect from contamination by dust.

Reagents

All reagents should be prepared from reagent-grade materials.

1. Concentrated nitric acid (16 N)

2. Anhydrous magnesium perchlorate

3. Tin(II) chloride solution

Dissolve 250 g tin(II) chloride dihydrate in 500 ml deionized water. Carefully add with stirring 500 ml concentrated hydrochloric acid. Transfer to a dispensing bottle, add a few pieces of mossy tin and refrigerate. This solution is stable for about 2 months.

4. 0.5 N potassium permanganate

Prepare as in Appendix I

5. 2.0 N sulfuric acid

Prepare as in Appendix I

6. Stock mercury solutions

A. Weigh 2 to 3 grams (about 0.15 ml) oxide-free reagent-grade mercury to the nearest 0.1 mg into a clean, dry, tared 10-ml beaker. Immediately transfer to a 1-liter volumetric flask containing 100 ml concentrated nitric acid. Wash the beaker with 4 or 5 five-ml rinses of concentrated nitric acid, adding the rinsings to the flask. Add 100 ml concentrated nitric acid and about 500 ml water. Swirl and allow to come to room temperature. Dilute to 1-liter, mix thoroughly, and transfer to a clean, dry, glass bottle. Seal tightly. This solution is stable for at least one year.

B. Transfer 25.00 ml of solution A to a 1-liter volumetric flask containing 500 ml water and 50 ml concentrated nitric

acid. Dilute to the mark and mix thoroughly. This solution is stable for at least two months.

7. Working Standard Solution

Prepare a dilution of 0.100 µg Hg/ml by transferring an appropriate aliquot of solution B to a 1-liter volumetric flask containing 25 ml concentrated nitric acid and about 300 ml water. Dilute to 1 liter and mix thoroughly. This solution must be made fresh daily.

Preparation of Standard Curve

1. Turn on the mercury-vapor detection instrument and allow to warm up for 30 minutes. Adjust the flow rate of the compressed air line to 2 liter/min and constantly purge the gas cell with mercury-free air.

2. Adjust the zero and full-scale span of the instrument.

3. To six 150-ml pear-shaped flasks add 25 ml of 0.5 N potassium permanganate with a 25-ml graduated cylinder.

4. Add 25 ml of 2.0 N sulfuric acid to each flask with a 25-ml graduated cylinder.

5. With a pipette, add 0.00, 1.00, 2.00, 3.00, 5.00, 7.00 ml of the 0.100 µg Hg/ml working standard. Swirl each flask.

6. Turn on the recorder and recheck the instrument zero and full-scale span. Integrate with the recorder. A stable, noise-free base line is necessary.

7. Add 10 ml tin(II) chloride solution to the first flask (control blank) from the dispensing bottle. Swirl and immediately insert into the analysis train. The solution should be colorless; if

it is not, prepare a new control blank by adding more tin(II) chloride solution.

8. Rotate the stopcock of the three-way valve to flush air through the flask and into the vapor detector. After 2-3 minutes, or when the recorder pen returns to the base line, remove the flask from the bubbler tube and rotate the stopcock so the gas cell is constantly being purged with air.

9. Repeat steps 7 and 8 for the rest of the mercury-spiked standards. The pen response to mercury vapors occurs in a few seconds, and the mercury is usually flushed in 2-3 minutes, indicated by return of the pen to the base line.

A complete set of standards must be run along with every set of air-sample scrubber solutions.

Analysis of Samples

It is recommended that samples be analyzed the same day they are collected. The analytical equipment arrangements used are shown in Figure XII-7.

1. Combine the two air-scrubber solutions from a single sampling in a 200-ml volumetric flask with washings. Dilute to the mark and mix thoroughly. If there is precipitate adhering to the flasks, it may be necessary to reduce the permanganate with a few milliliters of 10% hydrogen peroxide before transferring the solutions to the volumetric flask.

2. Transfer duplicate aliquots to pear-shaped flasks. If necessary, dilute to about 50 ml with water.

3. Follow steps 7, 8, and 9 in the standardization procedure. Larger aliquots may be necessary in some cases where the concentration of mercury in air is very low. Generally, the greater the area under the recorded curve, the higher the accuracy and precision.

Calculations

Determine the area under the curve representing the mercury-spiked standards and the air samples with a planimeter. Repeat and average the results. Plot the mean area against µg Hg per standard on normal graph paper connecting the points with a straight line. Determine the amount of mercury in each air sample solution aliquot from the standard curve.

$$\text{mg Hg/cubic meter of air} = \frac{\frac{200\ A}{B} - C}{DE}$$

Where A = Average µg Hg found by analysis of aliquots of air sample scrubber solution

Where B = Volume of aliquot in milliliters

Where C = µg Hg in the control blank

Where D = Time of sampling period in minutes

Where E = Flow rate of bubbler in liters/min.