

Determination of Mercury in Air

1. SCOPE

This method is applicable to elementary mercury present in air.

2. PRINCIPLES OF METHOD

Elementary mercury is taken up from the air by amalgamating with silver gauze. When heated, the mercury liberated is then flushed through the cell of a spectrophotometer.

3. REAGENTS

3.1 *Silver gauze*

The material used consisted of 24 wires per cm, each wire having a diameter of 0.165mm, the total surface area, per sq cm of gauze, being approximately 2.45 sq cm.

3.2 Magnesium perchlorate.

3.3 Mercury.

3.4 *Palladium chloride absorbent*

20 grams of 10-20 mesh firebrick is impregnated with a solution of 0.5 grams of palladium chloride and dried.

4. APPARATUS

4.1 Spectrophotometer with gas cell.

4.2 Recorder.

4.3 Gastight syringe, graduated in μl , 1,000 μl total capacity.

4.4 Silver absorber, see Figure 1. A piece of silver gauze, forming a double cylinder, mounted in a finger condenser of borosilicate glass or quartz (Page 168).

4.5 Muffle furnace.

5. PROCEDURE

5.1 *Sampling of air*

Before using the absorber, heat activate by means of a gas micro burner, meanwhile purging the silver element with a constant flow of clean air. Cool the absorber and seal the side arms with PVC tubing; the absorber can be stored for 10-20 days before sampling. Connect the absorber to the portable air sampler as shown in Figure 2. Direct the side arms downwards to avoid intake of dust. Start the pump, the flow rate of which has been adjusted to the desired value by calibration with a rotameter. Allow a known volume of air to pass through the silver absorber (Page 169).

Note: The sampling interval of the pump can be operated by a clockwork timer. If simultaneous sampling with several absorbers is desired, this can be effected by means of radio assemblies with remote control operation.

If the concentration of mercury in the air is high, the sampling method can be modified, see Figure 3. Draw air, purified by passage through a palladium chloride scrubber, through the silver absorber by means of a portable air sampler. Collect the air sample in an airtight disposable polyethylene syringe and inject into the air stream (Page 170).

5.2 *Determination of mercury*

Connect the silver absorber to the spectrophotometer (setting 253.7nm) as shown in Figure 4. Start the pump so that air flows into the apparatus through a palladium chloride scrubber, then through the silver absorber and then to the gas cell. Adjust the flow to 1,500ml/min. With the use of heating tape, heat the cell to 80°C continuously in order to prevent condensation of water or mercury vapour. Heat the silver absorber to 800°C so that the mercury vapour is transferred to the gas cell. The light absorption is registered by the recorder. Alternatively, by the use of a digital integrator as used in gas chromatography, offscale readings may be avoided. The accuracy of the measurement can be increased considerably, using the integrator, and the capacity of the silver absorber can be increased from 10ng to 100ng mercury.

5.3 *Calibration*

Store mercury metal in pyrex reagent bottles, fitted with serum caps, in a thermostated water bath, maintained at $\pm 0.1^\circ\text{C}$. After equilibration,

withdraw a volume of saturated mercury vapour by a gastight syringe. Inject the mercury vapour into the apparatus through the septum situated in front of the silver absorber. Heat the absorber to 800°C and record the light absorption. Cool the silver absorber and inject a new volume of mercury vapour. Convert the peak heights obtained into absorbance units. Plot the absorbance against mercury content to obtain a calibration graph. Convert the peak height obtained from the sample and determine the mercury content from the calibration graph.

6. EXPRESSION OF RESULTS

Using the spectrophotometer-integrator assembly it is possible to measure as little as 0.1 nanogram of mercury. Some values of precision obtainable are presented below:

<i>Mercury nanograms</i>	<i>Precision percent</i>
4.0	±2.29
1.0	±2.50
0.4	±6.90

MERCURY VAPOUR SAMPLER

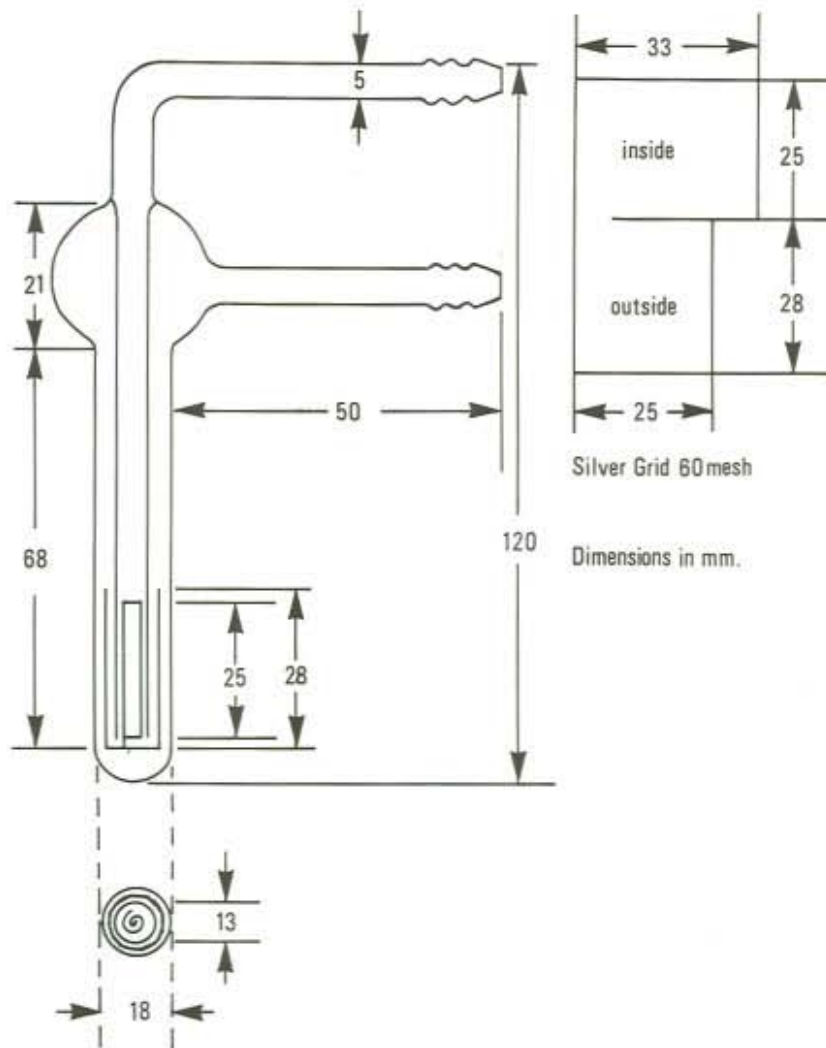


Fig. 1

AIR SAMPLER

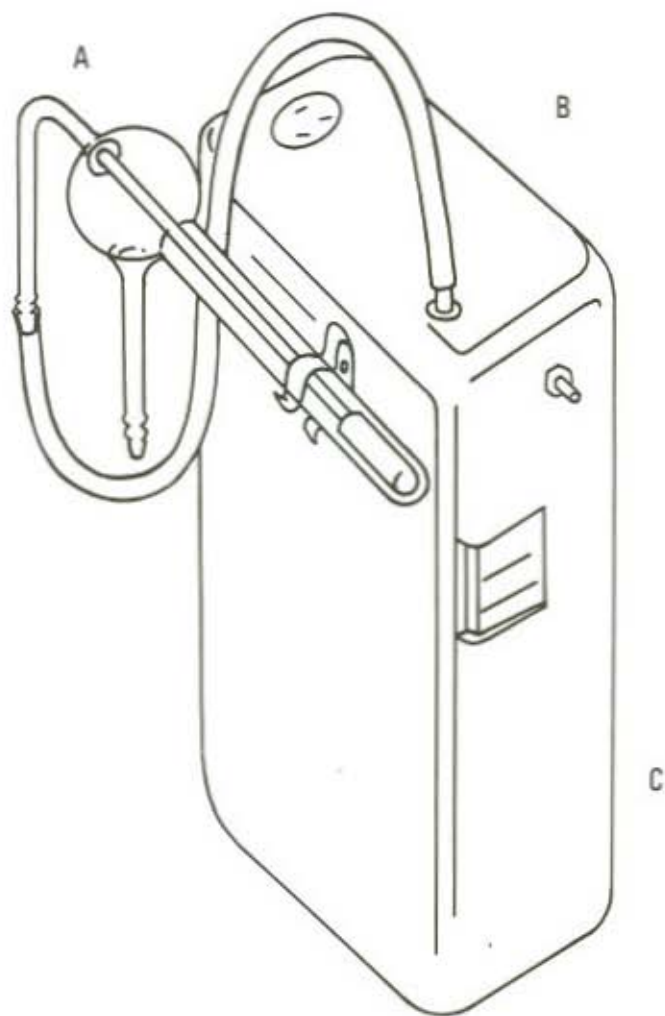


Fig. 2

- A Silver absorber
- B PVC tubing
- C Portable air sampler

SAMPLING FOR HIGH CONCENTRATIONS OF MERCURY

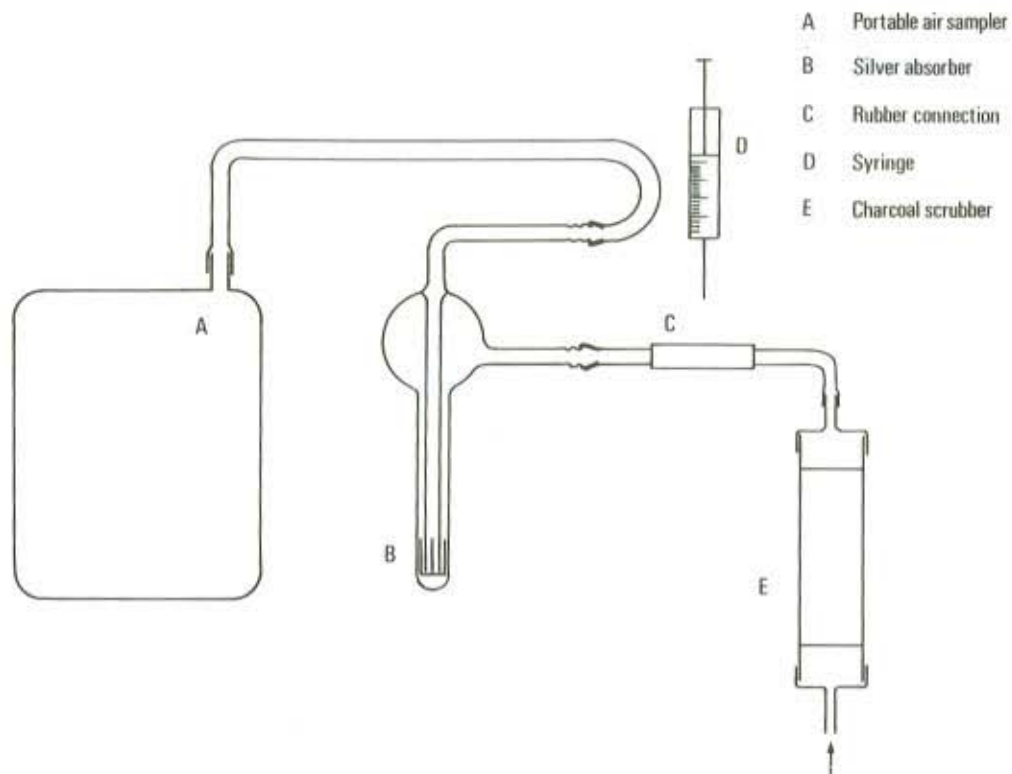


Fig. 3

ELEMENTAL MERCURY IN GAS SAMPLES

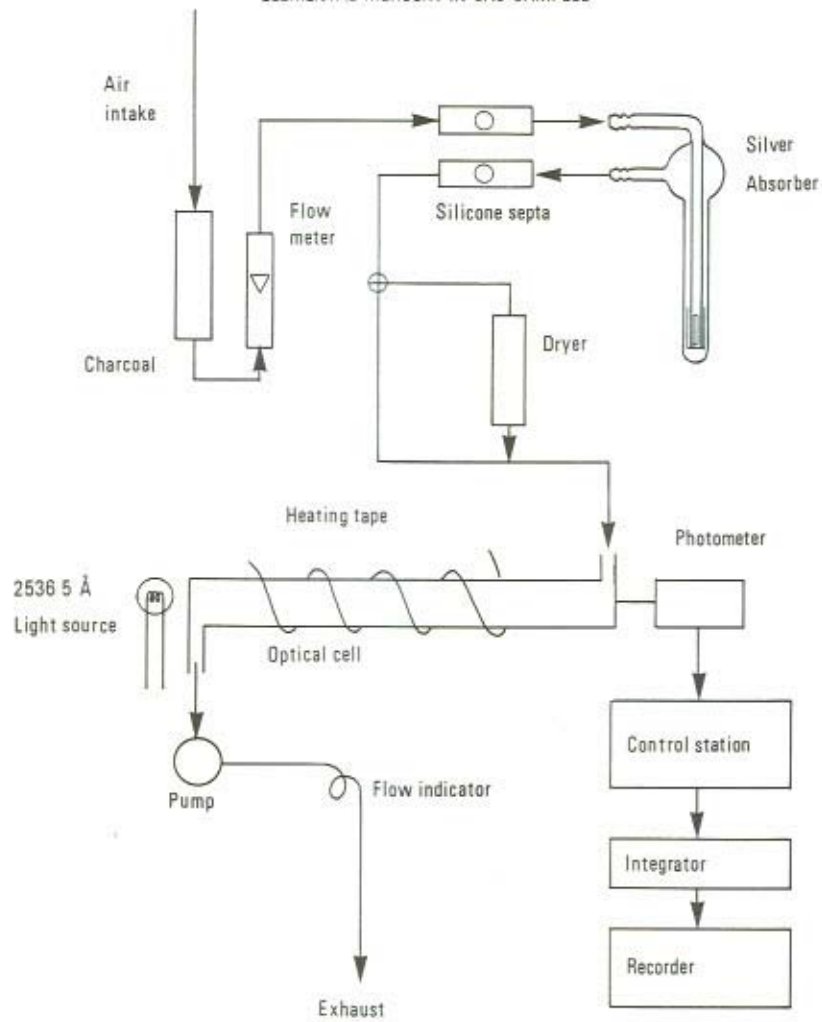


Fig. 4