84-025 TOTAL MERCURY in Soils

1. Application

1.1 The determination of mercury in soils in the µg/L range became feasible with the development of flameless atomic absorption spectrophotometry techniques. The procedure outlined was adopted from that of Malaiyandi and Barrette (1970) for biological materials. It is also suitable for the determination of Hg in plant materials.

2. Apparatus

2.1 Cold finger digestion assembly as shown in Fig. 10.

2.2 Hot plate.

2.3 Atomic absorption spectrophotometer, a recorder and a mercury lamp.

2.4 A gas absorption cell 210 mm long, 3 mm inside diameter made of pyrex with quartz end windows cemented in place with epoxy (Fig. 11).

2.5 Technicon autoanalyzer proportional pump equipped with platter prepared as in Fig. 11.

3. Reagents

3.1 Sulphuric acid, conc. H_2SO_4

3.2 Nitric acid, conc HNO_3.

3.3 Vanadium pentoxide.

3.4 Hydrogen peroxide 50%.

3.5 Dry ice.

3.6 Prepare a solution of hydroxylamine sulphate - sodium chloride by dissolving 15 g of hydroxylamine sulphate and 15 g of sodium chloride in 500 mL of distilled water. Make fresh daily as required.

3.7 Prepare a solution of stannous sulphate by dissolving 25 g of stannous sulphate and 5 g of sodium chloride in 250 mL of 2N sulphuric acid (conc H_2SO_4 55.5 mL/L). Add 1 g of mossy tin. This solution is stable for two weeks.

3.8 Prepare a wash solution by adding 120 mL of concentrated sulphuric acid and 80 mL of concentrated nitric acid to 800 mL of distilled water. Make fresh solution daily as required.

3.9 Prepare a 1000 µg/mL standard by dissolving 1.3535 g of mercuric chloride in 1 liter of 1N sulphuric acid (conc. H_2SO_4 27.8 mL/L). From this stock solution prepare fresh standards as required containing mercury concentrations of 2.5, 5 and 10 µg/L.

4. Procedure

4.1 Digestion.

4.1.1 Select a weight between 2 to 5 g of 2mm soil and place in a modified 200 mL Erlenmeyer flask (Fig. 10).

4.1.2 Add 0.1 g of vanadium pentoride (Do not weigh vanadium pentoxide on paper) and 3-4 glass beads to sample. Assemble the glassware and add powdered dry ice to cold finger.

4.1.3 Turn on condenser cooling water and add 5 mL of concentrated HNO_3 dropwise from dropping funnel.

4.1.4 Lower apparatus to preheated hot plates, 125°C for soils.

4.1.5 Heat samples gently for five minutes and add 5 mL of concentrated H_2SO_4 dropwise from dropping funnel. While adding the acid lift the digestion assembly from the hot plate and swirl while the exothermic reaction occurs. (a) keep the brown fumes (NO_2) and the bluish green nitrous anhydride (N_2O_3) at least one third below the top of the cold finger by keeping the cold finger well filled with dry ice and cold water running through the condensers.

4.1.6 Resume heating and reflux digest for about 30 minutes, maintaining the dry ice level.

4.1.7 Allow the digest to cool and add 3-4 drops of hydrogen peroxide (50%) while swirling.

4.1.8 When room temperature is reached remove the dry ice from the cold finger and wash the apparatus parts with a minimum of 2% aqueous sulphuric acid (30-40 mL), catching washings in the digestion flask.

4.1.9 Filter digest through prewashed glass wool into 100 mL volumetric flask and make to volume with 1.0N H_2SO_4.

4.2 Determination

4.2.1 Mercury wavelength 253.7 mµ, 5 mAmps.

4.2.2 Maximize signal from mercury lamps.

4.2.3 Align gas cell where the flame would normally be.

4.2.4 Start the proportional pump with reagent lines in place Fig. 11.

4.2.5 Manually input samples and standards for up to one minute.

NOTES:
(1) Clean glassware with tap water followed by rinsing with 10% nitric acid and 5-6 rinses with distilled water.
(2) Hydrochloric and Hydrofluoric acids contain small amounts of mercury.

5. Calculations

5.1 Prepare a standard curve from readings of standards.

5.2 Convert peaks for samples to µg/L Hg using standard graphs.

5.3 µg/L Hg in soils = µg/L in solution x $\frac{100}{\text{wt of sample (g)}}$

6. Precision

6.1 Insufficient data available.

7. References

7.1 Malaiyandi, M. and Barrette, J. 1970. Determination of sub micro quantities of mercury in biological materials. Anal. Lett. 3, 579-584.

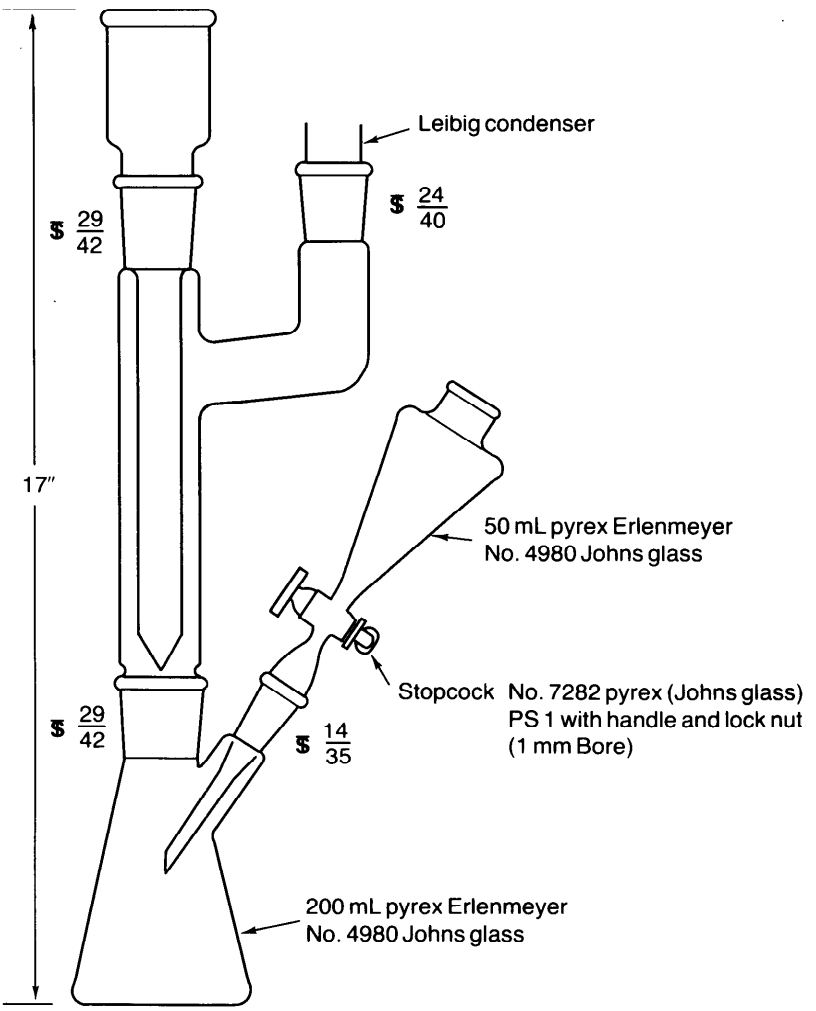
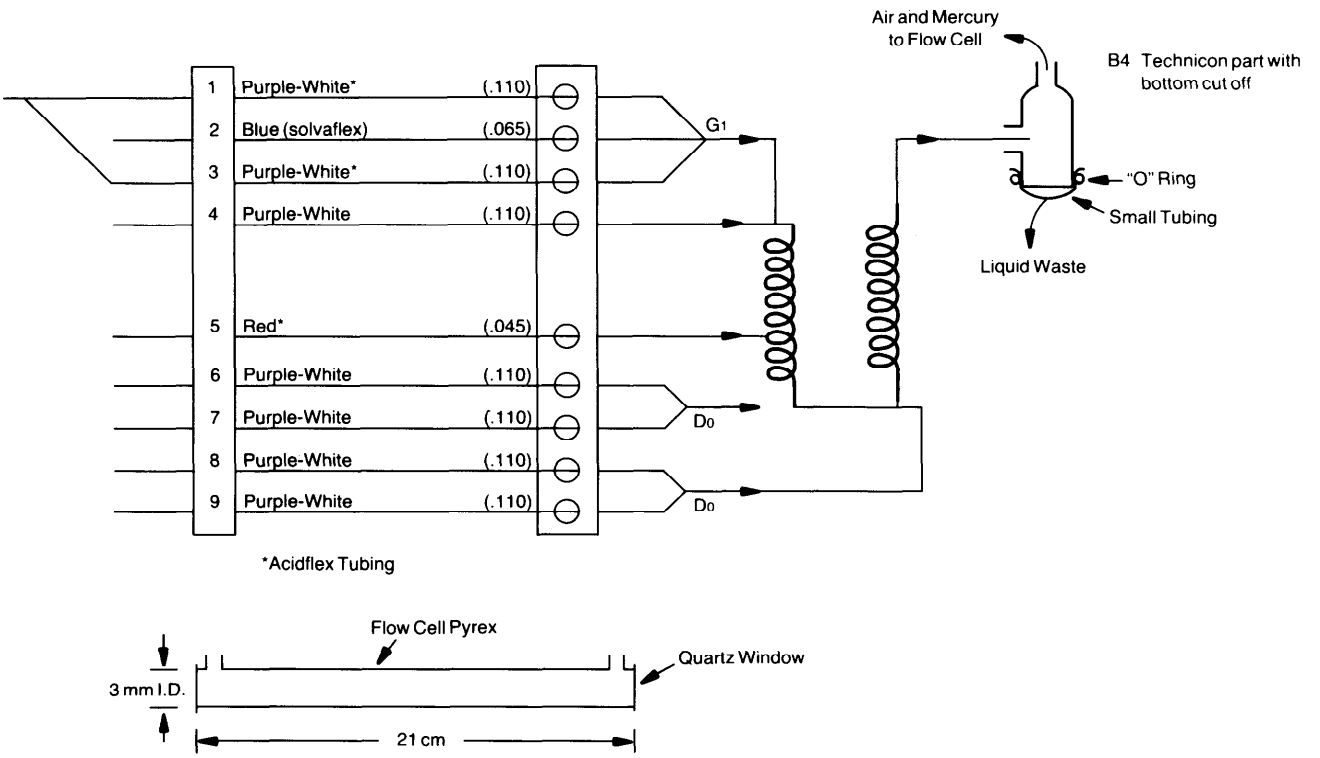


Fig. 10 Digestion assembly for Hg determination.

Sampling rate: 20/hr
Detection range: 0-10 μg/L



1,3. Sample (divided)
2. Hydroxylamine Sulphate
4. Air
5. Stannous Sulphate
6,7. Pull through from Cell
8,9. Air

Fig. 11 Flow diagram and flow cell for the determination of Hg.

Notes