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THE RAPID ESTIMATION OF MERCURY IN THE ATMOSPHERE OF WORKROOMS

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To measure the hazard to the worker it is often necessary to determine the atmosphere concentration of mercury in a workroom. It should be borne in mind that inhalation of mercury, though probably the most dangerous, is not the only way by which mercury enters the body. It follows that a measurement of the mercury content of the atmosphere does not give a full indication of the hazard. Air analyses are useful in determining the degree to which the workers are exposed; this is especially true of those not handling mercury, but working in the same atmosphere as those who do. Such analyses are also a means of keeping a check on the general good housekeeping of the workroom.

In the method described by Stock and Heller (1926) mercury vapour is removed from the atmosphere by trapping in chlorine water, while in the method described later by Stock and Cucuel (1934) mercury vapour is removed by passing the air through a tube cooled in liquid air. The mercury is then dissolved in chlorine water and deposited electrolytically on a copper wire. Fraser (1934) collected mercury from the air using solid carbon dioxide and ether or liquid nitrogen as his cooling agent. Mercury vapour may also be collected in alkaline hypobromite (Buckell, Hunter, Milton, and Perry, 1946; Milton and Duffield, 1947), in a solution of iodine in potassium iodide (Barnes, 1946), or in a mixture of potassium permanganate and sulphuric acid (Kuziatina, 1939). Dusts of mercury compounds have been collected in nitric acid in an impinger or a Palmer dust machine (Jacobs, 1941), or on a filter paper followed by digestion of the filter paper (Milton and Duffield, 1947; Buckell and others, 1946).

An examination of some of these techniques revealed certain practical drawbacks. The methods

of Stock and Heller, Stock and Cucuel, and Fraser all require electrolytic separation and are therefore too time-consuming for field use. The method described by Barnes proved insufficiently sensitive. Hypobromite solution as a trapping agent as described by Milton and Duffield, and by Buckell and others has proved less efficient than the acid permanganate of Kuziatina.

The method described below is the outcome of the experimental work and field experience gained over the last six years. Its advantages are that it is quick and easily carried out under field conditions with standard laboratory apparatus. It uses the acid permanganate scrubbing solution of Kuziatina but determines the trapped mercury by extractive titration with standardized dithizone in chloroform instead of allowing the sample solution to stand with the reagent. Using an extractive titration, interference from copper is minimized. Barnes (1947) has shown that the speed of extraction of copper by dithizone in chloroform increases with increase of pH but is always much slower than that of mercury. At a pH of less than 2 copper is extracted only on prolonged shaking. Using carbon tetrachloride the speed of extraction of copper is of the same order of that of mercury. The method is sensitive to mercurial dusts as well as to mercury vapour, and so for most purposes it is an improvement on the electronic mercury vapour detector. The titration gives the mercury content of the sample examined to the nearest microgram. The lower limit of the concentration that can be detected is governed by the volume of the sample taken.

Method and Materials

Principle.—A known volume of the atmosphere to be tested is sucked through a scrubbing bubbler containing

an acid permanganate solution. The permanganate is destroyed with oxalic acid and the mercury trapped in the solution is determined by a dithizone titration.

Reagents.—The following reagents are used.

Trapping Solution.—Potassium permanganate, 0.05, N in 5% sulphuric acid.

Oxalic Acid Solution, 10%.

Dithizone Stock Solution.—The solution is made up of 50 mg. % in chloroform, and diluted 1 in 100 with chloroform before use.

Standard Mercury Solution.—Mercuric chloride, 0.06723 g., is made up to 100 ml. with glass-distilled water, and diluted 1 in 100 before use to give a solution containing 5 µg. mercury per ml.

Apparatus.—Bubblers containing glass beads or the type with a sintered glass disc on the inlet tube are equally satisfactory. The air is sucked through for a known time at a known flow rate. The flow rate may be governed by a critical orifice placed between the bubbler and the pump, or a flowmeter can be used and the suction from the pump regulated by means of a leak and a screw clip.

Method.—The dilute dithizone solution is standardized against the standard mercury solution as follows: 50 ml. of 5% sulphuric acid is placed in a separating funnel and 2 ml. of the dilute standard mercuric chloride solution is added; 1 ml. of the dithizone solution is run in from a burette and the funnel is shaken for 30 seconds. The colour of the dithizone changes from green to orange. The chloroform layer is allowed to separate and drawn off, a further 1 ml. of dithizone is added, and the process repeated until there is no change in the dithizone colour. The amount of dithizone used is equivalent to 10 µg. of mercury and should be about 10 ml. The dithizone solution must be kept in the dark

when it is not in use and must be standardized immediately before each set of determinations.

There should be no mercury in any of the reagents, other than the standard. To check this, 20 ml. of the trapping solution is placed in a beaker, 4 ml. of 10% oxalic acid solution is added, and the beaker warmed until the contents are colourless. The solution is cooled and transferred to a separating funnel and titrated with standardized dithizone solution.

The bubbler is cleaned with nitric acid, well washed out, and rinsed with glass-distilled water. To make sure that the bubbler is clean 20 ml. of 5% sulphuric acid is allowed to stand in it for half an hour, then rinsed out into a separating funnel and titrated with dithizone. If mercury is present the bubbler is cleaned again and the process repeated.

Trapping solution, 20 ml., is placed in the clean bubbler and a known volume of air is drawn through. The contents of the bubbler are washed out and decolorized with oxalic acid as described above, then made up to 250 ml. in a graduated flask and a suitable fraction taken for titration.

Using 20 ml. of liquid in each of a series of three bubblers and a flow rate of 10 litres per minute, it was found in a set of six experiments that 96.2 to 98.5% of the mercury found was retained in the first bubbler. In three out of six experiments no mercury was found in the third bubbler. Since wide variations are to be expected in factory atmosphere conditions only one bubbler was used in subsequent work.

REFERENCES

- Barnes, E. C. (1946). *J. industr. Hyg.*, 28, 257.
 Barnes, H. (1947). *Analyst*, 72, 469.
 Buckell, M., Hunter, D., Milton, R., and Perry, K. M. A. (1946). *British Journal of Industrial Medicine*, 3, 55.
 Fraser, A. M. (1934). *J. industr. Hyg.*, 16, 67.
 Jacobs, M. B. (1941). "Analytical Chemistry of Industrial Poisons: Hazards and Solvents." New York.
 Kuziatina, N. S. (1939). *Zavodskaya Lab.*, 8, 174.
 Milton, R. F., and Duffield, W. D. (1947). *Analyst*, 72, 11.
 Stock, A., and Cucuel, F. (1934). *Ber. dtsch. chem. Ges.*, 67B, 122.
 —, and Heller, R. (1926). *Z. angew. Chem.*, 39, 466.



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