[CONTRIBUTION FROM THE CHEMISTRY DEPARTMENT OF IOWA STATE COLLEGE]

A SIMPLE AUTOMATIC MERCURY PUMP

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In the progress of a research it became desirable to collect and to measure accurately gas evolved slowly at low pressures. There are several vacuum pumps that permit the collection of the exhaust gas.[1]

Some of these are automatic. The valve designed by Maass[2] may be attached to any pump of the Toepler or Antropoff type to make it automatic. Without increasing the difficulties of construction, the simple Toepler pump has been modified so that it operates continuously and automatically, and returns the mercury to the reservoir after each stroke. The only auxiliary apparatus needed is a pump which will produce a pressure as low as 5 cm. of mercury. An ordinary water pump is satisfactory.

Description of the Pump

The tubes A and C and the bulb B (Fig. 1) are an ordinary Toepler pump without the movable mercury reservoir and rubber tube connection. The lower end of A is turned up and surrounded by the mercury cup H to facilitate the collection of the exhaust gas.

Instead of the movable reservoir connected to the bottom of C, a fixed reservoir E surrounds C from just below its junction with the bulb B to its lower end, a distance which must exceed 76 cm. The upper part of E is large so that B may be filled without a great change in level of the mercury in E. The lower end of E is sealed. The upper end is closed by a rubber stopper through which pass three glass tubes, C, D which leads to the auxiliary pump, and F which extends into the mercury cup H. The tube D is fitted with a 3-way stopcock K from which extends the tube L. The tube F, which is fitted with a stopcock G, is supported by the rubber stopper only, so that it hangs lower when filled with mercury than when empty. Its lower end should be between 1 cm. and 2 cm. above

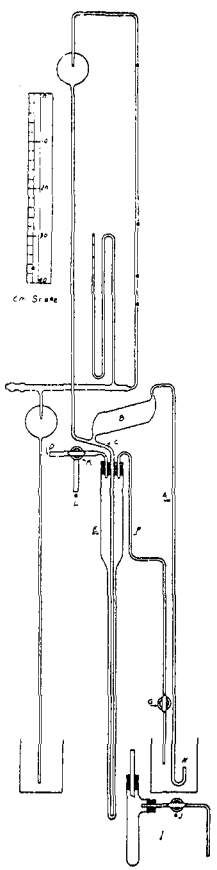

Fig. 1.

[1] Antropoff, *Z. Elektrochem.*, **25**, 269 (1919). Bauer, *Z. physik. chem. Unterricht*, **23**, 91 (1911). Beutell and Oberhoffer, *Chem.-Ztg.* **43**, 705 (1919). Chen, *Sci.*, **58**, 18 (1923). Johnston, THIS JOURNAL, **34**, 909 (1912). Pamfil, *Bull. soc. chim. Romania*, **4**, 57 (1922). Porter, *Ind. Eng. Chem.*, **16**, 731 (1924). Stock, *Z. Elektrochem.*, **23**, 35 (1920). Steele, *Chem. News*, **102**, 53 (1910); *Phil. Mag.*, **19**, 863 (1911).

[2] Maass, THIS JOURNAL, **41**, 53 (1919).

the upturned end of A. The upper part of the drawing shows a convenient arrangement of mercury traps, safety tubes, and manometer. The whole apparatus can be mounted on a board 1.8 m. long and 30 cm. wide.

When the pump is assembled the cup H is filled with mercury to a level just below the lower end of F. The reservoir E is filled with mercury to a level as high as possible without danger of drawing mercury out through D into the auxiliary pump.

Operation

A preliminary evacuation is necessary. The highest pressure p_0 at which the pump will work automatically is expressed by the equation $p_0V = (P-h)v$, where V is the volume of bulb B, P is the atmospheric pressure, h is the difference in level between the mercury in E when B is filled, and the highest point in the downfall tube A, and v is the volume of the gas when it is compressed in the upper part of A just as the mercury sweeps past the highest point of A. The bulb B can be made of such size that the auxiliary pump will give a sufficiently low preliminary pressure by direct evacuation.

The preliminary evacuation is made by connecting A to K by means of a piece of pressure tubing and evacuating both E and B, while the stopcock G is closed. When a pressure as low as p_0 is reached, K is set to evacuate E only, the rubber tubing is removed from A, and G is opened. The air rushes through F, presses on the mercury in E and forces it up through C and B, and down through A until the lower end of F is covered. Then as E is evacuated the mercury rises in F and falls in B. Mercury begins to flow from F into E about the time B fills with gas from C. When the lower end of F is exposed another stroke begins.

If B is so large that the auxiliary pump cannot produce a pressure as low as p_0, the device I may be inserted in the pressure tubing between A and L. The stopcock J is placed as close to A as possible. When the auxiliary pump has produced as low a pressure as possible in E and B, K is set to evacuate I, but not E, and G is opened. The suction through I aids in making the stroke. As soon as mercury begins to flow into I, J is closed. Then G is closed and E evacuated until B is in communication with C. Stopcock K is set to evacuate I, but not E, and G is opened. After the mercury breaks the communication between B and C, J is opened and a second stroke made. This device must be used until p_0 is reached.

A pump in which the capacity of B is 150 cc. was operated by a small water pump. The modified Toepler made one stroke per minute and ran continuously for 96 hours without attention. The capacity of the pump and the final pressure attainable are the same as for any other Toepler pump.

Summary

The Toepler pump has been so modified that, with the aid of a common water pump, it will operate continuously and automatically, return the exhaust mercury to the reservoir, permit the collection of the exhaust gas, and guard against its contamination. The modified pump is no more difficult to construct than the simple Toepler pump. It will operate over long periods of time without attention.

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THE VOLUMETRIC DETERMINATION OF SMALL QUANTITIES OF CARBON IN TUNGSTEN BY COMBUSTION

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The description of the volumetric method for the determination of carbon in tungsten by combustion, presented in this paper, is applicable to samples in which the total carbon is quite small (less than 0.2 mg.). The modifications in apparatus and technique have been developed for the analysis of carbon in tungsten filaments of incandescent lamps. A sensitive method is necessary, not only because the amount of carbon in the filaments is small (less than 0.05%) but because the weight of sample is frequently limited to a few milligrams of filaments or occasionally to the weight of a single filament.

Cain,[1] Brady,[2] Truog[3] and Hibbard[4] describe methods for the determination of carbon or carbon dioxide which depend upon the absorption of carbon dioxide in barium hydroxide solution and the titration of the excess of alkali with hydrochloric acid using phenolphthalein as indicator. These authors show that this adaptation of the titrimetric method using 0.1 to 0.5 *N* barium hydroxide is reliable for the larger amounts of carbon occurring in common steels and carbonate samples. No application of the method to small amounts of carbon is mentioned.

The present method of analysis depends upon the combustion of tungsten in oxygen, passage of the products of combustion into a special absorption apparatus containing hot 0.01 *N* barium hydroxide solution and titration of the cold, filtered solution with 0.01 *N* hydrochloric acid using thymolphthalein as indicator. Oxygen is purified by ignition over platinized porcelain and washing in concd. potassium hydroxide solution. A mercury seal and flexible plunger make possible the use of a closed system so

[1] Cain, *J. Ind. Eng. Chem.*, **6**, 465 (1914).

[2] Brady, *ibid.*, **6**, 843 (1914).

[3] Truog, *ibid.*, **7**, 1045 (1915).

[4] Hibbard, *ibid.*, **11**, 941 (1919).