

APPARATUS FOR EVOLUTION METHODS OF ANALYSIS.

By E. R. DOVEY.

THE apparatus shown in Fig. 1 was found to be exceedingly convenient for the examination of pyrolusite, and has been since used for various evolution methods, such as the estimation of sulphur in steel by the ammoniacal cadmium sulphate process.

The diagram is self-explanatory: the bulk of the absorbing liquid is placed in the large test-tube C, a smaller quantity in the U-tube E. It is easy to charge, and

* The American Society for Testing Materials Specification for Raw Tung-oil, mentioned above, allows a maximum of 3 per cent. free fatty-acid. The writer has seen very few wood-oils of this high acidity which would pass the twelve-minute limit with the heating test.

is proof against sucking back, as on the slightest diminution of pressure in the flask A air bubbles in through the tube B. It is found in practice that in most cases complete absorption takes place in the tube C, which at the end of the distillation is very easily detached and washed out.

The apparatus shown in Fig. 2 was primarily designed for the estimation of carbon in steel by the wet combustion method, absorbing the CO_2 in barium hydroxide solution, and afterwards titrating the barium carbonate. It, however, has been used for other evolution methods where it is desirable to have a condenser above the boiling flask. It consists of a boiling flask, A, fitted with a short Liebig

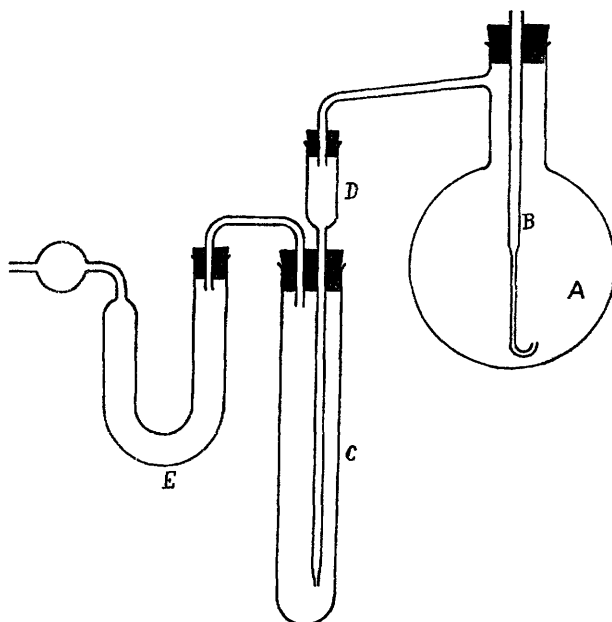


FIG. 1.

condenser, B, through which passes the long stem of a tap funnel, C. The whole is connected, as shown, with a 100 c.c. burette containing a Young's rod and disc, which fits loosely, and extends about two-thirds of the length of the burette. The top of the latter is attached by a three-way tap to an aspirator. For the carbon estimations, after the apparatus has been washed out with a stream of CO_2 -free air, 30 c.c. of barium hydroxide solution are sucked up into the burette, the flask A charged, and the estimation proceeded with, controlling the flow of gas by means of the burette stopcock, sufficient head of water being maintained in the aspirator all the time to prevent any escape of the barium hydroxide through the lower tap. When the absorption is complete, the tip of the burette is passed through a rubber stopper fitted into a wide filter-tube, the liquid run straight from the burette through the filter, the burette and precipitate being washed by allowing the filter-pump to draw

portions of the aspirator water through the apparatus. In this way the barium carbonate can be filtered off and washed without any contact with the atmosphere.

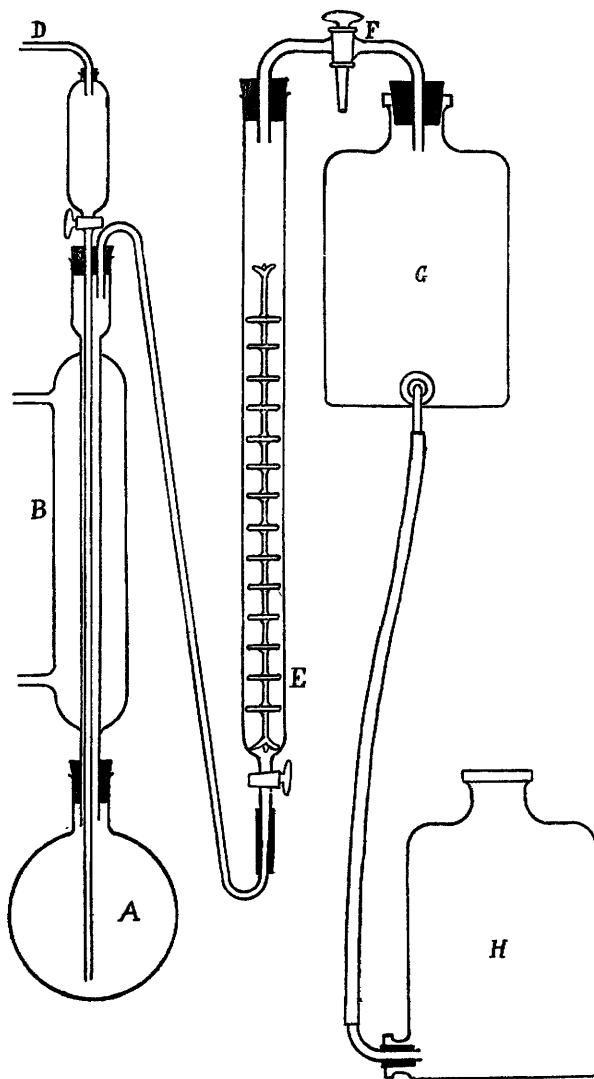


FIG. 2.

The burette fitted with the rod and discs forms a very efficient absorption apparatus, and may be used for many purposes.

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AN IMPROVED FORM OF U-TUBE.

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THE form of U-tube shown in the sketch has been found to be more easily cleaned and charged, and also much less fragile than the ordinary form. The two limbs are brought together so that the rims at the top are in contact. A slight squeeze when holding the tube during charging or connecting is much less likely to result in fracture than in the ordinary form. Two perforated Gooch discs rest at the bottom of each limb, keeping the bent portion free from absorbent material.

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