

## IMPROVED ORSAT APPARATUS.

BY A. BEMENT.

Received August 12, 1905.

THE instrument shown in the illustrations, was designed by the author for greater convenience of working than is possible with the regular Orsat apparatus. The distinguishing features are, that gas may be aspirated through the burette; also that absorption in the pipettes may be accomplished without displacement of the gas volume by means of the leveling bottle as is necessary with the usual pipette, so that one introduction of the gas to the pipette suffices, agitation of the reagent being accomplished by means of a simple form of pump, which consists of an orifice formed by a glass tube, to the lower end of which is attached a rubber connection, compression of which causes the reagent to be projected into the confined gas, the pipette containing no glass tubes. Babb<sup>1</sup> has recently described an apparatus employing a pipette wherein the gas is bubbled through the reagent. The pipette presented by the author works in an opposite manner, the reagent being sprayed or projected into the gas.

Referring to Fig. 1, gas enters by a rubber connection marked *A*, passing through a cotton filter shown, which is attached by tubing to a fitting, *B*, which takes any strain brought upon the connection. From this point the gas flows to cock *C*, which is provided with the regular three-way openings, and across the capillary connection it flows down through the burette, passes a three-way cock, *D*, at the bottom, and out by way of the connection *E*. The flow of gas through the instrument may be produced by any usual means. In this way all of the old gas is expelled and an entirely new sample is secured within the burette, after which, by manipulation of cocks *C* and *D*, it is immediately confined, and by turning cock *D* to the left-hand horizontal position, the passageway to *E* is closed and a corresponding connection established between the burette and the aspirator bottle. Then manipulation is precisely the same as with the Orsat apparatus as far as leveling and manipulation of the gas by the aspirator bottle is concerned. When the cock *D* is in position for the flow of gas by way of the passage *E*, it, of course, is closed off in the direction of the aspirator bottle. When the

<sup>1</sup> This Journal, 27, 156.

sample of 100 cc. has been secured, the leveling bottle is raised and the pinch-cock at the neck of one of the pipettes is opened,

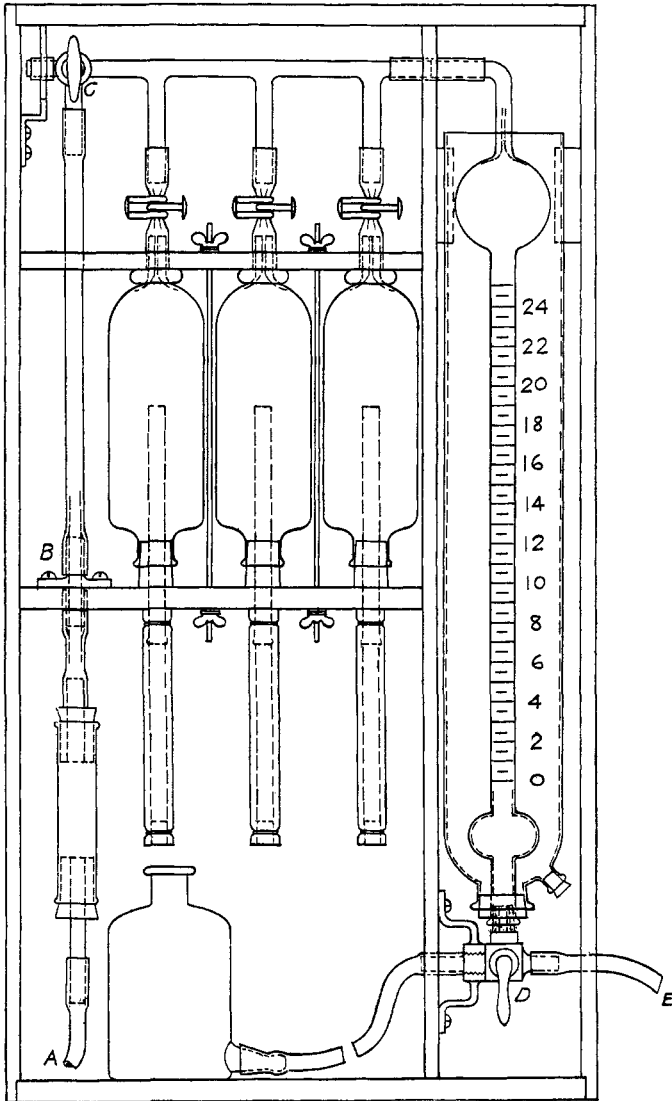


Fig. 1. Improved Orsat apparatus.

the gas being forced therein, after which the pinch-cock is closed, confining the gas to the pipette, then by compression of the

rubber projection below, the reagent is projected in jet form into the gas, which may be continued as long as necessary, after which the gas is withdrawn and measured. By this means it is often possible to secure complete absorption by one exposure in the pipette, and it is sometimes convenient when the attendant has other matters requiring his attention, to allow the gas to remain in the pipette while he gives momentary attention to other things, or the manipulation of the absorption process may continue, even if his attention is not directly upon it, which is something impossible with the usual form of pipette.

Fig. 2 is a cross-sectional elevation of the pipette and its associated features. *F* is a shelf upon which it rests, and *G* a binding-

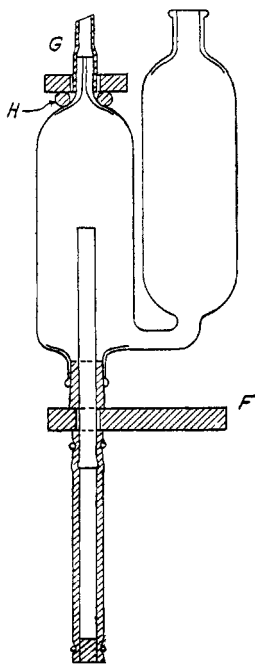


Fig. 2. Section through pipette.

piece held down by means of rods and thumb nuts, this binding-piece being kept in place by guides located at its ends and fastened to the sides of the instrument case. *H* is a rubber washer located between the top of the pipette and the binding-piece. As will be observed, the bottom end of the pipette is closed by a pierced rubber stopper, through which a glass tube projects. The lower end of this tube has fastened upon it a piece of  $\frac{3}{8}$  inch or  $\frac{7}{16}$  inch rubber tubing closed at the lower end by a plug as indicated. The positions of the glass tubes are such that there is always a sufficient seal of the reagent over their ends. Compression of the rubber extension causes a corresponding quantity of reagent to be discharged through the tube and into the gas, and when the rubber extension is released, a similar quantity of reagent returns to its interior.

The burette is surrounded by a water jacket as indicated, having at the bottom an opening provided with a stopper for drainage. The burette at its lower end has an enlargement; this is to insure that a considerable quantity of gas larger than 100 cc. is first obtained, a matter which is of considerable importance in those cases where gas may be drawn from a source at a

pressure below that of the atmosphere. In the particular instruments which the author has had made for combustion gases, the stem of the burette is of smaller diameter than usual, so that the entire contents are not greater than 28 or 30 per cent. of the burette. This makes possible finer divisions being graduated thereon, which read to 0.01 per cent.

The cock located at the lower end of the burette has in all cases been made of metal, and has proven more satisfactory than those of glass; those at the top of the pipette formerly employed, the author was compelled to replace with pinch-cocks, owing to the difficulty of obtaining carefully made and durable glass cocks.

215 DEARBORN ST., CHICAGO.

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[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY, No. 95.]

## THE USE OF THE ROTATING ANODE AND MERCURY CATHODE IN ELECTRO-ANALYSIS.

By LILY G. KOLLOCK AND EDGAR F. SMITH.

Received August 28, 1905.

(FIRST PAPER.)

SEVERAL investigations made in this laboratory have shown that when in electro-analysis the anode is rotated, high currents can be used and metals be precipitated completely in very short periods of time; further, by the use of mercury cathodes most interesting determinations and separations of metals are possible.<sup>1</sup> In the latter case, however, the anode has been stationary, and the electrolyte consequently not agitated. Then, of course, the precipitation of the metal has been comparatively slow. Observing the splendid results obtained with the rotating anode, when platinum was the cathode, we determined to use a combination of rotating anode and mercury cathode. This was accordingly done, and in some preliminary trials made last August (1904), the results of which were briefly alluded to in a communication published in this Journal, 26, 1614, mention was made that 0.4810 gram of copper could be precipitated in twenty-five minutes, and that this success could be had with other metals. Since then we have made additional experiments which we desire to record here. Not only is the time factor reduced for the metals studied, but the plan of combining a mercury cathode with the rotating anode

<sup>1</sup> This Journal, 25, 884; 26, 1124.