

CHEMICAL MANIPULATION. APPARATUS: GAS GENERATORS, MANOMETERS, ETC.—BY DOUGLAS MCINTOSH, M. A., D.Sc., Dalhousie University, Halifax, N. S.

(Presented 21 March 1927)

From time to time the student of chemistry employs some device or useful method of manipulation which he incorporates in a published article, where it frequently remains unnoticed, to be rediscovered by some other investigator. This could hardly occur if a description of his apparatus were given in a separate paper with a distinctive title. I am therefore describing a few pieces of apparatus which I have found convenient, and which I have used for a number of years. Some have been mentioned by me in various published papers; some doubtless have been used and described by others.

1. **Carbon dioxide generator.** A convenient substitute for the Kipp is a Dewar vessel with stopper and delivery tube, containing carbon dioxide snow. With the test-tube form which I ordinarily use, a "bag" of solid carbon dioxide (about 160 grams) gives a steady stream of gas for about 24 hours, while the same quantity in a litre flask lasts for nearly three days. The gas need not be washed or dried, for the vapour pressure of water at  $-78$  degrees is only 0.01 mm. Since a 50 pound cylinder yields from 24 to 28 "bags", the cost of the material is about 30 cents.

It is, of course, much cheaper to use a reducing valve and the gas direct from the cylinder, since the yield of snow is only some 15 per cent.

2. **Apparatus for the Purification of nitrogen.** The commercial gas from a cylinder can be easily freed from oxygen by bubbling it through molten phosphorus in a modified Drexel flask kept at  $100^{\circ}$  in a water-bath. Professor F. M. G. Johnson and the writer found this an excellent method in some experiments where a small amount of the lower oxides of phosphorus was not harmful.

3. **Generator for acetylene.** The carbide is placed in a filter-flask and covered with alcohol. Water is added to the alcohol by a dropping funnel, and the acetylene is purified and dried by chromic and sulphuric acids, potassium hydroxide and phosphoric anhydride, and is finally solidified in a tube surrounded with carbon dioxide boiling under reduced pressure.

4. **Apparatus to show change of liquid to solid acetylene.** Acetylene under atmospheric pressure is a solid like carbon dioxide, but unlike the latter, it can be liquefied by a pressure of a few centimeters. If the liquid be placed in a test-tube closed by the finger, it solidifies instantly on opening the tube. This pretty experiment can be repeated several times with a small amount of acetylene.<sup>1</sup>

For continuous demonstration the apparatus shown in Figure 1 is satisfactory. The tube containing the solid acetylene is warmed with the hand until the acetylene is liquefied. On opening "A" the liquid solidifies. When the tube is placed in liquid air the gas passes from the large bulb to the tube. Stop-cock "A" is then closed, and the tube is warmed again until liquefaction is complete. The experiment can then be repeated without loss of gas.

5. **Platinum resistance thermometers.** A platinum wire thermometer wound on glass has certain advantages over the ordinary form utilizing a mica frame. Dr. Barnes and the writer made a large number of these of a special pattern for boiling-point determinations, and several in which glass rods or closed tubes were used were made at the same time.<sup>2</sup> Since these have been recently described as something new, an account of the way in which the glass spiral was made may not be out of place.

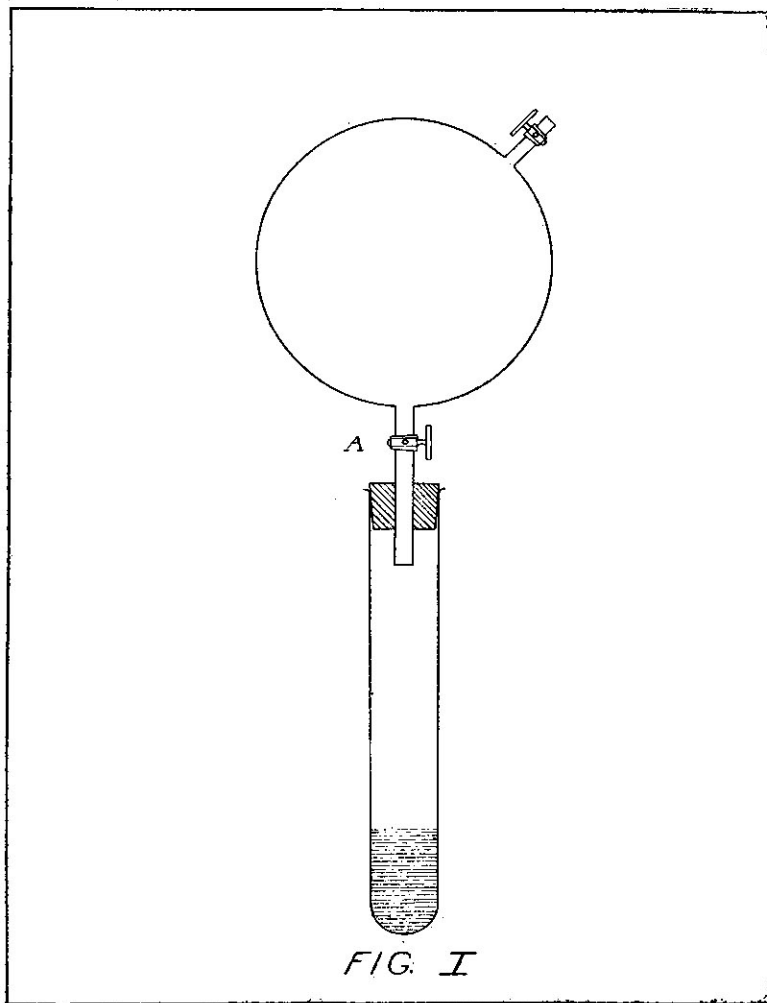
The tool for ruling the spirals was made from a hollow piece of brass holding a light spring. The cutting edge was

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<sup>1</sup> This was shown to me by Mr. E. Lesueur of Ottawa.

<sup>2</sup> Phil. Mag. 350, 1903.

kept by the spring from pressing too strongly on the glass. The tube or rod was coated with wax, centered in the lathe, and a double spiral cut in the wax. The spiral was then deeply etched with hydrofluoric acid solution. The resistance wire



is doubled and the loop attached at the lower end of the spiral by a drop of molten glass, wound in the depression, fastened at the upper end, and fused to the copper leads. The compensation leads are fixed in place, and the whole enclosed in a tightly fitting glass tube. A thermometer made in this way in 1903 is still in constant use.

6. **Manometers.** For pressures to one atmosphere the ordinary U shaped form (Figure II) is quite satisfactory. These can best be filled by exhausting and then distilling in the mercury. If made in this way the mercury remains clean; while, if filled with mercury and the air removed by boiling, a scum is often noticed on the surface. Professor Maass and I called attention to the advantage of filling manometers in this way in 1914.<sup>1</sup>

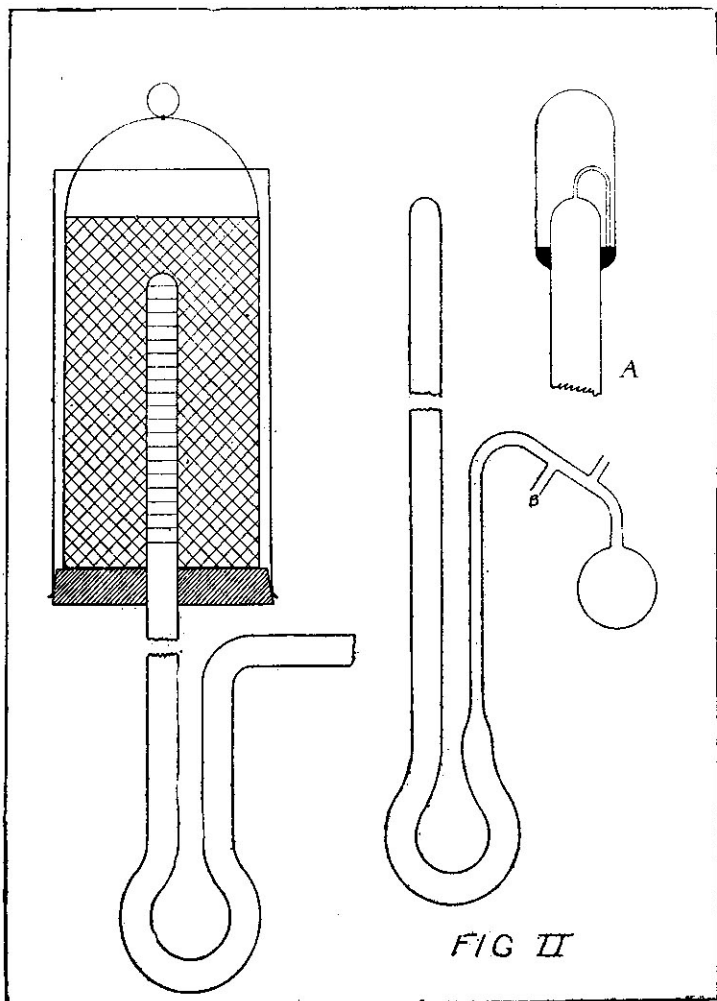
Pressures of from one to five or six atmospheres can be conveniently measured with an air or nitrogen filled manometer. The upper part of the manometer is graduated and carefully calibrated, and is then sealed to the U tube. After drying and exhausting, the gas is admitted and the proper amount of mercury distilled or poured in. If the high pressure manometer be now sealed to the low pressure one at "B", it can be calibrated while the low pressures are being measured. To prevent the mercury of the high pressure gauge from being drawn over at the low pressures, it is frozen by means of carbon di-oxide snow at the bend.

The calibrated part of the manometer is kept at constant temperature by means of an outer tube, through which water circulates, or by an ice-bath. In the latter case a basket of fine mesh, fitting the outer tube closely, is used. When a reading is to be made the basket is raised, and the gauge is easily read through the ice-water surrounding it. The diagram makes the method clear.

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<sup>1</sup> Trans. Royal Soc. Canada, 8, 66.

A neat device for the manometer of a hydrogen thermometer is shown in Figure II. If any gas enters the tube it may be pushed up to the reservoir at the top and trapped. Dr. B. D. Steele showed me this apparatus many years ago. I mention it since it has been "rediscovered" recently.



7. **Bell for delivering gases.** Figure III makes the principle clear. The bell is allowed to fill up slowly from a generator. When the gas reaches the level of the side tube it siphons out quickly. It is an easy matter to arrange this apparatus, so that the gas may be measured with some accuracy. I have no doubt that this has been used before, although I have never seen it, nor had Dr. Maass of McGill.

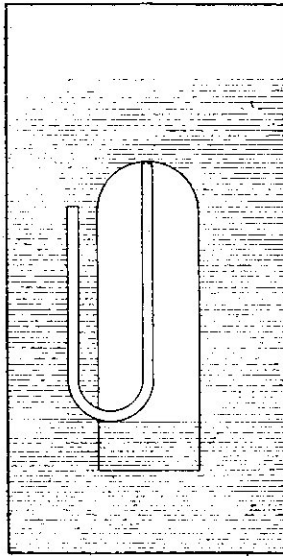
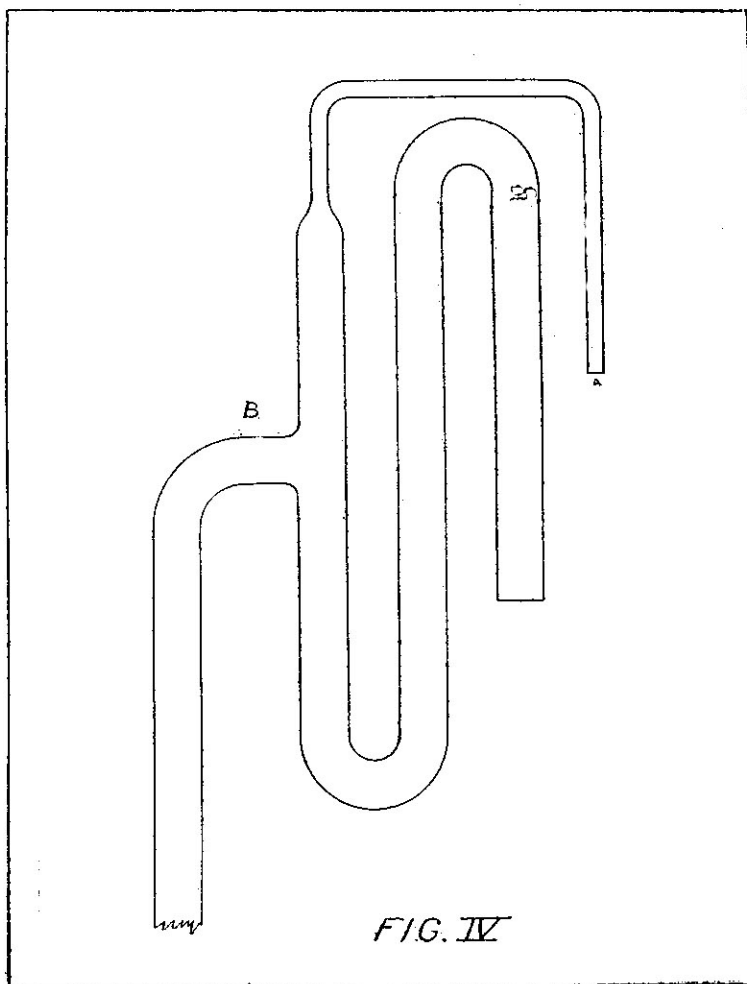


FIG. III

8. **An automatic siphon.** This differs from the ordinary form only in having the tube "A" (Figure IV) which admits the air, bent so that it is only a short distance above the level of the out flow tube. When the liquid to be transferred rises to "B" admission of air is stopped and the siphon runs quickly. The level of the liquid can be kept between "A" and "B".



9. **A device to regulate the swing of an analytical balance.** The diagram Fig. V, shows this simple arrangement. On pressing the rubber bulb a gentle current of air impinges on the bottom of the pan, making the balance swing through a few divisions. If on releasing the support the pointer moves over too large an arc, the swing may be easily damped by the air current.

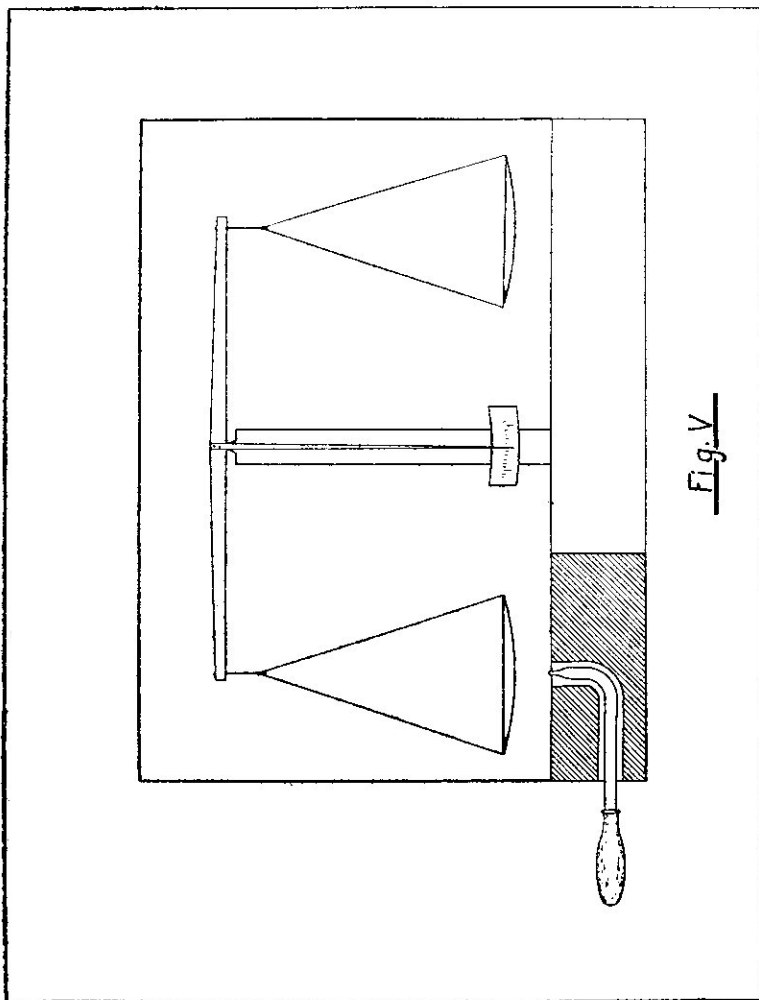


Fig. V