158. *Apparatus for Quantitative Catalytic Reduction.*

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IN the course of investigations in these laboratories it has been necessary to ascertain with some accuracy the amount of hydrogen absorbed by small quantities of substances isolated from plants or obtained in the course of degradation experiments on such products. The apparatus now described for use at atmospheric pressures has been used successfully for some years past (Henry and Paget, J., 1931, 30), whilst that for use at higher pressures has been devised recently owing to difficulty experienced in using quantitatively forms of such apparatus dependent on a dial gauge which at the best reads only to 0·5 lb. per sq. inch (Skita and Meyer, *Ber.*, 1912, **45**, 3589; Gough and King, *Chem. and Ind.*, 1928, **47**, 410).

The apparatus shown in Fig. 1, adapted from a Jowett and Carr nitrometer, is for use at atmospheric pressure. The substance and the catalyst are placed in *A*, which is attached by a butt joint covered by pressure tubing to the side tube *B* of the graduated limb *C*. Water is blown in to fill *C*, the 3-way cock *E* is opened to *A* and a water-pump applied at *F*; the air removed from *A* is expelled through *G*. Hydrogen is then led in through *D*, and is admitted to *A* after the small amount of water remaining at the top of *C* has been expelled. The operation is repeated to secure complete replacement of air by hydrogen, the levels adjusted and the volume noted. A cog-wheel constructed of cork and rotated rapidly by attachment to a small friction-geared motor is brought into contact with *A* to secure vigorous agitation. If necessary, *C* can be refilled with hydrogen without interrupting the experiment.

The apparatus for hydrogenation under pressure is shown in Fig. 2. The U-tube is made of stout glass tested to withstand 20 atmospheres and calibrated to read c.c. of gas in the left-hand limb. The substance and catalyst are placed in flask *A*, closed with a rubber bung carrying a Schrader bicycle tyre valve *A′* of a type known as a " French valve " (Schweinert, B.P. 15458/1914). The bung is forced well into the neck of the flask, and is secured in that position by a wired metal washer. The ends *E* and *F* of the U-tube are similarly closed by Schrader valves *E'*, *F'*. The side tube *B* carries a modified Schrader valve from which the original parts *K*, *N* (Fig. 3) have been unscrewed. Connexions to the valves are made with bicycle-pump tubing through an adaptor *M* sold for this purpose. Leakage is prevented by the application of a little rubber solution at the base of the screw-threads and of a trace of soft paraffin on the packing at *N*; it is important to keep the parts of the valves quite free from dirt. The valves *E'*, *F'* are opened to admit or withdraw hydrogen as required by pressing down the plunger *K* with a rod; in the case of *A'*, where the valve is less accessible, this is done by means of a short metal tube *H*, made from a large hypodermic needle carrying a ball of solder on its point and a collar at *O* which fits tightly into the rubber tubing.

FIG. 1.

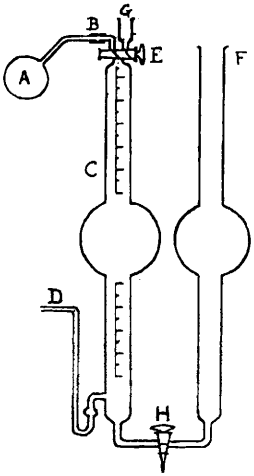


FIG. 2.

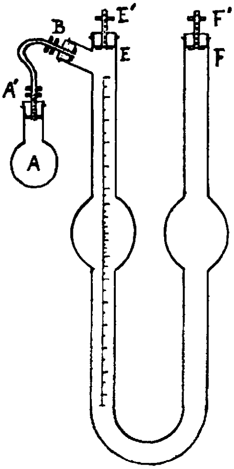
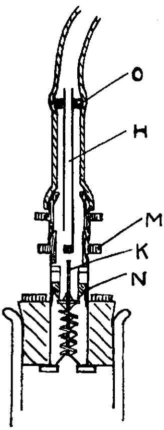


FIG. 3.



A is detached, evacuated, and filled with hydrogen. The graduated limb is filled through *B* by displacement of water into the right-hand limb. *A* is then connected to *B*, valves *A'* and *F'* are depressed to equalise the pressure throughout the apparatus, and the total volume of gas is read and corrected for the difference in level of the water in the two limbs.

An ordinary bicycle-tyre pump is now attached to *F'*, and the pressure raised by the desired amount. The pump is detached, and *F'* opened, thereby releasing the pressure in the U-tube while that in *A* is retained by the automatic closing of *A'*. The difference between the residual volume and the volume of hydrogen admitted to the apparatus is the amount compressed into *A*. By a suitable form of shaker, *A* can be vigorously agitated, while attached to the apparatus, or the tube connecting *A′* with *B* can be closed by a stop or pinch-clip and *A* removed and shaken separately. At the conclusion of the reaction, the gas under pressure in *A* is released into the apparatus by depressing *A′* and *F′* and the amount absorbed is calculated. If necessary, the gas can again be compressed and the experiment continued until no further contraction of volume takes place; before the final volume is read, the flask *A* is shaken for a few minutes at reduced pressure in order to liberate the hydrogen dissolved in the solution.

The following results were obtained, about 5 mg. of platinum oxide catalyst (Adams, " Organic Syntheses," Vol. VIII, p. 92) being used in each case.

Substance.	Amount, g.	Pressure, atm.	Hydrogen absorbed, c.c. at *N.T.P.*	Calc., c.c.
Chaulmoogric acid ...	0·4830	4	38	38·6
Maleic acid	0·4270	4·3	82	82·4

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