NOTES

A Greaseless Magnetic Valve for High Vacuum Systems

By Don L. Armstrong

Although high vacuum technique has become highly developed in recent years, there is yet need for an easily constructed valve which avoids the presence of any substance other than glass and which may be heated without injury. Several mechanically or magnetically operated valves have been described in the literature,[1-12] but each fails to meet one or more of the above requirements. It is believed that the valve described in Fig. 1 satisfies all of these conditions.

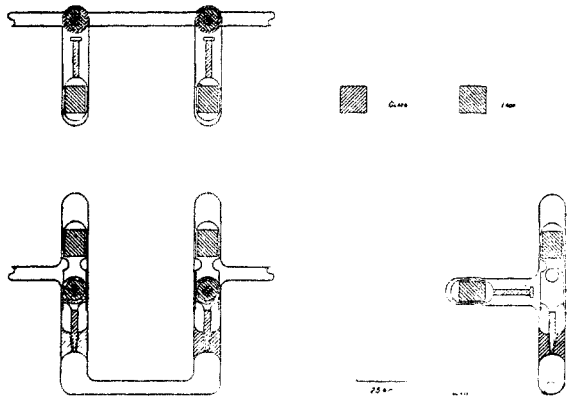

Fig. 1.

The valve is opened by placing a solenoid around one of the vertical tubes containing a valve stem within which is sealed a small piece of iron rod. While the valve stem is thus raised, another solenoid is placed about the corresponding horizontal tube holding the valve support, and the latter is moved toward the stem. The current through both solenoids is now shut off. This allows the valve stem to drop so that the support collar engages the support. This process is repeated with the other half of the valve. While the valve is open, it may be heated by a flame to assist the passage of difficultly volatile materials.

The valve seats are formed with the aid of a carbon rod, accurately tapered at an angle of 7° to the axis of the rod. The valve-plugs are made by drawing out the ends of solid rods to nearly the same taper, and grinding them into the seats to produce a high polish. The grinding is done before the excess length of rod is cut off.

The valve described here was able to maintain a pressure of 900 mm. on one side and a vacuum on the other with less than 0.05 cc. (gas at S.C.) leakage in ten minutes. Such performance could be obtained only when the valve seat and valve-plug were perfectly clean. Sutton and Mayer[11] made a much more modest claim for a one-way valve of related design. The difference is probably due to the double-plug arrangement of the two-way valve here described.

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The Synthesis of 6-Diethylamino-1-aminohexane[1]

By David S. Breslow and Charles R. Hauser

In connection with another problem a relatively large amount of 6-diethylamino-1-aminohexane was needed. This diamine has previously been synthesized apparently only from ϵ-benzoylaminoamyl chloride, which itself is rather difficult to prepare, in a yield of only 9%.[2] We have prepared the diamine from hexamethylene bromide and diethylamine using the Gabriel synthesis and from ϵ-bromocaproic acid by the following series of reactions.

$$\mathrm{Br(CH_2)_5COOH} \xrightarrow[NH_3]{SOCl_2} \underset{(64\%)}{\mathrm{Br(CH_2)_5CONH_2}} \xrightarrow{SOCl_2}$$

$$\underset{(76\%)}{\mathrm{Br(CH_2)_5CN}} \xrightarrow{(C_2H_5)_2NH} \underset{(90\%)}{(C_2H_5)_2N(CH_2)_5CN} \xrightarrow{[H]} \underset{(70\%)}{(C_2H_5)_2N(CH_2)_6NH_2}$$

The yield (45%) by the Gabriel method is satisfactory but we prefer the latter method, since the reactions are readily carried out and the yields (given in parentheses) are satisfactory. We have made no attempt to obtain optimum yields. The ϵ-bromocaproic acid is readily prepared from cyclohexanone by oxidation with potassium persulfate followed by hydrolysis of the resulting lactone with hydrogen bromide.[3]

(1) The work described in this paper was done under a contract, recommended by the Committee on Medical Research, between the Office of Scientific Research and Development and Duke University.
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