9.8 and 19.0%. In the general case, of course, results are expressed as moles per unit weight.

The writer is indebted to Professor James B. Conant, who suggested carrying out the above tests.

Summary

The measurement of the boiling points of saturated solutions is suggested as an aid in identifying and testing the purity of compounds to which the familiar melting point procedure is not applicable because of decomposition. Examples of the method are given.

Negative results in the tests for impurities are not necessarily conclusive. Any of the procedures is subject to essentially the same limitations as the analogous test by the melting point method.

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NOTES

A Diaphragm Valve.—In connection with some experiments in which a greaseless device for regulating gas flows was needed, the diaphragm valve described in this note was developed. It consists of an aluminum diaphragm A, about 0.3 mm. in thickness, resting on the flange of a glass cup B, which has an outlet tube C, and a capillary inlet tube D, as shown in Fig. 1. The upper end of tube D and the flange are ground flat with fine carborundum powder. Around the edge of the diaphragm and the flange of the cup is a rubber gasket, E. These parts are held together by a brass frame F. The position of the diaphragm is adjusted by a differential screw G. The lower surface of the diaphragm is roughened with very fine emery paper, so as to have better contact with the ground end of the capillary tube for the regulation of the low flows.

In cases where the gas used is likely to react with or corrode aluminum, a thin piece of mica is cemented with Duco Household Cement across the entire lower surface of the aluminum diaphragm. The mica surfaces should first be roughened with fine emery paper.

Fig. 1.

By varying the diameter of the inlet and outlet tubes, and the size and thickness of the diaphragm, this type of valve can be adapted to a wide range of gas flows. In connection with some experiments in this Laboratory, this type of valve was used successfully to regulate gas flows ranging from a few tenths of a cubic centimeter to several liters of gas per minute.
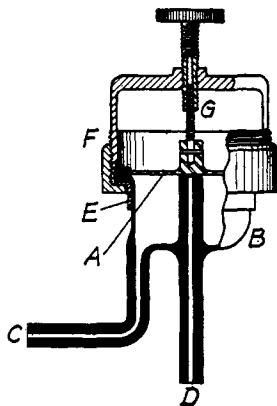
This valve is especially useful in controlling very slow flows where an ordinary stopcock is inadequate.

The writers wish to express their thanks to Mr. L. Testa for making the glass cup for the valve.

J. Y. YEE
J. REUTER

A Method of Winding Helical Quartz Springs and of Constructing Glass Sorption Buckets.

—The construction and characteristics of the quartz helix sorption balance have been described in detail by McBain and Bakr[1] and the technique of its application to sorption studies by McBain and co-workers.[2] The author feels that the method of winding such helices which is presented here is an improvement upon that described by McBain and Bakr as regards ease of manipulation and uniformity of the springs produced.

The fibers are drawn from a quartz rod 3–5 mm. in diameter which is rotated in an oxyhydrogen flame until the quartz is evenly heated. The rod is then removed from the flame and drawn out immediately. The desirable fibers are about 0.2 mm. in diameter and about 100 cm. long. With some practice it is possible to draw fibers, a fair percentage of which will be of a useful diameter.
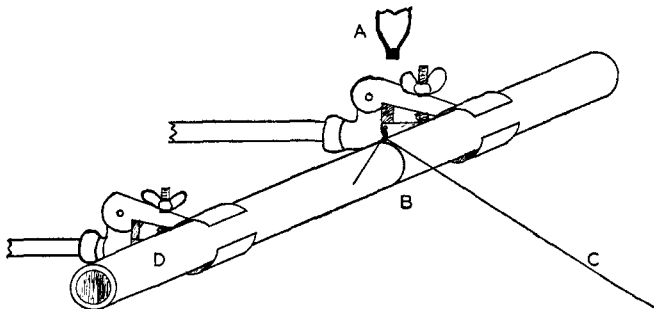

Fig. 1.

The method used in winding the helices is illustrated in Fig. 1. A section of quartz combustion tubing, D, 1.5 cm. in diameter and 30 cm. long is mounted horizontally with two buret clamps holding it loosely so that it can be moved horizontally and rotated freely. A small hand torch, A, is mounted vertically about 2 cm. above the axis of the tube. The author

¹ McBain and Bakr, THIS JOURNAL, 48, 690 (1926).

² McBain and Britton, ibid., 52, 2198 (1930); McBain, Lucas and Chapman, ibid., 52, 2668 (1930); McBain, Jackman, Bakr and Smith, J. Phys. Chem., 34, 1439 (1930).

prepared a small glass hand torch for this purpose. A piece of heavy nichrome wire, B, is twisted around the tube to provide a point of attachment for the fiber to be wound. A gas flame about 6 cm. in length is fed an air–oxygen mixture until the inner cone does not impinge upon the tube. The end of the fiber, C, is bent and hooked around the wire post and the tube slid over so that the fiber is in the flame. The tube is then rotated with one hand and the fiber guided with the other.

The amount of oxygen fed to the flame is rather critical. Too hot a flame may melt the fiber or cause it to adhere to the tube. Too cold a flame will result in poor bending and the resulting spring will not be cylindrical. A trial or two will give the proper flame adjustment and the winding may then be quite rapid. If one tires, or if the tube overheats, the spring may be slid out from the flame, and the winding resumed at leisure.

When cold, the hook is broken off and the helix loosened from the tube by gently rotating the ends in opposite directions with the tips of the fingers. It may then be bunched up and slid off the tube. A tube with a slight taper is very advantageous.

Helical springs prepared in this manner are free from corners, and show excellent mechanical strength. The small hand torch with an air–oxygen-gas flame is very conveniently used to bend the ends of the helices to form suspension hooks. McBain and co-workers recommend that the completed helices be annealed at 400° for about forty-eight hours.

McBain and co-workers employed buckets of platinum or gold foil to hold their sorbent. The author has prepared glass buckets for the same purpose. Since these buckets cannot be
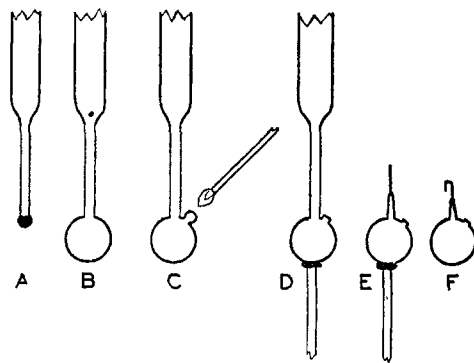

Fig. 2.

used to hold weights while the springs are being calibrated, a small pan of mica may be employed. Such a pan need weigh only 0.03 g. The stages in the preparation of the glass buckets are shown in Fig. 2.

A piece of Pyrex tubing, about 1 cm. in diameter, is heated and drawn out to give a capillary about 1.5 mm. in diameter. This capillary is touched to the edge of an air–oxygen-gas flame until a small ball of glass has collected. This ball, A, is then blown out to form a bulb, B, with very thin walls. A tiny gas flame is then touched to the top as shown in C, and a small bulb blown out, broken and the edges melted down. A second capillary is then fastened to the bottom of the bulb with a drop of melted wax as shown in D, and the original capillary drawn off as rapidly as

possible in a hot needle flame.   The glass stem remaining is then bent into a hook, using the tiny gas flame.   The wax is then melted off and the bulb cleaned with an appropriate solvent.   A bituminous base wax, such as Piceïn, is recommended over a shellac wax, such as deKhotinsky, because it is readily removed with carbon disulfide or carbon tetrachloride.

Buckets prepared in this manner may be made in any desired size.   The author has prepared a number which are more than 1 cm. in diameter and which weigh less than 0.1 g.   The weight of such buckets is usually about 0.07 g.   These buckets will withstand a baking-out temperature of 450°.

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RECEIVED MAY 13, 1931
PUBLISHED JULY 8, 1931

ALPHA-NAPHTHOLSULFONIC ACIDS AND DERIVATIVES.   II

BY E. GEBAUER-FUELNEGG AND ECKHARD HAEMMERLE

In a series of articles it has been shown that phenols can be converted in phenolpolysulfonyl chlorides[1] by the action of chlorosulfonic acid. It also was noticed that the reagent caused four distinct types of reaction: namely, sulfonation, sulfochlorination, chlorination and oxidation.   In certain cases the hydroxyl group of the phenol was found to be esterified prior to sulfonation, while in other instances condensation products such as the sulfonylides were isolated.   The predominance of any one of the above-mentioned reaction types will depend on the time, temperature, quantity of reagent and the nature of the phenols.

The most striking aspect of the reaction between unsubstituted phenols and chlorosulfonic acid is the fact that so far it has been impossible to obtain the respective monosulfonyl chlorides.

From these facts a reaction mechanism for the action of chlorosulfonic acid on phenols (and phenolsulfonic acids) was postulated comprising as the first step the primary formation of the phenolsulfonic acid ester, and as the second step its rearrangement to form a phenolsulfonic acid;   this is followed by reësterification of the hydroxy group and a second rearrangement to form the phenoldisulfonic acid in presence of sufficient amounts of the reagent.[2]   Only when the phenoldisulfonic acid is formed and further

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[1] J. Pollak and E. Gebauer-Fuelnegg, *Monatsh.*, 46, 383 (1925);   46, 499 (1925); 47, 109 (1926);   47, 511 (1926);   49, 187 (1928);   O. Litvay, E. Riesz and L. Landau, *Ber.*, 62, 1863 (1929);   E. Katscher, *Monatsh.*, 56, 381 (1930).

[2] E. Gebauer-Fuelnegg and A. Schlesinger, *Ber.*, 61, 781 (1928).