IN THE LABORATORY

An Apparatus for the Quantitative Separation of Volatile Substances by Fractionation and Distillation

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An apparatus that has proved useful for the quantitative separation of small quantities of volatile materials in complex pharmaceutical products is here described. As shown in Fig. 1, the apparatus is a combination unit composed of a fractionating column joined to a condenser by way of sealed glass connections and provided with standard ground-glass joints at the distillation and condenser ends. A small, sealed-in well at the top of the fractionating column, when filled with water, serves to increase the reflux at this point and offers a convenient place in which to insert a small thermometer for measuring the approximate distilling temperature. A 125- or 250-ml. iodine flask (⅋22 ⅋ neck) serves as the distillation flask, while a long-stemmed adapter and a long-necked receiving vessel (cooled with ice) are employed at the condenser end. Ceramic Berl Saddle column packing (6 mm.) is employed in the fractionating column.

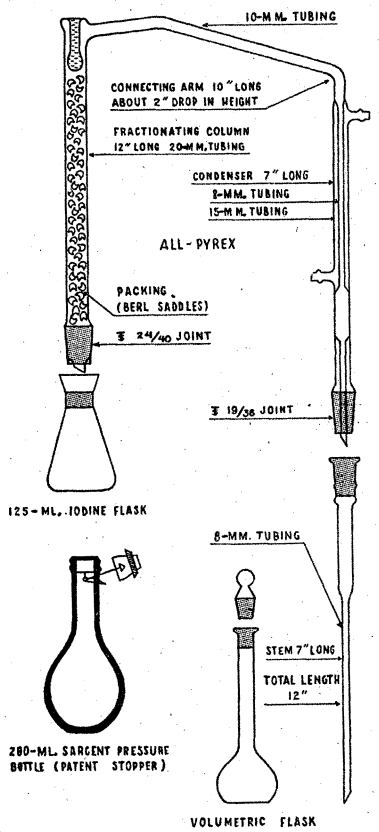

FIG. 1

Before operation of the apparatus, the flared mouth of the iodine flask is filled with water or some appropriate sealing fluid, and the lower tip of the adapter is submerged about ¼ inch in the solvent or reagent placed in the receiving vessel. The ground-glass joints, of course, should be wet before being connected.

The efficiency of the apparatus is largely dependent on the partial vacuum produced during distillation when an appropriate solvent is provided in the receiving flask. This vacuum, caused by the reduction of vapor pressure obtained at the surface of the solvent, is sustained until the adapter is disconnected. As long as ebullition is taking place at a fairly constant rate, an adequate partial vacuum is maintained. Bumping in the distilling flask, prevented mainly by the use of glass beads, etc., causes small variations in the partial vacuum, but the enlarged upper portion of the adapter acts as a safety valve in preventing the distillate from rising too high.

The apparatus has the following advantages: (1) Rapid and quantitative separation of volatile components is obtained. (2) Losses are at a minimum, due to the partial vacuum obtained during distillation, absence of joints in connecting arm, and air-tight connections at distillation and receiving ends. (3) Small amounts of volatile components can be recovered. (4) Standard taper connections permit the use of interchangeable flasks and adapters.

This apparatus has been successfully employed for over two years for the quantitative separation of alcohol, alcohol and ether, and chloroform from pharmaceutical products.

Stability of Penicillin in Glycerin and in Glycols

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Penicillin stability in mixtures containing glycerin (*2, 4*), propylene glycol (*5*), or Carbowax (*1, 3*) (a solid, water-miscible polyethylene glycol) may be less than recent reports would indicate. Since these compounds are major components of such mixtures, penicillin stability in glycerin or glycols alone was determined in order to prognosticate, if possible, stability in preparations containing them.

The activity of commercial calcium (CaP), sodium amorphous (NaP), and sodium crystalline (NaP cryst.) salts[1] was determined, before and after storage at 5°, 23°, and 37°C., in glycerin, propylene glycol, and Carbowax,[2] by the Food and

[1] Pfizer, Heyden, and Commercial Solvents Corporation, respectively.

[2] Glycerin USP; propylene glycol and Carbowax 1500, Carbide and Carbon Chemicals Corporation.