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A RAPID VACUUM EVAPORATION SYSTEM

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The prodigious use of solvents in our section of the Eastern Regional Research Laboratory has required the development of a fast but efficient vacuum evaporation system. Since large quantities of solvents are utilized in our work on extractions and fractionations of tobacco extracts, the commercially available evaporators have not proven satisfactory or adequate. The design, shown in figure 1, was developed

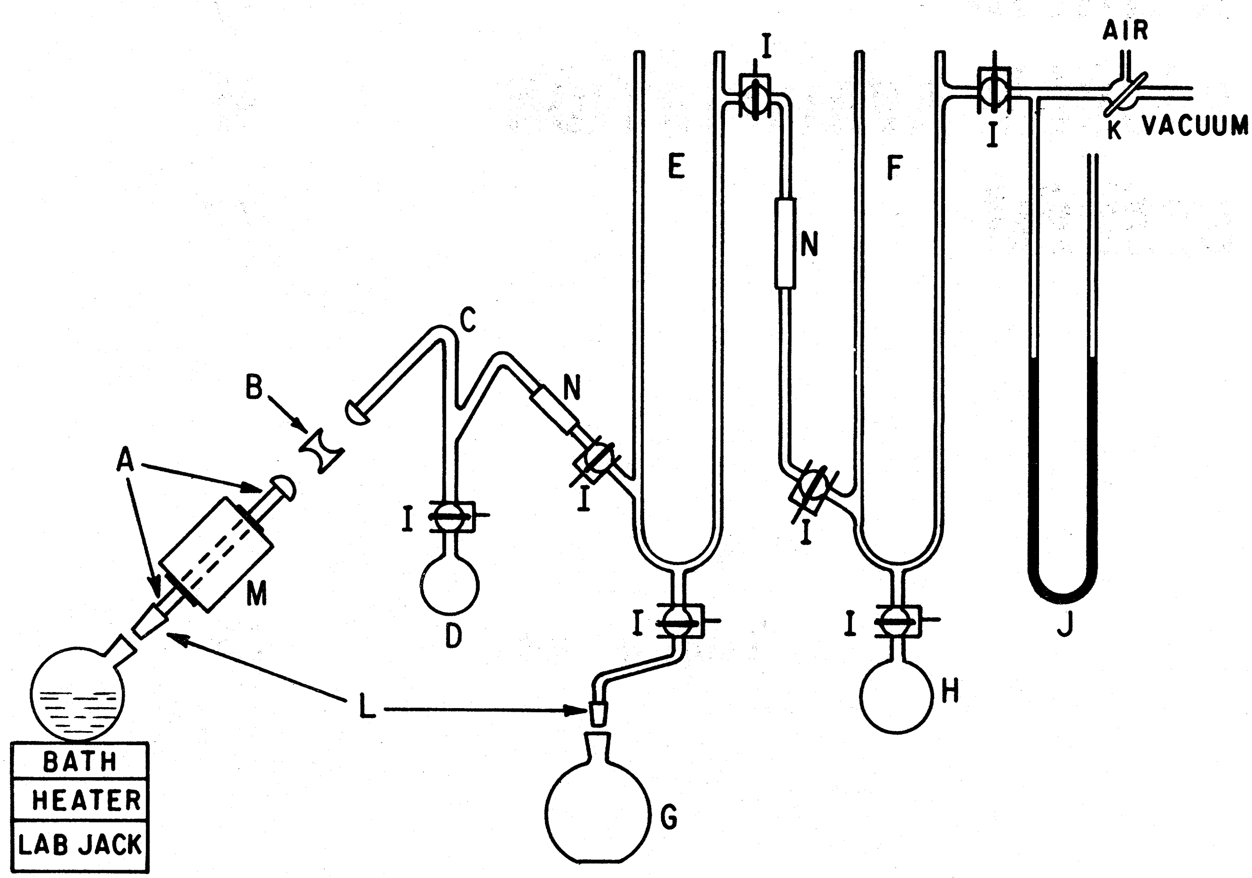

Figure 1.--An efficient evaporation system. (See text for explanation of the parts.)

over the course of several years. It is based on the rotary evaporator method as combined with dry-ice traps. This all-glass system avoids sample contamination from grease, metal, or rubber seals.

The evaporator is constructed of the following units. A motor (M) rotates the glass sleeve (A), which has a 24/40 joint (L) on one end and a ball joint (28/15) on the other end. It is joined to the stationary elbow adaptor (C) through a double-socket, rotating Teflon joint (B). (The motor, sleeve, and Teflon joint were obtained from Buchler Instruments.)1/ It is not necessary to clamp this joint, as the vacuum will hold it sufficiently tight. Two dry-ice traps (E and F) serve to condense the solvent vapors into flasks (G, H). The second trap is connected through a T-joint to a mercury manometer (J) and a three-way stopcock (K), which in turn leads to the vacuum pump or air inlet. Where possible, all units are connected with (28/15) ball joints and socket joints (I) and clamps. The short pieces of rubber tubing (N) alleviate any strain in the system.

Since the system is designed for rapid evaporation of large volumes of sample solution, some sample may inadvertently boil over when the vacuum is increased too rapidly. The elbow adaptor (C) was designed to catch and trap any of this sample in the overflow flask (D). In other evaporators, this material would become mixed with the removed solvent. In this system, the sample is recovered and the system does not have to be dismantled for cleaning and recovery of material. Solvent vapors condense slightly on the sleeve and elbow and gradually wash the material back into the flask and overflow trap (D).

Condensation with dry-ice traps is certainly the most efficient method. Rotary evaporators that use water condensors liberate significant amounts of solvent into the laboratory atmosphere. The first dry-ice trap is usually sufficient to condense most of the solvent, provided the dry-ice cavity is sufficiently large (8 cm. diameter, 45 cm. deep) and the space between the cavity and outer wall does not exceed 1 cm. The solvent is collected in the large round-bottom flask (G). To determine the appropriate vacuum at which a solvent is evaporated, an old-fashioned manometer is attached to the system. Obviously, at the same temperature, methanol requires a greater vacuum than acetone. Good control of the vacuum is an integral part of rapid evaporation. Thus, the manometer is an essential part of the system.

The system operates in the following manner. A round-bottom flask containing solution is joined to the glass sleeve (A) and held in position while the water bath is raised to give the flask partial support. The stopcock to the vacuum pump connection is opened and the drop in vacuum is observed on the manometer (J). After a drop of about 15 to 20 cm. in pressure, the motor (M) is turned on and the vacuum is allowed to rise slowly until the solution boils or a steady stream of solvent is running from the first trap. The stopcock is then occasionally adjusted to maintain the proper vacuum.

1/ Trade or company names are used in this publication solely for the purpose of providing specific information. Mention of a trade name does not constitute a guarantee or warranty of the product by the U. S. Department of Agriculture or an endorsement by the Department over other products not mentioned.

Despite the numerous joints, vacuum down to 1 mm. of mercury can be easily obtained with a good pump. In terms of efficiency, about 5 to 7 liters of organic solvent can be evaporated per hour. At this rate, the condensed solvent runs off the dry-ice trap as a small "stream." The evaporation of water is usually more indicative of the efficacy of a system. With the vacuum "wide-open" and a water bath at a temperature of 55°C; the evaporator removes 1 liter of water per hour from dilute aqueous solutions. We believe that these results substantiate the claimed efficiency of our evaporator. The relatively large diameter of the entire exhaust system (about 1 cm. inside diameter), as compared to the smaller diameters of many commercial evaporators, contributes to the high rate of solvent removal. Three of these units have been constructed and are being used in our laboratory.