SCIENTIFIC APPARATUS AND LABORATORY METHODS

LYOPHILIZATION APPARATUS

THE accompanying diagram (Fig. 1) illustrates a simple, rugged, inexpensive lyophilization apparatus which has been used successfully for moderately large-scale laboratory work.

Approximately 50 pounds of dry ice, broken into small lumps by means of an ice chopper, are required to fill the insulated chamber. Effective cooling is thus maintained without organic solvents and without further attention for at least 24 hours of operation. For shorter periods of lyophilization, the dry-ice requirement can be reduced by a false bottom in the dry-ice chamber.

The cylindrical welded steel receiver is unbreakable and has a condensing capacity of approximately 6 liters. The manifold is constructed of pyrex glass and is connected to the receiver by means of a rubber stopper. Obviously, the design of the manifold and the number of outlets can be varied to suit individual requirements. A useful design consists of four outlets spaced at 90° intervals to which round bottom pyrex-glass flasks of 1- or 2-liters capacity can be attached either by ground-glass joints or rubber stoppers. The flasks are filled to about one third of their

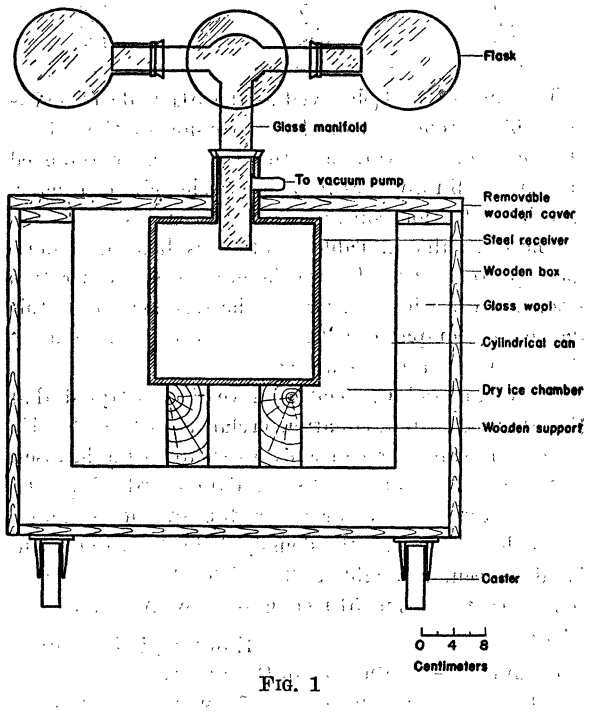

FIG. 1

capacity, and before they are attached to the manifold, are rotated in a dry-ice-solvent mixture to freeze the material in a thin layer.

A pressure of approximately 0.2 mm can be attained rapidly and maintained by means of a mechanical high-vacuum pump of suitable capacity. Approximately 1,200 ml of water (300 ml in each of 4 one-liter flasks) can be removed in 20 to 24 hours.

As suggested by Campbell and Pressman[1] a piece of cotton gauze cemented over the manifold opening effectively prevents loss of small amounts of dry material during lyophilization. Small leaks which may develop in the rubber or ground-glass joints when the system is subjected to high vacuum can be prevented by the liberal application of a sealing compound such as Apiezon Sealing Compound Q.[2] A sealing compound of this type, which is plastic at room temperature, can be applied quickly and easily and can be stripped off and reused. Furthermore, sealing connections from the outside in this manner obviates the use of greases, which may contaminate the material during lyophilization or during removal of the dry material from the flasks.

The usefulness of the apparatus can be increased by providing the cabinet with casters so that it may be moved as required. The vacuum pump, pressure gauge and a cold trap for protecting the pump can also be mounted on a caster-equipped table so that it can be used either with the lyophilization apparatus or with other equipment.

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[1] D. H. Campbell and D. Pressman, SCIENCE, 99: 285, 1944.

[2] May be obtained from the James G. Biddle Company, Philadelphia.

[3] On military leave.

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