A RAPID CONCENTRATING STILL.

W. E. VAN HEYNINGEN.

From the Sir William Dunn School of Pathology, Oxford University.

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IN the apparatus described here aqueous solutions can be concentrated at rates of 12–14 l./hr. at temperatures not exceeding 36° C. Under these conditions many enzymes, bacterial toxins and other substances can be recovered in high yield. No novelty is claimed for this apparatus; it is a laboratory application of the climbing film still used in industry, and it is closely modelled on an apparatus described by Mitchell, Shildneck and Dustin (1944). See also Kemmerer (1945), Ames (1946), Muirhead (1946) and Reavell (1946*a*, *b*). The apparatus is described here because it can be made from parts (mainly standard) supplied by a British firm, and because it is thought that a detailed description of its operation would be useful to those concerned with the isolation of small amounts of biologically active substances from large volumes of solution.

Construction.

The glass part of the apparatus (Fig. 1) is constructed of "Pyrex" industrial glassware supplied by Messrs. James A. Jobling & Co., Ltd., of Sunderland (their Drawing No. 7338/2). *A* is a standard 1 in. Y-piece. (All dimensions are internal.) *B* is a standard Davies type double wall condenser, 35 in. long, with pressed ends of 1 in. bore; it is used as heat exchanger, and steam is passed through the inner and outer water jackets. The heating surface has an area of 235 sq. in. *C* is a standard 1 in. 90° elbow with a $\frac{5}{16}$ in. side tube for holding a thermometer. *D* was especially made from a 5 l. flask with a $1\frac{1}{2}$ in. top neck, a 1 in. bottom neck, and a 1 in. neck entering horizontally at a tangent with its centre 1 in. above the middle of the flask. *E* is a 20 in. length of 1 in. pipe line. *F* is a standard 1 in. T-joint. *G* is a standard 1 in. 45° elbow with a $\frac{5}{16}$ in. side tube; this side tube is generally sealed with a rubber bung. *H* is a $7\frac{7}{8}$ in. length of 1 in. pipe line. *I* is a standard $1\frac{1}{2}$ in. 90° elbow. *J* was especially made from a 1 l. flask with a 2 in. top neck, a 1 in. bottom neck, and a $1\frac{1}{2}$ in. neck entering the flask horizontally at a tangent in the middle. *K* was especially made from $\frac{3}{8}$ in. tube with pressed ends of 1 in. bore; for ease in assembly the vertical part of it is cut into three equal lengths, which are then connected with rubber tubing (not shown on the diagram). *L* is a standard 2 in. U-bend. *M* is a standard 3 in. to 2 in. reducer. *N* is a standard 42 in. length of 3 in. pipe line. All the parts listed above are held together in the usual way by bolts passing through metal joint flanges fitted with graphited asbestos inserts; rubber interface joint gaskets are placed between each pair of pressed ends.

A copper condenser, obtainable from T. J. Maskell, 30A, Jericho Street, Oxford, drawn in Fig. 2 on four times the scale of Fig. 1, is inserted in tube *N*. It consists of four 42 in. copper tubes of $2\frac{1}{2}$ in., 2 in., $1\frac{1}{2}$ in. and 1 in. diameter

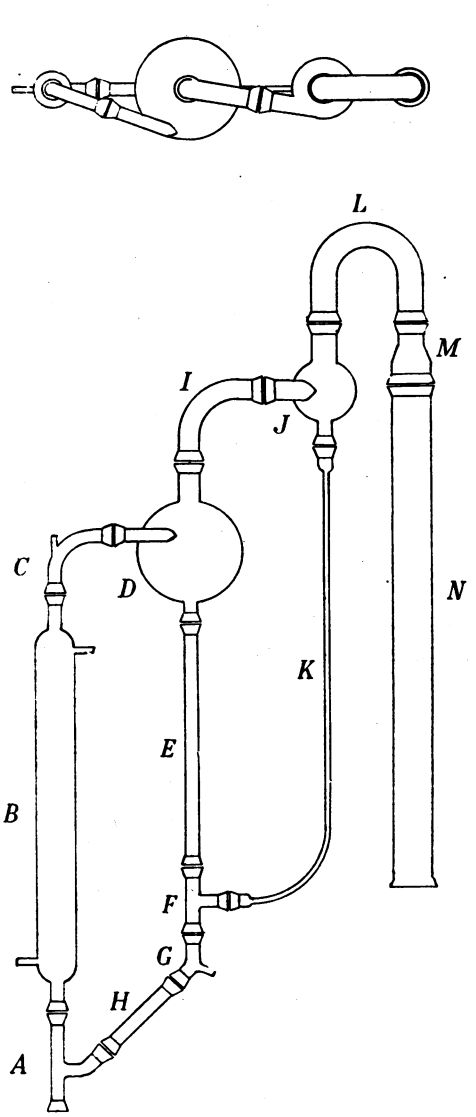

FIG. 1.

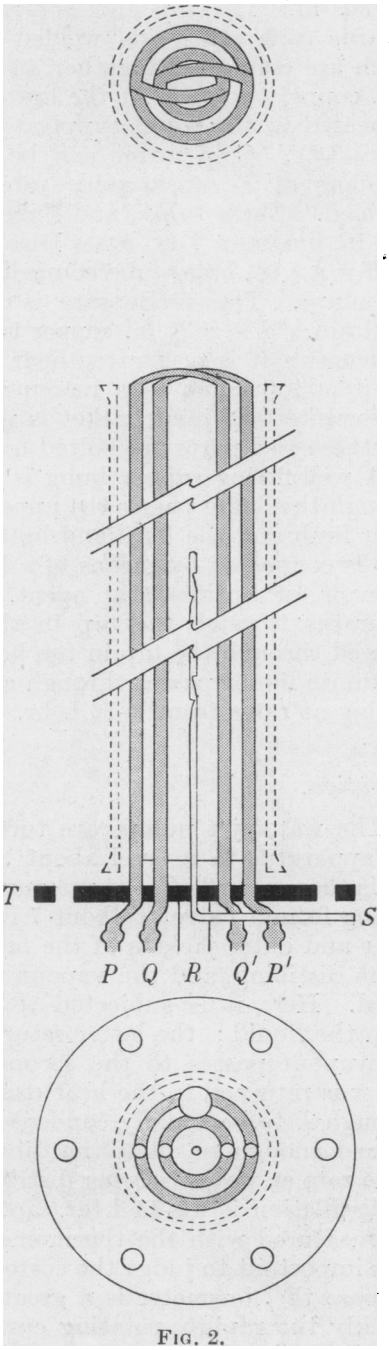

FIG. 2.

arranged concentrically to give four cooling surfaces with a combined area of 920 sq. in. The top and bottom ends of adjacent pairs of tubes are turned towards each other and welded together to form two hollow-walled cylinders which are connected together at the top, as shown, by two welded-in lengths of $\frac{1}{4}$ in. copper tubing. At the bottom ends the hollow walls of these cylinders are connected to the water supply by $\frac{1}{4}$ in. copper Tubes PP' and to the drain by Tubes QQ'. Cold water can be passed through the hollow walls of the two cylinders at a considerable rate and very efficient condensing can thus be attained. These tubes, and Tubes R and S, pass through, and are welded on to, a $6\frac{1}{2}$ in. diameter $\frac{1}{4}$ in. brass Disc T.

R is a $\frac{1}{2}$ in. brass tube connected by means of a Y-piece to two efficient water jet pumps. The condensate is discharged via this tube and the pumps into the drain. S is a $\frac{3}{16}$ in. copper tube reaching about 18 in. into the lumen of the condenser; it is perforated near the top at the condenser end, and at the other end it is connected to a mercury manometer and a vacuum release valve. A rubber interface joint gasket is placed between the end of N and the top of T, and these two parts are bolted together in the usual way.

A well-fitting rubber bung is inserted in the bottom of the Y-piece A, and through this bung passes the upper limb of a + piece of $\frac{5}{16}$ in. glass tubing. The lower limb and the left-hand limb are fitted with $\frac{1}{4}$ in. taps, and the right-hand limb is connected by means of a heavy wall capillary tube fitted with a tap to a reservoir of antifrothing agent. The liquid to be concentrated enters the apparatus through the tap in the left-hand limb, and the concentrate is discharged through the tap in the bottom limb. Before passing into the apparatus the dilute liquor passes through a " Rotameter " flow-meter calibrated for water flowing at rates from 6–16 l./hr.

Operation.

The water jet pumps are turned on and the dilute liquor is drawn up into the apparatus to a level about 3 in. below the bottom of Bowl D. The inlet tap is then turned off, the pressure is brought down to 15–20 mm. Hg., and steam issuing from a valve at about 7 lb. pressure is allowed to pass freely through the inner and outer jackets of the heat exchanger B. The liquid in B immediately starts distilling, and the vapour thus formed forces liquid into Bowl D at great speed. Here it is subjected to centrifugal force which separates the vapour from the liquid; the latter returns to the heat exchanger via $E\ F\ G\ H\ A$ while the vapour passes to the second centrifugal separator J, where any residual liquor is returned to the heat exchanger through K. From J the vapour passes through L to be rapidly condensed in N. After about a minute the inlet tap is opened enough to allow the dilute liquor to enter the apparatus at about the same rate as water is being distilled off. With tapwater at about 15° C. the rate of distillation is 12–14 l./hr., and the temperature of the liquid being distilled (as measured with the thermometer in the elbow C) does not rise above 36° C. It is important to judge the correct rate of inflow of the dilute liquor, and for this purpose the flowmeter is a great advantage. If the dilute liquor is fed in too quickly the rapidly rotating current of water vapour will carry liquid up into the elbow I and constrict it and thus slow down the rate of evaporation. If the dilute liquor is fed in too slowly for a long time there is danger that the con-

centrate will dry in the heat exchanger. When the apparatus was first constructed the parts *E* and *F* were reversed so that the return arm *K* came in at a higher level, as it does in the apparatus of Mitchell *et al.* (1944). With this arrangement there was not a sufficient hydrostatic head to keep an unbroken column of liquid in *K*. The result of this was that vapour rushed through *K*, carrying liquid with it and splashing it up into *L*, whence it was carried over to the condenser by the stream of water vapour. This led to considerable " creep " loss. With the present arrangement no such difficulty is met.

The vapour from *D* enters *J* at tremendous speed—about 150 l. per sec.—which means that it passes through the 1½ in. elbow *I* at 280 miles per hour. The effect of this was seen when, for reasons now irrelevant, a heavy glass stopper was placed in the bottom of Bowl *J*. As soon as distillation started the stream of vapour lifted up the stopper and whirled it around at great velocity for a fraction of a second before hurling it through the side of the bowl.

The centrifugal action to which the distilling liquid is subjected in Bowl *D* breaks down considerable froth, but with very violently frothing liquids it is advisable to introduce 0·5 ml. of an anti-frothing agent, preferably a non-volatile one such as triamylcitrate, at the beginning of the distillation.

A given volume of liquid can be concentrated down to about 750 ml. provided the concentration of total solids is not high enough to render the concentrate unmanageably viscous. When dealing with volumes of the order of 100 l. (*ca.* 0·2 per cent total solids), or with liquids containing high concentrations of solids, it might be advisable continuously to draw off the concentrate through the side tube on *S*. This could be done by attaching a tap and a flask evacuated to a higher degree than the rest of the apparatus. This procedure has not been tried out by the author. It is probably more convenient to stop the distillation at suitable intervals and draw off the concentrate.

Distillation is stopped by first turning off the steam ; after about five minutes, when the heat exchanger has cooled down, the vacuum is released, the water jet pumps are turned off and the concentrate is drawn off. The apparatus can be cleaned as follows : Draw in water up to about half-way in Bowl *D* and reduce the pressure to about 60 mm. Hg. Then open the inlet tap. This results in a violent swirling motion of the wash water, which scours the walls of the apparatus. This is repeated several times with changes of water, and, if necessary, with hot water or with dilute acid or alkali or a detergent. Finally, fresh water is sucked through the entire apparatus for some time and allowed to discharge through the water jet pumps.

SUMMARY.

The construction and operation of a laboratory circulating evaporator which is capable of evaporating water at the rate of 12–14 l./hr. at 34–36° C. and 20–30 mm. Hg is described.

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