CXX. STUDIES ON VACUUM EVAPORATION AND DISTILLATION.

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IN many chemical processes, especially in connection with modern biochemical problems, the rapid evaporation of large amounts of dilute solutions which contain heat-labile substances is necessary. For this purpose vacuum apparatus such as those of Anschütz, Hausmann and others are not efficient enough.

In recent publications the author [1929; 1930, 1, 2; 1931, 1, 2] has described efficient vacuum apparatus for rapid evaporation at low temperature. These vacuum distillation plants¹, working with water-jet pumps and giving yields of 2-10 litres of aqueous distillate per hour at temperatures of 15-20° (see Figs. 1 and 2) have the following principles in common:

(1) A highly efficient cooling system. The good condensation so produced makes for low steam pressure and boiling-point in the evaporating vessel (Fig. 1 *a*, Fig. 2 *a*).

(2) A large diameter of the tube system connecting the evaporation flask and the pump. This decreases the friction, speed and pressure of the steam flow to the lowest possible amount (Fig. 1 *b*, Fig. 2 *b*).

(3) An automatic arrangement so that the solution to be evaporated or distilled is drawn up continuously (Fig. 1 *c*, Fig. 2 *c*) and the distillate is automatically removed (Fig. 1 *d*, Fig. 2 *d*).

(4) A resistant capillary of rustless steel which can be regulated and cleaned by means of a stilette wire (Fig. 1 *e*, Fig. 2 *e*). This prevents any delay in boiling.

The apparatus now described has the further improvement, that in addition to both functions mentioned, the pump water is used also for cooling the condenser. For this purpose there is a direct connection of the outflow pipe with the condenser (Fig. 1 *f*, Fig. 2 *f*). This gives good results and, owing to the syphon action of the condenser, there is no reduction in the efficiency of the pump, if a sufficient water pressure is available. The mechanical work of the pump is not increased, and therefore the additional load of the pump is confined to the greater frictional resistance of the walls in the condenser system. When the pump is stopped the water of the condenser jacket runs into the pump and out into the vessel which may have been connected to receive the distillate. Before stopping the pump therefore either this vessel is removed or a check valve (Fig. 2 *g*) can be fitted to prevent the water reflux. The special water-jet pump working continuously and automatically has therefore three functions: (1) it evacuates the apparatus; (2) it drains off the condensate; (3) it cools the system².

¹ All types of apparatus can be obtained from Messrs H. Cornbleet, 34, East Street, London, S.E. 17.

² Fischer [1931] considers the omission of oil pumps a disadvantage in my apparatus; but this is not in accord with my own observations as explained above. He admits, however, that the removal of the steam by oil pumps is difficult and uneconomical, and that in most cases a condenser cannot be dispensed with. Oil pumps in my opinion therefore should be confined to the domain of high vacuum distillation.

Efforts have been made in the past few years to utilise these advantages of the water-jet pump for industrial purposes as for example by Peterson and the Nederlandsche Installatie Maatschappij Therma [1925] in a British Patent for vacuum evaporation of hot inorganic salt solutions of, *e.g.*, MgSO_4 , Na_2SO_4 *etc.* There is a similarity in the construction principle, with the exception that the condenser is erected before the pump. This arrangement however seems not to be so good because the pre-heated pump water has a higher vapour pressure which prevents an optimum vacuum.

Fig. 1. Standard model of a vacuum evaporator and still for an efficiency of about 2 litres per hour at 20°.

Fig. 2. Vacuum evaporator with seven-tube condenser and a continuous automatic efficiency of 5-8 litres per hour at 20°.

The triple function of the water-jet pump is greatly extended by using a larger apparatus with seven-tube condenser (Fig. 2).

This apparatus consists of a special pump (Fig. 2 *h*) and two ordinary pumps (*i*, *k*) as auxiliary pumps, the outflow pipes of which are joined to a single tube connected to the condenser (*f*). When the evaporation is begun all three pumps are working together for the purpose of more rapid evacuation. When a suitable vacuum has been attained, the auxiliary pumps (*i*, *k*) are stopped and the evacuated system is blocked by a check valve (*g*), so that water reflux is prevented. The apparatus is then worked with this single special pump, which, notwithstanding its small size, is sufficient for fulfilling the three functions: maintaining the vacuum, draining off the condensate and cooling. The apparatus has an efficiency of 5 litres water per hour at 20°.

The economical working of the larger model is important in technical work, when it is desired to increase the evaporation efficiency to more than 5 litres per hour.

The condensation of 5 kg. of steam per hour with the use of only 300 litres of cooling water supplied by one water-jet pump is possible on account of the great surplus of the cooling surface; actually the maximum efficiency of the condenser is considerably higher. Experiments with a single copper tube condenser (Fig. 1 *a*) have shown that the condensation of 4–5 kg. of steam per hour is possible with 300 litres of cooling water. Since the larger condenser (Fig. 2 *a*) consists of seven copper tubes of the same diameter and length with a sectional cooling water surface approximately 19 times greater, an efficiency of about 30 litres per hour can be expected by using 2000 litres of cooling water. Under ordinary laboratory conditions this can hardly be effected owing to lack of sufficiently powerful heating, approximately 18,000 Cals. per hour being necessary for evaporating 30 litres water at 20°. By heat supplied directly by means of a large ring burner with 24 flames (Fig. 2 *l*) to a tinned iron boiler of 30 litres capacity, efficiencies of about 10 litres per hour have been attained.

Smaller improvements can be attained by tinning the inner surface of the condenser¹ or by using chromium-nickel-steel resistant to acid vapours and also by a conical intermediate piece connecting the evaporation vessel and the metal connecting tube (Fig. 2 *m*) as suggested independently by Hansen [1933]. As regards the boiling capillary made of rustless steel, it is to be noted that the alloy (V2a Krupp-Stahl) was corroded not only by hydrochloric acid, but also by more concentrated cold sulphuric acid. It has also been ascertained that a capillary tube can often be dispensed with when working with continuous inflow, since the inflowing solution contains sufficient absorbed air to prevent delay in boiling. After all the solution is in the vessel, the inflow tube can itself serve as a boiling capillary (Fig. 1 *c*, Fig. 2 *c*). But the correct regulation of the apparatus, especially at the moment when all the solution has run into the flask, demands considerable practice in order to prevent the sputtering of the solution consequent on the violent entrance of air. A boiling capillary is therefore preferable even on a continuously working apparatus.

SUMMARY.

The special water-jet pump for continuous automatic working previously described by the author has been improved, so that three functions, evacuating, cooling and removal of the condensate, are fulfilled by this one special pump.

A large apparatus has also been devised fitted with a seven-tube condenser and three pumps, in which, when a full vacuum has been obtained, a single special pump suffices for maintenance of the vacuum, cooling and draining off the condensate at an efficiency of 5 litres per hour at 20°.

The efficiency of this larger apparatus can be increased to 10 litres water per hour at 20° by using a vessel of 30 litres capacity which is heated directly. Efficiencies up to 30 litres per hour may be attained if sufficient increase of heat in proportion to the condenser capacity can be applied to the boiling vessel.

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¹ In one case the apparatus was silvered, which was scarcely more expensive but possibly more resistant than the tinning process.

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