

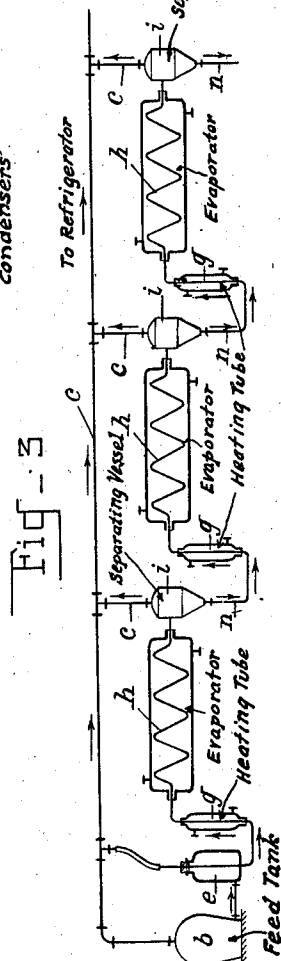
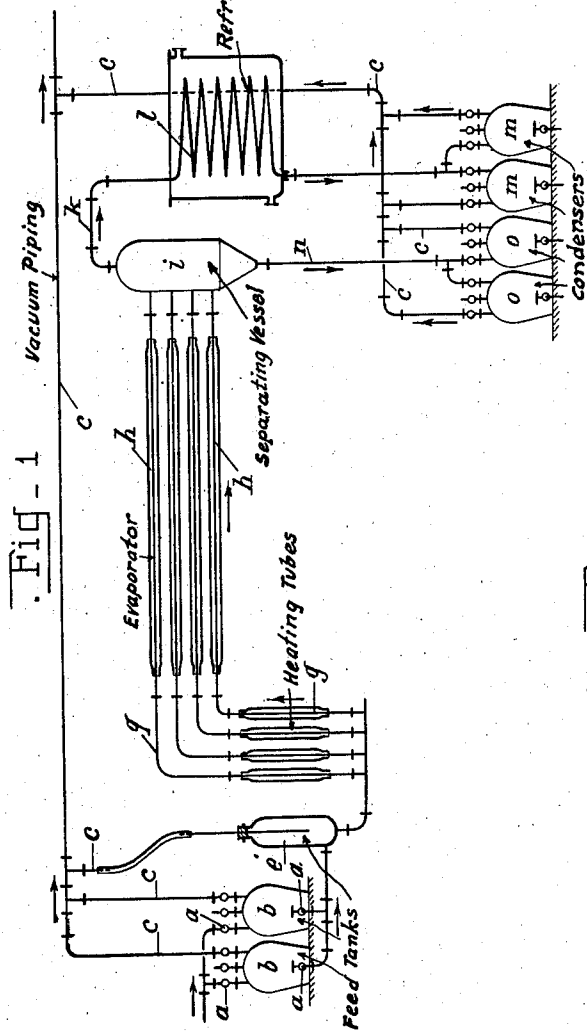
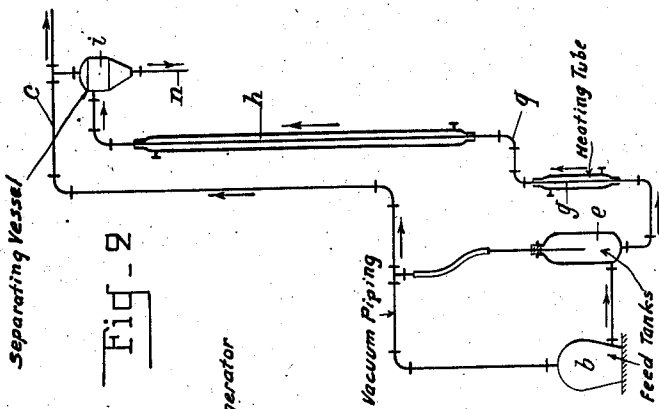
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PROCESS FOR VACUUM DISTILLATION OF HYDROGEN PEROXIDE

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PROCESS FOR VACUUM DISTILLATION OF
HYDROGEN PEROXIDE

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1 Claim. (Cl. 202—52)

The object of the present invention is a process for the vacuum distillation of easily decomposable liquids, together with an apparatus to perform the same.

5 This operation has always encountered serious difficulties in practise. In particular it has always been found extremely difficult to obtain hydrogen peroxide by vacuum distillation, when starting from aqueous solutions of compounds
10 containing active oxygen, due chiefly to the facility with which hydrogen peroxide decomposes in the presence of peracids.

The fact that the vapour pressure of hydrogen peroxide is lower than that of water also causes
5 grave difficulties. These have necessitated maintaining an optimum ratio between the quantities of water and hydrogen peroxide to be evaporated simultaneously. On the other hand it is very
10 important to prevent the solidification of the reagents owing to the evaporation of water proceeding too far.

In the processes hitherto known, in which the liquid to be distilled is sucked through the evaporator by means of vacuum, and its quantity regulated by means of injectors, cocks or capillary
25 tubes, the conditions prevailing in the evaporator are always irregular. These drawbacks are particularly applicable to the working of large industrial units formed of a great number of
30 evaporating tubes.

The present invention consists in a process of evaporation such that the same degree of vacuum is maintained at every point in the apparatus, all the parts of which (as for instance feed-tanks,
35 evaporators, collecting vessels etc.) are connected to the same vacuum pipe line. By this means the process is carried out in such a manner that the evaporator throughout the whole apparatus is always uniform.

40 In the preferred manner of putting the invention into execution, the movement of the liquid in the apparatus is not obtained as formerly by applying a difference of pressure between the inlet and the outlet, but by employing a difference of temperature. To effect this one employs
45 a system of communicating vessels, one of which is a reservoir of any desired shape and capacity, the other being one or more heated tubes. The difference in temperature thus established between the part of the apparatus which is heated
50 and the part not heated results in imparting a movement to the liquid of any desired speed simply by regulating the temperature of the hot tube or tubes which are situated between the
55 feed-tank and the evaporator. This speed is best

regulated to coincide with that which will give sufficient bubbles of steam to cause the solution to enter the evaporator in the form of an emulsion and not in the form of a liquid layer, thus in turn causing the entire surface of the evaporating
5 tubes to be filled with a small quantity of solution. According to the invention one maintains uniform conditions in the evaporator by maintaining the same reduced pressure at the inlet and the outlet of the said evaporator, by employ-
10 ing a speed for the circulation of the solution which is independent of the suction by vacuum, and lastly by maintaining the physical conditions of the solution homogeneous. The apparatus for putting the present invention into execution con-
15 sists essentially of an evaporator of any desired number of tubes placed in any desired position.

The following working examples will serve to make perfectly plain the preferred methods of putting the invention into execution.

Fig. 1 represents the main outlines of an apparatus according to the invention.

Fig. 2 represents the drawing of a portion of another mode of execution of the apparatus.

Fig. 3 represents the main outlines of a third
25 alternative.

In Figure 1 a uniform vacuum is obtained throughout the entire apparatus, due to the feed-tanks *b* and *e* and also the receivers *o* and *m* being joined to the same vacuum piping *c*. The
30 cocks closing the feed-tanks permit of one tank being connected up to the vacuum, while the other tank is shut off and refilled.

The feed-tanks may supply any desired number of evaporating tubes. Between the feed-tank and
35 the evaporator *h* is placed the system controlling the rate of feed, consisting of the vessel *e* and a series of tubes *g*. A constant level is maintained in the vessel *e* by any desired means, for example the vessel may be constructed on Mari-
40 otte's principle with the added peculiarity that the tube which dips into the vessel is connected through a flexible connection to the vacuum piping so that a regulation of the level in vessel *e* may be obtained by modifying the height of the
45 tube in the vessel.

As soon as one commences to heat the tubes *g* the liquid which is at a certain height begins to ascend and the emulsion enters the evaporator. The vapour separates from the liquid in the vessel
50 *i*, and then passes through piping *k* and the refrigerator *l* to the receivers *m* where the hydrogen peroxide is collected. The residual liquid flows from the vessel *i* into the receiver *o*, connected to the vacuum system operating the whole plant.

In Fig. 1 the connecting tubes *q* are shown diagrammatically one above the other. They may also be placed in the same horizontal plane or may again meet in a tubular bunch of cylindrical section.

Further the position of the evaporating tubes *h* need not be horizontal, for these tubes may, if so desired, be inclined vertically (as in Fig. 2), or even take the form of a coil.

In Fig. 2 the tube *g* destined to produce the circulation of the liquid is shown joined to the evaporating tube by an elbow *q*, but the connecting tube *q* can equally well be straight, or again be so formed that tube *q* and *h* are in one piece, the vacuum being applied both at the inlet and outlet of this single tube. At the same time it is considered generally preferable that the two tubes, one for producing the circulation and the other for the distillation, should be separate, as this increases the ease with which they can be maintained at different temperatures.

The present invention lends itself particularly well to distillation in which the vapour is eliminated at two or more separate points in the evaporator. This manner of working has the advantage of withdrawing the vapour formed away from the zone of heat, and out of contact with the solution.

As a result of the desired pressure under which the apparatus is working being identical at all points and the speed of circulation of the solution being regulated by the heat applied, one always obtains a regular distillation with a high yield in the different steps of this operation.

In Fig. 3 an apparatus is shown in which there are three exits for the vapour. The liquid coming from the feed-tank *b* passes through a first heating tube and a first condenser, exactly as in the apparatus outlined in Fig. 1. After having passed out through the separating vessel *i* the vapour passes through the piping *c* to the refrigerator, while the liquor enters the second stage

of the evaporation through piping *n* and a second heating tube *g*. This second stage of evaporation is then followed by a third or even further similar stages, each with a similar lay-out. Finally after the last separating vessel the liquid is sent to the receiver connected to the vacuum piping.

It will be seen that in all the above described embodiments the vacuum has absolutely no effect upon the movement of the liquid by reason of the fact that all of the vessels, for instance, *b*, *e*, *g*, *i*, *o* and *m* are connected to the vacuum *c*. Accordingly the same absolute pressure exists in the entirety of the arrangement and no effect of pressure or lack of pressure can play any part in order to cause a displacement of the liquid.

Thus the displacement of the liquid is produced by the sole effect of the temperature to which the tube *g* is subjected.

Having now particularly described and ascertained the nature of my invention and in what manner the same is to be performed, I declare that what I claim is:

Process for the preparation of hydrogen peroxide by vacuum distillation of solutions containing active oxygen, which comprises passing the solution to be distilled from a feed tank through ascending tubes into a tubular evaporator, the ascending tubes being heated in such a manner that a continuous movement of the liquid to be distilled from the feed tank into the tubular evaporator is engendered solely by the action of the heat difference between the feed tank and the ascending tubes, all these parts, as well as the rest of the apparatus being throughout subjected to substantially the same degree of reduced pressure, the elements at the two ends of the apparatus, namely, the receiver and the feed tank, being for this purpose connected separately and directly to the same vacuum system.

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