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APPARATUS FOR HIGH-VACUUM DISTILLATION

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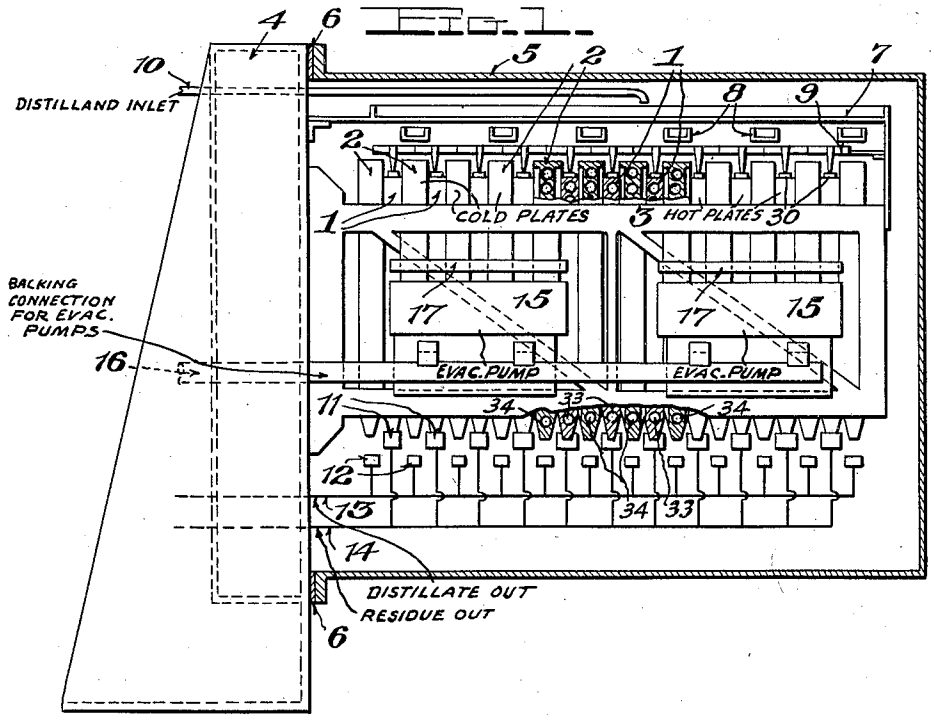

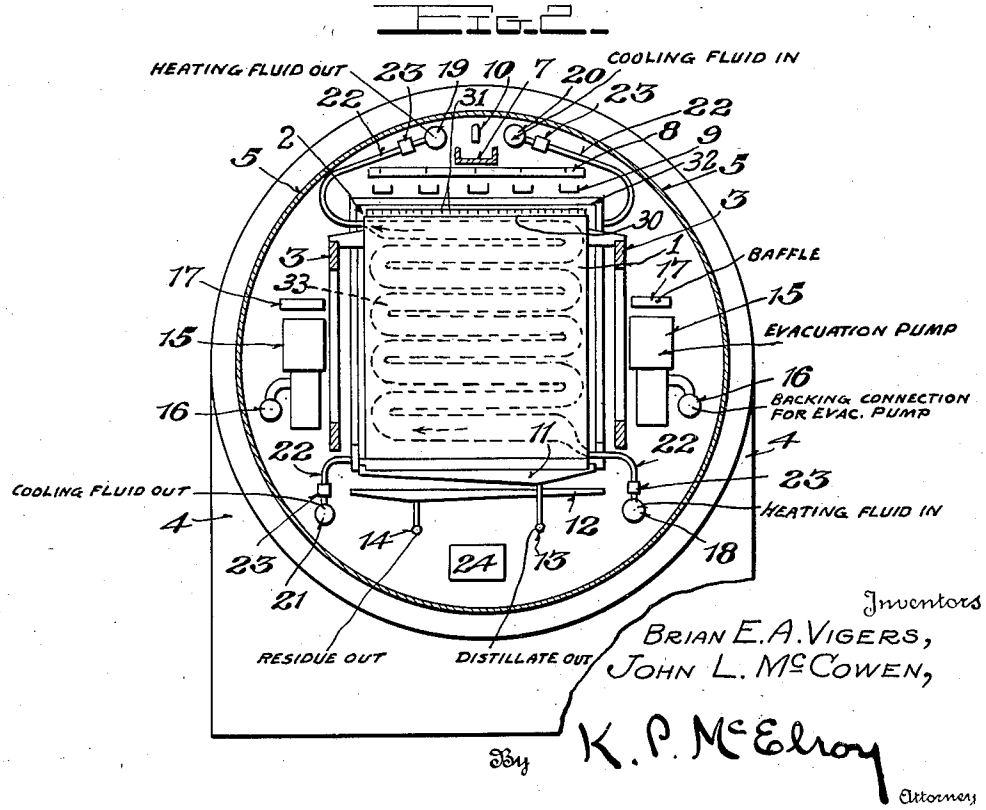

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APPARATUS FOR HIGH-VACUUM DISTILLATION

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7 Claims. (Cl. 202—205)

(Granted under the provisions of sec. 14, act of March 2, 1927; 357 O. G. 5)

The invention relates to an improved apparatus for carrying out short-path high-vacuum distillation.

The general conditions for carrying out distillations of this type are, firstly that the distillation is effected under an extremely high vacuum of the order of 10^{-2} to 10^{-6} mms. of mercury, and secondly that the evaporating and condensing surfaces inside the still are in close proximity, usually about 1 to 5 cms. apart. It is known to construct the evaporating and condensing surfaces as vertical surfaces, down which flow respectively the liquid to be distilled and the condensate, either by employing a concentric tube arrangement or by employing flat-surfaced plates or blocks. In such apparatus it is desirable that there should be ready access to the interior and working parts of the still including all joints subjected to high vacuum on one side, that the minimum number of such joints should be used, and that the various control points should be conveniently situated. The present invention provides a construction of apparatus which meets these desiderata, and is, in general, an improvement over existing types of still.

According to the invention, an apparatus for effecting short-path high-vacuum distillation comprises a number of parallel plates or blocks, the faces of which provide the evaporating and condensing surfaces, the said plates or blocks being supported in a vertical or inclined position, from a headstock, that is a combined support and closure member, through which substantially all pipe and other connections necessary for the working of the still are made, while the assembly of plates or blocks together with their attendant feeding and collecting apparatus is enclosed by a vacuum-tight casing, preferably a cylindrical casing, making a single vacuum-tight joint at the headstock. The plates or blocks are suitably formed with tubular or hollow passages in order to permit of the passage of heating or cooling media. Where the headstock is arranged vertically and the casing is horizontal, it may be mounted on wheels running on rails, so that it may be easily retracted for inspection.

An advantageous form of the invention will now be described with reference to the accompanying drawing in which Figures 1 and 2 are diagrammatic sectional side and end elevations respectively of the apparatus.

The evaporating plates 1 and the condensing plates 2 are carried in the desired positions by the cantilever truss 3, which is attached to the headstock frame 4. The whole assembly of plates is enclosed by a cylindrical casing 5, which makes a vacuum-tight joint at 6 with the headstock. The headstock thus serves as a closure for the casing as well as a support for the plates.

The arrangement of rectangular plates in a casing of circular cross-section provides four segmental spaces around the plate assembly. In the upper segmental space are provided troughs 7, 8 and 9. The material to be distilled is fed into the main trough 7 by the pipe 10 passing through the headstock 4, and is thence distributed by the troughs 8 and 9 to the tops of the evaporating plates 1. Troughs 30 are provided integral with or attached to the tops of the evaporating plates and the feed to the latter is by a series of weir notches 31 (Fig. 2) along each side of the top in order to provide even distribution over both faces of the plates. It is advantageous to have the condensing plates rather larger than the evaporating plates, the top edges of the former having ridges as indicated at 32 arranged to act as baffles to catch molecules escaping from the ends of the evaporating plates, and from the gap between the plates, into the evacuated space, which would not otherwise strike the cooling surfaces. This arrangement of the condensing plates also facilitates the attaining of the necessary high vacuum.

In the lower segmental space are provided collecting troughs 11 and 12 respectively for the residues from the evaporating plates and distillates from the condensing plates. These troughs are drained respectively by the residue manifold 13 and the distillate manifold 14, which are led through the headstock to extraction pumps (not shown). The troughs 11 are positioned close to the hot plates and the troughs 12 are located on a lower level in order to reduce heat transfer by radiation from the hot residue to the cold distillate. Radiation may be further reduced by interposing bright metal foil radiation screens between the troughs 11 and 12.

In the segmental spaces at the sides of the plate assembly the first stage or stages of evacuation pumps 15 may be arranged. Such pumps may be of the molecular diffusion or condensation type, supplemented by a backing pump, the backing connections 16 being carried out through the headstock and connected to an appropriate backing pump (not shown), for the purpose of producing as high a vacuum as possible within the apparatus. Cooled baffles 17 may be provided over the intakes of the pumps 15.

Hot oil for heating the evaporating plates is introduced by the pipe 18 and removed by the pipe 19. The plates are shown as having tubular

passages **33** for conducting the hot oil (Figs. 1 and 2). Cold water for cooling the condensing plates is introduced by the pipe **20** and removed by the pipe **21**. The condensing plates are similarly formed with tubular passages, **34**, for the cooling water. These pipes **18, 19, 20** and **21** come out through the headstock, although not shown in Figure 1. The heating and cooling media are conducted to their appropriate plates by pipes **22**, which are preferably of considerable length in order that their elasticity may permit of slight movements due to temperature changes. For ease of dismantling, joints **23**, preferably of the metallic lens ring type, may be introduced in the pipes **22**.

The above construction and assembly of distillation apparatus can be applied for the purpose of a multi-stage still, in which case the necessary pumping arrangements are provided for transferring distillate or residue or both from one stage to another, such pumps being located in the bottom segmental space at **24**. These pumps may be driven by electrical means, or by means of a common driving shaft brought out through the headstock by a gland and connected to a suitable source of power.

Various modifications of the apparatus hereinbefore described may be made in carrying out our invention, and all such modifications are intended to come within the scope of the appended claims insofar as they achieve to a useful degree the improvements and advantages hereinbefore disclosed.

We claim:—

1. Apparatus for carrying out short-path high vacuum distillation, comprising a plurality of plate-like members in parallel arrangement and having opposed substantially vertical parallel faces, means for heating alternate members so as to provide evaporating surfaces, means for cooling the members intermediate the heated members so as to provide condensing surfaces opposed to said evaporating surfaces, a casing and a closure member making a vacuum-tight joint with the casing, the casing and closure member defining a vacuum-tight chamber surrounding the plate-like members, means for supporting said plate-like members on said closure member, conduit means extending through the closure member into the chamber for feeding raw material to the upper portion of the heated members, and means for collecting distilled material from the cooled members.

2. Apparatus for carrying out short-path high vacuum distillation, comprising a plurality of rectangular plate-like members having opposed, parallel faces arranged for flow of liquids downward thereover by gravity, means for heating alternate members to provide evaporating surfaces, means for cooling intermediate members to provide condensing surfaces, a housing for said members, troughs provided above the evaporating members and arranged to deliver liquid to be distilled over the surfaces of the evaporating members, a closure member for the housing arranged to support said plate-like members, the closure member and housing defining a vacuum-tight chamber surrounding the plate-members, collecting troughs below the members arranged to collect separately the liquid condensate and residue from the condensing members and evaporating members respectively, and conduit means communicating with said troughs and passing through the closure member for separately withdrawing condensate and residue.

3. The matter of claim 2 wherein the troughs for collecting the condensate are located at a lower level than the troughs for collecting the residue, so as to reduce heat transfer by radiation from the hot residue to the cold distillate.

4. The apparatus of claim 1 wherein at least some of the plate-like members are provided with interior passages for circulation of temperature regulating fluid, and conduit means are provided extending through the closure member and inside the casing for supplying said plate-like members with the fluid.

5. The matter of claim 1 wherein the condensing members are provided with ridges at their top extending beyond the tops of the evaporating members and serving as baffles to catch molecules escaping from the ends of the evaporating members and from the gap between the members.

6. The apparatus of claim 1 wherein there are provided evacuating pumps in the casing adjacent the sides of the plate-like members and conduit connections for the pumps extending out through the closure member, and arranged to be put in communication with backing pumps.

7. The apparatus of claim 1 wherein there are provided evacuating pumps in the chamber adjacent the sides of the plate-like members, conduit connections for the pumps extending out through the closure member, for connection to backing pumps, and cooled baffles arranged over the intakes of said evacuating pumps.

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