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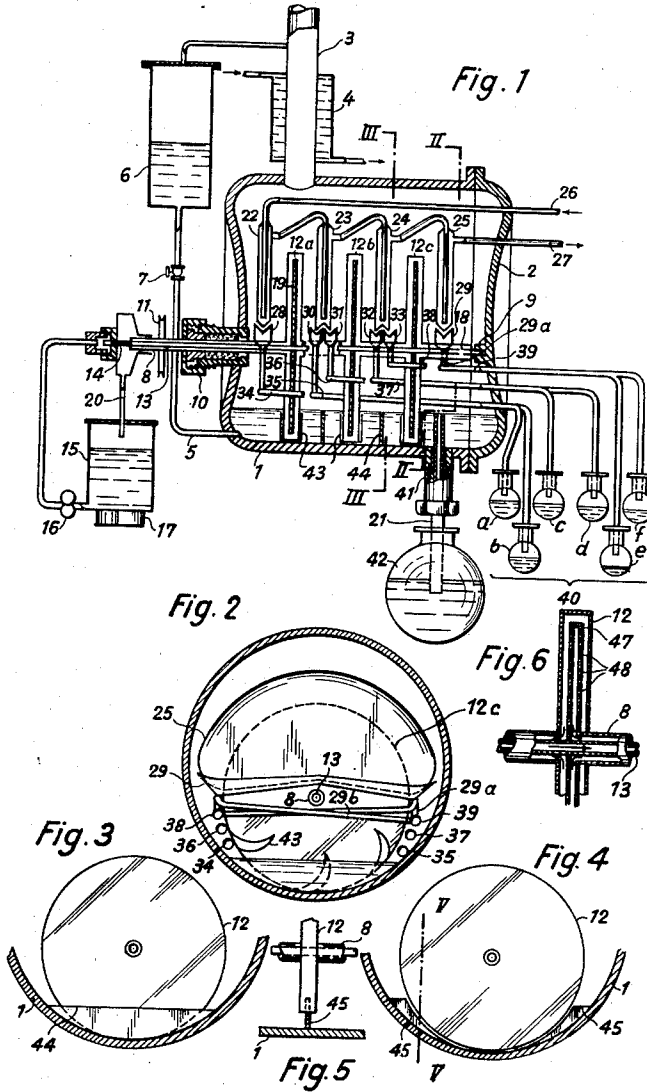
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HIGH VACUUM DISTILLATION APPARATUS

Filed April 30, 1956

2 Sheets-Sheet 1



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2 Sheets-Sheet 2

Fig. 7.

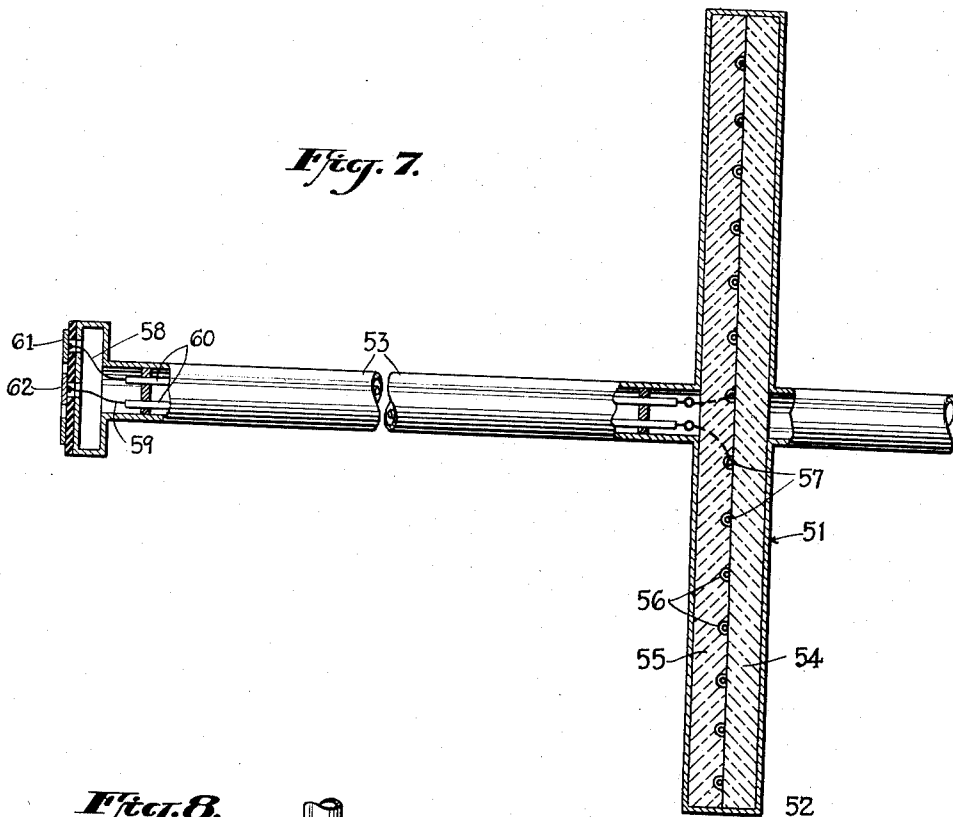
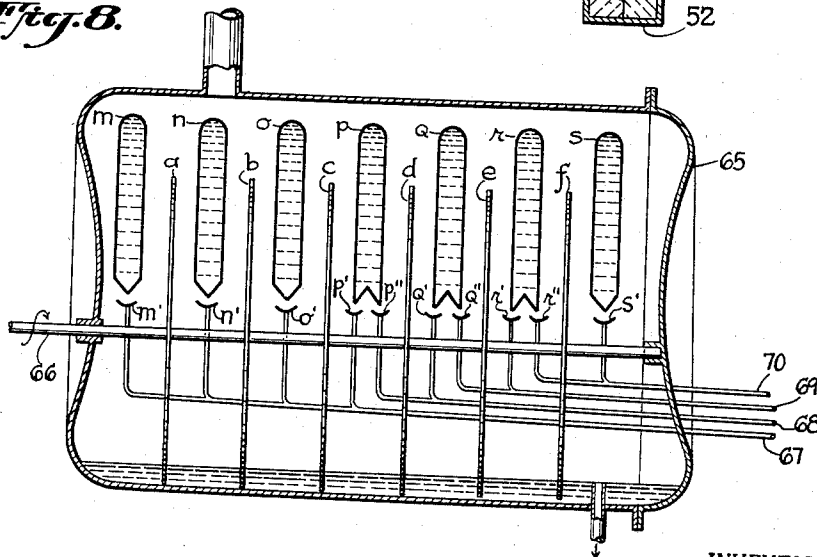


Fig. 8.



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## HIGH VACUUM DISTILLATION APPARATUS

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1 Claim. (Cl. 202—205)

The present invention relates to high vacuum distillation apparatus—particularly to apparatus for carrying out molecular distillations in which the distance between the evaporation surface and the condensation surface is smaller than the mean free path of the vapor molecules.

The invention is concerned with improvements of high vacuum distillation apparatus of the type in which a rotating horizontal shaft is provided inside a vacuum container, a plurality of discs being mounted on the shaft in spaced relation and at a right angle to the rotation axis of the shaft. The discs are at least partly heated to different temperatures and are partly immersed in the liquid to be distilled which is at the bottom or sump of the vacuum container for picking up the liquid upon rotation of the discs. The lateral surfaces of the discs form evaporation surfaces which are individually opposed to stationary condensation surfaces. The latter are formed by the lateral walls of pockets which are individually interposed between the aforesaid discs, a coolant flowing through the interior of the pockets. Means are provided for continuously supplying the substance to be distilled into the container and for removing the distillation residues from the container.

The invention is characterized by the provision of two troughs beneath the cooling pockets which are interposed between differently heated discs for individually receiving the distillate dripping from the lateral walls of the pockets. Preferably separate collecting and removing conduits are provided for the distillate collected in the individual troughs. In some cases it may be advisable to provide a single collecting conduit for the distillate received in two troughs from distilland evaporated on the two sides of the same disc. In an apparatus in which separating walls are provided in the sump of the vacuum container between the discs additional walls may be provided which walls envelop the marginal portions of the discs and form with the latter a clearance or gap, the additional walls and the separating walls dividing the spaces in the sump of the container between the discs into individual chambers.

The novel features which are considered characteristic of the invention are set forth with particularity in the appended claim. The invention itself however and additional objects and advantages thereof will best be understood from the following description of embodiments thereof when read in connection with the accompanying drawing in which

Fig. 1 is a schematic longitudinal sectional illustration of an apparatus according to the invention for molecular distillation;

Fig. 2 is a cross sectional view of the apparatus shown in Fig. 1, the section being taken along line II—II in Fig. 1;

Fig. 3 is a cross sectional view along line III—III of the lower part of the apparatus shown in Fig. 1;

Fig. 4 is a cross sectional view of a modified detail of the apparatus shown in Fig. 1;

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Fig. 5 is a sectional view of the detail shown in Fig. 4, the section being taken along line V—V in Fig. 4;

Fig. 6 is a large scale sectional view of an evaporating disc;

Fig. 7 is a large scale sectional view of a modified evaporating disc;

Fig. 8 is a diagrammatic longitudinal sectional view of a modified apparatus according to the invention.

The same numerals designate the same parts in all figures.

Substances having a high boiling point can be decomposed by high vacuum distillation at temperatures which are below the decomposition point of the substances. In processes known as molecular distillation, distillation is effected at such a vacuum that the length of the mean free path of the vapor molecules is greater than the distance between the evaporation surface and the condensation surface. These processes are based on the consideration that the maximal possible distillation velocity can be obtained, if provisions are made ensuring that each molecule leaving the evaporation surface reaches the condensation surface and is held thereat without bouncing into other molecules in the vapor space.

Figs. 1 and 2 illustrate an apparatus which is suitable for performing molecular distillation. A vacuum container including a horizontal cylindrical vessel 1 and a removable cover 2 is connected by a pipe 3 with a conventional vacuum pumping plant, not shown. The latter usually includes a diffusion pump upstream of which is a pre-vacuum pump and which must be adapted to maintain very low pressures, as a rule less than 10<sup>-2</sup> mm. Hg, within the vacuum container. The pipe 3 may be cooled by a cooler 4 so that vapors in the pipe 3 are condensed and prevented to enter the vacuum pumping plant. A pipe 5 terminates in the lower part of the vacuum container which will henceforth be called the sump. The pipe 5 which is provided with a valve 7 connects the interior of the vacuum container with a reservoir 6 for the liquid to be distilled; the latter will henceforth be called distilland. A discharge conduit for the distillation residues is vertically movable in a stuffing box 41 and terminates in a flask 42. By vertical displacement of the pipe 21 the level of the distilland in the container 1 can be changed.

A hollow shaft 8 having one end rotating in a bearing 9 extends through the interior of the vacuum container; the other end of the shaft 8 extends through the wall of the vessel 1 to the outside, a stuffing box 10 being provided for preventing entry of air into the vacuum container. The shaft 8 can be rotated by driving a drive wheel 11 mounted on the shaft 8 by conventional means, not shown.

A plurality of hollow discs 12a, 12b, 12c are mounted in spaced relation on the shaft 8, the discs being partly immersed in the distilland in the sump of the vacuum container. The discs 12 are provided with scoops 43. A heating liquid is pumped by a gear pump 16 from a reservoir 15 in the direction of arrow 14 through a pipe 13 located inside the hollow shaft 8. The liquid in the reservoir 15 is heated by a heater 17 to a desired temperature. The heating liquid enters the space between the pipe 13 and the hollow shaft 8 through apertures 18 in the right end of the pipe and flows into the interior of the hollow discs 12. Baffles 19 inside the discs 12 force the heating liquid to flow along the insides of the lateral walls of the discs 12. The heating liquid flows consecutively through the discs 12c, 12b, 12a and is returned to the reservoir 15 through a conduit 20.

Stationary cooling pockets 22, 23, 24, and 25 are interposed between and on the outside of the outermost evaporation discs 12. A coolant is conducted to the pockets

22 to 25 through a conduit 26 and removed therefrom through a conduit 27. The lower marginal portion of the pockets 22 and 25 is wedge shaped and extends into troughs 28 and 29, respectively, for catching the condensate formed on the surfaces of the pockets 22 and 25 which surfaces face the discs 12a and 12c, respectively. The pockets 23 and 24 which are interposed between the discs 12a, 12b and 12b, 12c, respectively, are provided with two spaced edges at their lower ends so that the distillate condensed at the two sides of the pockets is not mixed. A separate trough is provided for each edge, the troughs for the pocket 23 being designated by numerals 30 and 31 and the troughs for the two edges of the pocket 24 being designated by numerals 32 and 33. As seen in Fig. 2 the troughs have a relatively high elevation at the center and a lowest point at either end. These lowest points are connected with distillate collecting conduits. The distillate caught in the trough 29 which distillate comes from the side of the pocket 25 facing the disc 12c flows through conduits 29a and 29b into a collecting tube 39. Similarly, the condensate collected in the troughs or drip trays 28, 30, 31, 32, and 33 flows into collecting conduits 34, 35, 36, 37, and 38, respectively. The conduits 34 to 39 extend through the cover 2 to the outside and terminate in flasks 40a, 40b, 40c, 40d, 40e, and 40f.

The apparatus operates as follows: Upon rotation of the shaft 8 a layer or film of liquid is formed on the discs 12a, 12b, and 12c which are partly immersed in the distilland; this action is assisted by the scoops 43 which are arranged at the marginal portions of the discs. The sides of the latter act as evaporation surfaces. The vapor molecules released from the liquid film are caught by the side surfaces of the cooling pockets 22 to 25 which are opposed to the respective sides of the discs. The condensate flows into the drip trays and therefrom through the collecting conduits into the respective flasks.

The arrangement of the drip trays and connecting and collecting conduits according to the invention affords increased selectivity and differentiated action of the disc stages. Due to the reduction of the temperature of the heating agent while it consecutively flows through the individual evaporation discs the average temperatures of the lateral surfaces of the discs become lower and lower as they are heated by the flowing and cooling heating agent. The distilland flows consecutively through the spaces formed by the discs in the sump. The disc 12a scoops distilland which spreads over the lateral surfaces of the disc. The temperature of the left side of the disc 12a is lower than that of the right side. Therefore, the most volatile substances contained in the distilland evaporate at the left side of the disc 12a. The liquid film on the right side of the disc 12a is formed of somewhat less volatile substances whereby the percentage of the less volatile portion of the distilland to be evaporated on the right side of the disc 12a is increased. The liquid film on the right side of the disc contains less volatile substances than the matter evaporated at the left side of the disc. Due to the separate removal of the distillate condensed on the pocket 22 and on the left side of the pocket 23 a continuous distillation can be performed at a highly selective fractionation.

By suitable control of the flow of the heating agent inside the discs 12 the temperature of both sides of the discs will be substantially the same. Fig. 6 illustrates a structure in which, instead of a central baffle 19 a hollow body 47 is arranged the interior of which communicates with the hollow shaft 8. Apertures 48 in the lateral walls of the body 47 distribute the heating agent over the inside of the side walls of the disc 12 in such a manner that the temperature of the outside of both side walls is substantially the same. In this case a common collecting conduit can be provided for the distillate collected in two drip trays, for example 28 and 30 in Fig. 1, and resulting

from vapor produced on the side surfaces of the same disc, for example 12a. Since the temperature of both side surfaces is the same, the distillate collected in both trays is the same.

The discs may be heated electrically instead of by means of a heating fluid and means may be provided for heating both sides of the discs to the same temperature.

An apparatus of this type is shown in Figs. 7 and 8 in which numeral 51 designates a disc corresponding to one of the discs 12 in Fig. 1. The disc 51 includes two disc shaped ceramic insulation elements 54 and 55, the latter being provided with a spiral groove 56 into which an electric heating wire 57 is placed. The elements 54 and 55 are encased in an envelope 52 made of stainless chrome-nickel steel and mounted on a hollow shaft 53 which projects from a vacuum container 65 as does the shaft 8 project from the container 1 in Fig. 1. The ends of the wire 57 are connected with wires 58 and 59 which are enclosed in insulating tubes 60. The free ends of the wires 58 and 59 are connected with a contact ring 61 and a contact plate 62, respectively. The elements 61 and 62 are mounted on one end face of the shaft 53 and are electrically insulated therefrom. Current is supplied to the elements 61 and 62 in the conventional manner.

Fig. 8 shows an apparatus in which discs *a, b, c, d, e,* and *f* of the type shown in Fig. 7 are mounted on a shaft 66. The discs dip into the distilland in the bottom of the container 65. Cooling pockets *m, n, o, p, q, r,* and *s* are individually interposed between the discs. By controlling the current supply to the discs the temperature of the discs *a, b, c* is maintained the same and the temperature of the discs *d, e, f* is held higher than that of the discs *a, b, c*, the temperature of the disc *e* being higher than that of disc *d* and the temperature of the disc *f* being higher than that of the disc *c*. The temperatures of both side walls of each disc are the same. Since the side walls of the discs *a, b,* and *c* have the same temperature, the distillate collected in the trays *m', n',* and *o'* which are beneath the pockets *m, n,* and *o* has the same composition and can be removed in a common collecting pipe 67. The composition of the distillate condensed on the left side of the pocket *p* and collected in the tray *p'* is the same as that collected in the trays *m', n',* and *o'* and can also be conducted into the pipe 67. The distillate evaporated on the sides of the disc *d* which is condensed on the right side of the pocket *p* and on the left side of the pocket *q* and which is collected in the trays *p''* and *q'* has the same composition which is different from the composition of the distillate removed through the pipe 67. A separate pipe 68 is therefore provided for removing the distillate collected in the trays *p''* and *q'*. For the same reasons a separate pipe 69 is provided for removing the distillate from the trays *q''* and *r'*, and a pipe 70 is provided for removing the distillate from the trays *r''* and *s'*. Fig. 8 is diagrammatic and the means for supplying a coolant into the pockets *m, n, o, p, q, r,* and *s* are not shown. They are the same as the coolant supply means 26, 27 in Fig. 1.

It may be advisable to provide individual chambers in the sump of the vacuum container, one chamber for each side of an evaporating disc, in order to prevent mixing of the distilland to be picked up by the individual side surfaces constituting individual evaporating stages. As seen in Fig. 3 partitions 44 are arranged in the sump of the vacuum container between two consecutive discs 12 so that the sump is subdivided into individual chambers for the individual discs. The partitions form an overflow between the individual chambers. Each individual chamber formed by the partitions 44 is further divided by a separating wall 45 so that there is a sub-chamber for each side surface of each disc 12. The walls 45 (Figs. 4 and 5 extend to the circumference of the discs 12, leaving only a small clearance, throttling the flow of the distilland as the distilland passes through the sump of the vacuum container. The cooling pockets and the drip

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trays are not shown in Figs. 3 and 4. The arrangement of the partitions 44 and of the separating walls 45 increase the degree of separation and the distillation velocity.

What is claimed is:

An apparatus for distilling at a high vacuum comprising, in combination, a vacuum container, a horizontal shaft rotatable in said container, a plurality of discs mounted on said shaft and having side surfaces placed at a right angle to said shaft, means connected with said discs for heating same to different temperatures, said container having a bottom portion, means for supplying the fluid to be distilled into said bottom portion, said discs dipping into the fluid in said bottom portion and picking up fluid upon rotation of said shaft for evaporating part of the fluid from said side surfaces, a plurality of cooling means individually having side surfaces individually placed opposite individual side surfaces of said discs for condensing the part of the fluid evaporating from the side surfaces of said discs on the opposed side surfaces

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of said cooling means, a separate trough placed beneath each of the side surfaces of those of said cooling means whose side surfaces are placed between two neighboring discs for separately collecting the distillate dripping from the side surfaces of the cooling means which side surfaces are located opposite the neighboring side surfaces of two different discs, and partitions disposed in said bottom portion of said container between said discs for dividing said bottom portion into individual spaces pertaining to individual discs, and separating walls individually placed in said bottom portion beneath the individual discs for subdividing said spaces into individual spaces which are contiguous to the individual side surfaces of the discs beneath which said separating walls are placed.

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