March 7, 1944. K. C. D. HICKMAN 2,343,668

HIGH VACUUM STILL

Filed March 4, 1942

FIG. 1.

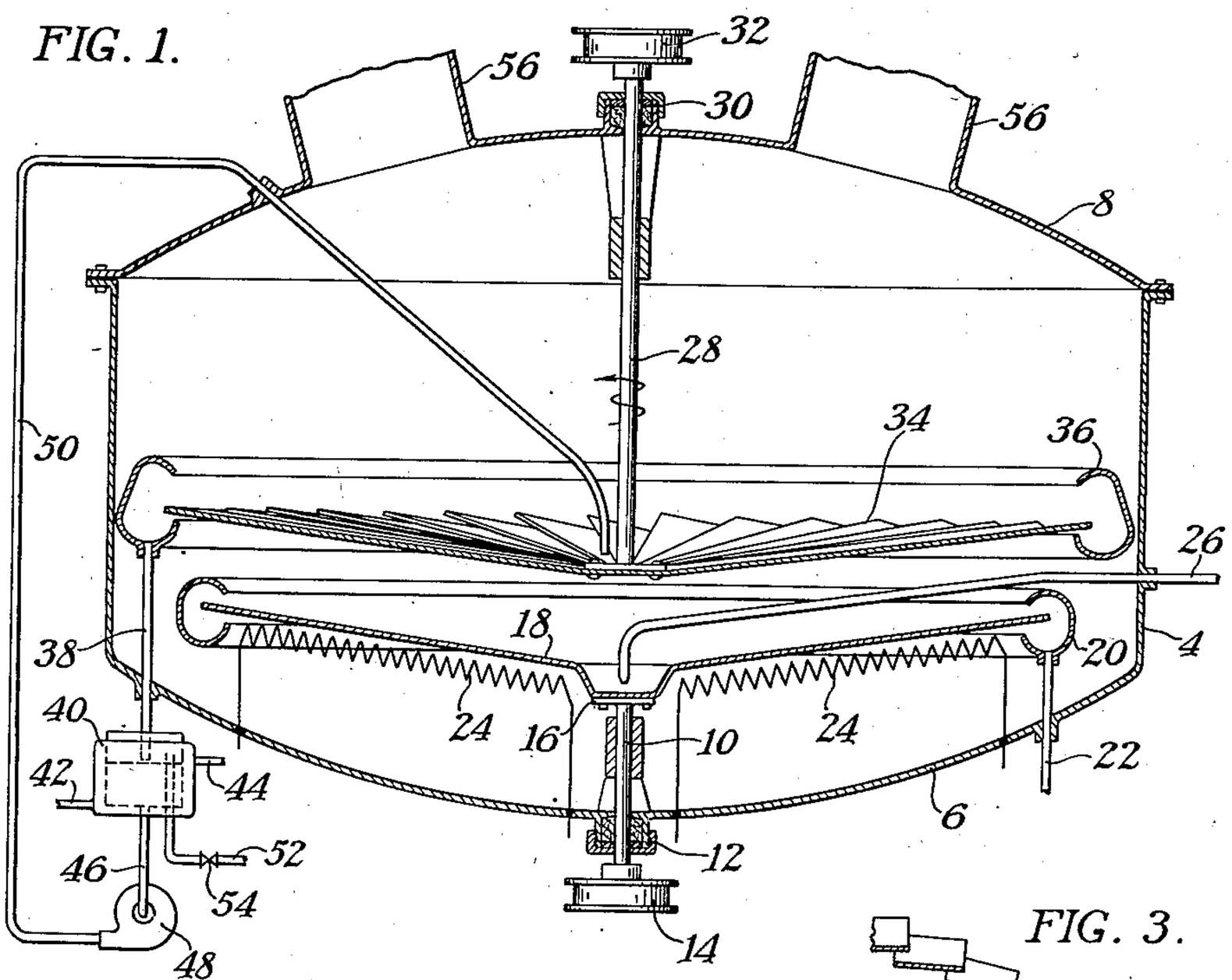

FIG. 3.

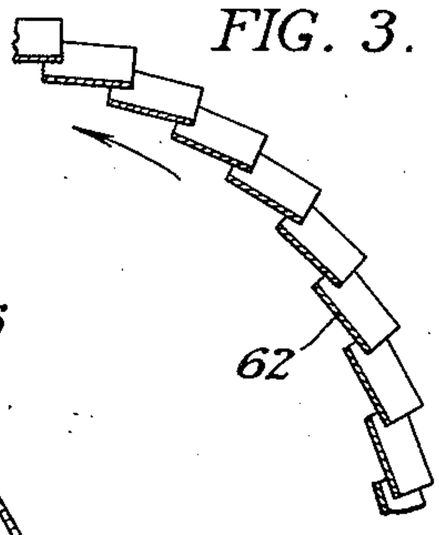

FIG. 2.

KENNETH C.D.HICKMAN
INVENTOR

BY Newton W. Perry
Warren H. Cannon
ATTORNEYS

Patented Mar. 7, 1944

2,343,668

UNITED STATES PATENT OFFICE

2,343,668

HIGH VACUUM STILL

Kenneth C. D. Hickman, Rochester, N. Y., assignor to Distillation Products, Inc., Rochester, N. Y., a corporation of Delaware

Application March 4, 1942, Serial No. 433,337

10 Claims. (Cl. 202—205)

This invention relates to improved high vacuum stills and particularly to improved high vacuum stills in which the distilland is distributed over the vaporizing surface by centrifugal force and the distilling vapors are condensed upon a rotating condensing surface.

In my Patent 2,210,928, August 13, 1940, I have described high vacuum unobstructed path centrifugal stills in which the distilland is introduced on to a rotating vaporizing surface and condensate is condensed upon a rotating condensing surface, the condensing surface being adapted to throw the liquid condensate by centrifugal force into a gutter arranged at the periphery of the condensing surface. Also, in Hickman and Hecker Patent 2,180,052, November 14, 1939, there is described similar stills provided with rotating condensing surfaces having slots to permit the escape of non-condensable gas. Cooling of the rotating condensing surface has been a difficult problem to solve. Introduction of distilland on to the condensing surface results in intermixture of condensate and distillate. Cooling by radiation from an adjacent cooling surface is frequently insufficient.

This invention has for its object to provide simple and economical means for efficiently cooling a rotating condensing surface of a centrifugal still. Another object is to make available a high vacuum unobstructed path still provided with a condensing surface which has slots so arranged that residual gas can pass through the condensing surface into the evacuating pumps, and which are also arranged so that rotation of the slotted condensing surface results in partial evacuation of the still. A still further object is to provide a high vacuum unobstructed path still having a rotating condensing surface which is slotted, the slots being so arranged that condensate and cooling fluid introduced thereon is transferred to the opposite side of the condensing surface from the vaporizing surface. Other objects will appear hereinafter.

These and other objects are accomplished by my invention which includes a high vacuum unobstructed path still having a rotating condensing surface and slots thereon so arranged that the condensate is thrown to the rear of the surface thence travels to an adjacent cool surface or cooler and the cooled liquid condensate introduced back on to the slotted rotating condensing surface. I have also found that the slots or vanes of the rotating condensing surface can be so positioned or arranged that during rotation evacuation of the distilling space takes place. My invention, therefore, includes stills provided with rotating condensing surfaces of this type.

In the following description I have given several of the preferred embodiments of my invention, but it is to be understood that these are set forth for the purpose of illustration and not in limitation thereof.

In the accompanying drawing I have illustrated two of the preferred embodiments of my invention wherein like numbers refer to like parts and wherein:

Fig. 1 is a vertical section of a centrifugal still having a rotating condensing surface and slots or vanes which are adapted to transfer the liquid thereon to the side of the condensing surface farthest from the vaporizing surface and which are adapted to evacuate the space between the vaporizing and condensing surfaces; and

Fig. 2 is a vertical section of a modification of the apparatus shown in Fig. 1, the condensing surface being bell-shaped to increase the effective condensing area; and

Fig. 3 is a horizontal section of the rotating condensing surface taken on line 3—3 of Fig. 2.

Referring to Fig. 1, numeral 4 designates a cylindrical still casing provided with a bell-shaped base plate 6 integral therewith and a bell-shaped removable cover 8. Numeral 10 designates a shaft rigidly mounted in a gas tight bearing 12 which shaft is provided at one end with a pulley 14 and at the other end with a flange 16. To the flange is rigidly mounted a circular vaporizing plate 18. The periphery of plate 18 extends into an annular gutter 20 to which is connected withdrawal conduit 22. Numeral 24 designates an electrical heating resistance unit adapted to heat plate 18 to distilling temperature. Numeral 26 designates a conduit for introducing distilland on to the approximate center of plate 18.

Numeral 28 designates a shaft rigidly held in a rotatable manner by gas tight packed bearing 30. The upper end of shaft 28 is provided with a driving pulley 32 and the opposite end of the shaft is integral with circular condensing surface 34. The condensing surface is constructed from a plurality of radical vanes which overlap in the same fashion as the blades of a windmill. The blades are spaced so as to allow passage of residual gas. The periphery of the condensing surface 34 extends into annular gutter 36 to which is connected withdrawal conduit 38. Numeral 40 designates a cooler and reservoir through which liquid from conduit 38 passes. Cooling takes place by introduction of cooling liquid into the cooling jacket

through conduit 42 and withdrawal through conduit 44. Numeral 46 designates a conduit serving to convey liquid from cooler 40 to pump 48. Numeral 50 designates a conduit for conveying liquid from the exhaust side of pump 48 to the approximate center of condensing surface 34. Numeral 52 designates a conduit provided with a valve 54 for withdrawing or bleeding off distillate from the system. Numeral 56 designates conduits connected to evacuating backing pumps (not shown).

Referring to Fig. 2 numeral 60 designates a bell-shaped still casing provided with a gas tight base 62 which can be removed when the still is disassembled. Numeral 62 designates a condensing surface constructed of a number of radial vanes which overlap as illustrated in Fig. 3. The vanes are assembled to form a unitary condensing surface having the bell shape illustrated. The base of each vane is provided with an outwardly turned edge 64 which protrudes into gutter 66.

During operation of the apparatus illustrated in Fig. 1 the still is evacuated by means of backing pumps connected to conduits 56. Heating resistance units 24 are put into operation and distilland is introduced on to the center of the vaporizing plate 18 by way of conduit 26. Power is applied to pulleys 14 and 32 to rotate the vaporizing and condensing surfaces. Condensing surface 34 is rotated in the direction indicated by the arrow. Distilland is caused to flow by centrifugal force over the surface of plate 18 as a thin film. Vapors are efficiently formed and pass to the condensing surface 34. Here they impinge upon the vanes due to the fact that they overlap. They are then condensed and due to the centrifugal action travel to the upper edge of each vane. In this position they cannot undesirably affect the thermal characteristics of the still. The condensate is then flung by centrifugal force into gutter 36. Liquid condensate then flows through conduit 38 into cooler 40 and is pumped by pump 48 through conduit 50 back on to the condensing surface 34. The liquid is then flung by centrifugal force over the various vanes of condensing surface 34 and is again recirculated through pump 48 and conduit 50. When sufficient distillate has accumulated in cooler 40 it is continuously or intermittently bled from the system through conduit 52 and valve 54. Undistilled residue is removed from the collecting gutter 20 by way of conduit 22. Gases in the distilling space are continuously removed by the rotating vanes and are caused to pass into conduits 56 from which they are removed by the backing pumps.

It has been found that organic liquids distilled in high vacuum stills are efficient absorbers and radiators of heat. For this reason it is desirable to maintain the liquid, both on the vaporizing and condensing surfaces, in a thin film. The construction shown in Fig. 1 performs this desirable function very efficiently. Both the initial condensate and the recirculated condensate are maintained on the upper edge of the condensing vanes where they cannot absorb heat from the vaporizing surface while condensate on the portions nearest the vaporizing surface is in such a thin film that it will not absorb heat. This is particularly advantageous when the liquids are introduced on to the condensing surface. Otherwise the cooling liquid would greatly increase the thickness of the condensate film on the condensing surface, hence increasing heat losses greatly. It is also desirable, but not necessary, to maintain the vaporizing and condensing surfaces in a highly polished condition. Highly polished surfaces in conjunction with thin films of condensate or distilland greatly decrease the heat losses which are otherwise considerable in high vacuum unobstructed path stills.

In operating the apparatus illustrated in Figs. 2 and 3 the still is evacuated and the vaporizing surface is put into operation as described in connection with Fig. 1. Condensing dome 62 is rotated counterclockwise. Condensate collecting on condensing dome 62 is at least partially flung through the spaces between the vanes on to the outside dome 60 which is air cooled. The condensate then partially drops from the upper part and especially the top part of dome 60, after being cooled, back on to the vanes and hence cools them to an efficient condensing temperature. The condensate eventually flows by gravity into gutter 66, but it may be transferred back and forth between the condensing surface 62 and cool dome 60 many times before eventually reaching gutter 66. The condensate is then withdrawn from the system through conduit 38. Parts of this withdrawn condensate may be cooled in 40 and recycled back to 62 by way of conduit 50 as described in connection with Fig. 1. This is advantageous when the shape of 62 and 60 does not result in sufficient return of cooled condensate to condenser 62. In addition to the other advantages mentioned this particular construction increases the condensing area, and the effective pumping capacity of the vanes.

High vacuum unobstructed path distillation involves distillation at pressures of below approximately 1 mm. and especially at pressures below .1 mm. such as .001 mm. A single backing pump cannot efficiently produce such low pressures. Also at these low pressures gases diffuse and cannot be sucked so that the removal of gases from the still depends upon their rate of diffusion. The construction shown in Figs. 1 and 2 enables improved removal of the gases from the system and particularly from the space between the vaporizing and condensing surfaces. Due to the positioning of the vanes they tend to force the gases in the space between the two surfaces toward the evacuating conduits 56 from which the gases can be more efficiently removed by the evacuating pumps. The faster the vanes rotate the better the pumping effect. Speeds of 100 to 10,0000 or more R. P. M. can be used but speeds of about 500 to 5,000 will be found to be generally most useful.

Many modifications can be made in the apparatus illustrated without departing from the spirit or scope of the invention. For instance, it would be perfectly feasible to construct the apparatus of Fig. 1 so that the vaporizing and condensing surfaces are vertical. Some modification would be required to do this in connection with the apparatus of Fig. 2 since return of the condensate to the condensing surface for cooling purposes requires gravity flow. However, this apparatus could be similarly modified by providing a circulating pump for the cool condensate.

The invention described constitutes a simple and economical method for cooling slotted rotating condensing surfaces. The construction illustrated not only enables efficient cooling, but at the same time permits residual gases to efficiently pass from the space between the condensing and vaporizing surfaces where they would otherwise harmfully affect the distillation. A particular advantage of the invention is that the stills are

made to perform a pumping action without any expenditure of energy in addition to that necessary to remove condensate. A further advantage is that the heat losses are reduced because of the transfer of condensate to the portion of the condensing surface farthest from the vaporizing surface and the maintenance of a very thin film of condensate, which does not absorb much heat on the parts of the condensing surface nearest the vaporizing surface.

What I claim is:

1. High vacuum distillation apparatus comprising a rotatable vaporizing surface, a rotatable condensing surface separated from the vaporizing surface by substantially unobstructed space, means for heating the vaporizing surface, a cooler for condensate positioned so that the condensing surface is between said cooler and the vaporizing surface, slots in the condensing surface so positioned that during rotation of the condensing surface the condensate is transferred to the side of the condensing surface opposite to the vaporizing surface and thence flung to the adjacent cooler and means for returning at least part of the cool condensate to the condensing surface.

2. High vacuum distillation apparatus comprising a rotatable, substantially horizontal vaporizing surface, and a rotatable condensing surface the axis of rotation of which is approximately at right angles to the vaporizing surface, the condensing surface being separated from the vaporizing surface by substantially unobstructed space, means for heating the vaporizing surface, a cooler for condensate positioned so that condensate flung from the condensing surface during rotation thereof impinges upon the cooler, slots in the condensing surface so positioned that during rotation thereof the condensate is transferred to the side of the condensing surface opposite to the vaporizing surface and thence flung to the adjacent cooler, and means for returning at least part of the cooled condensate to the condensing surface.

3. High vacuum distillation apparatus comprising an approximately horizontal rotatable vaporizing surface, a bell-shaped condensing surface separated from the vaporizing surface by substantially unobstructed space the axis of rotation of the condensing surface being approximately at right angles to the vaporizing surface, a bell-shaped cooler at least partially surrounding the condensing surface, slots in the condensing surface so positioned that during rotation of the condensing surface condensate is thrown to the side of the condensing surface away from the vaporizing surface thence to the bell-shaped cooler surrounding the condensing surface, said bell-shaped cooling surface being of such a shape that condensate thrown thereto drops by gravity back on to the condensing surface.

4. An evacuating high vacuum still comprising in combination a rotatable vaporizing surface, means for heating the vaporizing surface, means for introducing distilland on to the approximate center of the vaporizing surface, means for removing undistilled residue from the vaporizing surface, a rotatable condensing surface separated from the vaporizing surface by substantially unobstructed space, said condensing surface being provided with vanes and slots therebetween said vanes being so positioned that during rotation gases in the space between the vaporizing and condensing surfaces are caused to pass to the side of the condensing surface farthest from the vaporizing surface, an evacuating port communicating with that side of the condensing surface to which the gases are caused to pass, and means for removing condensate from the still.

5. An evacuating high vacuum still comprising in combination a rotatable vaporizing surface, means for heating the vaporizing surface, means for introducing distilland on to the approximate center of the vaporizing surface, means for removing undistilled residue from the vaporizing surface, a rotatable condensing surface separated from the vaporizing surface by substantially unobstructed space, said condensing surface being provided with vanes and openings therebetween, said vanes being positioned so that during rotation gases in the space between the vaporizing and condensing surfaces are caused to pass to the side of the condensing surface farthest from the vaporizing surface, and so positioned that during rotation liquid condensate thereon is transferred to the portion of the vanes farthest from the vaporizing surface, an evacuating port communicating with that side of said condensing surface to which the gases are caused to pass, and means for removing condensate from the still.

6. An evacuating high vacuum still comprising in combination a rotatable vaporizing surface, means for heating the vaporizing surface, means for introducing distilland on to the approximate center of the vaporizing surface, means for removing undistilled residue from the vaporizing surface, a rotatable condensing surface separated from the vaporizing surface by substantially unobstructed space, said rotatable condensing surface being constructed of a plurality of overlapping radial vanes with ample openings therebetween for passage of residual gas, and being positioned so as to pump gases when rotated, an evacuating port communicating with that side of the condensing surface opposite from the vaporizing surface, and means for removing condensate from the still.

7. The apparatus defined in claim 6 in which the radial vanes overlap so that distilling vapors must collide at least once therewith.

8. An evacuating high vacuum still comprising in combination a rotatable vaporizing surface, means for heating the vaporizing surface, means for introducing distilland on to the approximate center of the vaporizing surface, means for removing undistilled residue from the vaporizing surface, a rotatable condensing surface separated from the vaporizing surface by substantially unobstructed space, said condensing surface being provided with vanes and openings therebetween said vanes being so positioned that during rotation gases in the space between the vaporizing and condensing surfaces are caused to pass to the side of the condensing surface farthest from the vaporizing surface, means for collecting condensate, means for cooling the condensate, and means for returning at least part of the cooled condensate to the vanes of the condensing surface, an evacuating port communicating with that side of the condensing surface to which the gases are passed, and means for removing at least part of the condensate from the still.

9. An evacuating high vacuum still comprising in combination a rotatable vaporizing surface, means for heating the vaporizing surface, means for introducing distilland on to the approximate center of the vaporizing surface, means for removing undistilled residue from the vaporizing surface, a rotatable condensing surface separated from the vaporizing surface by substantially unobstructed space, said condensing surface being

provided with vanes and openings therebetween for the passage of residual gas, said vanes being so positioned that during rotation gases in the space between the vaporizing and condensing surfaces and condensate on the vanes are caused to pass to the side of the condensing surface farthest from the vaporizing surface, an evacuating port communicating with that side of said condensing surface to which the gases are caused to pass, and means for removing condensate from the still.

10. An evacuating high vacuum still comprising in combination a rotatable vaporizing surface, means for heating the vaporizing surface, means for introducing distilland onto the approximate center of the vaporizing surface, means for removing undistilled residue from the vaporizing surface, a rotatable condensing surface separated from the vaporizing surface by substantially unobstructed space, said condensing surface being provided with vanes and slots therebetween, said vanes being so positioned that during rotation distilling vapors impinge upon that side of the vanes opposite from the vaporizing surface and so that there is ample space for passage of residual gases through the slots, an evacuating port communicating with that side of the condensing surface opposite from the vaporizing surface, and means for removing condensate from the still.

KENNETH C. D. HICKMAN.