

July 15, 1941.

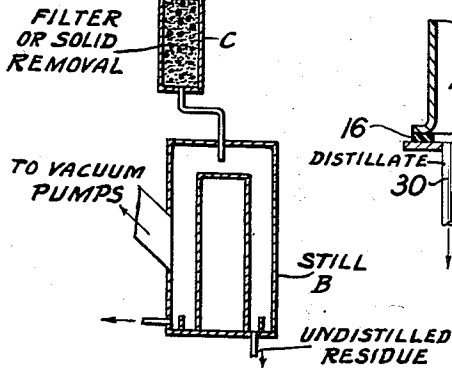
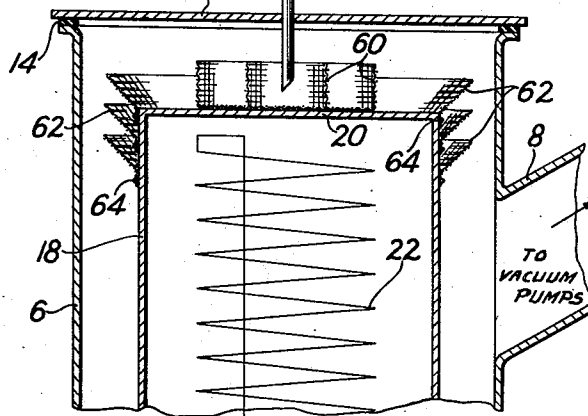
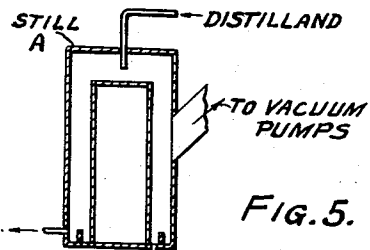
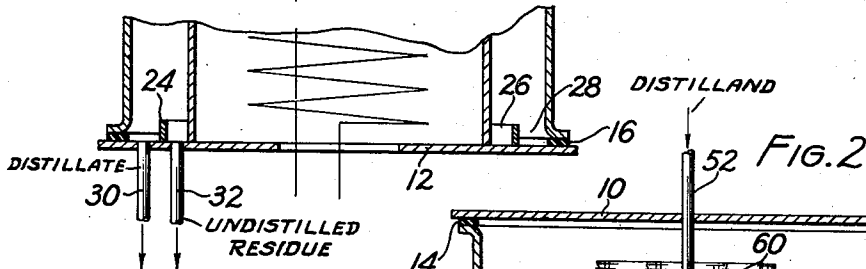
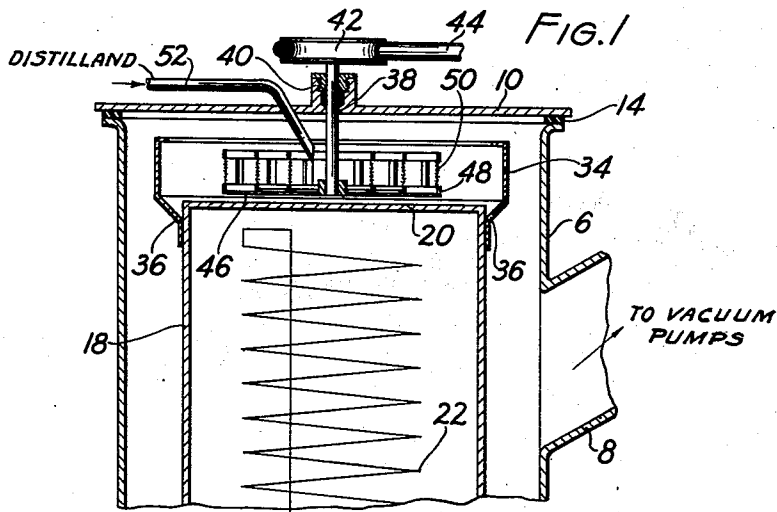
K. C. D. HICKMAN

2,249,526

VACUUM DISTILLATION

Filed June 9, 1939

2 Sheets-Sheet 1



KENNETH C. D. HICKMAN
INVENTOR

BY *Wm. M. P. P. P.*
Warren H. Cannon
ATTORNEYS

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FIG. 3

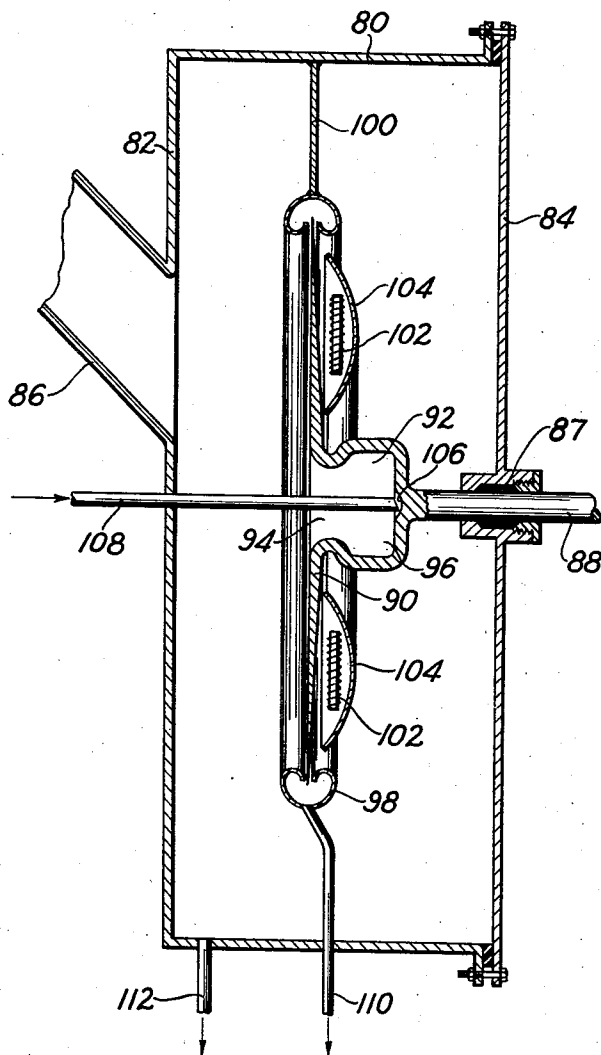
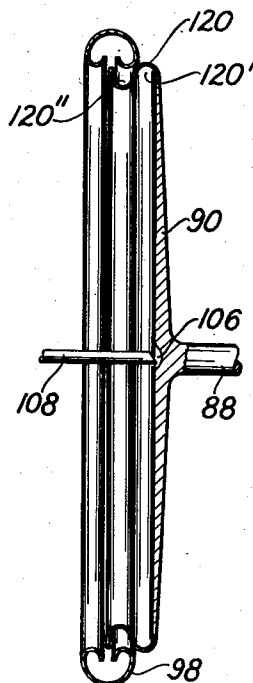


FIG. 4



KENNETH C. D. HICKMAN
INVENTOR

BY *Warren H. Cannon*
ATTORNEYS

UNITED STATES PATENT OFFICE

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VACUUM DISTILLATION

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

Application June 9, 1939, Serial No. 278,308

6 Claims. (Cl. 202—52)

This invention relates to improved method and apparatus for distilling liquid organic materials in the form of a thin film under vacuum conditions.

It is customary to vacuum distill organic liquids by causing them to flow in a thin film over a heated surface which heats them to distillation temperature. After prolonged operation of such vacuum, thin film still, the vaporizing surface may become dark and coated with a carbonaceous or other material which hinders distillation and lowers the yield through decomposition. This is true even when the organic distilland has been given a preliminary refining treatment to remove protein and suspended material. There are certain valuable substances contained in crude oils that make it undesirable to apply a preliminary refining treatment, but when the crude oils are passed through the vacuum still, the vaporizing surfaces become fouled from 5 to 100 times more quickly. It has previously been proposed to scrape the vaporizing surface in order to remove this deposit. However, this expedient is satisfactory only when the oil is subjected to a single passage over a distilling surface. Where multiple passages are involved the solids removed during scraping seriously contaminate the oil.

I have discovered that the deposition of solids on the vaporizing surface of an unobstructed path still can be counteracted and in effect substantially prevented if the distilland is filtered to remove solids therefrom between the various stages of the still. I have found that solids are first formed in the oil during heating on the vaporizing surface. If these solids which are suspended in the oil are permitted to remain during the subsequent distillations they will be precipitated on the vaporizing column and become carbonized thereon. However, if the solids are removed from the oil before they have an opportunity to adhere to the column, the vaporizing columns will remain bright for a much longer period of time.

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

The invention has its main application to multi-stage distillation wherein the distilland is caused to flow through a plurality of stills and wherein removal of different fractions takes place. In accordance with my invention, I treat the distilland as it flows from one still of the

series to the next, in order to remove solids therefrom before it is introduced onto the vaporizing column of the next still.

The removal of the solids from the liquid distilland can be accomplished by many different methods. A method which is highly satisfactory and preferred because of its simplicity is filtration whether by gravity flow or forced flow such as by centrifugal separation. The distilland containing solids may be conducted into a chamber containing a body of filtering materials, such as wire gauze, charcoal, or other comminuted material. The filtered oil is then conveyed to the next still of the series and it is preferred that this operation be carried out in such a manner that the oil is not cooled materially. The filters may be placed at the head of each vaporizing surface or at the foot of the preceding vaporizing surfaces. Alternatively the filters may be placed in the pipe lines between the stills and may be fed by gravity or by a forced pump. Centrifugal separation, centrifugal filtration, etc. may also be employed. Various chemical treatments may be employed to cause the solids to agglomerate and precipitate.

In the accompanying drawings wherein like numbers refer to like parts:

Fig. 1 is a vertical section partly in elevation of a gravity flow high vacuum unobstructed path still which is provided with centrifugal separating means for removing solids from the distilland;

Fig. 2 is a vertical section of a still unit substantially the same as that illustrated in Fig. 1 except that gravity flow filtering means are employed to remove solids from the distilland;

Fig. 3 is a vertical section of a centrifugal high vacuum, unobstructed path still provided with centrifugal filtration means integral with the rotating centrifugal vaporizing surface;

Fig. 4 is a vertical section of a centrifugal rotating vaporizing surface and gutter assembly, similar to that illustrated in Fig. 3, but provided with centrifugal filtering means for the solids at the periphery of the plate instead of at the center;

Fig. 5 illustrates diagrammatically the sequence of steps involved in practicing my invention, namely, distilling, filtering to remove solids, and again distilling the filtered liquid.

Referring to Fig. 1, numeral 6 designates a cylindrical still casing to which is connected a conduit 8 of wide admittance which leads to evacuating pumps (not shown). Numerals 10 and 12 designate top and base plates which close

the ends of the cylinder casing 6 in a gas-tight manner because of the interposed gaskets 14 and 16 respectively. Numeral 18 designates a cylindrical vaporizing column which is closed at the top by plate 20 and which is integral with base plate 12 at the bottom. Vaporizing cylinder 18 is disposed inside still casing 6 and approximately concentric therewith. It is heated by an internal electric resistance coil 22. A circular collar 24 is mounted upon base plate 12 and approximately concentric with vaporizing cylinder 18. Collar 24 in cooperation with the bottom portions of casing 6 and cylinder 18 forms annular gutters 26 and 28. Withdrawal conduit 30 is connected to gutter 28 and conduit 32 is connected to gutter 26. An annular flange 34 is mounted at the top of vaporizing cylinder 18 and integral therewith. The periphery of the flange at the point where it comes into contact with cylinder 18 is provided with a plurality of holes 36. A shaft 38 extends through packed gland 40 and is provided at one end with a pulley 42 driven by belt 44 and at the other end with a circular integral plate 46. Plate 46 is provided with a plurality of circular concentric projections 48 upon which are mounted a plurality of concentric gauze cylinders 50. Numeral 52 designates a conduit which serves to introduce distilland onto the approximate center of plate 46.

Referring to Fig. 2, numeral 60 designates a plurality of concentric gauze cylinders, the base portions of which are rigidly mounted upon plate 20. Numeral 62 designates a plurality of truncated cones constructed of wire gauze which are mounted near the top of vaporizing column 18 by means of a fastening wire 64. Numeral 66 designates an annular U-shaped piece of wire gauze which is fitted into gutter 26.

Referring to Fig. 3, numeral 80 designates a truncated cylinder which is closed at the ends by plates 82 and 84 so as to form a gas-tight cylindrical still casing. An evacuating conduit 86 is connected to plate 82. Plate 84 is provided with a gas-tight packed gland 88 through which passes shaft 88. A plate 90 is rigidly mounted upon the end of shaft 88 and the central portion of the plate 90 is provided with a well 92, the circular opening 94 of which has a smaller diameter than its well portion 96. The outside periphery of plate 90 extends into a circular split gutter 98 which is held in the position illustrated by a series of spaced spider supports 100. Gutter 98 is split to form two identical halves which can be securely fastened together. This mode of construction facilitates mounting it about the periphery of plate 90.

Plate 90 is heated by electrically heated radiant heater 102, the heat from which is reflected against plate 90 by annular reflector 104. Liquid to be distilled is introduced into depression 106 in shaft 88 by way of conduit 108. Conduit 110 connects to the lower portion of gutter 98 and serves to withdraw liquid therefrom. Conduit 112 connects to the lower portion of still casing 80.

Referring to Fig. 4, numeral 120 designates an S-shaped flange mounted upon the periphery of plate 90 and integral therewith. The flange is divided into two semi-circular portions designated as 120' and 120''. The outside edge of 120'' extends into close proximity with the split opening into gutter 98.

In operating the apparatus illustrated in Fig. 1, the system is evacuated to the required high

vacuum by pumps connected to conduit 8. Organic liquid distilland which is preferably in a degassed condition is introduced through conduit 52 and heating element 22 is put into operation. Shaft 38 and plate 46 are caused to rotate by force applied through pulley 42. Liquid distilland drops from the end of conduit 52 under the approximate center of plate 46. Due to the rotation of this plate the liquid is caused to flow, to the periphery of the plate and through the successive filtering screens 50. The liquid is flung from the periphery of plate 46 onto the inside of flange 34 and flows downward through openings 36 onto the surface of vaporizing cylinder 18. During passage through filtering screens 50 all solids are removed. Due to centrifugal distribution upon flange 34, the liquid is very evenly distributed on all sides of vaporizing column 18.

The filtered liquid distilland flows down the surface of column 18 in a thin film and is heated to distillation temperature. Vaporized fractions pass to the inside of still casing 6 which is air cooled and condensed thereon. The liquid condensate flows by gravity into gutter 28 and is withdrawn through conduit 30. Undistilled liquid residue flows from the bottom of vaporizing cylinder 18 into gutter 26 and is withdrawn through conduit 32.

The operation of Fig. 2 is substantially the same as that described in connection with Fig. 1. However, filtration takes place by ordinary gravitational flow. The liquid distilland flows through the sequence of filtering gauzes 60 and 62 and then flows downward over the heated vaporizing surface 18. The plurality of filtering gauzes 60 and 62 effectively remove solids suspended in the distilland. Undistilled residue which collects in gutter 26 is filtered by filtering gauze 66 before it is withdrawn from the still. This will serve to effectively prepare the oil for subsequent distillation if the filtering gauze is properly located so that all of the residue has to pass therethrough before passing into conduit 32.

In operating the apparatus of Fig. 3, the system is evacuated through conduit 86. Shaft 88 is caused to rotate at relatively high speed, such as 300 to 20,000 R. P. M. and heating element 102 is put into operation. Liquid distilland passes through conduit 108 into 106 and is forced by centrifugal force over the inside surface of well 92. It then passes over the edge of restricted opening 94 of well 92 and thence over the heated vaporizing surface 90 from which distillation takes place. Since solids suspended in the oil are thrown by centrifugal force into the portion of well 92 having the greatest diameter, and against the periphery of well 92, they are prevented from passing with the distilland over the vaporizing surface. The solids are, therefore, collected and held in this portion of the apparatus by centrifugal force and can be removed intermittently. Vapors derived from the distilland during its passage over plate 90 are condensed upon the various inside walls of casing 80 and plates 82 and 84, all of which are air cooled. The condensate flows to the base of casing 80 and is withdrawn through conduit 112. Undistilled residue is flung from the periphery of plate 90 into gutter 98, flows to the base thereof by gravity and is withdrawn through conduit 110.

During operation of a centrifugal still provided with a plate such as illustrated in Fig. 4, distilland introduced at the center of the plate passes to the periphery by centrifugal force and distil-

lation takes place during this passage. Solids formed during the distillation collect in 120'. Liquid residue flows therefrom over the edge of 120' and is flung thence into gutter 98. This residue is, therefore, filtered and free of solids and can be directly introduced onto the vaporizing surface of the next still in the series. Solids collected in 120' can be intermittently removed.

Referring to Fig. 5, liquid to be distilled is introduced into still A. A fraction is removed therein and undistilled residue containing solids flows into a solid removal device C which is shown in the form of a filter containing a filtering agent, such as a body of comminuted material. Solid matter is removed by filtration and the filtered liquid then flows into still B where a second fraction is removed.

Although I have illustrated only one still section in each of the figures, it will be understood that a plurality of units connected in series are contemplated. Each of the units in the series need not be identical and it is perfectly satisfactory to use any or all of the types illustrated in series or to employ analogous types. However, they should be so constructed that filtration takes place at the base of the previous unit or at the top of the next succeeding unit.

It is apparent that my invention can be carried out with particular ease in connection with centrifugal vaporizing stills because the centrifugal force employed to cause the distilland to flow in a thin film during vaporization can also be used to filter the distilland although such centrifugal apparatus is preferred it is obvious that separate centrifugal filtering or separating equipment may be used. Further details regarding centrifugal distillation are disclosed in my U. S. application 99,631, filed September 5, 1936, and application 194,901, filed March 9, 1938.

My invention is applicable to high vacuum, unobstructed path distillation procedure wherein the liquid distilland is caused to flow over the vaporizing surfaces in the form of a thin film. By unobstructed path is meant distillation wherein the vaporizing and condensing surfaces are separated by substantially unobstructed space so that the vapor molecules can freely pass from the vaporizing to the condensing surface. When the distance between these surfaces is short, the distillation is known as short path, high vacuum distillation and when the distance is less than the mean free path, the distillation is known as molecular. In distillations of this type the liquid passes over the vaporizing surfaces in a thin film and troubles due to carbonization and decomposition are usually encountered. For this reason my invention is applicable to these distillations as a class.

Troubles due to coating of the vaporizing surface are not peculiar to any particular distilland, and I, therefore, contemplate the application of my invention to the distillation of organic liquids which cause a troublesome deposition. Examples of such materials are fish oils containing fat soluble vitamins, such as cod-liver, halibut liver, menhaden, gray fish liver, pollack, etc. oils, petroleum and petroleum residues, animal refuse, etc. The invention is also applicable to

the distillation of edible oils and glyceride drying oils, such as linseed, corn, sesame, soy bean, etc. oils, and oils or oil-like extracts containing hormones, sterols, hydrocarbons, or natural anti-oxidants. The invention is of particular advantage in the distillation of fish oils to obtain vitamin concentrates.

What I claim is:

1. The process of multi-stage distillation which comprises in combination, subjecting a liquid organic material to high vacuum unobstructed path distillation while in the form of a thin film, treating undistilled liquid residue to mechanically separate and remove solids contained therein and formed by the distillation and then subjecting the treated liquid to high vacuum, unobstructed path distillation while in the form of a thin film.

2. The process of multi-stage distillation which comprises in combination subjecting a liquid organic material to high vacuum, unobstructed path distillation while in the form of a thin film, filtering undistilled liquid residue to remove solids contained therein and then subjecting the treated liquid to high vacuum, unobstructed path distillation while in the form of a thin film.

3. The process of multi-stage distillation which comprises in combination subjecting an oil to high vacuum unobstructed path distillation while in the form of a thin film, filtering undistilled oil to remove solids contained therein and then subjecting the treated liquid to high vacuum unobstructed path distillation while in the form of a thin film.

4. The process of multi-stage distillation which comprises in combination subjecting a fish oil which contains a fat soluble vitamin to high vacuum, unobstructed path distillation while in the form of a thin film, filtering undistilled oil to remove solids contained therein and then subjecting the treated oil to high vacuum, unobstructed path distillation while in the form of a thin film.

5. High vacuum distillation apparatus for carrying out the process of claim 1 comprising in combination a plurality of high vacuum, unobstructed path stills connected in series characterized by means for treating undistilled residue, to remove solids therefrom, as it passes from one still to the next still of the series.

6. High vacuum distillation apparatus comprising a plurality of high vacuum unobstructed path thin film stills connected in series, each still of the series comprising in combination a rotatable vaporizing surface whereby during operation distilland passes thereover in a thin film by centrifugal force, means for introducing liquid distilland onto the approximate center thereof, a filter for distilland mounted upon the surface so that distilland passes therethrough by centrifugal force when the surface is rotated means for removing unvaporized residue from the periphery of the surface, heating means for the surface, a condensing surface separated from the vaporizing surface by substantially unobstructed space and means for maintaining a high vacuum in the space between the two surfaces.

KENNETH C. D. HICKMAN.