Feb. 27, 1945. J. C. HECKER 2,370,462

VACUUM DISTILLATION PROCESS AND APPARATUS

Filed June 4, 1941 2 Sheets-Sheet 1

FIG. 1.

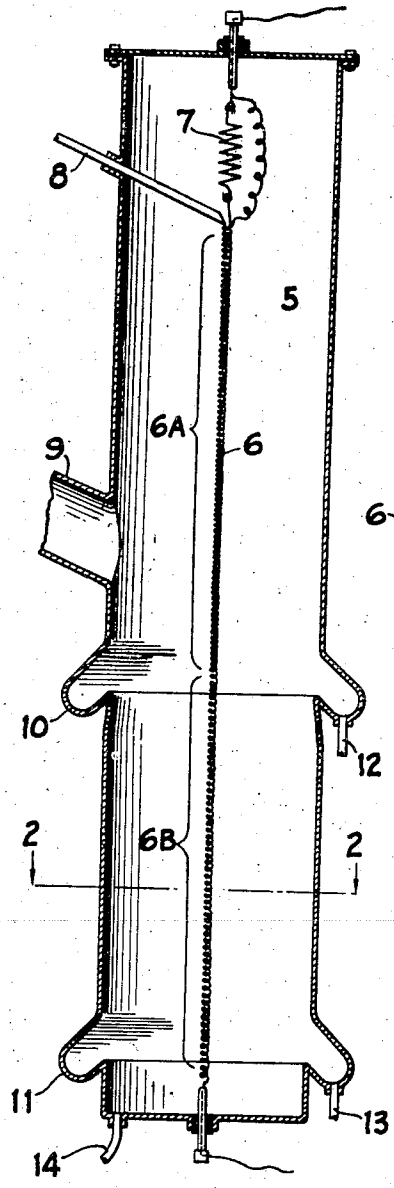


FIG. 3.

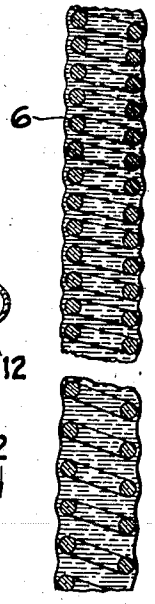


FIG. 4.

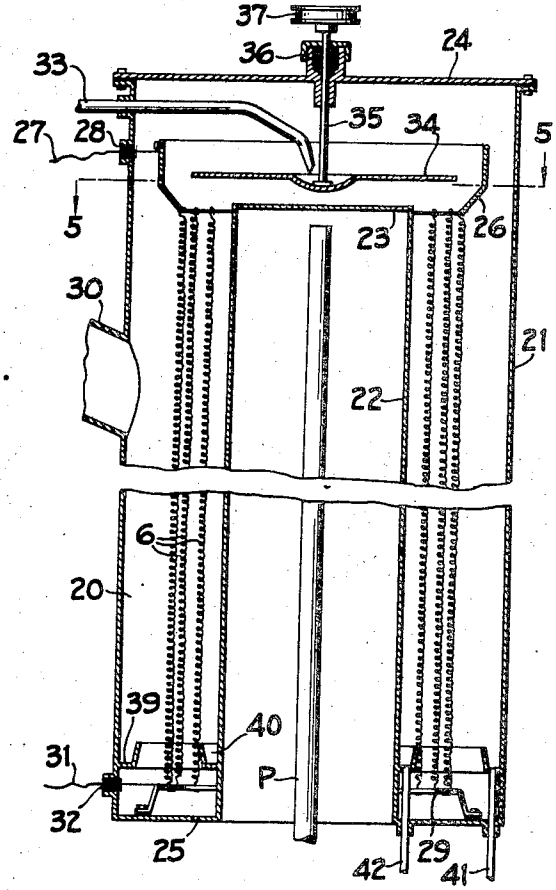


FIG. 5.

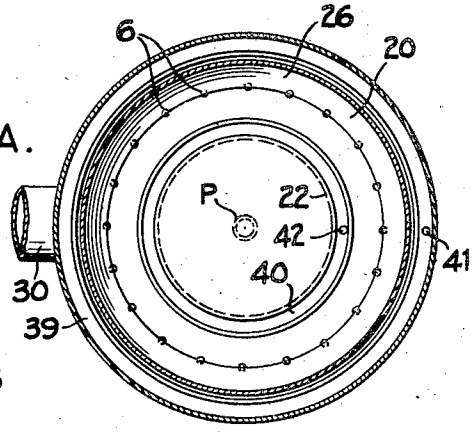


FIG. 2.

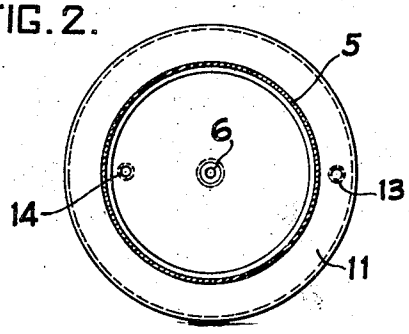
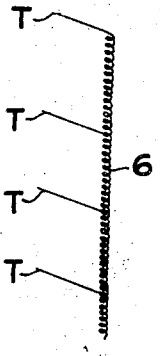


FIG. 3A.



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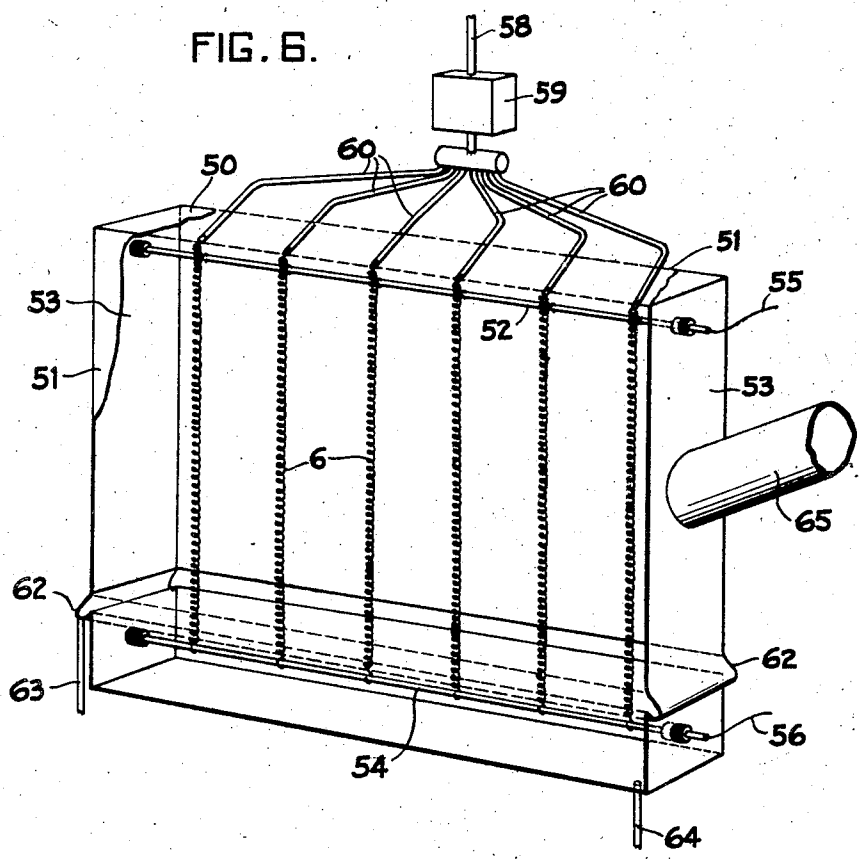
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FIG. 6.



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UNITED STATES PATENT OFFICE

2,370,462

VACUUM DISTILLATION PROCESS AND APPARATUS

John C. Hecker, Rochester, N. Y., assignor to Distillation Products, Inc., Rochester, N. Y., a corporation of Delaware

Application June 4, 1941, Serial No. 396,570

9 Claims. (Cl. 202—52)

This invention relates to a process of and to apparatus for effecting evaporation of a distilland and more particularly to the process of and to apparatus for high vacuum distillation.

In prior molecular stills and especially in evaporators in which there is no ebullition, it has been customary to effect evaporation by means of heated plates. Such plates are expensive as well as difficult to fabricate and because of their necessary mass, are subject to considerable heat lag which wastes much time during the starting and stopping of the apparatus. In addition, such evaporating plates must be provided with means to prevent the distilland from channeling, that is, the formation of rivulets of distilland on the evaporating surface with bare spaces in between. Also, such evaporating plates effect considerable heat radiation and, therefore, cause considerable thermal losses.

The main feature of the present invention relates to a still having an inexpensive evaporating element which is simple in construction and in which there is negligible thermal lag whereby the loss of time in starting and stopping evaporation is reduced to a minimum.

Another feature of the invention relates to an evaporating element which has small radiating area for thermal losses and in which the thermal efficiency will be high since the total amount of heat is applied directly to the distilland. In addition, the evaporating element will never be much above the evaporating temperature so that radiation losses will be small.

A further feature of the invention relates to a simple arrangement for altering the temperature gradient of the evaporating element. Since the evaporating element is in the form of a coil the heat supplied to the several unit lengths of the coil can easily be changed by varying the number of coil turns per unit length to satisfy the heat requirements in any given situation. Instead of varying the space between the turns of the coil, electrical taps can be connected to the coil at various lengths thereof to afford individual temperature adjustments to selected zones of the coil. By this arrangement, the heat input of the coil, near the top thereof, can be of higher value where the oil is cold and requires a quick temperature rise.

An additional feature of the invention relates to the use of one long heating coil adjacent various condensing zones spaced along its path so that it serves as a complete evaporator. Thus, a single distilling unit will have the advantage that evaporation can take place during the entire time that the oil is hot and obviates the need for transfer tubes and pipes wherein thermal decomposition takes place and wherein heat is lost by radiation.

Another feature of the invention relates to an evaporating unit, the temperature of which can be easily controlled. Since the evaporating element is in the form of a coil of wire, the resistance of which depends on temperature, the temperature of the element can be easily maintained by holding the resistance of the coil at a predetermined value such as by a bridge network.

For a clearer understanding of the invention, reference is made to the detailed description and claims when taken with the drawings in which:

Fig. 1 is a vertical section through a still made in accordance with the present invention;

Fig. 2 is a cross section of this still taken on the line 2—2 of Fig. 1;

Fig. 3 is an enlarged view of a piece of the evaporating element illustrating the distribution of the distilland thereon;

Fig. 3A is a fragmentary view of a modified form of evaporating element;

Fig. 4 is a vertical section thru a modified form of still incorporating the present invention; while

Fig. 5 is a cross section of this modified form taken substantially on line 5—5 of Fig. 4; and

Fig. 6 is a perspective view of a further modified form of the invention.

Referring to the still of Fig. 1, the numeral 5 designates a closed cylindrical chamber, the inner wall of which provides a condensing surface. The chamber is provided with an evaporating element 6 in the form of a helically wound, electrical heating coil of nichrome resistance wire or the like, which is insulatedly mounted at the top and bottom of chamber in such a manner that the coil extends along the principal axis of the chamber. As herein illustrated, the upper end of the coil is suspended from the tension spring 7 which tends to keep the coil taut at its axial position in the chamber. The space between the inner wall of the chamber and the heating coil is such that short path distillation can be effected in accordance with the disclosures of the Hickman & Hecker Patent 2,126,467 granted Aug. 9, 1938, and Hickman Patent 2,221,691 granted Nov. 12, 1940.

The upper portion of the chamber is provided with an inlet pipe 8 by which degassed oil or other distilland is supplied to the upper end of the evaporating coil. When the still is in op-

eration, a rarefied atmosphere is maintained within the chamber thru a conduit 9 which is connected to a suitable vacuum pump (not shown). While the invention is not limited to any particular degree of evacuation, it is primarily related to devices commonly referred to as high vacuum unobstructed path stills of the character disclosed in the mentioned patents.

In the arrangement of Fig. 1 the still is assumed to be of the multi-stage type wherein the evaporating element 6 has two zones 6A and 6B to be maintained at different temperatures during the operation of the still. The side wall of the cylindrical chamber is formed with annular gutters 10 and 11 located at the lower boundaries of the heating zones 6A and 6B respectively. Thus, the distillates condense on the wall of the chamber and accumulate in the respective gutters to flow away thru discharge pipes 12 and 13 communicating therewith. The bottom of the chamber is provided with an outlet pipe 14 through which the residue is discharged.

Although the invention is not limited to any particular diameter or length of coil, a satisfactory heating element has been made of #22 gauge nichrome resistance wire helically wound into a coil one-eighth of an inch in diameter and twenty-four inches long, the coil being stretched so that adjacent turns thereof were not in contact. With such a construction, the oil or other distilland flows down the coil in the form of a compact rod at the rate of one quart per hour even when the oil was heated to 200 degrees centigrade.

The limit of the size of the wire that can be used and the limit of the diameter of the resulting helical coil must be such that the coil will suspend the oil or distilland between two adjacent turns as shown in Fig. 3 and at least twenty-five percent of the space within the coil should preferably be occupied.

During operation of the still, the chamber 5 is evacuated to a very low value thru the pipe 9. The evaporating element 6 is included in series with a source of heating current, a suitable rheostat (not shown) being preferably included in series with this circuit so that the temperatures of the zones 6A and 6B of the heating element 6 can be closely regulated. Oil or other distilland is introduced through the pipe 8 to the heating coil 6 on which it flows down slowly because of capillary action due to the narrow spaces between the adjacent turns of the coil. The heated oil, as it flows down the coil, evaporates and the fractions of the distillate condense on the inner wall of the chamber where they accumulate and flow down into the respective gutters 10 and 11 being discharged therefrom through the discharge pipes 12 and 13. The fraction of the distilland which does not evaporate at the prevailing temperatures flows down to the bottom of the chamber where it accumulates as a residue which is discharged through the outlet pipe 14.

In the form of the invention just described, the different heating zones 6A and 6B of the evaporating element are obtained by a different amount of spacing between the turns of the coil. In the case of the zone 6A, the individual turns are closer together and, therefore, have a higher overall heating effect than where the spacing is farther apart as in the case of the zone 6B. Instead of varying the spacing between the coil turns to obtain the several temperature zones, the coil may be provided with one or more electrical taps T along its length (Fig. 3A) to give individual temperature adjustment for any selected zone.

Where it is desired to gain increased capacity, the evaporating coils can be arranged in parallel relation according to a cylindrical pattern, in a modified form of the still shown in Figs. 4 and 5. This still is provided with an upright cylindrical chamber 20 generally annular in cross section, the chamber being defined by an outer cylindrical casing 21 and a smaller cylindrical casing 22 coaxially positioned therein. The tops 23 and 24 of these cylindrical casings are closed and mounted in spaced relation to provide space for a radial distributor. The inner casing is open at the bottom for receiving cooling medium therein from pipe P but the lower edges of the inner and outer casings are sealed together by an annular plate 25. Provision is made for evacuating the annular chamber in the form of a pipe 30 communicating with the chamber and connected to a suitable vacuum pump (not shown). The distributor disc 34 may be mounted for rotation in any suitable manner such as by being concentrically secured to the lower end of a shaft 35. This shaft extends coaxially of the inner casing thru a stuffing box 36 and terminates in a driven pulley 37. The inner walls of the chamber 20 near the lower portion thereof are provided with gutters 39 and 40 in which the distillate collects being discharged thru the pipes 41 and 42 respectively.

It has been mentioned that the evaporating coils 6 are arranged in vertical parallel relation according to a cylindrical pattern which is coaxial with the chamber 20. While these coils may be thus arranged in various ways, it is preferred to suspend these coils from the lower edge of an annular or funnel-shaped conducting member 26. This conducting member is insulatedly mounted on the inner wall of the casing 21 with the edge of the member on a level with the top of the inner casing 22. A power lead 27 extends from the member 26 thru an insulating bushing 28 to the exterior of the chamber. Similarly, the lower ends of the coils 6 are secured to an annular conducting member 29 which is insulatedly mounted on the bottom plate 25 of the chamber. A second power lead 31 extends from member 29 thru an insulating bushing 32 in the wall of the outer casing.

The oil or other distilland is introduced into the chamber thru an inlet pipe 33. This pipe discharges the oil on a rotating, horizontal disc 34 serving to distribute the oil uniformly on the funnel-shaped member 26 from which the oil flows down the coils 6. The operation of this still will be obvious from the foregoing description.

In the modified form of the invention illustrated in Fig. 6, the still 50 is generally oblong in shape with its side walls 51 spaced apart a distance appropriate for short unobstructed path distillation. In this arrangement, the evaporating coils 6 are mounted in a vertical plane parallel to and preferably midway between the side walls 51 of the still. The coils are conductively joined at their upper ends to the horizontal conducting rod 52 insulatedly mounted in the end walls 53 of the still. Similarly the lower ends of the coils 6 are electrically connected to the horizontal conducting rod 54 insulatedly mounted in the end walls 53. The rods 52 and 54 are connected thru the power leads 55 and 56 to a source of electric current while the still is in operation.

The oil or other distilland is supplied to the still thru supply pipe 58 to a metering device 59 from which it flows to branch feed pipes 60 sealed into the top of the still and discharging on the upper ends of the coils 6.

The lower parts of the side and end walls of the still have a continuous gutter 62 formed therein in the region of the lower ends of the coils 6. This gutter has a discharge pipe 63 for distillate while the bottom of the still has a pipe 64 thru which residue is discharged.

Since this form of the still is intended for high vacuum distillation, the space within the still is evacuated thru pipe 65.

The operation of this modified still will be obvious from the description of the operation of the still disclosed in Fig. 1.

It should be appreciated that the evaporating coils are inexpensive to manufacture and can be easily replaced by fresh units if the need arises. Since each evaporating coil has small mass, it responds promptly to changes in heating current so that the temperature within the still can be easily regulated as desired. It will also be appreciated that radiation of heat is reduced to a minimum since each coil is surrounded by the flowing oil.

What I claim is:

1. The method of effecting distillation which comprises causing a distilland to flow slowly as a column with its entire outer surface exposed to a rarefied atmosphere, introducing heat into said column along an internal, line-like, helical path for the most part completely immersed in said column and extending lengthwise thereof, a substantial portion of the space within said path being filled with distilland, condensing the distillate adjacent said column, and discharging the distillate.

2. The method of effecting distillation which comprises causing a distilland to flow slowly in a substantially straight course as a column with its entire outer surface exposed to a rarefied atmosphere, introducing heat into said column along an internal line-like helical path for the most part completely immersed therein and extending lengthwise thereof, a substantial portion of the space within said path being filled with distilland, condensing the distillate adjacent said column, and discharging the distillate.

3. In an unobstructed free path distillation device, a downwardly extending coreless, helical coil of wire, said coil having its inner and outer surfaces completely exposed for the flow of distilland thereover, means for heating said coil, means for applying a stream of distilland to the upper end of said coil, a condensing surface extending in generally parallel short path relation to said coil, means for maintaining a rarefied atmosphere between said coil and said surface, and means for accumulating distillate and residue at spaced points in said device.

4. In a device of the class described, a closed, elongated, vertical chamber, means for maintaining a rarefied atmosphere in said chamber, a helical electrical heating coil extending along the principal axis of said chamber, said coil being coreless whereby distilland can flow over the inner surface and over the outer surface thereof, and means for applying a stream of distilland to the upper portion of said coil, said chamber having one portion to accumulate distillate and a second portion to accumulate residue.

5. In a device of the class described, at least one downwardly extending coreless, helical heating coil of wire suspended by its ends, means for heating different portions of said coil to different temperature values, means for applying a stream of distilland to the upper end of said coil, a condensing surface extending in generally parallel short path relation to said coil, means for maintaining a rarefied atmosphere between said coil and said surface, and means for accumulating distillate and residue at spaced points in said device.

6. In a device of the class described, at least one coreless, helical coil of resistance wire extending downward with its inner and outer surfaces exposed for the flow of distilland thereover, said coil having at least two zones, the spacing of the turns of said coil in one zone being different from the spacing of the turns in the other zone, means for applying an electric heating current to said coil, means for applying a stream of distilland to the upper end of said coil, a condensing surface extending in generally parallel short path relation to said coil, means for maintaining a rarefied atmosphere between said coil and said surface, and means for accumulating distillate and residue at spaced points in said device.

7. In a device of the class described at least one helical, coreless, resistance-type heating coil extending downward, said coil having electrical taps dividing the same into at least two zones and means for applying electric heating currents of different values to said taps, means for applying a stream of distilland to the upper end of said coil, a condensing surface extending in generally parallel short path relation to said coil, means for maintaining a rarefied atmosphere between said coil and said surface, and means for accumulating distillate and residue at spaced points in said device.

8. In a device of the class described, an upwardly extending chamber provided with spaced condensing walls, upwardly extending helical, coreless coils of the resistance heating type having their principal axes displaced from one another in side by side relation, said walls being spaced in condensing relation to said coils, means for maintaining a rarefied atmosphere in said chamber, means for applying electric heating currents to said coils, means for applying a distilland to said coils, and means for accumulating a distillate condensed on said walls.

9. In a device of the class described, an upwardly extending cylindrical chamber provided with condensing walls, coreless heating coils of wire mounted within the chamber with their principal axes extending in spaced upright relation according to a cylindrical pattern coaxial with said chamber, means for maintaining a rarefied atmosphere in said chamber, means for applying electric heating currents to said coils, means for applying a distilland to said coils, and means for accumulating distillate condensed on the walls of said chamber.

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