Nov. 14, 1939. K. C. D. HICKMAN 2,180,053

DISTILLATION APPARATUS

Filed June 29, 1938 2 Sheets-Sheet 1

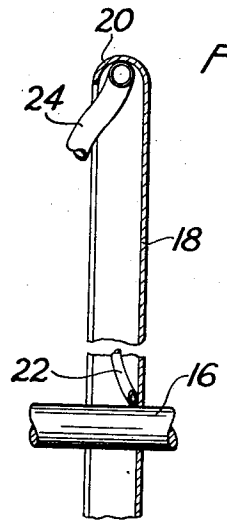


Fig. 1

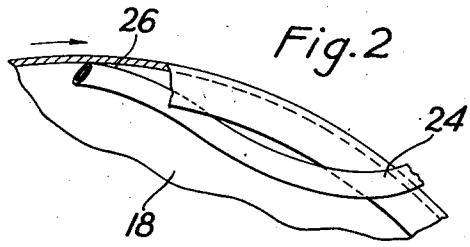


Fig. 2

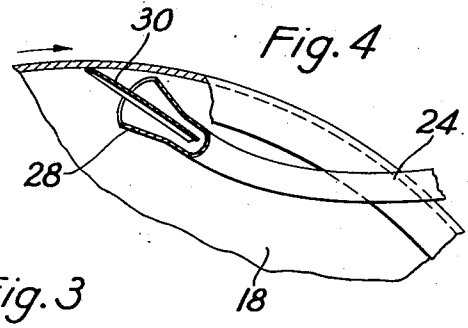


Fig. 4

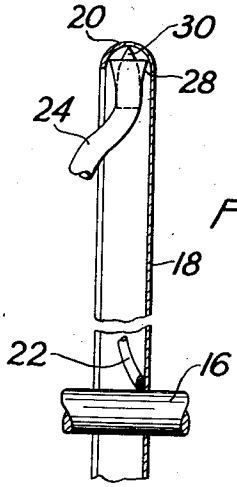


Fig. 3

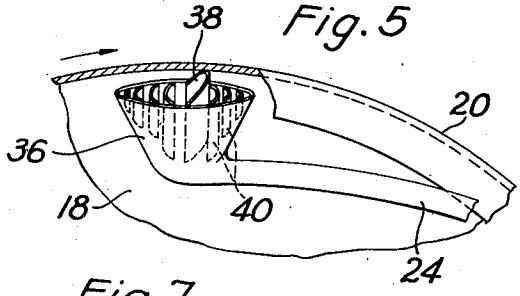


Fig. 5

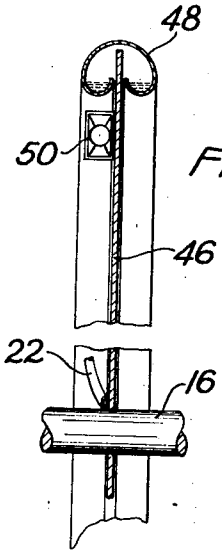


Fig. 6

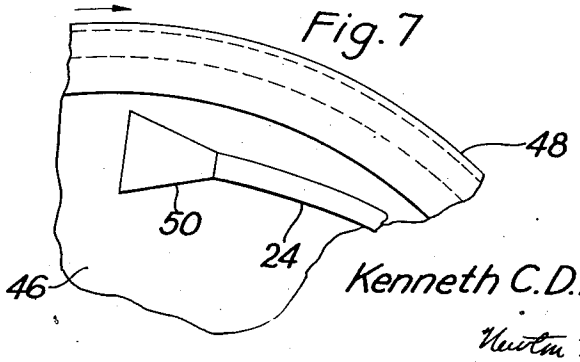


Fig. 7

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DISTILLATION APPARATUS

Filed June 29, 1938 2 Sheets-Sheet 2

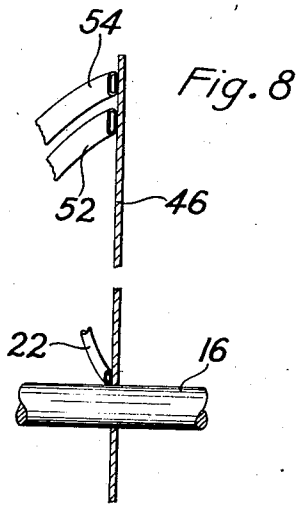


Fig. 8

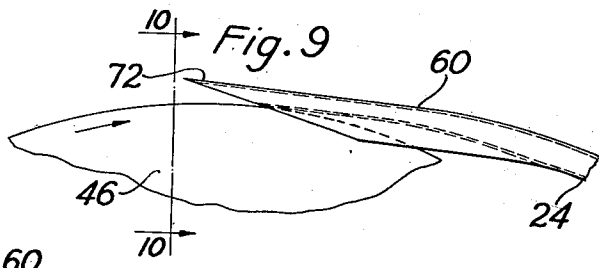


Fig. 9

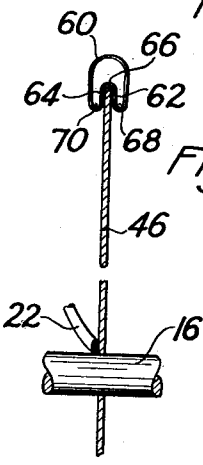


Fig. 10

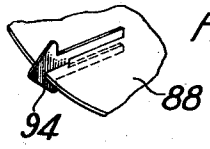


Fig. 13

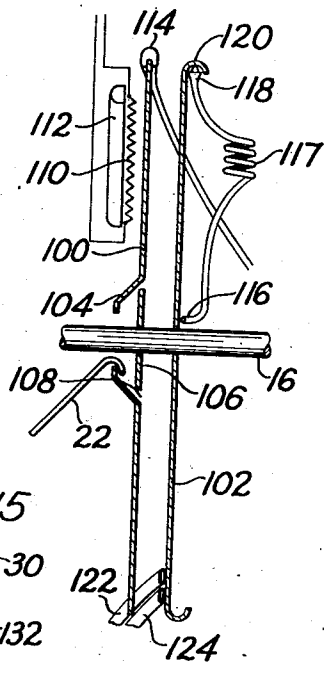


Fig. 14

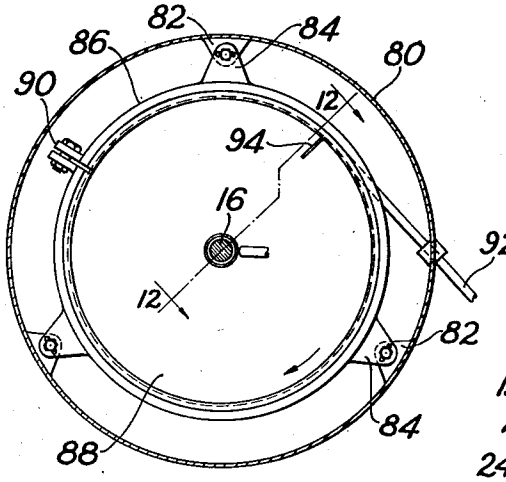


Fig. 11

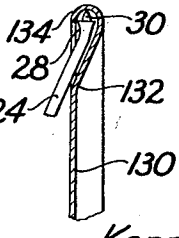


Fig. 15

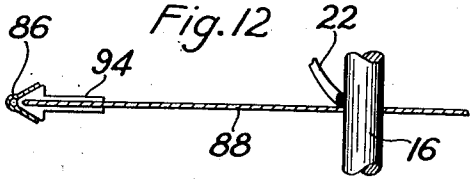


Fig. 12

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UNITED STATES PATENT OFFICE

2,180,053

DISTILLATION APPARATUS

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Application June 29, 1938, Serial No. 216,551
In Great Britain July 29, 1937

8 Claims. (Cl. 202—236)

This invention relates to improved means for distilling under reduced pressure. The invention in particular relates to improved high vacuum unobstructed path distillation apparatus.

In my United States application 99,632, filed September 5, 1936, I have described distillation apparatus wherein the distilland is caused to flow in the form of a thin film at high speed over the distillation or vaporizing surface of a high vacuum, evaporative or short path still. This is accomplished by introducing the distilland in liquid state onto a heated rapidly rotating surface. The liquid is caused to flow to the periphery of the heated surface by centrifugal force. In the same application I also describe the feature of employing a rotating condensing surface and causing the liquid condensate to flow to the periphery of the condensing surface by centrifugal force.

In both cases the liquid which was forced to the periphery by centrifugal action, was caught in a gutter suitably located around the spinning surface. The constructions have outstanding advantages over prior art methods of distillation.

This invention has for its object to provide improved vacuum distillation apparatus of this general nature. Another object is to provide improved centrifugal, short path distillation apparatus Another object is to provide improved methods of removing organic oils or liquids from rotating high vacuum distillation surfaces. Other objects will become apparent as the description proceeds.

In the drawings and description, I have set forth several of the preferred embodiments of my invention. However, it is to be understood that they are described for the purpose of illustration and that my invention is not limited thereto.

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

Figs. 1, 3 and 6 are fragmentary vertical sections of different liquid removal means;

Figs. 2, 4 and 7 are fragmentary perspective views, partly in section of the apparatus illustrated in Figs. 1, 3 and 6 respectively;

Fig. 5 is a fragmentary perspective partly in section of a modification of the liquid removal means illustrated in Figs. 3 and 4;

Fig. 8 is a vertical section of a modification of the removal means illustrated in Figs. 6 and 7;

Fig. 9 is a fragmentary elevation of another type of liquid removal means;

Fig. 10 is a view taken on line 10—10 of Fig. 9;

Fig. 11 is a vertical section through a still provided with a centrifugal distilling plate and a liquid removal device embodying the principles of my invention;

Fig. 12 is a view taken on line 12—12 of Fig. 11;

Fig. 13 is a fragmentary perspective of the scraping device of Fig. 11.

Fig. 14 is a vertical section of the moving parts of a centrifugal still provided with rotating vaporizing and condensing plates equipped with the various removal devices illustrated in Figs. 3, 8 and 9;

Fig. 15 is a fragmentary vertical section of a slight modification showing a turned edge construction which does not obstruct passage of gas from between the rotating plates.

In Figs. 1 and 2 numeral 16 designates an approximately horizontal shaft upon which is mounted a vertical circular plate 18, the periphery of which is turned back to form a gutter having an approximately semi-circular cross section as indicated at 20. Numeral 22 designates a conduit by means of which liquid distilland is introduced onto the center of plate 18. Numeral 24 indicates a conduit, the end 26 of which protrudes into the turned edge 20, and is in light contact with the bottom of the gutter formed by the turned edge.

During operation of a still provided with this equipment disc 18 is caused to rotate at relatively high speed by means of force applied to shaft 16 and liquid distilland is simultaneously introduced through conduit 22, the liquid flows onto the center of disc 18. It is then immediately forced to the periphery by centrifugal force. During the passage over the plate it is in the form of a very thin film. Plate 18 being a vaporizing surface will be heated by means (not shown) to distillation temperature. The thin film is therefore effectively distilled during its passage over the plate. Undistilled residual liquid passes into the gutter formed by turned edge 20. As it accumulates it is caught in the end 26 of conduit 24 and flows by its own inertia and/or gravity through the conduit and can be conveyed to any required remote point, or to another distilling plate.

The end 26 of conduit 24 is preferably in light contact with the inside of the turned edge 20. However, it can be at a slight distance therefrom and effectively remove liquid since the liquid will accumulate until it has a sufficient depth to be caught by the end of the conduit.

The construction shown in Figs. 3 and 4 is similar to that shown in Figs. 1 and 2, the dif-

ference being in the construction of the scoop or liquid pick-up means. In this construction conduit 24 is enlarged at the end as indicated at 28. A small scoop or scraper 30 is mounted in the open end of the conduit and extends into the gutter formed by turned edge 20. This scoop is shaped somewhat similar to the hollow end of a pen point or a spoon. It points in a direction opposite to that in which the disc 18 rotates. It may be in actual contact with the turned edge 20 or may merely be in close proximity thereto.

During rotation of plate 18, liquid which passes into 20 is effectively caught by the scooping action of point 30 and is conveyed into removal conduit 24. The enlarged portion 28 effectively catches any small amount of spray or droplets.

Referring to Fig. 5, numeral 36 designates a funnel shaped enlargement at the end of conduit 24. The upper open portion of 36 is oval and extends into close proximity to the turned edge 20. A semi-circular scoop 38 protrudes from the center and is in light contact with the gutter. A plurality of semi-circular vanes 40 are mounted within 36.

The operation and construction of the apparatus of Fig. 5 is quite the same as that of Figs. 3 and 4. However, the construction of member 36 and provision of vanes 40 more effectively assures that all splashings are caught and removed by the conduit 24.

In Figs. 6 and 7, numeral 46 designates the centrifugal plate of a high vacuum still. The periphery is not turned as in the previous figures, but extends into an annular gutter 48. Numeral 50 designates a scoop or scraper which is preferably in light contact with the surface of disk 46 and which is connected to removal conduit 24.

During operation liquid introduced through conduit 22 is spun to the periphery of plate 36 by centrifugal action. As soon as the liquid reaches scoop 50 it is scraped or wiped up from the surface of the disc. The removed liquid then flows into conduit 24 connected to the scoop. Any liquid which is not removed by the scoop is caught in gutter 48 from which it may be removed by a conduit (not shown) connected to the base thereof. This is used only as a secondary guard to catch the small portions of distilland which escapes the main collecting mechanism 50. Contact between scoop 50 and the plate should be resilient and can best be maintained by a spring.

It has been found that with speeds of about 3000 R. P. M. the liquid will proceed outward at speeds of between 1 and 100 cm. per second according to the rate of supply and the dimensions of the p'ate. Manifestly if the collecting tool 50 has a width greater than the distance of travel of the distilland during one rotation, substantially the whole quantity of liquid residue will be caught in the collecting scoop.

Instead of using one device 50, a plurality of such devices may be used. This expedient is illustrated in Fig. 8 where two scoops 52 and 54, similar to 50 of Fig. 6, are employed. The use of a plurality of scoops or of a single extra wide scoop, preferably divided by partitions, more surely removes residual liquid and therefore makes guard ring 48 unnecessary.

Referring to Figs. 9 and 10, numeral 46 indicates a plate having a flat periphery as described in connection with Figs. 6–8. Numeral 60 designates an enlargement at the end of conduit 24, the central portion of which is depressed. The walls 62 and 64 and the bottom 66 of this depression are so shaped as to come into intimate contact with the sides and end of the plate 46. Two small gutters 68 and 70 are thus formed. The enlargement extends beyond the depression at 72 to form a shield.

During operation the plate 46 is rotated in the directon indicated. Liquid is introduced onto the center of the plate through conduit 22. Undistilled liquid is thrown to the periphery by centrifugal force where it is scraped or wiped from the plate by one or more of the surfaces 62, 64 and 66. This removed liquid flows along the gutters 68 and 70 and into conduit 24. Droplets are caught by shield 72 and conveyed to conduit 24 in a similar manner.

Referring to Figs. 11, 12 and 13, numeral 80 designates a closed cylindrical still casing and numeral 82 a plurality of lugs integral therewith. Similar lugs 84 support an adjustable ring 86. This ring is provided with an annular V-shaped gutter which surrounds vaporizing plate 88 and is close to its periphery. Ring 86 is split at 90 so that the clearance between it and the periphery of plate 88 can be varied. A conduit 92 communicates with the V-shaped gutter at a tangent and serves to carry away liquid contained therein. Numeral 94 designates a cleft scraper, the cleft portion of which slips over plate 88 and the corresponding uncleft portion of which fits into a slot in the periphery of plate 88. The head of scraper 94 is the same shape as the V-shaped gutter in ring 86 and during rotation extends into this gutter and makes resilient contact therewith.

During operation the plate 88 is caused to rotate and the ring 86 is adjusted at 90 so that liquid introduced at conduit 22 is received into ring 86 without splashing and so that scraper 94 smoothly engages with the V-shaped gutter. The liquid is partly distilled in its travel over plate 88 as described above. Undistilled residue is thrown into the V-shaped gutter. The liquid in the gutter is scraped or wiped up by each rotation of scraper 94 and pushed or thrown into removal conduit 92.

The action of scraper 94 is not definitely understood but it is thought that a minute wave of liquid is formed in front of the scraper and that this small body of liquid passes into the conduit 92 as it passes over the opening between the gutter and the conduit.

In Fig. 14 reference numeral 100 designates a vaporizing surface and 102 a condensing surface located at a short distance therefrom. Surface 100 is punched out at the center to form a truncated cone 104 into which extends conduit 22. A plate 106 integral with shaft 16 is of smaller diameter than the punched out portion of plate 100 so as to leave a space 108 therebetween. Plate 106 supports plate 100 by a plurality of supports extending across space 108. These supports have not been shown in the drawings because it would impair their clearness. One side of the plate 100 is heated by means of an electrical heating element 110, the heat of which is reflected against the plate by reflector 112. Numeral 114 designates a scoop device similar to that illustrated in detail in Figs. 9 and 10. Numeral 117 designates a cooling coil, one end 116 of which terminates near the center of plate 102 and the other end of which is provided with the removal device 118 illustrated in Figs. 3 and 4, which extends into the turned edge 120. Numerals 122 and 124 designate scoop removal devices similar to those illustrated in Fig. 8.

In operating the apparatus illustrated in Fig. 4 the system in which the plates are located is evacuated to the desired low pressure. Shaft 16 is then caused to rotate at a suitable speed. Heating element 110 is put into operation and liquid distilland is introduced through conduit 22 onto the inside of cone 104. The liquid travels through space 108 and over the surface of plate 100. Since the plate is heated to distillation temperature the liquid distilland is distilled during its passage thereover. Undistilled residue is removed by means of device 114. Vapors pass to plate 102 and are condenser thereon. The liquid condensate is caused to pass to the periphery of plate 102 due to its high speed of rotation. As the liquid arrives at the periphery of the plate it is caught in scoops 122 and 124 and removed from the still.

The condensing plate 102 must be maintained at a rather low temperature. This is accomplished by introducing a cooling fluid into coil 117 which is located outside the still where it can be cooled. The fluid flows by gravity through conduit 116 to the center of plate 102, is then thrown by centrifugal force into turned edge 120, is then caught in scoop 118 and returned to cooling coil 117 by gravity. This cooling liquid should be of a suitable low vapor pressure so that it will not interfere with the high vacuum distillation.

In connection with Fig. 14 it will be noted that if both plates were provided with edges turned back towards each other there would be little space for removal of gas from the space between them.

Fig. 15 illustrates a turned back edge which overcomes this problem of gas removal. The plate 130 is bent backwards or offset at 132 so that the turned back edge at 134 is on about the same plane as the plate 130. When two such plates are used in combination there will be ample space for removal of gases.

In the interests of clarity I have shown only those parts of a still necessary to illustrate how my invention is applied thereto. In a complete working setup the plates will be mounted in a closed system which can be evacuated by a conduit connected thereto. Several pairs of plates can be mounted in the same system or chamber.

While many of the figures are limited to vaporizing surfaces the same expedients are suitable for use in connection with the removal of condensate and/or cooling fluid from rotating condensing surfaces. Rotating condensing surfaces need not be used. The whole still assembly has not been illustrated since its construction is obvious. The surfaces are preferably non-horizontal to permit the taking advantage of the pumping effect of the invention. The invention is applicable to horizontal surfaces with many attendant advantages. When solids are condensed a rotating condensing surface can be employed in conjunction with a scraper which will remove the solid condensate which can then be permitted to whirl away into large annular chambers at the periphery of the plate.

The procedure is particularly useful in high vacuum unobstructed path distillations, i. e., where the condensing and vaporizing surfaces are separated by substantially unobstructed space and a high vacuum such as a pressure below approximately 1 mm. exists therebetween. High vacuum short path conditions are almost generally satisfactory, namely, distillation as described where the distance between the two surfaces is short and of the order of less than 1 foot and preferably about ½ to 6 inches. When the distance between the surfaces is less than the mean free path the distillation is of a type widely known as molecular distillation.

From a consideration of the figures it will be noted that this method of liquid removal has the advantage that a pumping action is simultaneously performed. The liquid is introduced at the center of the plate and is removed at the top of the plate. The liquid therefore has been lifted a distance which corresponds to the radius of the plate. This is advantageous where a plurality of plates are employed in one still unit. The liquid removed at the top of the plate can be allowed to flow to the center of the next plate where it is again spun to the top of the plate. This can be repeated any required number of times without necessitating the provision of expensive lifting pumps.

The collecting tools have a further advantage. When liquids are collected in gutters, they flow with considerable slowness to the withdrawal conduit connected to the gutter and tend to accumulate in rather large amounts. If the liquid is hot when it is removed from the plate considerable thermal-decomposition can take place during the period of time that it remains in the gutter. Also cooling and corresponding heat loss take place. In addition the invention has the decided advantage that condensing and vaporizing plates can be placed much closer together than is possible with gutter collecting means. Placing gutters at the peripheries of the plates materially restricts the space for removal of gases from between the plates. By eliminating the gutters, the plates can be placed very close together. This results in extremely high efficiency. Spraying and splashing is also greatly reduced.

What I claim is:

1. Vacuum distillation apparatus comprising in combination within a closed system a non-horizontal rotatable disc, means for heating the disc, means for introducing liquid distilland onto the approximate center of the disc, means located at approximately the highest point of the disc for removing undistilled liquid residue from the periphery of the disc before it has been thrown from the disc by centrifugal force, a condensing surface located in close proximity to the disc and separated therefrom by substantially unobstructed space and means for applying a suction to the closed system whereby the pressure within the system and between the disc and the condensing surface can be reduced.

2. Vacuum distillation apparatus comprising in combination within a closed system, an approximately vertical rotatable disc, heating means located on one side of the disc, means for introducing liquid distilland onto the approximate center of the opposite side of the disc, a scraper located at the periphery of the disc and above the axis of the disc which is adapted to remove the liquid undistilled residue from the surface of the disc, a conduit connected to the scraper whereby liquid removed is carried away, a condensing surface located opposite the side of the disc upon which the distilland is introduced, which is in close proximity to the disc and separated therefrom by substantially unobstructed space, means for removing condensate from the condensing surface and a conduit for removing gases from the system and the space between the disc and the condensing surface.

3. Vacuum distillation apparatus comprising in

combination within a closed system, a rotatable disc-shaped vaporizing surface, means for heating the surface, means for introducing liquid distilland onto the approximate center of the surface, means for removing undistilled liquid residue from the periphery of the surface where it is caused to flow by centrifugal force, a condensing surface located in close proximity to the vaporizing surface and separated therefrom by substantially unobstructed space and means for applying a suction to the closed system whereby the pressure within the system and between the disc and the condensing surface can be reduced.

4. Vacuum distillation apparatus comprising in combination within a closed system, a rotatable disc-shaped vaporizing surface heating means located on one side of the surface, means for introducing liquid distilland onto the approximate center of the opposite side of the surface, a device located at the periphery of the surface which is adapted to remove the liquid undistilled residue from the surface, a conduit connected to this device whereby liquid removed is carried away, a condensing surface located opposite that side of the vaporizing surface upon which the distilland is introduced, which is in close proximity to the vaporizing surface and separated therefrom by substantially unobstructed space, means for removing condensate from the condensing surface and a conduit for removing gases from the system and the space between the disc and the condensing surface.

5. Vacuum distillation apparatus comprising in combination within a closed system, a rotatable disc, a heating element located on one side of the disc and adapted to heat the disc by radiation to distillation temperature, means for introducing liquid distilland onto the approximate center of the opposite side of the disc, a scraping device in light contact with the same side of the disc at its approximate periphery which is adapted to remove liquid undistilled residue from the disc, a conduit connected to the scraping device to carry away liquid thus removed from the disc, a rotatable condensing surface located at a short distance from the disc and separated therefrom by substantially unobstructed space, a scraping device in light contact with the condensing surface at its approximate periphery which is adapted to remove liquid condensate therefrom, a conduit connected to the scraper to carry away liquid condensate thus removed and a conduit for evacuating the system and the space between the disc and the condensing surface.

6. Vacuum distillation apparatus comprising in combination within a closed system an approximately vertical rotatable disc, a heating element located on one side of the disc and adapted to heat the disc to distillation temperature by radiation, means for introducing liquid distilland onto the approximate center of the opposite side of the disc, a scraping device in light contact with the same side of the disc located at a point above the axis of the disc and at its approximate periphery, which device is adapted to remove liquid undistilled residue from the disc, a conduit connected to the scraping device to carry away liquid thus removed from the disc, a rotatable condensing surface located approximately parallel to and a short distance from the disc and separated therefrom by substantially unobstructed space, a scraping device in light contact with the condensing surface which is located at a point above the axis of the condensing surface and at its approximate periphery and which is adapted to remove liquid condensate therefrom, a conduit connected to the scraper to carry away liquid condensate thus removed and a conduit for evacuating the system and the space between the disc and the condensing surface.

7. Vacuum distillation apparatus comprising in combination within a closed system, a rotatable disc means for heating the disc, a conduit for introducing liquid distilland onto the approximate center of the disc, an annular gutter surrounding the periphery of the disc and in close proximity thereto, a scraper mounted upon the periphery of the disc so as to make resilient contact with the bottom of the gutter, a conduit connected to the gutter at a tangent in such a manner as to carry away liquid deposited therein by the scraper, a condensing surface located in close proximity to the disc and separated therefrom by substantially unobstructed space, and a conduit for removing gases from the system and the space between the disc and the condensing surface.

8. Vacuum distillation apparatus comprising in combination within a closed system, a rotatable disc the edge of which is turned to form an annular gutter having an approximately semicircular cross section, means for heating the disc, means for introducing liquid distilland onto the approximate center of the disc, a scoop mounted so that it protrudes into the gutter at the periphery of the disc whereby liquid therein is removed, a conduit connected to the scoop for carrying away the liquid removed from the disc, a condensing surface located near the disc and separated therefrom by substantially unobstructed space, means for removing condensate from the condensing surface and a conduit for removing gases from the system and the space between the disc and the condensing surface.

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