May 28, 1940. M. H. ITTNER 2,202,008

DISTILLATION OF FATTY ACIDS

Original Filed June 11, 1936 3 Sheets-Sheet 1

Fig. 1.

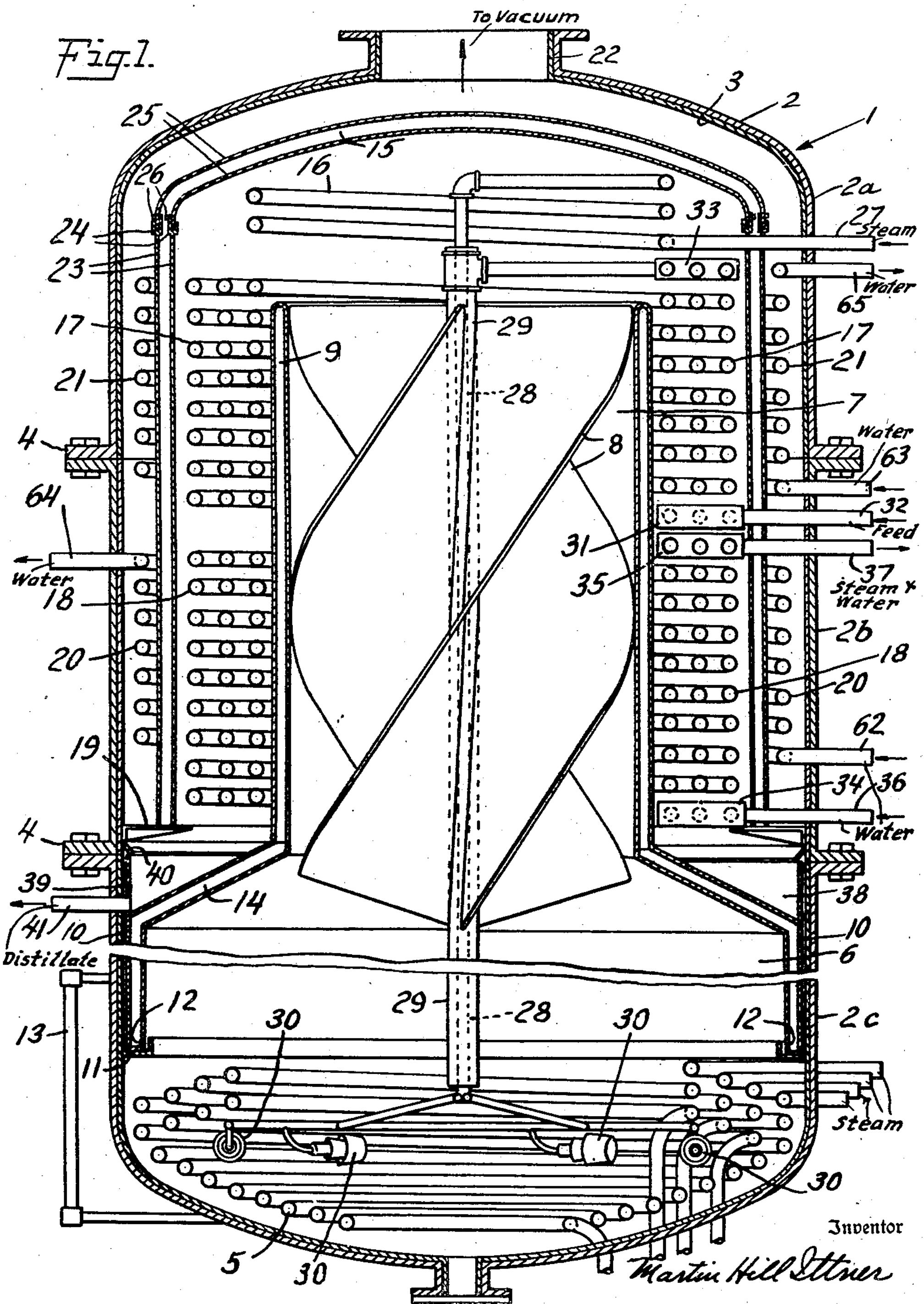

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Fig. 2.

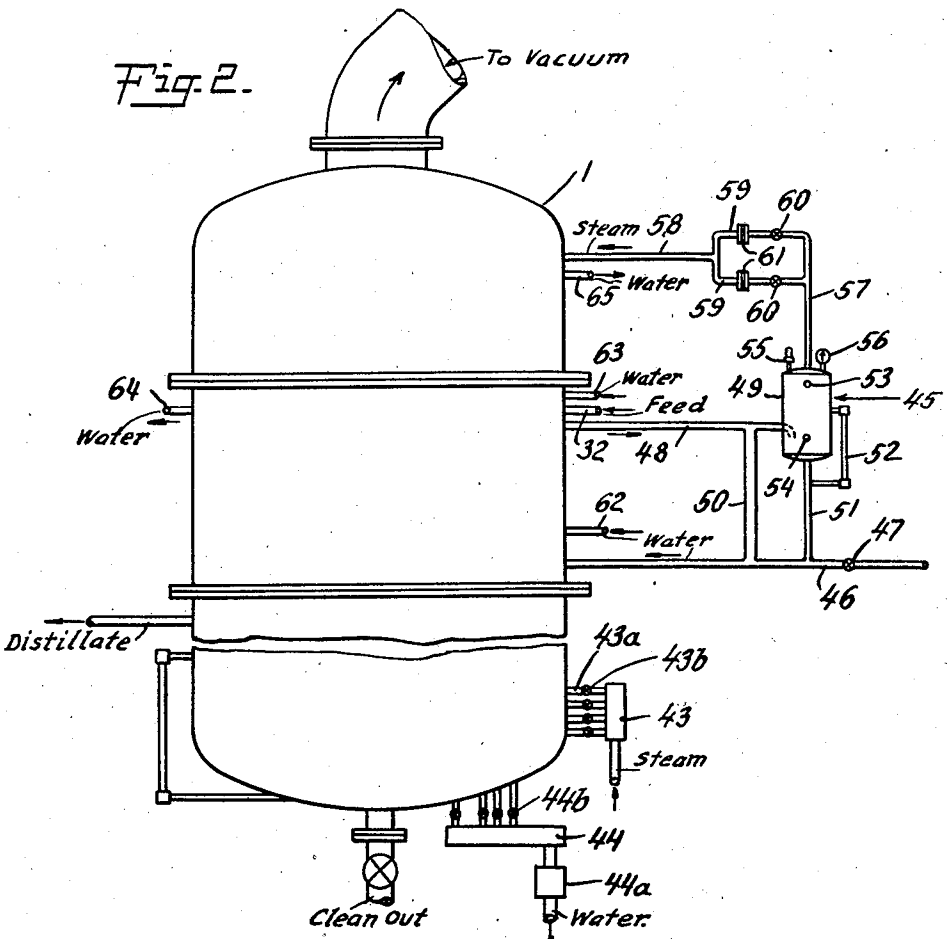
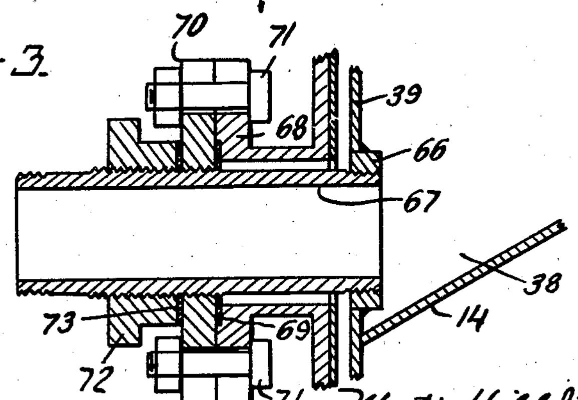


Fig. 3.



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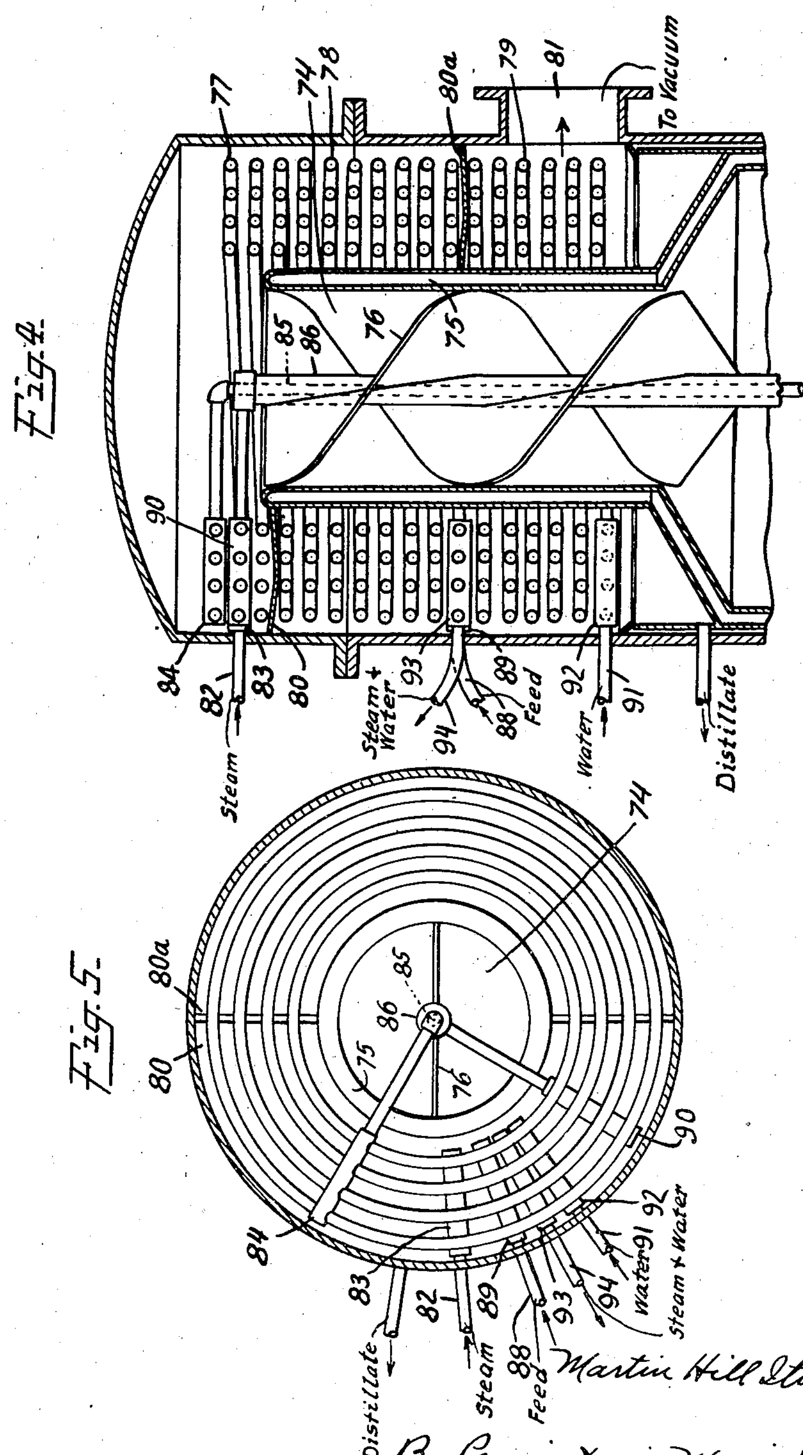

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UNITED STATES PATENT OFFICE

2,202,008

DISTILLATION OF FATTY ACIDS

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19 Claims. (Cl. 202—177)

This invention relates to improvements in apparatus for the distillation of high boiling liquids, and more particularly to improvements in apparatus for the distillation of fatty acids.

The improved apparatus of the present invention makes possible the distillation of fatty acids and other high boiling liquids with a radically smaller heat consumption than has heretofore been possible, while at the same time the quality of the fatty acids distilled is improved, and the losses in distillation, due to the formation of tars and to decomposition, are substantially lessened.

The apparatus of the present invention, while adapted for, and useful for, the distillation of high boiling liquids generally, particularly such high boiling liquids as are not miscible with water and are advantageously distilled by a vacuum-steam process, such as high boiling petroleum fractions, is particularly adapted for, and particularly designed for, the distillation of fatty acids. The term "fatty acids," as used herein, and as commonly used in industry, includes not only the true fatty acids, which are saturated, but also the related unsaturated acids with varying degrees of unsaturation. These fatty acids are commonly obtained by the saponification of natural fats and oils, which consist of triglycerides of the fatty acids, and are generally obtained as mixtures of various saturated fatty acids and various unsaturated fatty acids. In general, the saturated fatty acids are more stable than the unsaturated fatty acids, and are more resistant to decomposition by heat and to oxidation.

The improved apparatus of the present invention makes it possible to distill fatty acids with a radically lowered heat consumption because it permits the recovery of the heat from condensation of the fatty acid vapors as well as sensible heat of the fatty acid vapors and the hot condensed fatty acids, and, where jet steam is used to aid in the distillation, much of the sensible heat of the water vapor, and the utilization of such heat for preheating the fatty acids and producing the jet steam used in the distillation, and also increases the economy of the operation by decreasing the losses due to decomposition and tar formation. This apparatus permits the use of much higher vacua, or lower absolute pressures, within the still and in the vapor space over the liquid being distilled and thereby reduces the amount of jet steam required to aid in the distillation and also lowers the temperature at which the distillation may be carried out in a commercial manner.

The apparatus as a whole in its preferred embodiment includes a heating zone, in which the fatty acids being distilled are heated and in which the volatilization takes place, a vapor space above the heating zone, a separator, in which the vapors are separated from entrained material and a suitable cooling zone, in which the fatty acid vapors are condensed and the accompanying water vapors cooled, all within a single air-tight shell similar to the shells of stills now commonly provided. The apparatus is also provided with suitable means for supplying heat to the material being distilled, such as high pressure steam coils, and with ejector jets for mixing the contents of the still with superheated steam if desired, the jets being so located as to insure effective and uniform heating of the still contents by the heating coils; suitable insulating means for preventing the passage of heat from the separating zone, through which the vapors pass directly from the vapor space above the still contents, to the cooling zone, or zones, which is or are annular and surrounding the separator, the insulating device advantageously defining the separator; and a suitable connection to a vacuum device to permit the flow of water vapor from the still and to insure the maintenance of a low pressure in the still.

In the apparatus, all passages through which the vapors must pass, that is, the vapor space above the still contents, the separator, and the cooling zone or zones are of such substantial size that very little resistance to flow of the vapors is offered, and the pressure difference between different parts of the still is very slight, a pressure difference as small as a fraction of a millimeter of mercury between the vapor space just above the still contents and the vacuum device outside of the still used to maintain the vacuum within the still being easily obtainable. Because of this small pressure drop, the apparatus may be operated, and the process carried out, while pressures as low as three millimeters of mercury are maintained within the still while using open jet steam. The low pressure which may be maintained within the still is distinctly advantageous, as it greatly decreases the temperature at which effective and rapid distillation of the fatty acids can take place, and thus reduces to a substantial extent the decomposition and tar formation which ordinarily occurs.

The invention will be further illustrated and explained in connection with the accompanying drawings, which show, in a diagrammatic fashion, apparatus which embodies the invention, but the invention is not limited thereto.

In the drawings:

Fig. 1 is a vertical section, with parts in elevation, of a preferred embodiment of the still;

Fig. 2 is an elevation showing a suitable arrangement of the various parts of the apparatus;

Fig. 3 is an enlarged section of a suitable device for drawing fatty acids from the still;

Fig. 4 is a vertical section of a portion of a still, showing a modified arrangement of the cooling zones; and

Fig. 5 is a horizontal section of the still of Fig. 4.

In Fig. 1 the still 1 is shown as provided with an outer shell 2, with a lining 3 of a suitable corrosion-resistant metal, such as stainless steel, attached thereto. The shell is advantageously made in three sections, *2a*, *2b*, *2c*, bolted together by flanges 4. The interior of the still is divided into several zones or sections in which the operations of the process take place. At the bottom of the still are provided coils 5 which may be provided with high pressure steam, or other suitable heating medium, to provide the heat necessary for the volatilization of the fatty acids. The level of the liquid being distilled is normally maintained somewhat above the heating coils. Above the heating coils, and above the liquid, is a vapor space 6 leading directly into the separating zone 7, which is of large diameter and is provided with the helical or spiral fins 8. The vapor space and the separator are defined by the double-walled partition 9 which is advantageously made of relatively thin metal and with polished surfaces. Small holes 10 are provided in the outer sheet of this insulating partition to provide communication between the still and the interior of the partition so that the same pressure exists within the partition as exists within the still proper, these holes communicating with a relatively cool portion of the still, so that no condensable vapors, which might condense between the walls of the partition, and impair its insulating efficiency, can enter through them into the space between the walls and condense. This partition rests upon the lipped ledge 11, which provides a small ledge in which liquid fatty acids collect and form a liquid seal to prevent the transfer of vapors between the still wall and the insulating partition. A few small holes 12 may be provided at the bottom of the inner wall of this partition to permit the drainage of any material which may get within the partition. The level of the liquid being distilled is normally maintained a little above the ledge 11. A gauge glass 13 is provided to determine the level of the liquid.

The insulating partition defines the separating zone, which is of a diameter approximately one-half the diameter of the shell, and below the separating zone flares out to the diameter a little less than that of the shell, as shown at 14, forming the vapor space above the liquid, and providing a throat leading the vapors into the separating zone, thus serving not only to insulate the sides of the separating zone, but also to insulate the vapor space and prevent or minimize losses of heat through the sides of the still adjacent to the vapor space. Above and surrounding the upper portion of this insulating partition is a bell-shaped partition 15 which is double-walled, and which serves to deflect the vapors which pass upwardly through the separating zone down past the series of coils 16, 17 and 18, which constitute the primary cooling agencies, where the vapors are cooled and a large proportion of the fatty acids condensed. This bell-shaped deflector is supported on brackets 19, and the vapors after passing down between this bell and the insulating partition pass up between the bell and the wall of the still, past the coils 20 and 21 and then pass out through the pipe 22 to a suitable vacuum device.

The deflector bell is advantageously double-walled, and is made of a suitable corrosion-resistant metal. To simplify the assembling of the still, the bell-deflector is advantageously made in sections, the lower section consisting of the two concentric cylinders 23, provided with double lip gutters 24 at the top, and the upper section consisting of an inner and an outer dome 25, which rest in the gutters and are held in place by bolts 26. Suitable holes are provided in the bell-deflector for the passage of various tubes leading to the coils which are placed between the insulating partition and the bell-deflector.

Three sets of coils 16, 17 and 18 are provided between the partition and the bell-deflector. Of these, the upper section 16, with an inlet 27 is used for superheating the steam. The steam passes through this coil where it is superheated and then passes down the inner pipe 28 of the double pipe 29 provided in the center of the coil and passes out of this pipe through the jets 30, which are preferably arranged so that the discharging steam and the fatty acids which are intimately admixed with it in the jets have a horizontal path, and are forced into intimate contact with the heating pipes 5 thus insuring the uniform and effective heating of the liquid, and its intimate admixture with the superheated jet steam.

The second set of coils 17 leads from the manifold 31 into which the fatty acids to be distilled are introduced through inlet 32 to the manifold 33, and thence to the outer pipe of the double pipe 29 which runs down the center of the still and discharges the heated fatty acids into the liquid at the bottom of the still somewhat below the upper surface.

The third set of coils 18 leads from the manifold 34 to the manifold 35 and these coils are used to vaporize the water and form the steam which is subsequently superheated and used as jet steam in the distillation, water being introduced through inlet 36, and hot water and steam being taken from outlet 37. The vapors thus pass over the three sets of coils in series, superheating the steam, preheating the fatty acids, and producing the steam which is used in the distillation. These coils are so arranged that any fatty acids which may be deposited upon them by condensation are not returned through the separating zone to the liquid in the still, but drip down and enter the trough 38 which is formed by the flaring portion of the partition 14, the cylindrical dam 39, formed by extending upwardly the outer wall of the insulating partition, and the guide lip 40, which is fastened to the inner lining of the outer shell and which deflects any fatty acids which may condense on the wall of the still down into the trough. Between the bell-deflector and the outer wall or shell of the still there are provided cooling coils 20 and 21, through which cold water or other cooling agent may be circulated to further cool the vapors after they have been partially cooled by the other coils, and thus to condense the last traces of fatty acid and to insure that the vapors which pass out of the still are substantially free from fatty acids. In the apparatus illustrated, two such cooling coils are provided; but one cooling coil may be used if desired, or more than two cooling coils may be used.

An outlet 41 is provided at the side of the still from the trough for the removal of the fatty acids which condense and pass down into the trough where they are collected. At the bottom of the still there is an outlet 42 for the discharge of still residues.

The still illustrated in Fig. 1 may be made in various sizes, and with wide variations in the proportions of the various zones and parts of the apparatus. For a still designed to distill about 2400 pounds of fatty acids per hour, a still having an outer shell about 8 feet in diameter may be provided, which may be about 15 feet high. The separating zone, which should be quite large, may be about 4 feet in diameter and about 6 feet high, the vapor space beneath the separator may be about 3 feet high, with the heating zone where the liquid fatty acids are heated being about 3 feet deep. The deflector-bell may be about 7 feet in diameter and about 7 feet high. A still of such dimensions provides adequately large vapor spaces or passages which offer no substantial resistance to the vapor flow. The cooling coils, and the heating coils, may be made of 2 inch tubing, of a suitable corrosion-resistant metal.

Fig. 2 is a diagrammatic sketch of the exterior of the still, showing the general arrangement of the exterior parts. In this figure, the still is shown at 1 with the heating coils connected, by pipes 43a provided with valves 43b, to a suitable manifold 43 connected to a high pressure steam supply to supply the steam required for heating, and with the manifold 44 and trap 44a into which the heating coils discharge through valves 44b, this trap being such that only water is allowed to pass, so that none of the steam is removed from the coils until it has given up its heat from condensation.

There is also shown a suitable device 45 for supplying cooling water to the coils 18 and for separating the steam which is formed in the coil from the water. The cooling water may be brought into pipe 46 from an outside source, the flow being controlled by the needle valve 47. Distilled water is preferably used. The water flows through the tube 48 and connection 36 and manifold 34 into the coils, where a part of it becomes converted into steam by the heat absorbed from the fatty acid vapors which flow over the coils and the heated cooling water and the steam generated rise through the coil and flow out through the connection 37 and the tube 48. Most of the water returns to tube 46 through connection 50, while the steam and the rest of the water go into separator 49, in which the water may return to tube 46 through connection 51. The level of the water in the separator is maintained a little below tube 48 at the level of outlet 54, and is indicated by the gauge glass 52.

A suitable connection 53 is provided at the top of the separator for supplying steam from an outside source when starting operation, and with a suitable pressure relief valve 54 for the discharge of excess steam and water. Another pressure relief valve 55 may be provided at the top of the separator. A pressure gauge 56 may be provided at the top of the separator to show the pressure maintained within the chamber. The chamber is preferably maintained at a superatmospheric pressure, e. g. 5 pounds gauge or less, to avoid the possibility of air leaking into the separator and into the still. At the top of the separator is provided an outlet 57 for the separated steam which is connected to tube 58 which leads through connection 27 to the superheater coil by the parallel tubes 59, each provided with a valve 60, and with an orifice 61, which may be of the same size or of different sizes, and which enable the flow of steam from the separator to the superheater coil to be adjusted to a predetermined, desired amount.

The cooling coils 20 and 21, between the bell-deflector and the outer wall of the still may be supplied with cold water through inlets 62 and 63 and may discharge through outlets 64 and 65, the cold water thus flowing concurrently with the outgoing vapors, or the direction may be reversed.

In Fig. 3 is illustrated a suitable connection for the removal of liquid condensed fatty acid from the trough 38 (Fig. 1). As illustrated, a bushing 66 is welded into the lower part of the dam 39 of the trough, and a special nipple 67 is provided to be screwed into this bushing. A flanged nipple 68 with an inner diameter somewhat greater than the outer diameter of nipple 67 is welded to the shell, so that the nipple 67 when connected to the bushing 66 is centrally located within the flanged nipple 68. Radially slotted bolt holes are provided around the flange of the nipple 68. The nipple 67 is threaded on each end with standard pipe threads and along a portion of its outer surface with machine threads having the same pitch as the pipe threads but having a larger outer diameter. A small recess is provided in the face of the flanged nipple 68 for a gasket 69. A smooth-faced flange 70 provided with radial slots corresponding to those in nipple 68 is provided with an inner hole threaded to match the machine threads on the nipple 67. Nipple 67 is then screwed into bushing 66 and the flange screwed down until it presses upon the gasket. The flanges are then bolted together with bolts 71 and tightened so as to make a tight joint between them. A follower 72 is also screwed upon the outer part of nipple 67 with a grommet 73 between it and the outer face of flange 70 so as to make a tight joint around the pipe threads. Other means of connecting the fatty acid outlet to the still may be used. Similar devices may be used for making the connections to the cooling coils to insure that these connections are vacuum-tight and that air does not leak into the still at these points.

In Figs. 4 and 5 there is illustrated the upper part of a still showing a modified arrangement of the cooling zone, with a modified vapor passage, which may be used. In the modification of the apparatus illustrated in these figures, there is provided a separating zone 74, defined by an insulating partition 75 of the same construction as that shown in Fig. 1, within which are the helical or spiral vanes 76, two in number, which serve to remove entrained matter from the vapors. In this modification, no deflector-bell is used, the vapors passing from the top of the separating zone and being deflected by the top of the still. Three sets of cooling coils, 77, 78 and 79, are provided in the zone between the insulating partition and the wall of the still, the vapors after being deflected from the top of the still passing over these coils. These coils are used for much the same purposes as those provided in the apparatus described in Fig. 1, the upper coil being used to superheat jet steam, the central coil to preheat fatty acids, and the lower coil to generate steam. There are also provided two helical baffles 80 and 80a having about the same pitch as the cooling coils and each extending a little more than one-half of the way around the insulating partition 75, which deflect the vapors so that instead of flowing downwardly over the cooling coils, they flow downwardly and around the insulating partition in a helical or spiral path, finally passing out of the still through the opening 81 to the vacuum devices.

In the arrangement shown in Figs. 4 and 5, care should be taken that the cooling coils have an adequate area to cool the vapors to an extent sufficient to insure that substantially all of the fatty acids are condensed within the still, so that no substantial amount passes on to the vacuum devices. The fatty acids condense on the cooling coils, and drop down into a collecting trough, which is of the same construction as shown in Fig. 1.

In the arrangement shown in these figures, instead of a single superheating coil being used, a plurality of coils is provided, the saturated steam entering through inlet 82, into manifold 83 passing through the coils 77 to be superheated and into manifold 84 and then into the inner pipe 85 of double pipe 86 and to the jets. The fatty acids enter the coils 78 through inlet 88 and manifold 89, and pass spirally upward through the coils into manifold 90 and then pass into the outer pipe of the central double pipe. Cooling water is supplied through inlet 91 to manifold 92 from where it passes spirally upward through coils 79 into manifold 93 and then passes into a separator such as described in connection with Fig. 2 through outlet 94.

In the apparatus illustrated in Fig. 1, no insulation is provided on the still. It will be understood that insulation is provided at the lower part of the still, that is, around the portion of the still where the liquid material is maintained and somewhat up the sides of the still at the vapor space. It is not necessary to provide much insulation at the level of the vapor space, as the insulating partition effectively serves to insulate this portion of the still and to prevent any substantial loss of heat by radiation. Above the vapor space, no exterior insulation need be provided, and in fact, such insulation is generally undesirable. At this portion of the still, it is desirable to cool the vapors, and provision is even made for the introduction of cold water to provide for the complete cooling of the vapors. The walls of the still above the vapor space normally are quite cool, and, in any case, any heat which might be lost by radiation by these portions of the still does not rob heat from the heating zone, and does not lessen the thermal efficiency of the still, but may add to its condensing efficiency.

In operating the still illustrated in Fig. 1, a body of liquid fatty acids is maintained in the bottom of the still at a level somewhat above the top of the heating coils, and is heated by the high pressure steam in the coils. A high vacuum is produced in the still by a suitable vacuum device (not shown). Superheated jet steam is introduced through the jets into the liquid material, insuring the intimate contact of the liquid material with the heating coils and insuring the effective and uniform heating of the material. The vapors of the fatty acids, admixed with the water vapor, pass up from the liquid material into the vapor space 6 and then through the throat into the separating zone 7. Here the vapors are deflected by the helical vanes 8. The attenuated fatty acid vapors and water vapor are deflected readily by the vanes, and no substantial resistance to the flow of these vapors through the separating zone is offered. Any entrained liquid or non-volatilized material which is carried along with the vapors through the vapor space into the separating zone, being many times heavier than the vapors, is not diverted from its path and comes into contact either with the vanes of the separator or the insulating partition and flows down and drips back into the liquid in the bottom of the still. Any material which is so deposited on the vanes will flow downward along the line of greatest slope, which is toward the center and down the center of the vanes. This separator may be provided with any suitable number of vanes, four vanes being suitable, as the size of the passageway for the vapors is so great that no substantial resistance to their flow is offered. The vapors after passing up through the separating zone are deflected by the bell-deflector 15 and pass down over the three sets of coils between this bell and the insulating partition 9. In the lower set of coils, cooling water enters through tube 36 into the manifold 34 and passes upwardly through the spiral coils into the upper manifold 35, a large part of the water being converted into saturated steam, and the admixed water and steam being separated by connection 50 and separator 49, the water being returned to the coils and part of the steam being passed through one or the other, or both, of the orifices 61 in controlled amounts, and then passing through the upper coil 16 in which it is superheated. The saturated steam to be superheated thus comes into indirect contact with the hottest fatty acid vapors coming from the separating zone and is superheated to a temperature approximating the temperature of the liquid material in the still. The steam after being superheated passes from the coil downwardly through the inner pipe of the double pipe and is conducted by suitable connections to a plurality of steam jets in the lower part of the still where it is discharged as jet steam to assist in the distillation.

The bell-deflector 15 serves not only to deflect the vapors downwardly and over the cooling coils but also serves to prevent loss of heat from the vapors into the space above the bell-deflector and close to the top of the still. The vapors in this upper space are cool, and any heat supplied to them from the hot vapors from the separating zone would, of course, be wasted and lessen the efficiency of the condensation in the space between the bell-deflector and the outer still shell. The double-walled deflector-bell, which is advantageously made of polished metal, and which is highly evacuated, being in direct communication with the still, serves effectively as an insulating agent to prevent the transfer of heat from the hot vapors to the cooler vapors above, so that the vapors which come into contact with the superheater coils, and with the preheating coils, are substantially at the temperature of the liquid contents of the still.

The fatty acids which are fed to the still are preheated in the second set of coils 17 by the fatty acid vapors and the water vapor that accompanies them. The fatty acids, preferably dry, are introduced into the coils 17 by the tube 32 connecting with manifold 31 which distributes the fatty acids through the coils whence they pass spirally upward and discharge into the manifold 33. The fatty acids are thus preheated by heat exchange with countercurrent flow of the fatty acids and of the hot vapors, the fatty acids being fed to the still absorbing heat from the vapors by condensing the fatty acid vapors and cooling the hot condensed liquid and cooling the hot vapors. The preheating which takes place in these coils is sufficient to heat the fatty acids being fed to the still substantially to the temperature maintained within the still, as the hot vapors when they first come in contact with the preheater coils are almost at the still temperature, the amount of heat absorbed by the superheater coil being very small and not lessening the temperature of the fatty acid vapors to any substantial extent. The preheated fatty acids, substantially at the temperature of the still contents, are led down between the inner pipe and the outer pipe of the double pipe to a point below the level of the liquid in the heating zone and near its center. The amount of feed is regulated by the rate of distillation and is such as to maintain the liquid level in the still at its most effective point, the gauge glass indicating this level.

The heat exchange coils which condense fatty acids and cool the liquid condensed fatty acids and cool the fatty-vapors and the water vapor and generate and superheat the steam used as jet steam and preheat the fatty acids fed to the still are so placed that none of the fatty acids which may condense on their surfaces may drop into the separating zone and thence to the bottom of the still but all must run downward between the insulating partition and the bell and be collected in the trough. If desired, instead of collecting all of the condensed material in a single collector, separate collectors may be placed at various locations within this cooling zone to permit fractionation or fractional condensation of the hot vapors. For example, if fractional condensation of hot fatty acid vapors is desired, or if the still is used for the distillation of high boiling petroleum fractions where fractional condensation is desired, the still may be provided with a series of collectors along the cooling zone, so that as the vapors become progressively cooled, and as the condensate becomes more and more volatile, the more volatile constituents may be separated from the less volatile constituents.

After the vapors have passed down between the space between the insulating partition and the bell-deflector, they pass upwardly between the bell-deflector and the wall of the still, the supplementary cooling coils 20 and 21 being provided here to insure the complete condensation of fatty acid vapors before removing the water vapor from the still through the upper outlet which leads to the vacuum device.

Thus the fatty acids condense within the cooling zone with substantial completeness and run down into the collecting trough 38 from which they are removed through the outlet 41, which may be constructed as described above, by a pump or drop-leg or other suitable means.

Although the apparatus illustrated is shown with condensing coils, it is obvious that other equivalent means for indirect heat transfer and simultaneous condensation may be employed in place thereof. Although two general arrangements of the cooling means have been illustrated, it is obvious that other arrangements therefor and for deflecting the vapors in heat exchange relation may be employed without departing from the principles of the invention.

The amount of heat which is available for accomplishing the preheating operation, when the still is properly constructed and the process is properly carried out, is considerably in excess of that required. Thus in condensing the fatty acid vapors and in cooling the resulting liquid fatty acids and cooling the water vapor considerably more heat is given off than is necessary to generate and superheat the open jet steam employed and to preheat the fatty acid feed. This excess heat is absorbed in part by the supplementary cooling coils located between the bell-deflector and the wall of the still and is partly radiated from the outer part of the shell. Advantage is taken of the fact that there is excess heat available in the construction of the still, and in its operation. In the preheating operation, about 54% of the heat required is required for preheating the feed, about 42% is required for generating steam, and about 4% for superheating the generated steam. Because excess heat is available for these purposes, it is not necessary to proportion the area of coil surface for each of the sets of coils, as it is only necessary to provide each set of coils with a reasonable excess of surface over and above that required to enable the transmission to the material being heated in the coils the amount of heat required to accomplish the heating operation for which the coil is designed. The excess of coil surface provided in each set of coils cannot operate to cause one set of coils to absorb more heat than it requires, or to prevent another set from receiving the necessary heat, as in each set of coils only a limited amount of heat can be absorbed. Thus in the superheater coil, only sufficient heat to superheat the steam to about the still temperature can be absorbed. Similarly, in the coils provided for the preheating of the fatty acid feed, only sufficient heat can be absorbed to heat the fatty acids to about still temperature, and, as the coils used for the generation of steam are preferably located for absorbing heat after the vapors have passed the superheating and preheating coils, any heat absorbed by these steam-generating coils cannot deprive the others of heat. These last coils absorb more heat, and thus generate more steam, than is necessary for the supply of superheated jet steam to the still, and this excess steam can of course be used for other purposes. Similarly, the area of the surface of the cooling coils provided between the bell-deflector and the outer shell of the still does not have to be carefully proportioned, as it is merely necessary to insure that these coils sufficiently cool the outgoing vapors to insure the condensation of all of the fatty acids.

The outlet pipe through which the water vapor is conducted after the fatty acid vapors have been condensed and which leads to the vacuum devices does not have to be as large as the other vapor passages in the still, as the vapor which passes through this pipe has a volume which is much less than the volume of the vapors which pass, for example, through the separating zone. In the separating zone, and at the upper portions of the primary cooling zone, the vapors consist of a mixture of fatty acid vapors and water vapor, and if, as in a typical case, the amount of superheated jet steam used is about 10% of the weight of the fatty acids distilled, the ratio of the volumes of water vapor and fatty acid vapors is about 3 to 2. Thus when the fatty acid vapors are removed, the volume of the vapors which must flow through the apparatus is reduced by about two-fifths, and with the concomitant cooling of the water vapor, the volume is still more reduced, so that the volume of vapor which passes through the outlet is much less than that which passes through the separating zone.

In operating the apparatus as described, I find that it is possible to carry out the distillation with pressures in the still ranging from 3 mm. to 6 mm. of mercury, or even less. These low pressures within the still are made possible because the resistance to flow of the vapors offered in the various parts of the apparatus is extremely small, large passageways being provided for the vapors, so that the difference in pressure between the vapor space, for example, in the still and the portion of the outlet pipe adjacent to the vacuum devices may be as small as a fraction of a millimeter. Thus with a vacuum device capable of reducing the pressure in the outlet pipe to about 3 mm. of mercury, the amount of pressure required to force the vapors from the vapor space through the various parts of the apparatus and up to the vacuum devices may be as small as a fraction of a millimeter, with the result that the distillation itself is carried out within the apparatus and at a pressure of 4 mm. of mercury or less. This lowered pressure which is maintained within the still enables the use of considerably lower temperatures in the distillation, with consequent decrease in the decomposition of the fatty acids, particularly the unsaturated fatty acids which are always present in commercial fatty acids, and increases the yield of distilled acids and improves the quality of the distilled acids.

The only source of outside or external heat which need be used in operating the apparatus is the high pressure steam supplied to the heating coils at the bottom of the still. The amount of heat which is required to be supplied by these heating coils is radically less than the amount of heat required to be supplied to carry out a similar distilling operation in such stills as have heretofore been provided, being about 30% of the heat heretofore required, or even less. The only heat required to be supplied is the heat to vaporize the fatty acids at about the temperature at which they are vaporized, in other words, the latent heat of vaporization of the fatty acids, and the small amount of heat which is lost by radiation from the lower portion of the still, that is, the portion of the still below the vapor space. This radiation loss is only about one-fourth, or possibly a little less than one-fourth, of the radiation loss ordinarily encountered in stills of similar capacity.

In similar distillation operations as heretofore carried out, it has been necessary to preheat the fatty acids and to generate the steam and superheat the steam by outside sources of heat; the heat content of the vapors has never been made available for these purposes before. The amount of heat which has heretofore been required for preheating and for generating steam and superheating it has been about twice the amount of heat required to supply the latent heat of vaporization of the fatty acids or somewhat more, and as the radiation losses from such stills as have heretofore been provided have been about four times the radiation loss from the still of the present invention, it is apparent that in operating the apparatus of the present invention, the amount of heat which must be supplied to carry out the distillation is about one-third, or less, of the amount of heat which has heretofore been required. This substantial saving in the amount of heat required, coupled with the greater yield and improved quality of the distilled acids are advantages which flow from the present invention.

As an illustration of the heat economy which can be obtained by the present invention, the following comparison of the heat consumption of a still constructed and operated in accordance with the present invention, and a still constructed and operated in accordance with common practice is given. This heat balance is based upon the assumption that both stills are operating on the same material and are distilling it at equal rates, and the further assumptions that the stills are operated at such a rate that 2400 pounds of distillate per hour, from a feed containing 99% free fatty acids, are obtained; that the average still temperature is 428° F.; that the specific heat of the feed and fatty acids is 0.6; that the latent heat of vaporization of the fatty acids is 115 B. t. u. per pound; that the fatty acids are fed to the stills at 200° F.; that the distillate leaves the still at 140° F.; that the water supplied is at 122° F.; and that the superheated jet steam used is 240 pounds per hour, or 10% of the weight of the fatty acids distilled. It is also assumed that the still constructed and operated according to common practice is insulated in accordance with accepted practice over its entire surface, over the goose-neck, and over the separator, and that the still operated and constructed in accordance with the present invention is insulated over the bottom and up the sides to a point slightly over the level of liquid within the still. All of the assumptions made above are well within the range of actual practice, and any variations in these assumptions will affect the results but slightly provided the assumptions are properly applied to both types of stills alike, with the actual advantages of the still of the present invention being taken into account.

Based upon these assumptions, the following table shows the heat required for the operation of a still constructed and operated as in common practice for a period of one hour.

	B. t. u.
Heat for preheating 2424 lbs. (99%) feed	331,600
Heat for generating and superheating 240 lbs. jet steam	278,900
Latent heat of 2400 lbs. fatty acid vapors	276,000
Radiation loss	130,000
Heat required per hour	1,016,500

The following table shows the amount of heat required for operating a still constructed in accordance with the present invention and distilling at the same rate for a period of one hour.

	B. t. u.
Latent heat of 2400 lbs. fatty acid vapors	276,000
Radiation loss	32,500
Heat required per hour	308,500

It will be noted that in the second table, no heat input is required for generating the steam or for superheating it or for preheating the feed, because, as pointed out above, there is more heat available from the vapors than is required for these purposes. It might be pointed out that the amount of steam required to carry out the distillation is somewhat less when the improved apparatus of the present invention is used than has heretofore been required, and that less jet steam can be used with equally efficient distillation, largely because of the fact that the apparatus offers almost no resistance to flow of the vapors and allows the use of a smaller pressure differential between the vapor space and the vacuum device, and a higher vacuum within the still. However, this decreased amount of steam which may be used would not affect the heat balance given above, as there is more than enough heat available to generate and superheat 10% of steam based upon the weight of the fatty acids, or even more. In any event, less jet steam is required when operating in accordance with the present invention and the still may be operated with less jet steam than other types of stills, while giving equivalent performance. The resulting lesser amount of water vapor produces an important economy and improvement in vacuum in the operation of the vacuum device.

While the apparatus has been thus far described with particular reference to the vacuum-steam distillation of fatty acids, it is also applicable to the distillation of other high boiling liquids, such as high boiling petroleum fractions, glycerin, and the like. Where desired, the cooling space provided may be separated into various zones for the fractional condensation of the hot vapors, thus permitting the fractionation of the distillate. Also, the apparatus is advantageous for the dry distillation of high boiling liquids, including the dry distillation of fatty acids, such dry distillation being advantageous for various high boiling liquids which are incompatible with water vapor.

Where the apparatus is used for the dry distillation of such high boiling liquids, and no supply of superheated jet steam is required, the heat given up by the hot vapors in the cooling zone may be used to preheat the feed, and also may be used to supply steam to other apparatus, or may be used for heating other liquids as desired. Nevertheless, the invention is particularly applicable to the vacuum-steam distillation of high boiling liquids. For example, in the distillation of fatty acids from cocoanut oil, the use of jet steam enables the distillation to be carried out at a temperature as much as 25° F. or more, lower than the temperature required if the jet steam is not used, and simple dry distillation is used, and the drop in the temperature when distilling fatty acids derived from tallow is even greater. Furthermore, the steam aids in bringing about intimate contact of the liquid being distilled with the heating coils and insures the intimate and effective contact of the liquid with the heating coils thus greatly increasing the rapidity of the distillation.

In the operation as thus far described, the heat content of the hot vapors which is made available for heating purposes has been utilized for the generation and superheating of the steam used in the distillation, and for preheating the fatty acids. This is a particularly advantageous combination and method of operation, but the invention in its broader aspects is not limited thereto, as, considering the distillation proper, the superheated steam used in the distillation may be obtained from other sources, and the preheating of the feed may be accomplished by other means, the operation of the still itself, with its improved distillation of the fatty acids, being advantageous. Also, the cooling coils provided within the still, instead of being used for preheating and for the production of the superheated steam, may be used as sources of heat for other purposes if desired.

So also, the arrangement of the parts within the still, with the cooling zone annularly disposed around the separating zone, and with an efficient double-walled insulating partition between, is particularly advantageous; but the invention in its broader aspects is not limited thereto, as other arrangements of the separating zone and the cooling zone, or other means of arranging the heat interchangers, the heating zone and the other zones of the apparatus may be employed. The provision of the double-walled insulating partition which defines the separating zone and which prevents or minimizes the flow of heat from the vapor space above the distilling liquid and from the separating zone into the cooling zone or into other parts of the apparatus or into the wall of the still, and which, when the still is operated at a high vacuum, provides thermal insulation approximating in efficiency a Dewar bulb, is a particularly advantageous feature of the present invention. The separating zone, insulated by the insulating partition against the transfer of heat and placed directly above the vapor space, and provided with helical vanes for the removal of entrained material from the outgoing vapors, of large dimensions such that it affords almost no resistance to the flow of the vapors and in which the vapors are not measurably cooled, is another advantageous feature of the invention. These features of the invention are applicable not only to the vacuum-steam distillation of high boiling liquids, particularly fatty acids, but also to their dry distillation under a vacuum, and also to ordinary distillation operations. The conservation of heat which is obtained by efficient insulation provided by the double-walled evacuated insulating partition enables a substantial saving in heat in all such distilling operations.

I do not, in this application, claim the new process of distillation described herein, as it is claimed in my application Serial No. 84,626, filed June 11, 1936, of which this application is a division.

I claim:

1. A still comprising, in one shell, a heating chamber, a separating chamber above and connecting with the vapor space of said heating chamber, an annular vapor-cooling chamber provided with cooling surfaces, and located around said separating chamber and communicating therewith but thermally insulated therefrom, means for supplying heat to said heating chamber and means for cooling said surfaces within said vapor-cooling chamber and means for collecting liquid condensed within said vapor-cooling chamber, said still being substantially vacuum-tight and provided with a vapor outlet connected to a vacuum line, said vapor outlet being so positioned, with respect to the path of vapor flow, that the vapors pass over the cooling surfaces before reaching the vapor outlet.

2. A still comprising, in one shell, a heating chamber including a vapor space, a separating chamber directly communicating with said vapor space, an annular vapor-cooling chamber provided with cooling surfaces and located around said separating chamber and thermally insulated from said vapor space and said separating chamber by a double-walled insulating partition defining said vapor space and said separating chamber, and means for deflecting vapors passing through said separating chamber into and through said annular vapor-cooling chamber, means for collecting liquid condensed within said

vapor-cooling chamber, said still being substantially vacuum-tight and provided with a vapor outlet connected to a vacuum line, said vapor outlet being so positioned, with respect to the path of vapor flow, that the vapors pass over the cooling surfaces before reaching the vapor outlet.

3. A still comprising a heating chamber including a vapor space for heating a liquid to be distilled, a separating chamber above said vapor space and directly communicating therewith, means in said separating chamber for deflecting all the vapors as they pass therethrough and removing entrained material from said vapors by contact, a thermally insulating double-walled partition around and defining said separating chamber and said vapor space, said still being provided with cooling surfaces placed, with respect to vapor flow, subsequent to said separating chamber, said still being substantially vacuum-tight and provided with a vapor outlet connected to a vacuum line, said vapor outlet being so positioned, with respect to the path of vapor flow, that the vapors pass over the cooling surfaces before reaching the vapor outlet.

4. A still comprising, in one shell, a heating chamber including a vapor space, a separating chamber directly above and communicating with said vapor space, an annular cooling chamber around said separating chamber, said vapor space and said separating chamber being thermally insulated from said cooling chamber by a double-walled insulating partition, the space between the walls of which communicates with, and is in pressure equilibrium with, the interior of the still, the opening providing communication between said space and the interior of the still being in the outer wall of the partition and so located as to prevent access of condensing vapors to said space, said partition defining said vapor space and said separating chamber, and means for deflecting vapors passing through said separating chamber into and through said annular cooling chamber, means for collecting liquid condensed within said cooling chamber.

5. In a still, a separator positioned within the still and directly communicating with the vaporizing chamber, said separator being defined by a thermally insulating partition comprising two concentric walls with a space between said walls, with openings in one or both of said walls providing communication between said space and the interior of the still, said openings being so located as to prevent access of condensing vapors to said space, said separator being provided with helical vanes to deflect all the vapors as they pass through said separator while removing entrained material from said vapors.

6. A still adapted for the high vacuum distillation of liquids comprising a heating chamber including a vapor space for vaporizing said liquids, said vapor space being defined and surrounded by thermally insulating means having two concentric walls with a space between said walls, with openings in the outer of said walls providing communication from the space included between the walls to the interior of the still, said openings being so located as to substantially prevent the access of condensible vapors to the space between the walls, whereby said space may be maintained in pressure equilibrium with the interior of the still.

7. A still adapted for the high vacuum distillation of liquids comprising a heating chamber including a vapor space for vaporizing said liquids, and a separating chamber above and directly communicating with said vapor space, said vapor space and said separating chamber being defined and surrounded by a thermally insulating partition having two concentric walls with a space between said walls, with openings in the outer of said walls providing communication from the space included between the walls to the interior of the still, said openings being so located as to substantially prevent the access of condensible vapors to the space between the walls, whereby said space may be maintained in pressure equilibrium with the interior of the still.

8. A still adapted for the high vacuum distillation of liquids comprising a heating chamber including a vapor space for vaporizing said liquids, a separating chamber above and directly communicating with said vapor space, said vapor space and said separating chamber being defined and surrounded by a thermally insulating partition having two concentric walls with a space between said walls, with openings in the outer of said walls providing communication from the space included between the walls to the interior of the still, said openings being so located as to substantially prevent the access of condensible vapors to the space between the walls, whereby said space may be maintained in pressure equilibrium with the interior of the still, and an annular cooling chamber around said separating chamber and communicating therewith, means for collecting liquid condensed in said cooling chamber and means for withdrawing said condensed liquid.

9. A still adapted for the high vacuum distillation of liquids comprising a heating chamberber including a vapor space for vaporizing said liquids, a separating chamber above and directly communicating with said vapor space, said vapor space and said separating chamber being defined and surrounded by a thermally insulating partition having two concentric walls with a space between said walls, with openings in the outer of said walls providing communication from the space included between the walls to the interior of the still, said openings being so located as to substantially prevent the access of condensible vapors to the space between the walls, whereby said space may be maintained in pressure equilibrium with the interior of the still, an annular cooling chamber around said separating chamber, means for deflecting vapors from said separating chamber into said cooling chamber, and means for collecting liquid condensed in said cooling chamber.

10. A still adapted for the high vacuum distillation of liquids comprising a heating chamber including a vapor space for vaporizing said liquids, a separating chamber above and directly communicating with said vapor space, said vapor space and said separating chamber being defined and surrounded by a thermally insulating partition having two concentric walls with a space between said walls, with openings in the outer of said walls providing communication from the space included between the walls to the interior of the still, said openings being so located as to substantially prevent the access of condensible vapors to the space between the walls, whereby said space may be maintained in pressure equilibrium with the interior of the still, an annular cooling chamber around said separating chamber and communicating therewith, means for supplying heat to said heating chamber, and means, including at

least one coil for preheating feed, for cooling said cooling chamber.

11. A still adapted for the high vacuum distillation of liquids comprising a heating chamber including a vapor space for vaporizing said liquids, a separating chamber above and directly communicating with said vapor space, said vapor space and said separating chamber being defined and surrounded by a thermally insulating partition having two concentric walls with a space between said walls, with openings in the outer of said walls providing communication from the space included between the walls to the interior of the still, said openings being so located as to substantially prevent the access of condensible vapors to the space between the walls, whereby said space may be maintained in pressure equilibrium with the interior of the still, an annular cooling chamber around said separating chamber and communicating therewith, means for supplying heat to said heating chamber, and means, including at least one coil for preheating feed, and coils for generating steam, for cooling said cooling chamber while condensing vapors.

12. A still adapted for the high vacuum distillation of liquids comprising a heating chamber including a vapor space for vaporizing said liquids, a separating chamber above and directly communicating with said vapor space, said vapor space and said separating chamber being defined and surrounded by a thermally insulating partition having two concentric walls with a space between said walls, with openings in the outer of said walls providing communication from the space included between the walls to the interior of the still, said openings being so located as to substantially prevent the access of condensible vapors to the space between the walls, whereby said space may be maintained in pressure equilibrium with the interior of the still, an annular cooling chamber around said separating chamber and communicating therewith, means for supplying heat to said heating chamber, and means, including at least one coil for preheating feed, and coils for generating and superheating steam, for cooling said cooling chamber for condensing vapors.

13. A still adapted for the high vacuum distillation of liquids comprising a heating chamber including a vapor space for vaporizing said liquids, a separating chamber above and directly communicating with said vapor space, said vapor space and said separating chamber being defined and surrounded by a thermally insulating partition having two concentric walls with a space between said walls, with openings in the outer of said walls providing communication from the space included between the walls to the interior of the still, said openings being so located as to substantially prevent the access of condensible vapors to the space between the walls, whereby said space may be maintained in pressure equilibrium with the interior of the still, an annular cooling chamber around said separating chamber, means for supplying heat to said heating chamber, and means, including at least one coil for preheating feed, and coils for generating and superheating steam, for cooling said cooling chamber for condensing vapors, and a double-walled bell-deflector over said separating chamber for deflecting vapors into contact with said coils.

14. A still as in claim 12, in which means is provided in the heating chamber for bringing steam into intimate contact with the liquid being distilled, said means being connected to said coils for generating and superheating steam.

15. A still, adapted for vacuum distillation of high-boiling liquids comprising a heating chamber including a vapor space, a separating chamber above and directly communicating with said vapor space, a double-walled insulating partition defining said separating chamber and flaring out below it to define said vapor space, said partition comprising two concentric walls with a space between and having openings in the outer of said walls, said openings being so located as to substantially prevent access of condensible vapors to said space, whereby said space is maintained in pressure equilibrium with the interior of the still, an annular cooling chamber around said separating chamber and communicating therewith but thermally insulated therefrom by said double-walled partition, a thermally insulating bell-deflector for deflecting vapors from said separating chamber into said cooling chamber, cooling means, including means for preheating feed and generating and superheating steam between said double-walled partition and said bell-deflector, at least one other cooling means between said bell-deflector and the wall of the still, at least one trough above the flaring portion of the double-walled partition for collecting condensed liquid, means for withdrawing condensed liquid from said trough or troughs, and means for reducing the pressure in said still whereby the vapors flow through the separating chamber and over the cooling means, preheating the feed and generating and superheating steam while substantially all of the vapors of the high-boiling liquid may be condensed on said cooling means and withdrawn from the still.

16. A still as in claim 15, in which helical vanes are provided in said separating chamber to cause the vapors to follow a spiral path therethrough and to separate entrained material by impingement.

17. A still as in claim 15, in which means are provided in the heating chamber for bringing steam into intimate contact with the hot, high-boiling liquid, said means being connected to the means for generating and superheating steam provided in the cooling chamber, whereby the open steam used in the distillation is generated and superheated by heat absorbed from the hot vapors and hot condensed liquid.

18. A still as in claim 15, provided with cooling means effecting differential cooling and a plurality of troughs for collecting and separating the liquids condensed by said differential cooling means.

19. A still as in claim 15, in which means are provided in the heating chamber for bringing steam into intimate contact with the hot, high-boiling liquid, said means being connected to the means for generating and superheating steam provided in the cooling chamber, whereby the open steam used in the distillation is generated and superheated by heat absorbed from the hot vapors and hot condensed liquid, and provided with cooling means effecting differential cooling and a plurality of troughs for collecting and separating the liquids condensed by said differential cooling means.

MARTIN HILL ITTNER.

CERTIFICATE OF CORRECTION.

Patent No. 2,202,008. May 28, 1940.

MARTIN HILL ITTNER.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 5, first column, line 26, after the word "fatty" insert --acid--; page 8, second column, line 36-37, claim 9, for "chamberber" read --chamber--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 6th day of August, A. D. 1940.

Henry Van Arsdale,
Acting Commissioner of Patents.

(Seal)