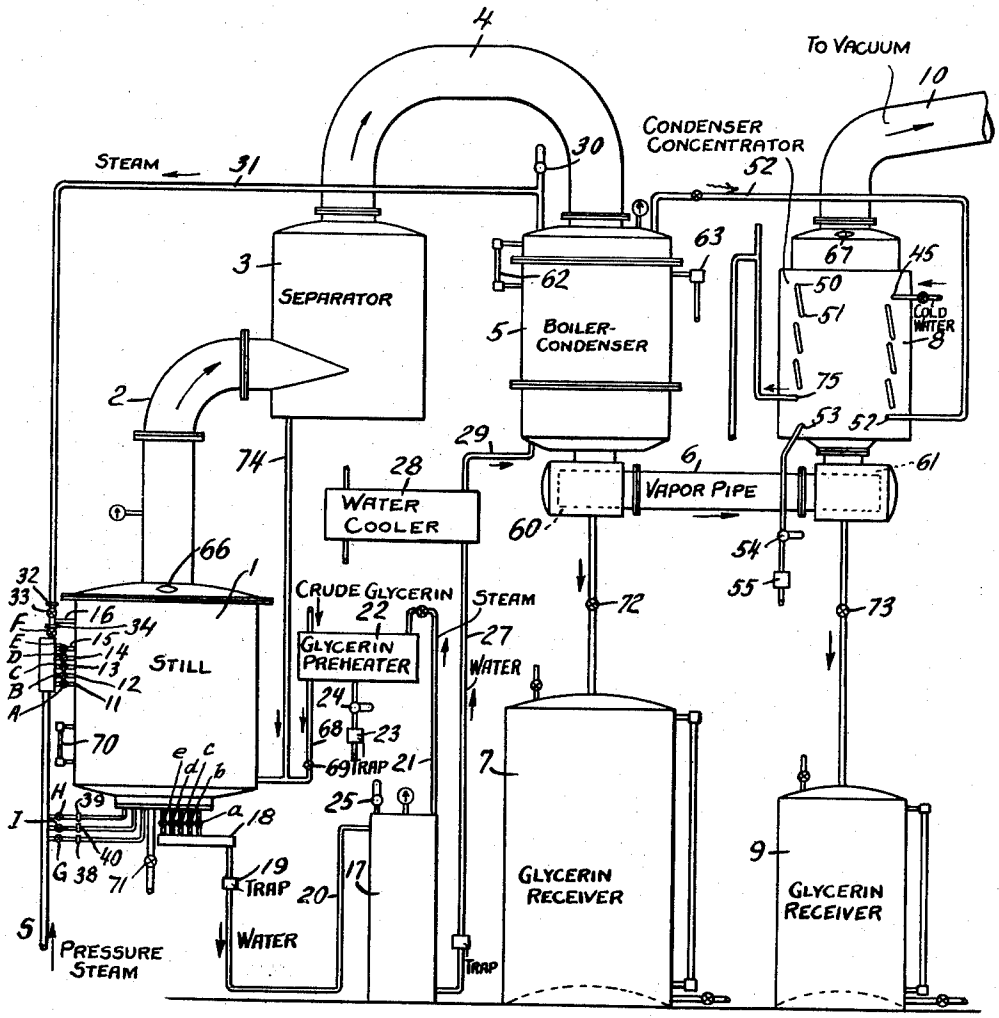
June 27, 1939. M. H. ITTNER 2,164,275

DISTILLATION OF GLYCERIN

Original Filed Dec. 23, 1935 6 Sheets-Sheet 1

Fig. 1.



Martin Hill Ittner Inventor

By Pennie Davis Marvin & Edmonds

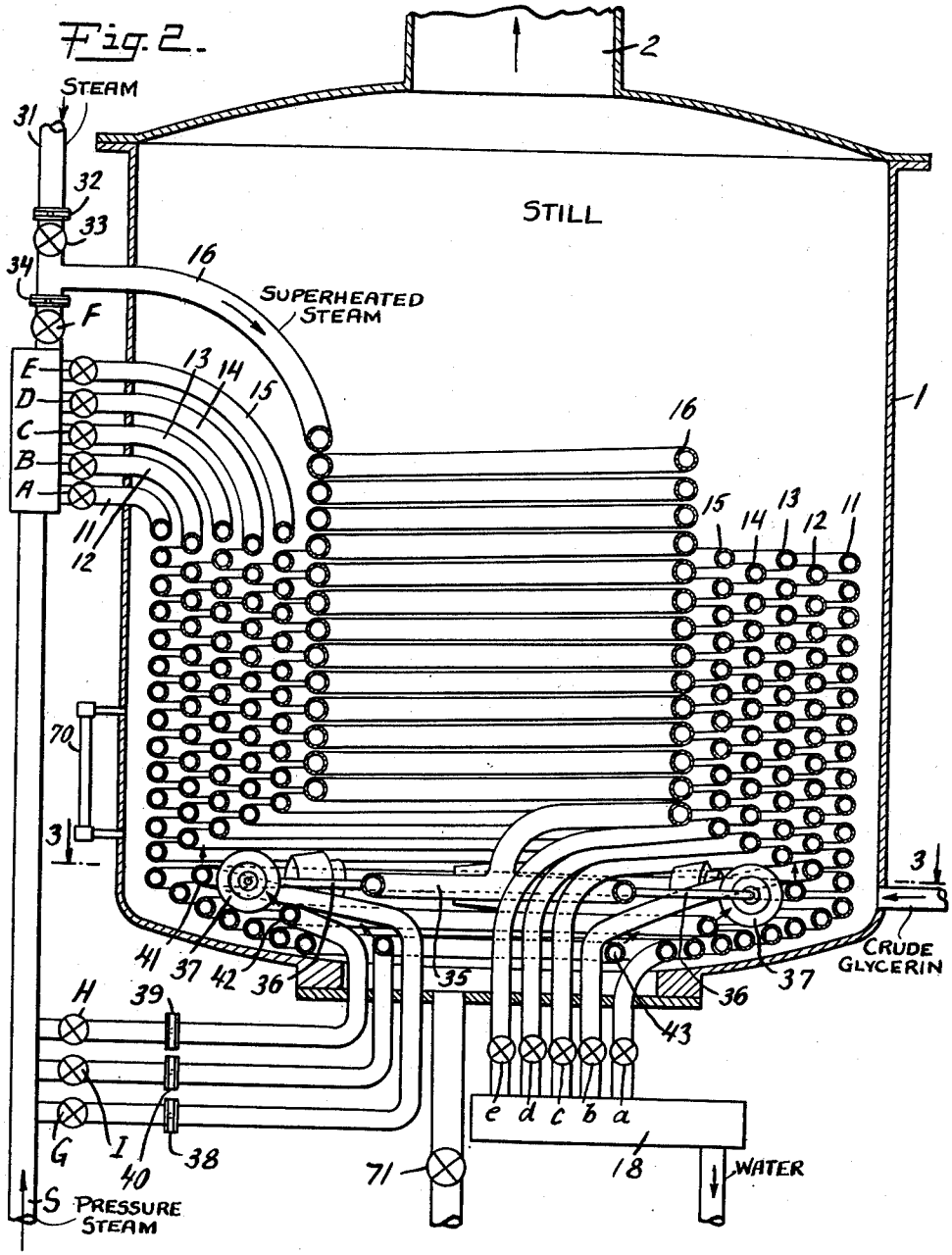
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DISTILLATION OF GLYCERIN

Original Filed Dec. 23, 1935 6 Sheets-Sheet 2

Fig. 2.



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DISTILLATION OF GLYCERIN

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Fig. 3.

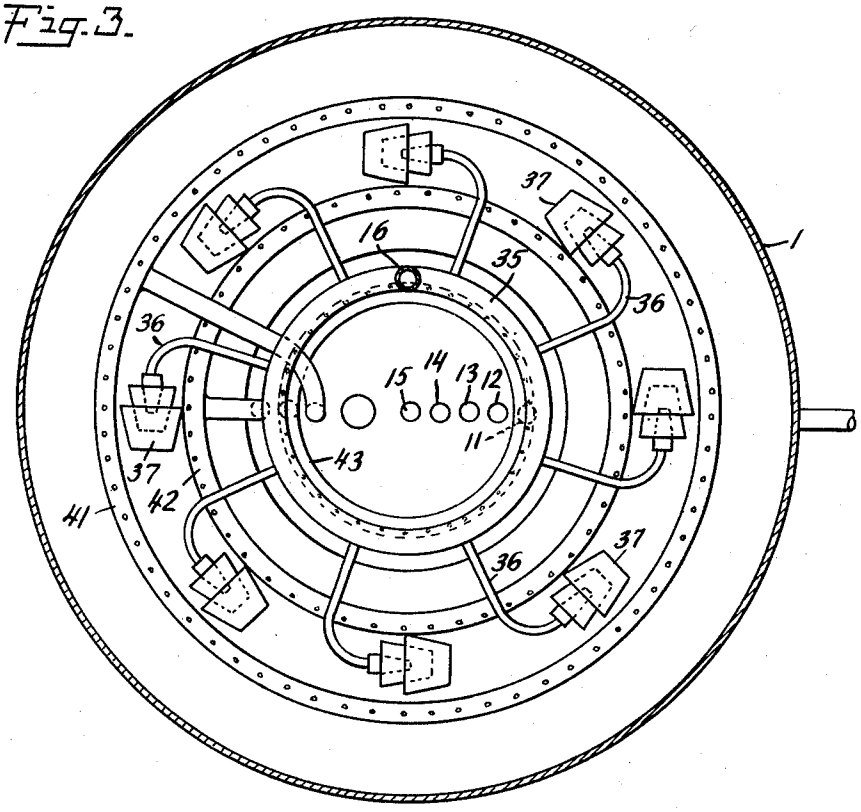
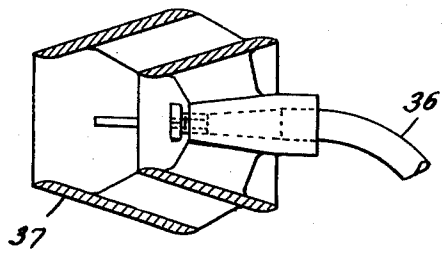


Fig. 4.



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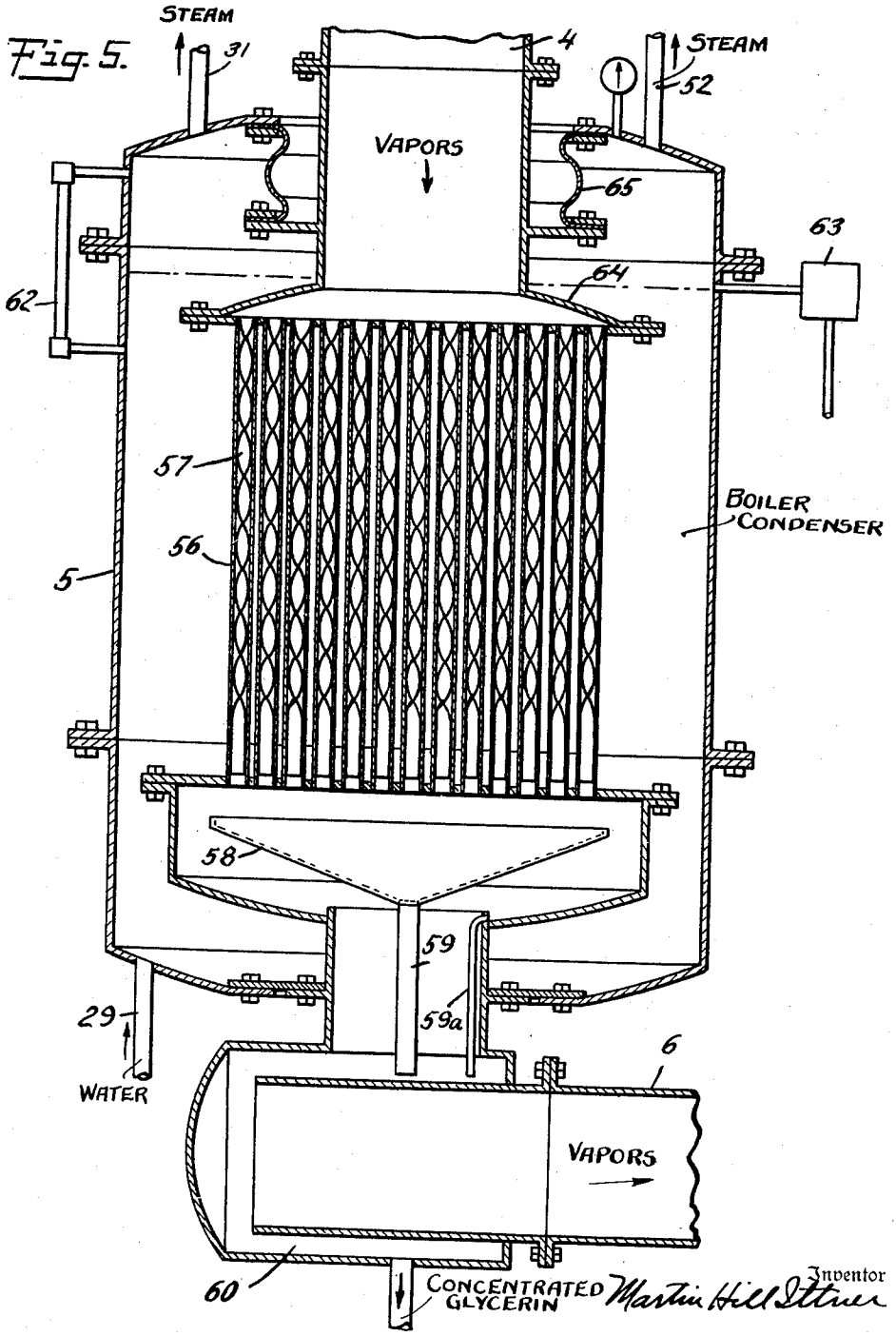
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DISTILLATION OF GLYCERIN

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Fig. 5.



Inventor
Martin Hill Ittner

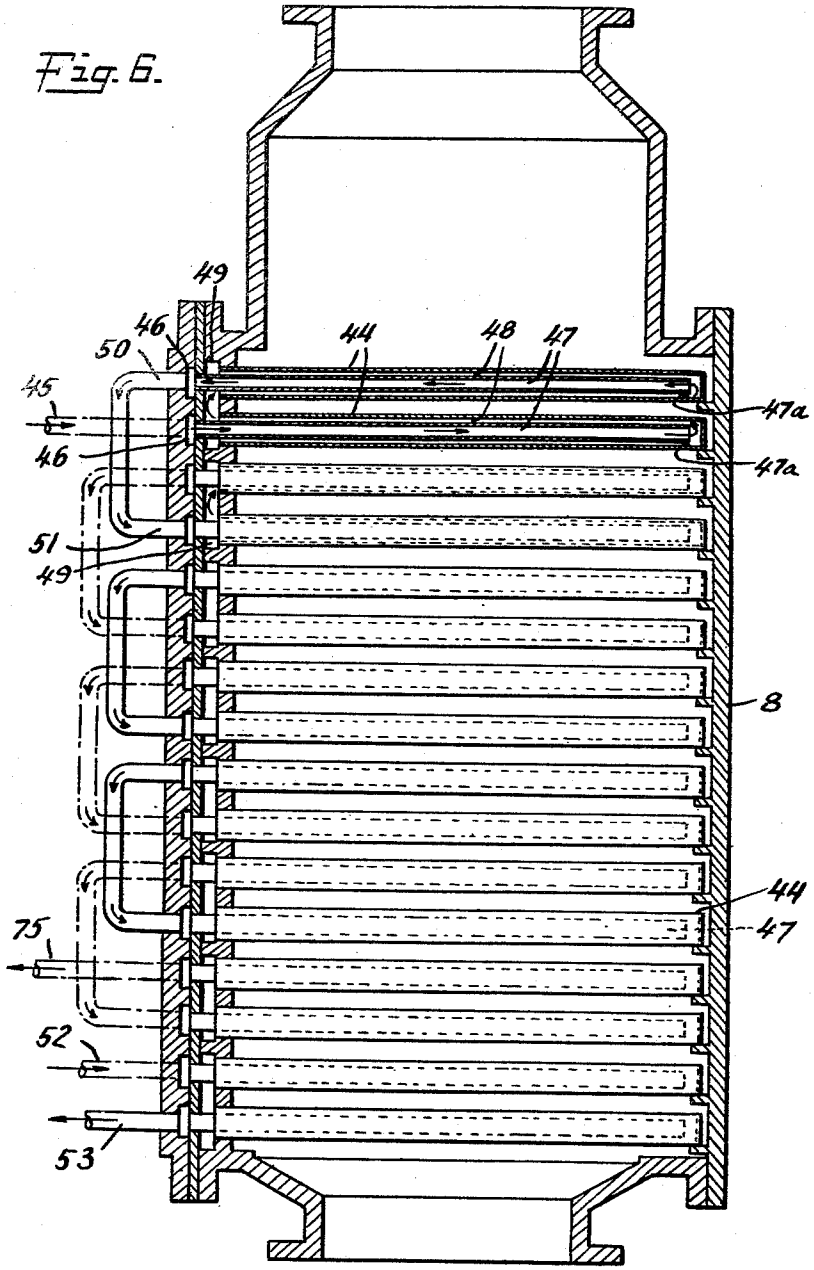
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DISTILLATION OF GLYCERIN

Original Filed Dec. 23, 1935 6 Sheets-Sheet 5

Fig. 6.



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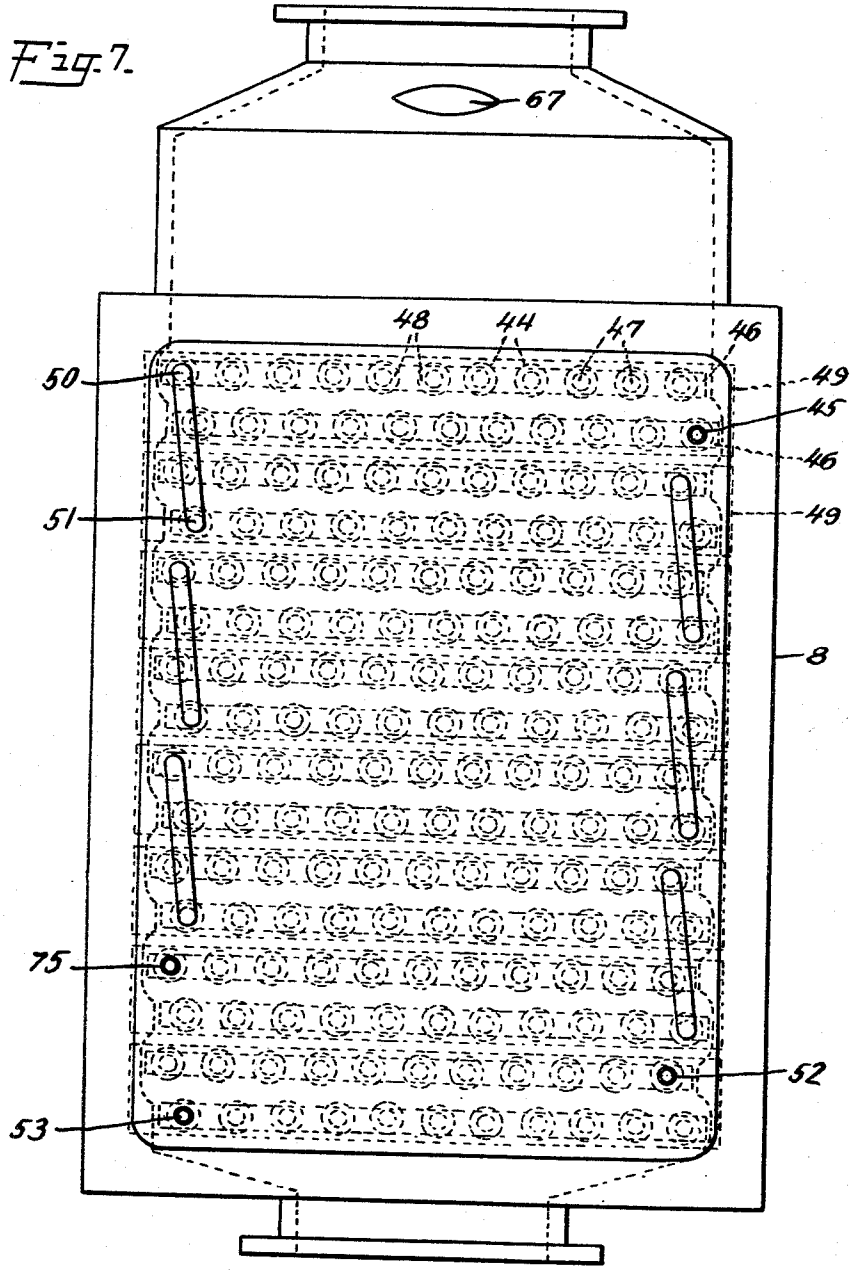
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DISTILLATION OF GLYCERIN

Original Filed Dec. 23, 1935 6 Sheets-Sheet 6

Fig. 7.



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2,164,275

UNITED STATES PATENT OFFICE

2,164,275

DISTILLATION OF GLYCERIN

Martin Hill Ittner, Jersey City, N. J., assignor to Colgate-Palmolive-Peet Company, Jersey City, N. J., a corporation of Delaware

Original application December 23, 1935, Serial No. 55,783. Divided and this application June 11, 1936, Serial No. 84,740

6 Claims. (Cl. 202—177)

This invention relates to improved apparatus for the distillation of glycerin.

This application is a division of my application Serial No. 55,783, filed December 23, 1935.

The apparatus of the present invention, when properly operated, gives greatly increased distilling capacity over that heretofore obtainable from known equipment of equal size, so far as I am aware, and yields the entire distillate in the form of high grade refined glycerin ready for sale and use without further evaporation or concentration or redistillation. By means of the present invention, it is possible to subject crude glycerin made from soap lyes to a single distillation and nevertheless convert the greater part of the distillate directly into the highest grade of C. P. glycerin, with less difficulty than is ordinarily experienced in making C. P. glycerin out of twice distilled glycerin. This apparatus includes the construction and combination of a number of pieces of equipment, the uses and operations of which are interdependent, and which, taken together, make a smooth-running economical whole which gives a better and more economical result than has ever heretofore been obtained, so far as I am aware. In addition, certain parts of the equipment, and certain features and sub-combinations of the apparatus are themselves new and useful, and form valuable features and sub-combinations of the invention, without considering the whole equipment; although the entire equipment, considered as an operating whole, is particularly advantageous.

The apparatus of the invention as a whole includes a still provided with a plurality of closed steam-heating coils located in the lower outer part of the still for heating the contents of the still with high pressure steam; steam operated ejector jets for mixing the contents of the still with steam, the jets being so located as to discharge a mixture of said still contents with steam against said heating coils beneath the normal operating level of said still contents to bring about uniform and effective heating of said still contents and the production of glycerin vapors; a superheating coil located within the still for superheating the steam before it is discharged into the still contents through said jets; vapor pipes for conducting the vapors from said still through an entrainment separator to a primary condenser for the glycerin; a primary condenser in the form of a combined boiler, condenser and heat interchanger for the simultaneous condensation of the glycerin and the generation of steam in the boiler by the boiling of distilled water; means for collecting the condensed water from the closed steam-heating coils and of utilizing the same and for supplying the same or a portion thereof to the combined boiler and condenser to be there converted into steam; means for supplying steam from said boiler-condenser to the superheater coil located in the still and thence to the jets; means for collecting the glycerin condensed in said boiler-condenser; a condenser-concentrator for condensing the balance of the glycerin from the vapors of said still as glycerins and aqueous glycerin solutions of various concentrations, with means for concentrating the more dilute glycerins and aqueous glycerin solutions condensed within said condenser-concentrator to produce concentrated glycerin with means for drawing off and collecting said concentrated glycerin; and means for conveying excess water vapors from said condenser-concentrator to a suitable vacuum apparatus so as to maintain a suitable partial vacuum within the still and condenser. The invention also includes further specific features of construction and arrangement, as well as individual features and subcombinations of features, of the apparatus thus referred to.

The improved apparatus of the present invention makes it possible to distill glycerin with greater economy of steam consumption than has heretofore been realizable while at the same time giving a distillate of improved quality without the need of redistillation, and with substantially complete recovery of the glycerin as concentrated refined glycerin, and with direct recovery of the greater part of the glycerin directly as the highest grade of C. P. glycerin.

I have made a study of the concentrations of mixtures of pure glycerin and water that will exist in the presence of an excess of water vapor under various conditions of heat and pressure unfavorable to the liquefaction of pure water vapor, and the concentrations of glycerin and glycerin solutions that will condense from a mixture of glycerin and water vapors under such conditions, and have found that in each case the concentration of the liquid glycerin or aqueous glycerin solution that will exist in a state of equilibrium in the presence of excess water vapor is very definite and is dependent solely on the pressure of the vapors present and temperature of the glycerin or glycerin solution regardless of the excess of water vapor that may be present.

I have also found that in a properly constructed still the mixture of glycerin and water vapors leaves the still in a condition of delicate equilib-

rium with respect to temperature and pressure, which equilibrium will be maintained without sensible precipitation of glycerin through the goose-neck of the still and an entrainment separator when these are properly covered with good heat insulating material, and that thereafter any drop in the temperature of the mixed vapors will cause a precipitation of glycerin, the concentration of which will depend on the pressure of the surrounding vapors and the temperature of the precipitated glycerin regardless of the amount of excess water vapor present.

I have thus found it practicable to condense most of the glycerin passing from my still at a carefully controlled comparatively high temperature which is sufficient at the pressure of operation to give pure glycerin direct of high concentration, 99% to over 99.5%, and under conditions so that substantially all of the volatile impurities present in the vapors remain in a volatile condition and pass off with the water vapor without contaminating the glycerin thus condensed.

I have also devised a simple means for condensing the glycerin distillate so that substantially 80%, or more, of the heat of the live steam furnished to run the still, exclusive of that required to overcome heat lost by radiation, may be recovered and utilized in the process.

I have also devised a system of natural thermal circulation of the still contents for intimately mixing the open jet steam used, in an extremely fine state of division, with the still contents in the zone of maximum heat supply, so that a balanced condition of still contents, glycerin vapor and water vapor is easily maintained within the still with a low proportion of water vapor. So intimate is the mixing of the still contents and the open jet steam that accurate measurements of the temperature of the still contents, superheated steam for the open jet, and the mixed glycerin and water vapors in the goose-neck issuing from the still at any given time have been found to be within about one-half degree of one another, and the still contents and issuing vapors are never superheated.

The invention will be further described in connection with the accompanying drawings, which show, in a somewhat conventional and diagrammatic manner, apparatus illustrating and embodying the invention, but it will be understood that the invention is not limited thereto.

In the accompanying drawings:

Fig. 1 is an elevation, showing one arrangement of the various parts of the apparatus;

Fig. 2 is a vertical section, with parts in elevation, of the still proper;

Fig. 3 is a horizontal section taken on the line 3—3 of Fig. 2, and with certain parts omitted for simplicity of illustration;

Fig. 4 is an enlarged section of one form of steam jet;

Fig. 5 is a vertical section with parts in elevation of the primary condenser or combined boiler-condenser;

Fig. 6 is a vertical section of the condenser-concentrator, with parts in elevation; and

Fig. 7 is an elevation of the condenser-concentrator, taken at right angles to that of Fig. 6.

In Fig. 1, the still 1, in which the glycerin is distilled, is shown as provided with a goose-neck 2 for the transfer of mixed glycerin and water vapors to the separator 3 to remove entrainment. Leading from the separator is another vapor pipe 4 leading to the primary condenser or boiler-condenser 5 which serves the dual purpose of condensing the greater part of the glycerin and of recovering the heat therefrom for generating steam by the evaporation of distilled water, which steam is utilized directly in the still to supply pure open jet steam, and the balance of which is utilized elsewhere in the system as will be subsequently explained. Connected with the lower part of the boiler-condenser 5 is a vapor pipe 6 for conveying water vapors mixed with glycerin vapors not condensed in the boiler-condenser 5, together with substantially all the volatile impurities from the still, to the combined condenser-concentrator 8. Located below the boiler-condenser 5 is a tank 7 for the reception of the pure glycerin condensed in this condenser; while a tank 9 is likewise arranged below the condenser-concentrator 8 to receive the glycerin concentrated from the condensates produced in the combined condenser and concentrator. The outlet pipe 10 is provided for conveying the water vapors and substantially all of the volatile impurities to the vacuum equipment (not shown) for removing the water and impurities and for maintaining the necessary vacuum. The outlet pipe 10 is shown as having an upward inclination of about 10° from the horizontal, so that any condensate condensed therein will be returned to the condenser-concentrator. The entrainment separator 3 may be of any suitable construction, for example, such as the Webre separator, but it should not offer any obstruction to the free flow of vapors therethrough.

As illustrated in Fig. 2, the still 1 is heated by closed steam heating coils 11, 12, 13, 14 and 15, controlled by inlet valves A, B, C, D and E and outlet valves *a*, *b*, *c*, *d*, and *e*. The inlets to these closed coils are connected to the pipe S through which steam is supplied at a suitable pressure, e. g., of around 150 gauge pressure from a source of high pressure steam (not shown). Saturated steam, supplied under pressure, serves to heat the contents of the still. The water condensed from the steam under pressure is withdrawn through the outlet valves *a*, *b*, *c*, *d* and *e* to the header 18 and thence through a trap 19 which permits only water, and no steam, to pass therethrough, so that escape of high pressure steam from the heating coils is prevented. The outlet of the trap 19 is connected by pipe 20 to the receiver 17, which has a suitable pressure relief valve 25 therein which determines the pressure maintained in the receiver, e. g., a pressure of about 25 pounds. The hot water entering the trap under the high pressure, e. g., of around 150 pounds, is discharged from the trap to a zone of lower pressure, i. e., that in the receiver 17.

The condensed steam is still at a high temperature when the pressure is released upon it at the trap 19 to the lower pressure controlled by the valve 25, and the reduction in pressure causes vaporization of a part of the hot water at the lower pressure prevailing in the receiver 17, part of the heat of the hot water being utilized to vaporize a portion of the water and generate steam therefrom at a lower pressure, the remainder of the water being still hot and at a temperature corresponding to the temperature of saturated steam at the lower pressure maintained in the receiver 17. For example, if the high pressure steam used in the heating coils is supplied at a pressure of around 150 pounds gauge and at a temperature around 366° F. or 185.5° C. the condensed water leaving the coils and passing to the receiver 17 will be at a temperature not greatly below that mentioned at

the time the pressure is released. The temperature of saturated steam at a pressure of e. g., 25 pounds gauge maintained in the receiver 17 is around 267° F. or 130.5° C. The heat contained in the hot water at the higher temperature and pressure is utilized in volatilizing part of the water and generating steam therefrom on reduction of pressure; and both the resulting steam and the remaining unvaporized hot water, at the somewhat lower temperature and pressure, are advantageously utilized in the process.

Leading from the top of the receiver 17 is a steam pipe 21 which conveys the steam to a crude glycerin heater 22 for preheating the crude glycerin before entering the still. The outlet from this heating chamber 22 has a trap 23 therein to permit the removal of condensed water but no steam and may have a relief valve 24 to blow off at some desired pressure as for instance about 25 pounds gauge pressure, and preferably at a slightly higher pressure than relief valve 25 on receiver 17. The outlet line 27 from the bottom of the receiver 17 has a trap 26 therein to prevent passage of steam while permitting passage of water and this outlet pipe 27 may lead to the water cooler 28 for cooling the distilled water from the receiver 17 to a lower temperature, when desired, for example, to around 190° F. before it is supplied through the pipe 29 to the boiler 5.

The boiler 5 is provided with a relief valve 30 through which excess steam may be discharged automatically to prevent accumulation of excessive pressure in the boiler and is also provided with a steam pipe 31 for conducting steam generated in the boiler to the top of the superheater coil 16, the steam pipe having a regulating orifice 32 and valve 33 therein. The top of the superheater coil 16 is also connected with the high pressure steam line S through the orifice 34 and valve F so that the high pressure steam can be used, with suitable reduction in pressure, in the superheater coil at such times as steam from the boiler 5 is not available or as a separate and independent source of steam to the superheater coil.

The superheater coil 16 as shown in Fig. 2 is arranged inside the closed heating coils 11 to 15 and extends somewhat higher in the still than these heating coils. It is connected at its lower end with a ring manifold 35 which in turn is connected through pipes 36 with ejector jets 37, one form of which is shown in Fig. 4. These ejector jets 37 are shown as arranged tangentially near the outer lower part of the still so that the discharge through them will give a lively swirl and stirring action to the contents of the still, thereby effecting continuous and rapid circulation of the contents of the still over the heating coils. The arrangement is such that the steam discharged through these jets, and the intimately admixed liquid, will rise through the coils 11, 12, 13, 14 and 15 and will carry the still contents upwardly over and among these heating coils, causing effective agitation and circulation of the contents of the still upwardly between and around these heating coils and downwardly through the central portion of the still inside the superheater coil 16.

As shown in Figs. 2 and 3, perforated steam rings 41, 42 and 43 are located near the bottom of the still and are connected with the high pressure steam line S through separate connecting pipes having valves G, H and I and orifices 38, 39 and 40. Rings 41 and 42 are shown so placed and with the orifices so arranged that the steam will be discharged therefrom upwardly through the heating coils 11 to 15 while the ring 43 is shown placed near the center and bottom of the still for use in washing out the still foots or residue after distillation.

The still is also shown as provided with peep glasses 66 on the cover of the still, with gauge glass 70 on the side of the still to indicate the level of the contents and a run-off outlet with valve 71 therein for emptying the still.

The boiler-condenser 5 of Fig. 1 is illustrated in greater detail in Fig. 5. This boiler-condenser is constructed as an indirect heat interchanger since it is essential that the cooling water and the vapors cooled shall be kept separate from each other. This boiler-condenser serves as a condenser for glycerin vapors and as a boiler for the generation of steam, the water in the boiler serving as cooling water for cooling and condensing the glycerin vapors, and the heat of the glycerin vapors serving for the evaporation of the distilled water in the boiler under a suitable pressure, preferably superatmospheric pressure.

The boiler is supplied with distilled water from the receiver 17, at a regulated temperature, through the inlet pipe 29, and the boiler has a relief valve 30 and steam pipe 31 leading to the superheater coil 16, as already described. The inlet for the glycerin vapors entering through the pipe 4 is shown as connected to the top of the boiler-condenser and a plurality of tubes 56 are provided through which the glycerin and water vapors from the still pass downwardly, these tubes being surrounded and cooled by the distilled water in the boiler which is converted into steam by the heat absorbed from the glycerin and water vapors in condensing the glycerin therefrom. The tubes 56 are shown as provided with spiral or helical inserts 57. These spirals may be made from thin metal twisted slightly so as to assume a helical or screw shape and it is desirable that these spirals shall have one or more turns during their length so that the vapors cannot pass through the tubes without being deflected continually toward the sides of the tubes. While the boiler may be constructed so that the vapors of glycerin and water vapor pass upwardly through the tubes 56, there are distinct advantages in passing them downwardly through these tubes. With the helical insert shown, the line of greatest slope from any part of the tube in contact with the edge of the spiral is rapidly toward the center of the spiral and down the center of the spiral so that the condensed glycerin is kept near the center of the spiral and away from the tubes, rendering the tube condensing surfaces more effective, while the helical shape of the spiral causes the downwardly directed vapors to be continuously deflected toward the sides of the tubes. The arrangement is thus such as promotes effective heat transfer to the surrounding water from the vapors passing over the interior surfaces of the tubes, while the condensed glycerin is largely removed from these surfaces by the spiral and flows downwardly on the spiral near the center of the tubes. The spiral inserts are shown as having their lower portions straight at their lower ends for a few inches so that the downwardly flowing vapors will leave the tubes in a straight direction rather than with a strong swirl that would tend to break up the glycerin streams into tiny droplets and thus increase entrainment. These spiral inserts are also shown

as stopping a short distance above the lower ends of the tubes.

Arranged below the tubes **56** is a pan **58** for catching and collecting the condensed glycerin. This condensed glycerin is in the form of a multitude of streams flowing from the lower ends of the tubes and is thus collected and separated from the onrushing mixed glycerin and water vapors which pass laterally and around the pan **58**. The condensed glycerin that falls into the pan **58** is conveyed down the pipe **59** and is delivered on to the top of the end of the cross pipe **6** around which it flows and then passes downwardly into receiving tank **7** for removal and use, and the connecting pipe having a valve **72** therein. The balance of the glycerin which is condensed in the boiler-condenser **5** and does not fall into the pan **58** is conducted downwardly through the opening **59***a* so that it also flows over the pipe **6** along with that flowing down through the pipe **59**.

The mixed glycerin and water vapors leaving the boiler-condenser **5** pass downwardly to the space **60**, surrounding the end of the pipe **6**, and then through the cross pipe **6** and the space **61** surrounding the other end of this pipe and thence upwardly into the condenser-concentrator **8**.

The boiler-condenser is shown as provided with a gauge glass **62** and an overflow trap **63** to maintain the level of the distilled water in the boiler so that it will always cover the inner dome **64**. The trap **63** as shown is arranged to remove boiling water at the water-steam level; or it may be located at the desired water level on a pipe (not shown) leading from the hot water inlet **29** to the top of the boiler, in which case it will remove some of the water entering the boiler where this entering water is in excess of that required in the boiler. I also find it advantageous to provide an expansion joint **65** between the upper portion of the inner condensing section of the boiler and the outer shell of the boiler to prevent the formation of undue strains in the tubes **56** or ground sheets due to uneven expansion between the boiler shell and the condensing parts.

The boiler **5** is shown as having a steam outlet pipe **52** leading to the bottom rows of tubes in the condenser-concentrator **8** for heating the same, as hereafter described, and the outlet **53** from these tubes has a pressure relief valve **54** and a trap **55**, as shown in Fig. 1, the pressure relief valve **54** controlling the pressure in the boiler **5** and being set at the desired pressure, e. g., 20 pounds absolute, while the trap **55** permits water condensed from the steam to escape without permitting escape of steam.

The boiler-condenser **5** may be so designed that the condensing surface will be sufficient to condense substantially all of the glycerin; but if this is done the glycerin thus obtained will contain an appreciably higher percentage of the objectionable volatile impurities, and, in order to make the condensation substantially complete, it would be desirable if not necessary to reduce the temperature of the water in the boiler either by operating at a reduced pressure or by constructing the boiler so that it would have a lower cooling section. In either event the concentration of the glycerin that would be condensed would be appreciably lower and less desirable than the concentrations which I have obtained, and the quality of the glycerin would be lower owing to a greater absorption of objectionable volatile impurities at the lower temperature and lower concentrations.

In order to obtain glycerin of high purity and sufficiently free from objectionable volatile impurities I find it advantageous to design the boiler-condenser **5** so that the condensing surface will not be sufficient to condense all the glycerin, but so that a large part of the glycerin will nevertheless be condensed of high purity; and I find it advantageous to operate the primary condenser at a temperature such that the water in the boiler will be vaporized and will generate steam at a suitable pressure for use in the ejector jets of the still, as previously explained.

I have operated my apparatus in an advantageous manner so as to condense glycerin in the primary condenser **5** in amounts ranging from less than 75% to more than 90% of the distillate; but I have found it advantageous, all things considered, to operate so that about 80% of the glycerin is condensed in the primary condenser **5** at about 99 to over 99.5% concentration.

The combined condenser and concentrator **8**, shown in Fig. 1 and illustrated in more detail in Figs. 6 and 7, is provided with a plurality of banks of condensing and concentrating pipes **44**. These are cooled near the top of the condenser with cold water entering at **45**. The condenser-concentrator **8** is so designed that the cold water entering at the top will flow gradually downward, instead of upward as is customary in surface condensers. In order that the condensing and concentrating pipes **44** may at all times be filled with water in that portion of the apparatus through which condensing water is allowed to flow, and not accumulate air or steam in pockets, I have devised an arrangement of the pipes so that the flow of water will be locally upward through the tubes **44**, though generally downward. This is accomplished by permitting the cold condensing water to enter at the inlet **45** which is at the second bank of tubes from the top, and causing the water to flow first through the tubes of this bank and then through the tubes of the top row and out at the outlet **50** from the top row to an outlet which connects with an inlet **51** to the fourth row from the top, so that the water flows through the fourth row and then through the third row from the top, then downwardly to the sixth bank or row of tubes and upward to the fifth bank of tubes, and so on.

By reference to Figs. 6 and 7, it will be seen that there are smaller tubes **47** located within the larger tubes **44**, and that the small inner tubes **47** connect with slotted spaces **46** at their ends, while the outer larger tubes **44** have slotted spaces **49** at one end connecting the ends of two adjacent rows of tubes. The smaller tubes, at their inner ends, have a small spur **47***a* on their under sides for support and open into the larger tubes, as shown. The arrangement is such that the water which enters the second slotted space **46** leading to the inner portions of the inner tubes **47** of the second bank of tubes, passes through these tubes and into the space **48** between the smaller tubes **47** and the larger tubes **44** of the second row of tubes, then out of the ends of the larger tubes into the space **49** and then to the ends of the larger tubes of the top row, then through these large tubes and back through the smaller tubes to the slotted space **46** of the top row of tubes and then out at **50**. Since the outlet is from the upper row of tubes, any air or vapors are forced outwardly with the water and down into the inlet of the fourth row of tubes. The arrangement is such that the water from the upper row of tubes passes down into the slotted

space at the ends of the small tubes of this fourth row, in the manner described, and then upwardly to the third row and through the third row of tubes, and then outwardly and down to the inlet of the sixth row of tubes, and then through the sixth row and the fifth row, and so on. In the alternate rows of tubes, e. g., the second, fourth, and sixth rows, etc., the flow is inwardly from the slotted spaces 46 to the inner small tubes 47, then back through the space 48 between the inner tubes and the outer tubes, then upwardly through the slotted space 49 to the inlet of the larger tubes of the next row, then through the space between the larger tubes and the inner tubes, and back through the inner tubes to the slotted space 46 of this next higher row, e. g., the first, third, and fifth rows, etc.

The water may thus be led through all of the banks of tubes and be withdrawn from the outlet at the next to the bottom row of tubes; or the condensing water can be withdrawn before permitting it to traverse all of the banks of tubes. This latter arrangement is an advantageous one since the lower rows of tubes can then be utilized for heating the lower part of the condenser-concentrator. In the apparatus illustrated in Figs. 1, 6 and 7, the water outlet is from the fourth row of tubes from the bottom, through the outlet pipe 75.

The lower two rows of tubes in the condenser-concentrator 8 are shown as connected with a steam supply for heating these tubes. The steam line 52 from the boiler 5 enters the next to the bottom row of tubes so that the steam can pass through this row of tubes and then through the lower row of tubes and then to the outlet 53, above referred to. Such an arrangement provides for heating one or more pairs of rows of tubes at the bottom of the condenser-concentrator with steam to increase the concentrating effect instead of depending on hot water gradually heated by the heat absorbed in its downward flow through the tubes of this apparatus. In the arrangement illustrated in Fig. 1, steam is supplied to the two bottom rows of tubes by utilizing part of the excess steam generated in the boiler 5, in excess of that required for supplying the open jet steam required in the still 1 for distilling glycerin.

By reference to Fig. 7 it will be seen that the tubes of the different rows or banks in the condenser-concentrator 8 are not placed directly under the tubes of the next higher row or bank, nor are they placed directly under the spaces between the tubes of the next higher row or bank. Instead, the tubes of each bank are shifted slightly side-wise from the tubes of the bank immediately above and below, with the result that vapors may not travel directly through the banks of tubes without being diverted in their course, and with the further result that water and dilute glycerin solutions condensing on the upper colder tubes may not drip off through the open spaces of the tubes beneath but will fall so as to strike the tubes of the next lower bank. This arrangement is an advantageous one, since, in this way, the more dilute glycerin solutions condensing on the upper, colder tubes fall in their downward course to successively hotter and hotter tubes, and there lose a portion of their water and become progressively more and more concentrated until they reach the bottom tubes which are at the highest temperature, and where it is desirable to heat the tubes with steam from an outside source or hot water that may be permitted to reach the boiling point through heat accumulated by the condensation of aqueous glycerin solutions in this part of the apparatus and by absorbing heat in cooling the hot vapors entering at the bottom of the condenser-concentrator 8.

The tube surface maintained in the condenser-concentrator 8 is sufficient to condense all of the glycerin as aqueous glycerin solutions of various concentrations, depending on the temperatures of the water in the various banks of tubes; but the total tube surface is preferably not sufficient to condense more water as water or dilute glycerin than can be re-evaporated by the excess heat of the vapors entering at the bottom and absorbed by the tubes and accumulated in the lower banks of tubes, or by excess heat in the form of steam supplied to the lower banks of tubes. The arrangement is advantageously such that the glycerin is condensed with substantial completeness before the cooled vapors leave the upper rows of tubes, so that practically glycerin-free water vapor, etc., leaves the top of the apparatus; while the heat interchange and heating of the condensed glycerin is advantageously such that the glycerin which finally flows from the bottom rows of tubes, after concentration thereon, is concentrated glycerin.

In the drawings, no heat insulation is shown on the apparatus, but it will be understood that, in actual construction and operation, good heat insulated covering will be provided on the still 1, the goose-neck 2, separator 3, vapor pipes 4 and 6, boiler 5 and tanks 7 and 9, as well as on all outside steam pipes and the receiver 17 and the connecting pipes between the boiler 5 and receiving tank 7 and also the connections between the condenser-concentrator 8 and the tank 9. It is also desirable to cover the crude glycerin heater and the pipe 68 for conveying preheated glycerin to the still, with heat insulation. This supply of preheated crude glycerin is regulated in flow by the valve 69.

A valve 72 is provided in the pipe that conducts glycerin from the condenser-boiler 5 to the tank 7 and a valve 73 is provided in the pipe that conducts glycerin from the condenser-concentrator 8 to the tank 9. Tanks 7 and 9 are also provided with gauge glasses for indicating the height of glycerin therein, and also suitable run-off pipes and valves and vacuum breaks, and connections to auxiliary vacuum apparatus (not shown), which may be employed in emptying the tanks 7 and 9 without interrupting the progress of the distillation.

It will be understood that vacuum gauges and pressure gauges will be supplied where necessary on the apparatus. Thus the goose-neck 2 may be provided with a suitable vacuum gauge, conventionally shown, and the boiler 5 with a pressure gauge, similarly shown. It will also be understood that thermometers will be provided, where necessary, to show the temperature of the liquid or vapors in different parts of the apparatus, for example, the temperature of the hot distilled water entering the boiler 5, the temperature of the hot water flowing from the lower part of the condenser-concentrator 8, etc.

The entrainment separator 3, which may, for example, be a Webre separator, is shown as provided with a drain line 74 leading back to the still 1. This line should also be carefully covered with insulating material.

Suitable peep glasses may be provided on different parts of the apparatus to permit observation of the progress of the distillation and con-

densation such as the peep glass 66 on the still cover and the peep glass 67 on the dome of the condenser-concentrator.

When the apparatus is in operation, and distillation is progressing, the water in the boiler 5 is kept boiling, and it will remain hot during short shut-downs. In starting distillation, after a long shut-down, as for instance over the week-end, the water in the boiler 5 may be brought quickly to the boiling point by running in live steam directly into the boiler from a pipe and valve (not shown).

The combined area of the high pressure heating coils 11 to 15 in the still 1 is purposely greatly in excess of what is needed for continuous distillation, in order that the still contents may be brought rapidly to the boiling point at the start of the distillation and thus minimize the time of shut-downs.

In the distillation of glycerin in the apparatus, I maintain a suitable vacuum in the apparatus which may be, for example, at an absolute pressure of 15 millimeters of mercury. It is not necessary to maintain this particular vacuum, as the distillation can be carried out satisfactorily with either a better or poorer vacuum and over a fairly wide variation of absolute pressure.

In starting the operation of the apparatus, all outside valves are first shut and a partial vacuum is induced through the vapor line 10 by suitable vacuum equipment (not shown). As soon as the vacuum has improved to approximately a so-called 25 inch vacuum, or an absolute pressure of about 5 inches of mercury, I begin to run crude glycerin rapidly into the still. When glycerin begins to show in the gauge glass 70 located near the bottom of the side of the still with its lower end slightly above the center of the ejector jets 37 within the still, I open at once valves A, B, C, D and E, and valves *a*, *b*, *c*, *d* and *e*, and also valve F leading from the high pressure steam line through a suitable orifice 34 to superheater coil 16, ring manifold 35, pipes 36, and ejector jets 37. During this time valve 33 is closed. In this way the high pressure steam is utilized in the closed heating coils for rapidly heating the apparatus, and the same source of steam is also utilized, with suitable reduction in pressure, in the ejector jets 37 for bringing about thorough and effective circulation and agitation of the liquid contents of the still.

Coincident with the starting of the vacuum, when the water in the boiler 5 is cold, I open a live steam line (not shown) and admit live steam into the distilled water in this boiler to bring this water to the boiling point, after which the live steam is shut off and no longer needed.

With the large excess of heating surface in the coils 11 to 15, the contents of the still are brought to a boil in a few minutes at the partial vacuum then maintaining within the still and even before the final vacuum is built up. The agitation of the contents of the still by the steam entering through jets 37, and the expansion of the finely divided steam particles released through the jets 37 cause the still contents to rise and cover all of the closed steam coils and to flow rapidly upwards through these coils, with an inward side-wise motion such that the flow is through the superheater coil 16 and downwardly within the space inside this coil 16 to the bottom of the still, rapid circulation of the contents of the still being thus accomplished and promoting the transfer of heat to the still contents.

The level of the still contents within the inner bounds of the superheater coil 16 is normally a foot or more below the level of the still contents rising through and over the coils 11 to 15 and pouring side-wise through and downwardly over the inner portion of the superheater coil 16. The circulation of the contents is so rapid, and the space between the turns of the superheater coil is so restricted that the general circulation is upwardly through the heating coils on the exterior of the superheater coil, and largely downward inside the superheater coil, with some flowing side-wise between the turns of this coil. So perfect and uniform is the mixing of the steam and the still contents, brought about by the ejector jets 37, that the contents of the still do not bump or surge or swell higher than about the top of the superheater coil 16, and there is no danger of any of the contents swelling over or spitting or bumping over, as the contents of the still at no time become locally superheated.

As a result of this rapid and uniform and effective circulation of the entire still contents, and the intimate contact of the jet steam with the liquid, and the rapid flow of the contents over the heating coils, there is only a small difference at most between the temperature of the liquid contents of the still, the temperature of the vapors leaving the still, and the temperature to which the steam is superheated in the superheater coil before it is discharged into the liquid contents of the still. The superheating of the steam, in the superheater coil 16, is thus accomplished by the liquid contents of the still, at a higher temperature than that at which the saturated steam is supplied to the coil, so that this steam is superheated to practically the temperature of the liquid contents of the still before it is introduced through the ejector jets into such contents.

By starting a still in the manner described I have been able to start a still that had been emptied and opened to the air so that it was distilling glycerin at a rapid rate within fifteen minutes after starting the vacuum apparatus. The glycerin vapor thus formed and passing through the goose-neck 2, separator 3 and pipe 4, enters boiler 5 and begins to make steam therein, due to the latent heat given up by the glycerin vapor in condensing to a liquid. The steam thus formed begins to build up a pressure in the boiler 5, and as soon as this pressure reaches about 4 pounds per square inch gauge pressure, the valve F from the high pressure steam supply line is closed and the valve 33 is opened to permit the steam generated in the boiler to enter the superheater coil 16 through orifice 32. The orifice 32 is somewhat larger than orifice 34, as it is supplied with steam at a lower pressure than that which passes through the orifice 34. The sizes of both of these orifices are so calculated as to take these different steam pressures into account and to deliver the desired amount of open jet steam. If the steam generated in the boiler 5 is maintained by means of the release valve 54 at about 5 pounds gauge pressure, or about 20 pounds absolute, there will be about 18 pounds differential pressure between the steam entering the orifice 32 and the still contents, causing a rapid blast of steam to pass through the ejector jets 37.

The jet steam, at the moment of entering the still contents, expands fully ten-fold, due to the sudden decrease in pressure, and causes an enormous amount of the liquid still contents to pass through the ejector jets 37, due to the ejector action. This liquid becomes most intimately mixed with this expanding steam at the instant

that the mixture is being discharged upwardly through the heating coils 11 to 15.

Operating in the manner described, I have found it possible to condense 80% or more of the glycerin distilled in the boiler 5 and collect this glycerin in the tank 7 in a high degree of purity. The rapidity of distillation may be observed by watching the change in height of glycerin in the gauge glasses located in the sides of the tanks 7 and 9, thus indicating the levels of distilled glycerin within these tanks.

Varying amounts of open jet steam may be used in operating the apparatus. I have operated, for example, with the use of 400 pounds of open jet steam per hour in the still while producing about 1000 to 2000 pounds of glycerin distillate per hour. This amount of open jet steam is considerably less than has been customarily used heretofore. I have found that a somewhat smaller or larger amount of open jet steam can also be used with good results. The orifice 34, or the orifice 32, through which the steam is supplied to the superheater coil and to the ejector jet, are of such a size that they will deliver a proper amount of steam, e. g., 400 pounds per hour, and the amount of open jet steam thus supplied can thus be regulated and controlled.

I have found that the distilling capacity of the apparatus of my invention is very flexible, as I have been able to distill soap lye crude glycerin at rates ranging from 1000 pounds to 2000 pounds of glycerin per hour in a still only about 6 feet in diameter and 6 feet high on the sides, while producing the highest grade of glycerin. This distilling capacity is greatly in excess of that of other types of glycerin stills heretofore used.

Most of the stills heretofore in use have been provided with large heating coils extending upward along the sides of the stills and above the liquid level to the very top of the still. This arrangement of coils, in contrast with the one which I use, is a misuse of heating surface, superheats the vapors without any advantage being derived therefrom and necessitates the use of more condensing surface. In stills of the former types the liquid within the still is subject to spasmodic superheating due to inadequate and irregular mixing which results regularly in spitovers so that it has always been necessary to provide a large first catchall to return not only spitovers to the still but also a substantial portion of the distillate. In ordinary stills entrainment frequently carries non-volatile impurities in appreciable amounts through all catchboxes, regularly necessitating redistillation, whereas the even boiling brought about by the ejector jets and arrangement of coils in my still induces practically no entrainment of still contents and the distillate collected is at all times substantially free from even traces of salt or non-volatile impurities even when distilling salt crude glycerin at the rates mentioned, while the separator 3 is at all times substantially free from liquid entrainment as may be seen through peep glasses (not shown) located in the top of the separator.

In the operation of the apparatus of the present invention, after the still has begun to operate in the manner previously described, it may be desirable to shut off part of the steam entering through the coils 11 to 15. This can be done by closing one or more of the valves A, B, C, D and E and the corresponding valves *a*, *b*, *c*, *d* and *e*. I have found that the heat supplied by a single coil would usually be sufficient to maintain a fair rate of distillation, but that it is preferable to use two or three coils, and, toward the end of the distillation, to turn on additional coils in order to maintain a high rate of distillation at all times.

The heat for the distillation is supplied mainly by the closed heating coils, and at the high temperature obtainable with high pressure steam, but local overheating of the still contents is avoided by the uniform and thorough circulation and agitation of the still contents by the use of the ejector jets for the direct jet steam.

I have found that a separate outside superheater for superheating the open jet steam is not necessary. The heat necessary for superheating the steam is relatively small, requiring only about two or three percent of the heat furnished to the still by the heating coils 11 to 15. By arranging the superheater coil in the still where it does not absorb heat from the still contents in the zone where heat supply is most needed, or lessen the capacity of the still, this superheater coil will readily absorb sufficient heat from the still contents to give the desired superheat, and to bring the steam to practically the temperature of the still contents, without the need of an outside superheater. The general circulation within the still, previously described, is such that vaporization takes place largely from that portion of the still outside the superheater coil, and around and above the heating coils 11 to 15, and the circulation of the liquid, from which the vapors are largely removed, inwardly and downwardly over and through the superheater coil, results in effective heating of this coil by the still contents, without objectionable cooling of the still contents or interference with effective distillation thereof. Heat losses from radiation with the use of an outside superheater, which are considerable, are thus avoided or minimized.

In distilling soap lye crude glycerin which contains an appreciable percentage of salt it becomes necessary to provide means for the removal of the accumulated salt and other non-volatile impurities known as "foots". This may be done once or twice a day or at any desired intervals by removal of the foots after distilling most of the glycerins from the still residue. To accomplish this, when the feed of crude glycerin to the still is discontinued, the distillation is allowed to proceed until the level of the still contents drops to the bottom of the gauge glass. All valves A, B, C, D and E and *a*, *b*, *c*, *d* and *e* may then be opened wide and valve 33 closed. At the same time valves G and H are opened wide permitting open jet steam from the high pressure main S to enter through orifices 38 and 39 to ring jets 41 and 42. Orifices 38 and 39 are of such a size that together they will permit a properly controlled amount of steam to pass, say about 400 pounds per hour.

In this way the greater part of the glycerin may be rapidly distilled from the still residue. The distillation may then be stopped and the vacuum may be broken and the still foots may be washed out with hot water in a suitable manner. The still can thus be made ready in a short time for further distillation. At the same time it is advantageous to remove the distillate from tanks 7 and 9. In beginning a new distillation closely following another, no auxiliary heating of the distilled water in boiler 5 is necessary.

After a distillation is started, as described, the distillation is almost automatic and one operator has plenty of time to attend to several stills, the starting times of which may be staggered. During the course of distillation the operator sees

that the level of the still contents is maintained substantially constant as indicated by gauge glass 70, by the feed control valve 69 and that the temperature of the hot water entering boiler 5 and the hot water coming from condenser-concentrator are maintained near the desired temperature. He will also observe that a good flow of cold water enters the top banks of tubes of condenser-concentrator 8 and that the vapors passing out of vapor pipe 10 are not permitted to become hot enough to carry any glycerin with them.

Careful tests of the absolute glycerin fed to my still and of the absolute glycerin distilled and accounted for show that there are substantially no unknown losses in the use of my still, such as might be due to decomposition or vapor losses.

In operating the apparatus, as thus far described, the open jet steam utilized in the still for aiding in the circulation and distillation, is produced by the evaporation of distilled water in the boiler-condenser 5 by means of the latent heat of the glycerin recovered in the condenser in condensing glycerin vapors to a liquid; and the distilled water so vaporized is itself derived from the condensation of steam in the closed heating system used to furnish heat in the still for the volatilization of the glycerin during distillation. This is a particularly advantageous combination and arrangement and sequence of steps and operations. The high pressure steam utilized for furnishing heat in the still is itself condensed, giving pure distilled water which is supplied to the boiler at a temperature approximating its boiling point and there serves to cool and condense the glycerin, while it is itself revaporized to give the open jet steam used in the still. The steam serves, finally, for carrying volatile impurities from the glycerin out of the system through vapor pipe 10. The same steam and distilled water thus serve a quadruple function in the process, and with notable economy in steam consumption, as well as a particularly advantageous utilization of the steam and water condensed therefrom.

In certain of its broader aspects, however, and considering the distillation operation in the still proper as an operation separate from the combined distillation and condensation, the distillation can be carried out with steam derived from some other source, such as by reduction in pressure of the high pressure steam supplied through a suitable controlling valve or orifice, such as provided in the valve F and orifice 34. So also, from the standpoint of generation of steam in the boiler 5, the operation is similar if the distilled water is supplied from some other source than the condensed water from the high pressure steam coils. Accordingly, I do not limit myself, in the broader aspects of the invention, to the use of open jet steam derived from the distillation of distilled water in the boiler-condenser; nor do I limit myself to the utilization of the condensed water in said boiler, when the open jet steam is to be generated therein. But I nevertheless claim the apparatus specifically when the open jet steam is derived in the boiler 5, and when the distilled water supplied thereto is recovered from the high pressure steam, since there are distinct advantages in the combined operations, considered as a whole. The utilization of the condensed water gives a proper supply of pure water for feeding to the boiler, at a temperature approximating the boiling point in the boiler, so that this water requires little or no heating to raise it to the boiling point; while it is free from objectionable volatile impurities, and the open jet steam produced therefrom is likewise free from objectionable volatile constituents. Moreover, the water itself, being pure condensed water, is free from non-volatile matter such as would leave accumulative and objectionable deposits on the heating surface of the boiler, and thereby impair its efficiency.

Another advantageous feature of the invention is the provision of means for the utilization of the flash steam recovered from the condensed water from the heating coils 11 to 15, generated on reduction of pressure on the hot condensate, for preheating the impure glycerin fed to the still 1. This flash steam is capable of utilization elsewhere, and the crude glycerin can be otherwise preheated, but the flash steam is nevertheless a convenient and advantageous heating medium for preheating the glycerin, and, by its use, added heat economy is obtained, and the heat contained in the condensed water from the heating coils is thereby further utilized to advantage in the process.

So also, in the operation of the apparatus, as heretofore described, the steam generated in the boiler-condenser 5, from heat recovered during the condensation of the glycerin, is advantageously used both for open jet steam in the still and for heating the lower rows of tubes in the condenser-concentrator 8, for concentrating the glycerin condensed therein, although steam of equal quality from other sources may be used. In certain of its broader aspects, the invention is not limited to the use of steam so produced; but steam of equal quality derived from other sources could be similarly utilized without departing from the spirit of my invention, in its broader aspects, even though the utilization of steam otherwise derived would not result in the economies and advantages which I obtain by utilizing heat recovered in the process in generating steam for use in various steps of the process; and I consider the process as a whole, with its various interrelated steps and operations, a particularly advantageous one, and one presenting important advantages from the standpoint of heat economy and quality of the glycerin produced.

The improved distilling apparatus of the present invention is distinguished in important respects from distilling apparatus in which bubble towers, or rectifying columns with horizontal plates and bubble caps, or towers packed with marbles, rings, or other filling material aimed to give a large amount of distributing surface, are employed. Bubble towers regularly produce a sensible hydrostatic head that is not ordinarily considered objectionable, as such towers are commonly operated at atmospheric or superatmospheric pressure. However, any hydrostatic head is highly objectionable in a distilling apparatus operating under a good vacuum, as it lessens very appreciably the vacuum that can be maintained and causes the ebullition in the still to be uneven and to surge as vapors pass through the liquid and result in a temporary drop in the hydrostatic head, with a temporary sudden improvement in the vacuum. Packed towers have the same objections as bubble towers but with the difference that their action is more irregular, due to irregular reflux. They are also subject to the objection that they are continually subject to irregular channeling which permits irregular flow of vapors through the towers.

My glycerin distilling apparatus is distinguished in important respects from such bubble towers or

packed towers. In contrast with such distilling apparatus, in which bubble towers or packed towers are employed, with objections such as those above mentioned, my distilling apparatus has no obstruction at any time to the free passage of the vapors in any part of the apparatus, as there is a full and unobstructed and substantially unconstricted passage for vapors from the still to the vacuum equipment, through the various parts of the apparatus and such passage remains open and unobstructed at all times.

Thus, in the entrainment separator **3**, while I intentionally divert the course of the vapors so as to remove any possible non-volatile impurity from them, there is no obstruction to the free passage of the vapors therethrough. Similarly, in the boiler-condenser **5**, while I provide means for deflecting the vapors against the condensing surfaces, and for removing the condensed glycerin from the condensing surface, there is free and unobstructed flow of the vapors through the spiral passages in the condenser tubes, so that uncondensed vapors will have a free and unobstructed passage through the boiler-condenser.

The arrangement of condensing and concentrating tubes in my condenser-concentrator is also such as to leave free passages for uncondensed vapors between these tubes. As already explained I stagger the tubes above one another in such a way that vapors will necessarily be deflected in their passage through the condenser-concentrator, and so that condensed aqueous glycerin solutions dripping from one set of tubes will fall successively to tubes of lower banks which are successively hotter and which successively evaporate additional quantities of water from the refluxing glycerin solutions until the reflux drips from the lowest steam heated or very hot tubes as highly concentrated refined glycerin. It can thus be seen that the condensation and concentration in my condenser-concentrator are at all times capable of accurate control and can be made complete inasmuch as the upper tubes are at all times kept cold, while the bottom tubes are always kept hot and the intermediate tubes are all substantially evenly graduated in temperature.

From the foregoing description it will be evident that the invention presents many features and advantages, both when considered as a whole, and when different parts and features are considered by themselves. The still itself, the boiler-condenser, and the condenser-concentrator each have distinct advantages in themselves, but they cooperate in a particularly advantageous manner, with notable economy, and with the direct recovery of concentrated glycerin, without the need of redistillation.

In the still proper, the indirect heating of the glycerin by high pressure steam coils suitably arranged near the outer portion of the still, the use of open jet steam introduced through ejector jets so introduced as to create a rapid circulation of the still contents and to force continuously a considerable portion of the still contents, intimately mixed with the finely divided sprayed steam from the jets, directly against and over that portion of the heating surface having the highest temperature, in such a way as to effect the maximum volatilization of glycerin while avoiding completely the superheating of the still contents, and the use of a superheated steam coil for the steam jets which is within the still and so located that it does not rob heat from the still in the zone where most of the glycerin is volatilized, provides a particularly valuable means of distilling glycerin.

The boiler-condenser enables heat to be recovered in an advantageous and effective manner from the glycerin vapors, while condensing these vapors to give a recovery of most of the glycerin in a state of high purity such that further distillation is not required. At the same time the heat thus recovered is utilized to advantage in the volatilization of distilled water and the production of steam free from objectionable volatile impurities which is advantageously supplied as the open jet steam in the vaporization of the glycerin; and the condensed water, condensed in the high pressure heating coils, is advantageously utilized, with reduction in pressure and partial self-vaporization, for preheating the crude glycerin supplied to the still and for supplying hot distilled water to the boiler-condenser at a high temperature. Part of the steam generated in the boiler is also advantageously employed in the subsequent condenser-concentrator for aiding in the concentration of the condensed glycerin therein.

The condenser-concentrator will effectively condense and concentrate all of the glycerin that is passed through the primary condenser (or boiler-condenser). It condenses glycerin of all concentrations at the same time from the most dilute, which may be practically of zero concentration in the upper part of the condenser-concentrator, to glycerin of around 99% concentration or over in the lower heated portion of the concentrator. This condenser-concentrator is so constructed and operated that the more dilute glycerin solutions condensed in the upper portion, falling downwardly from one bank of pipes to the next hotter bank, become gradually concentrated to 99% or over, so that highly concentrated refined glycerin is drawn off from the bottom of the condenser-concentrator at the same time that the upper colder banks of tubes prevent the volatilization and loss of any appreciable amounts of glycerin from the apparatus.

The following example of the distillation of glycerin by means of the improved apparatus will illustrate temperatures and other conditions which may obtain in different parts of the apparatus during the carrying out of the process:

Assuming that high pressure steam is used at about 150 pounds gauge pressure or at a temperature of about 366° F., the glycerin undergoing distillation in the still may be maintained, for example, under a vacuum of about 15 mm. mercury and at a temperature of around 320° F., being heated by the high pressure steam coils. The glycerin vapors leave the still and pass to the boiler-condenser at about the same temperature, i. e., at around 320° F.

The hot condensed water drawn off from the high pressure heating coils has its temperature reduced by partial self-vaporization, on reduction of pressure, so that the water in the receiver has a temperature of e. g., around 267° F. and the steam formed has a similar temperature. The steam used to preheat the glycerin preheats it to a temperature approximating that of the steam used, depending on the intimacy and efficiency of heat transfer.

The boiler-condenser has the boiler portion containing the condensed water at a pressure of e. g. 5 pounds gauge and with the steam generated at a temperature of about 228° F. This steam is superheated in the superheater coil, be-

fore it is discharged into the glycerin through the ejector jets, to approximately the temperature of the glycerin in the still, e. g., about 320° F.

The admixed glycerin and steam vapors, etc. enter the top of the boiler-condenser at approximately the temperature at which they leave the still, e. g., at about 320° F., and leave the bottom of this condenser at a lower temperature, e. g., around 257° F. The condensed water from the receiver 17 enters the boiler-condenser at a temperature which may approximate that of the water in the receiver 17 or at a lower temperature. If cooled to, e. g., 190° F. before it enters the boiler-condenser and introduced at that temperature it will have a somewhat greater cooling and condensing effect on the glycerin than it would if introduced into the still at a higher temperature, nearer that of the water in the receiver 17.

The temperature of the admixed glycerin and steam vapors, etc. entering the bottom of the condenser-concentrator may be approximately that of the vapors leaving the boiler-condenser, e. g., around 257°, more or less. The vapors of water and impurities leaving the top of the condenser-concentrator will vary somewhat in operation but may be for example around 120° F., more or less. The temperature of the cold water entering the condenser-concentrator will vary somewhat with climatic and other conditions and may be, e. g., around 60° F. and may be heated in the tubes of the condenser-concentrator to approximately the boiling point or somewhat below the boiling point, e. g., around 150° F., depending upon the amount of water supplied and other considerations. Where the lower rows of coils in the condenser-concentrator are heated by steam the steam will enter at a temperature, e. g., around 228° F., which may be below the temperature of the entering vapors of glycerin and water and will condense some glycerin and will assist these vapors in heating the condensed glycerin which drips down from the higher tubes on to these lower steam-heated tubes.

By operating the apparatus as a whole, I have found that it is possible to recover substantially the entire glycerin distillate in a concentration of 99 to over 99.5% glycerin, without appreciable loss of glycerin through volatilization, and without subsequent redistillation of the glycerin in separate distilling operations.

While the apparatus has been described in connection with the distillation of glycerin, apparatus and operations involving the same principles of construction and operation may be advantageously utilized in the distillation and purification of other volatile substances having a considerably higher boiling point than water and which are miscible with water in their higher and lower concentrations.

I claim:

1. An apparatus for distilling glycerin comprising a still provided with a plurality of closed steam heating coils located in the lower part of the still, steam operated ejector jets for mixing the contents of the still with steam, said jets being so located as to discharge the mixture of said still contents with steam against and among said heating coils for producing glycerin vapors, means for regulating the steam flow through said heating coils, an entrainment separator, vapor pipes for conducting the glycerin vapors from the still to the separator and from the separator to a boiler-condenser, a boiler-condenser for the simultaneous production of condensed glycerin and the generation of steam from boiling water, means for conveying steam so generated in said boiler-condenser to the ejector jets in the still to serve as open jet steam in distilling glycerin from the contents of the still, means for regulating the flow of jet steam to the still, means for drawing off condensed water from the closed steam heating coils and for reducing the pressure thereon, means for supplying such water at a reduced pressure to the boiler-condenser for the generation of pure steam therefrom, a superheater coil located in the still for superheating the steam passing from the boiler-condenser to the ejector jets, means for collecting the glycerin condensed in said boiler-condenser, a condenser-concentrator connected to and following said boiler-condenser for condensing the balance of the glycerin from the vapors of said still as glycerin and aqueous glycerin solutions of varying concentrations, means for conveying uncondensed vapors from said boiler-condenser to said condenser-concentrator, means in said condenser-concentrator for concentrating the more dilute glycerin and aqueous glycerin solutions condensed therein to produce concentrated glycerin, means for collecting said concentrated glycerin, and means for conveying excess water vapors substantially free from glycerin from said condenser-concentrator to a suitable vacuum apparatus so as to maintain a suitable partial vacuum within said still.

2. In an apparatus for distilling glycerin, a still having closed steam coils therein to furnish heat for the volatilization of the glycerin during distillation, a boiler-condenser connected to said still for receiving and condensing glycerin volatilized in said still and for the generation of steam from boiling water by utilization of the heat of condensation from the condensing glycerin for converting the boiling water into steam, and means for drawing off condensed water from the closed steam coils and supplying the same to the boiler-condenser for converting into steam, and means for collecting the said condensed glycerin.

3. An apparatus as set forth in claim 2 having ejector jets in the still for introducing open jet steam therein, a connection for conveying the steam generated in said boiler-condenser to said jets, and means for regulating said jet steam.

4. In an apparatus for the distillation of glycerin, a still having closed steam coils therein to furnish heat for the volatilization of the glycerin during distillation, a source of high pressure steam connected to said coils, means for drawing off water condensed from such steam under pressure and substantially free from high pressure steam and means for reducing the pressure on the condensed water while at a high temperature to effect partial volatilization thereof to steam, means for collecting the steam produced by such reduction in pressure and the remaining unvaporized hot water, a glycerin preheater for preheating the glycerin supplied to the still, and means for heating the glycerin in said preheater by the steam produced by said reduction in pressure.

5. In an apparatus for the distillation of glycerin with the aid of open jet steam and diminished pressure, a still of generally cylindrical shape, a plurality of closed steam heating coils arranged in the lower portion of the still with a generally concentric arrangement, a plurality

of ejector jets located in the lower portion of the still and constructed and arranged to cause rapid circulation of the contents of the still over and among the heating coils and intimate admixture of steam with the liquid to effect even and rapid circulation, said heating coils and ejector jets being located below the operating liquid level in the still whereby superheating of the vapors in the still is avoided, a superheater coil substantially concentrically arranged within the heating coils and connected with the ejector jets to supply steam thereto, a boiler-condenser connected to said still for receiving and condensing glycerin volatilized in said still by indirect contact with boiling water and with resulting generation of steam from the boiling water, means for supplying steam so generated to the superheater coil and ejector jets, means for supplying condensed water from the heating coils to the boiler-condenser, and the arrangement of the heating coils, the superheater coil, and the ejector jets being such as to maintain substantially uniform temperature of the liquid in the still and circulation of the liquid over the heating coils and superheater coil, and means for collecting the condensed glycerin.

6. In an apparatus for the distillation of glycerin with the aid of open jet steam and diminished pressure, a still having controlled steam heating surfaces forming a closed system within the lower part of the still for heating the contents of the still with steam under pressure, ejector jets for introducing steam into the still and effecting intimate intermixture of the steam and liquid contents of the still, and for circulation of the still contents, said steam heating surfaces and said ejector jets being situated within the lower portion of the still and below the operating liquid level therein and being so arranged as to provide for rapid mixing of the steam and still contents through the ejector jets and for projecting a mixture thus formed against said heating surfaces while maintaining an even and rapid circulation of steam and still contents over and among such steam heating surfaces during the volatilization of the glycerin, and also so arranged as to avoid local overheating or superheating of the still contents or of volatilized vapors, means connected to said still for receiving and condensing volatilized glycerin by indirect contact with boiling water to effect generation of steam therefrom, means for supplying steam so generated to the ejector jets, means for regulating steam so supplied, and means for supplying water condensed in the closed steam heating system as the boiling water to be utilized in the generation of steam supplied to said ejector jets and means for collecting the condensed glycerin.

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