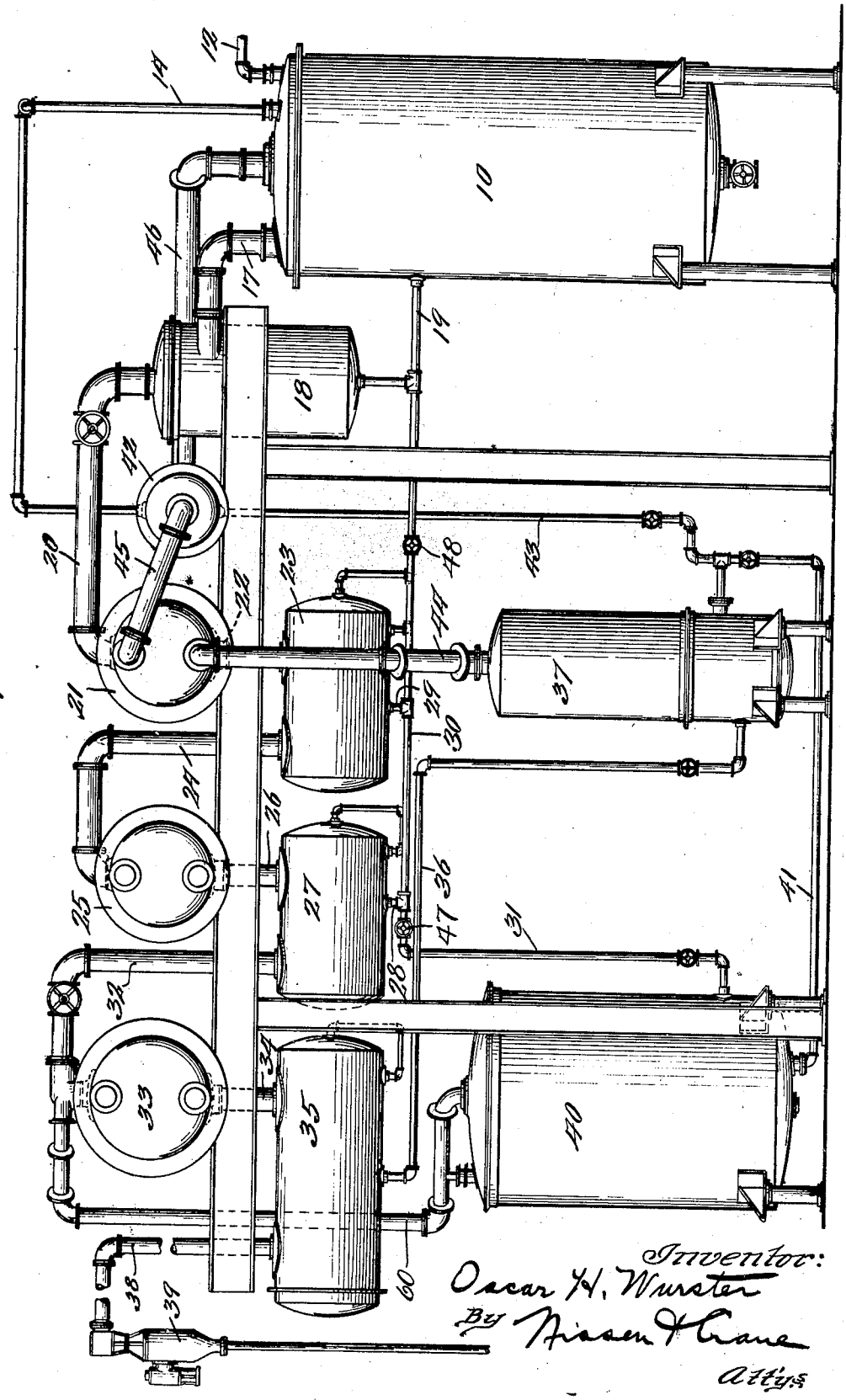
Nov. 1, 1932. O. H. WURSTER 1,886,256

PROCESS OF AND APPARATUS FOR DISTILLING LIQUIDS

Filed Feb. 13, 1928 3 Sheets-Sheet 1

Fig. 1.



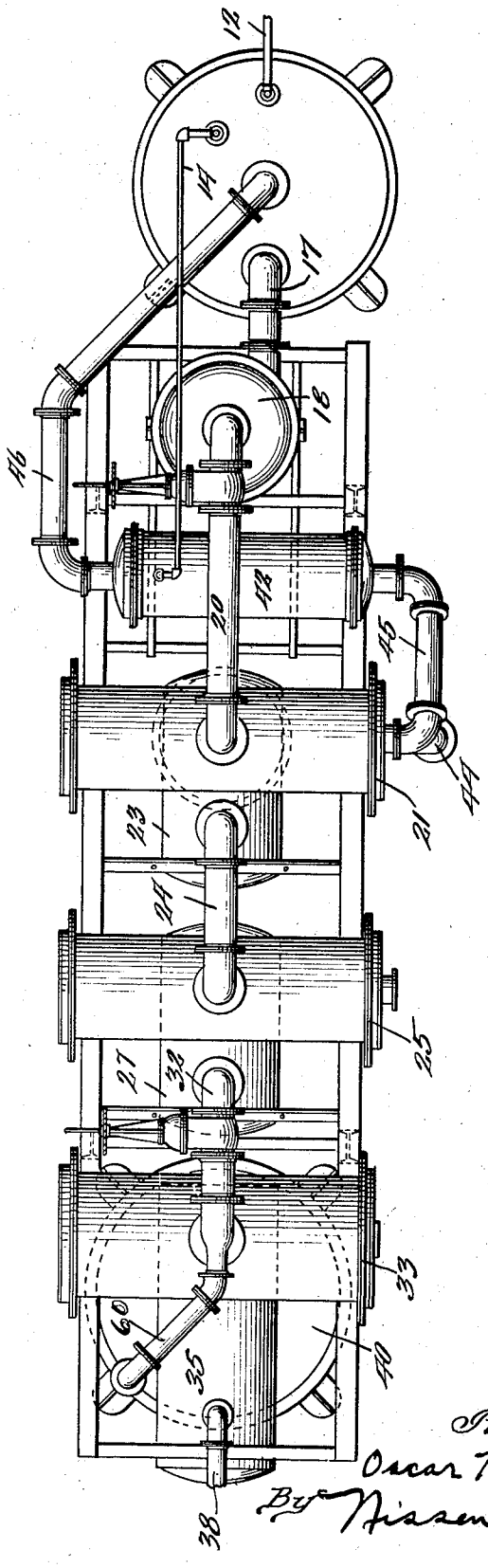
Inventor:
Oscar H. Wurster
By Mason & Crane
Attys

Nov. 1, 1932. O. H. WURSTER 1,886,256

PROCESS OF AND APPARATUS FOR DISTILLING LIQUIDS

Filed Feb. 13, 1928 3 Sheets-Sheet 2

Fig. 2.



Inventor:
Oscar H. Wurster
By Nissen & Crane
Attys.

Nov. 1, 1932. O. H. WURSTER 1,886,256

PROCESS OF AND APPARATUS FOR DISTILLING LIQUIDS

Filed Feb. 13, 1928 3 Sheets-Sheet 3

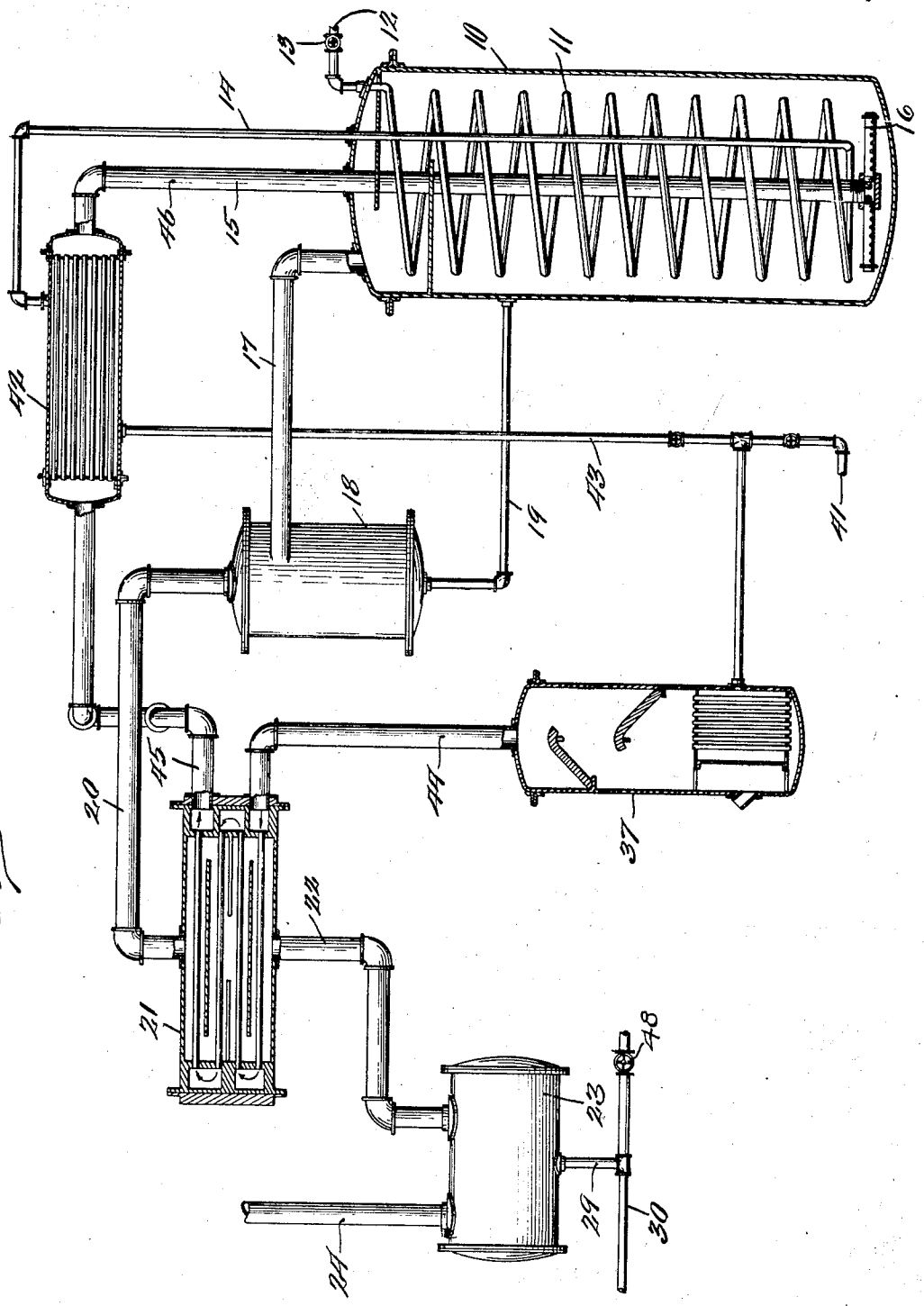


Fig. 3.

Inventor:
Oscar H. Wurster
By Nissen & Crane
Attys

Patented Nov. 1, 1932 1,886,256

UNITED STATES PATENT OFFICE

OSCAR H. WURSTER, OF CHICAGO, ILLINOIS

PROCESS OF AND APPARATUS FOR DISTILLING LIQUIDS

Application filed February 13, 1928. Serial No. 253,826.

My invention relates to new and useful improvements in distilling processes and apparatus for distilling various liquids and the same is particularly well adapted for use in connection with liquids which show a tendency to partially decompose and for liquids which do partially decompose when distilled directly or alone at normal atmospheric pressure. This invention relates to a process and apparatus similar to that disclosed in my co-pending application, Serial No. 253,827, filed on even date therewith. Such liquids are distilled in a partial vacuum, whereby the boiling points of the liquids is lowered in accordance with the degree of exhaustion. A gas or vapor, such as for example steam, may be injected into the liquid to be distilled so that a condition is brought about in which the sum of the vapor pressure of the liquid to be distilled and that of the vapor, or gas, which is introduced to facilitate the distillation is greater at a given temperature than the vapor pressure of the liquid to be distilled, and in this manner the boiling point is lowered enabling the distillation process to be carried out at a lower temperature and without harmful effect upon the liquid to be distilled. The two methods referred to above, namely, carrying out the distillation under partial vacuum and introducing a vapor to increase the vapor pressure and lower the boiling point of the mixture, may be combined for the purpose of preventing a partial decomposition of the liquids. I have improved the combined process of distillation under vacuum with injected gas, as will be described.

In many distillation processes, it is desirable to use superheated steam as the fluid to be injected into the liquid to be distilled. When steam is the injected fluid, it is desired to superheat it in cases where the temperature of distillation under the given conditions is above the temperature of saturated steam at the vacuum existing in the still.

In some former processes of distillation, steam from a boiler was expanded and superheated in a special vessel, and this steam was then injected into the liquid, such as glycerine. This process was improved by generating steam under vacuum from the dilute liquor obtained from the condensation of the steam used in a previous distillation. This steam so generated under vacuum was then superheated in a heat exchanger by the vapors of the distilling liquid and the superheated steam was then injected into the body of the liquid to be distilled to aid in the distillation thereof. This was a desirable improvement insofar as the conservation of heat was concerned. The steam for distilling was generated under vacuum and at relatively low temperature, and could then be generated by means of the exhaust heat from other parts of the process such as the still, the concentrator and the pumps. The low temperature steam was superheated by vapors from the distilling liquid, thus absorbing heat while at the same time cooling and partially condensing these vapors. In the operation of these older processes, I found the following objections and short-comings coupled with their specific advantages. The process which used steam from the boiler, expanding and superheating the steam to be injected, while giving expanded and superheated steam of high temperature, had a high steam consumption, and was therefore costly in operation. The high temperature, however, resulted in a high recovery of product from the still residue. The process which evaporated by means of waste heat the previously condensed dilute liquors and superheated the resulting steam by the vapors arising from the distilling liquid, was economical in steam consumption, but did not give still temperatures high enough to be conducive to a high yield of distilled liquid from the crude liquid. That this would be so, can be clearly understood on a consideration of the conditions. The steam to be injected being heated by the vapors from the distilling liquid, said superheated steam would not, in practice, in passing through the superheater, reach the temperature of the distilled vapors. Furthermore, the vapors arising from the distilling liquid are in practice at a lower temperature than the impure liquid itself. Therefore, the injected superheated steam in this older process, being colder than the liquid in the still,

has the actual effect of cooling the liquid in the still. The heat required for distillation must therefore be made up from the heat transmitted from the high pressure steam in the closed coil in the still. There is, therefore, a retarding effect on the process of distillation which becomes more and more pronounced as the process proceeds and the residue accumulating in the still becomes heavier and thicker.

High still temperatures are desired at the finish of a run to effect a high recovery of the liquid, such as glycerine, from the still residue or foots. But as the end of a run is approached, the amount of liquid vapors passing over with the steam becomes less and less. Therefore, the steam to be injected has in the older process less heat transferred to itself and goes to the still at a lower temperature. As the residue in the still becomes thick and heavy the heat from the still coils is conducted through the mass less readily and less effectively. The results of these conditions are that the capacity of the apparatus is reduced and it becomes impossible to remove and recover as much of the liquid, such as glycerine, from the residue or foots as can be recovered at higher still temperatures. Such liquid, as glycerine, is then lost in the residue or foots, or must be recovered so far as practical in some subsequent recovery process, adding to the cost and trouble of the operation.

To make the above conditions clear, they can be illustrated by the following example. With an average still temperature of 350° F. it has been found that the vapors of the distilling liquid would only heat the steam coming from the evaporator from an average temperature of about 120° F. to a temperature of 285° to 300° F. It is obvious, therefore, that in this instance the injected steam had to absorb sufficient heat from the liquid in the still to have its temperature raised 50° to 75° F. or to the still temperature before it could distill the liquid, and in having its own temperature raised, the injected steam must necessarily cool off the adjacent portions of the liquid.

I found that it would be desirable and would improve the process of distillation and increase the yield if the expanded injected steam would be brought to a temperature above the temperature of the vapors in the still and equal to or higher than the temperature of the liquid in the still, so that the injected steam would impart heat to the process of distillation instead of absorb heat as in the former process last described.

It is an object of my invention to economize heat in my novel process and at the same time to bring the temperature of the steam which is to be injected into the still to a temperature equal to or above the temperature of the liquid in the still prior to its injection.

Other objects of my invention are to obtain as pure a distillate as possible from the impure or crude liquid distilled, to obtain as high a yield of pure liquid as possible from the raw, crude, or impure liquid, and to have the apparatus so designed that the above desired results will be brought about as efficiently and as simply as possible, and at the same time with as low steam or heat consumption as possible.

My invention is exemplified in the combination and arrangement of parts shown in the accompanying drawings and described in the following specification and in the steps of the process disclosed therein.

The numbers on each drawing refer to the same or to corresponding parts of the equipment.

Fig. 1 shows one general arrangement in elevation of a complete distilling unit to which my invention applies;

Fig. 2 shows one plan of such a distilling unit;

Fig. 3 is a diagrammatic elevation of the still, catchall, preheater, receiver, evaporator, superheater, and connecting piping with parts in section showing one form of apparatus comprised in the present invention and suitable for carrying out the process as described.

One of the liquids for which my process and apparatus is well adapted is crude glycerine. It will, of course, be obvious that my process and apparatus is not limited in its application to the distillation of glycerine, but that the same may be used in distilling or fractionating other liquids which are distilled by steam or water vapors and particularly such liquids which show a tendency to partially decompose when distilled directly or alone at normal pressure.

My process and apparatus consists in installing a high temperature superheater in the steam line between the still and the heat-exchanger in which the steam from the evaporator or from the concentrator is preheated by the vapors arising from the liquid in the still. In this superheater the partially superheated steam which is below the temperature of the liquid in the still is further superheated to a temperature equal to or above the temperature of the liquid in the still.

This superheater may be heated by any suitable means such as by being oil or gas fired or steam heated. A means of heating which is readily controlled is naturally to be desired. I have found as an especially desirable form of superheater one in which the partially superheated steam is heated to its desired temperature by high pressure steam from the same source as the steam which is used in the closed coil of the still for heating the still. I have also found as desirable the method of connecting this final superheater in such a way that the heating steam from the

outlet of the still heating coil passes next through the superheater. The heating steam outlet of the superheater can then be connected to the heating steam inlet of the evaporator, thus effecting an efficient use of the available heat.

As shown in the drawings, the numeral 10 designates a still. This still is provided with a heating coil 11 with steam line 12 controlled by valve 13 leading to the coil and steam line 14 leading away from the still heating coil. The still is further provided with expanded and superheated steam inlet pipe 15 leading to open spider 16 from which the steam is injected into the liquid, such as glycerine, to be distilled. The mixed vapors arising from the liquid pass out of the still through pipe 17 through catchall 18. Entrainment separated in this separator returns to the still through pipe 19. The hot still vapors pass from the separator through pipe 20 to the preheater-condenser 21. In the preheater-condenser the mixed vapors are partially cooled and some of the glycerine is condensed. The remaining vapors and condensed liquid pass through pipe 22 to preheater-receiver 23 where the liquid is separated from the vapors. The vapors pass through pipe 24 to cooler-condenser 25. Here most of the remaining glycerine is condensed from the mixed vapors and passes through pipe 26 to cooler-receiver 27 where the condensed glycerine liquid is separated from the remaining water and glycerine vapors. The condensed glycerine from the preheater-receiver 23 and the cooler-receiver 27 passes through pipes 28, 29, and 30, back to the still 10, or through pipe 31 to the concentrator 40, as desired. Suitable control valves 47 and 48 are provided for this purpose. The water vapor containing a small amount of glycerine vapor and non-condensable gases from cooler-receiver 27 pass through pipe 32 to the sweet-water condenser 33. Here the water vapor and remaining glycerine are condensed and passing through pipe 34 to sweet-water receiver 35 flows through pipe 36 to sweet-water evaporator 37. The separated non-condensable gases pass through pipe 38 to vacuum pump 39.

The sweet-water evaporator 37 may be heated by steam from any suitable source, but as shown the exhaust steam from the high pressure coil in the concentrator 40 passes through pipe 41 to the steam inlet of the sweet-water evaporator and the exhaust steam from the high pressure steam chamber of the superheater 42 passes through pipe 43 to the steam inlet of the sweet-water evaporator.

The steam or vapor passes off from the evaporator under a vacuum of 24″ to 26″ corresponding to temperatures of approximately 140° to 125° F., through pipe 44 to the preheater-condenser 21, which is a special form of counter-current heat exchanger. Here the vapor or steam from the evaporator is superheated to a temperature somewhat below the temperature of the vapors coming from the still. This is so because the superheating is done by the vapors arising from the liquid in the still. When distilling glycerine the temperature to which the steam is superheated in the pre-heater condenser has been observed to be from 285° to 300° F.

The partially superheated steam from the preheater-condenser 21 passes through pipe 45 to superheater 42. This superheater is heated by suitable means such as high pressure steam from the outlet pipe 14 of the still heating coil 11. With steam at 200 pounds gauge pressure and at the corresponding temperature of saturated steam, the super-heater may thus be heated to a temperature of approximately 387° F. and the steam which has already been superheated to about 285° to 300° F. in the preheater-condenser is further superheated to a temperature above the temperature of the vapors rising from the still and to a temperature equal to or higher than the temperature of the liquid in the still. In the case of distilling glycerine the temperature of the superheated steam may be raised in the superheater to a temperature of from 350° to 375° F. using heating steam at 200 pounds pressure and to higher temperatures with heating steam at higher pressure or with the heating steam superheated.

The superheated injection steam, prepared as described above, next passes through pipe 46 into the still pipe 15 and into the open steam distributing spider 16 in the bottom of still 10. The expanded and superheated steam is here injected into the body of the liquid which is under a vacuum of above 27″ and facilitates the distillation thereof.

By preheating and further superheating the expanded injection steam as described above, my invention brings about a continuous system of distillation effecting a considerable economy in the use of steam and at the same time accomplishing the important result of giving a more complete recovery of the liquid, such as glycerine, from the still residue or foots.

It will be seen that I economize heat in my novel process and at the same time bring the temperature of the injected steam to a temperature equal to or above the temperature of the liquid in the still prior to its injection. By this means, I not only effect all of the economies inherent in using waste heat from various sources for generating the steam under vacuum and of transferring heat from the vapors of the distilling liquid to the steam to be injected and by this means partially cooling and condensing said vapors from the distilling liquid, but I also increase and maintain the rate of distillation in my apparatus because of this increase of tem-

perature of my injected superheated steam and I thus make it possible to have a high temperature in the still at and near the end of the run thus permitting a higher recovery of liquid, such as glycerine, from the still residue or foots. With suitably high temperatures and high vacuum this final recovery of the liquid, such as glycerine, from the foots may be carried to such a degree of completion that the residue or foots may be discarded as practically useless and thus eliminate the cost and trouble of any further recovery process.

It should be noted that my invention is not limited strictly to the various details of the process and of the apparatus which have been particularly described but that the same is as broad as is indicated by the accompanying claims.

I claim:—

1. A process of distilling liquids under vacuum and in an atmosphere of steam which comprises the injection of superheated steam into the liquid to be distilled, condensing the mixture of steam and vapor from the distilling liquid, evaporating a fraction of said condensate under vacuum and preheating the vapor therefrom by the vapors from the distilling liquid, and superheating said preheated vapors by high temperature and pressure steam to form the said superheated steam injected into the liquid to be distilled, said condensate being evaporated by the said high pressure steam after being used to superheat the said preheated vapors.

2. A process of distilling liquids which comprises passing steam through a superheater, injecting the superheated steam into the liquid to be distilled, condensing the mixture of steam and vapor from the liquid, maintaining the distilling liquid and condensate under vacuum, evaporating the water in the condensate by exhaust steam from said superheater, preheating the vapor or steam from the condensate by the vapors from the distilling liquid, and superheating the preheated steam by said superheater to form said superheated steam injected into the liquid.

3. A process of distilling liquids which comprises heating the liquid to be distilled with a steam coil, injecting superheated steam into the liquid, condensing the mixture of steam and vapor from the distilling liquid, evaporating a fraction of the condensate, maintaining the distilling liquid and condensate under vacuum, preheating the vapors from the condensate by the vapors from the distilling liquid, and superheating the preheated vapors above the temperature of the distilling liquid by the steam passing through the still steam coil to form the said superheated steam injected into the liquid to be distilled.

4. An apparatus for distilling liquids comprising a still, an evaporator, means for maintaining a vacuum in said evaporator and still, means for maintaining said still at distilling temperature, a preheating condenser having a conduit connecting said evaporator with said still and a conduit connecting said still with said preheating condenser to preheat the vapor from said evaporator by the vapor from said still, a preheater receiver connected with said preheater condenser to separate the vapors from the condensate, a final condenser, a receiver connected with said final condenser and having vacuum means to remove the uncondensed vapors, a liquid conduit connecting said last-mentioned receiver with said evaporator, a concentrator, liquid conduits connecting said preheater receiver with said still and concentrator, valves in said last-mentioned conduit, one of said valves being between said preheater receiver and said still and the other being between said preheater receiver and concentrator, said valves permitting the flow of liquid from said preheater receiver to be directed into said concentrator or into said still, as desired, and a superheater between said preheating condenser and still for heating the vapors in the conduit connecting said evaporator and still.

5. An apparatus for distilling liquids comprising a still, an evaporator, means for maintaining a vacuum in said evaporator and still, means for maintaining said still at distilling temperature, a preheating condenser having a conduit connecting said evaporator with said still and a conduit connecting said still with said preheating condenser to preheat the vapor from said evaporator by the vapor from said still, a superheater between said preheating condenser and still for heating the vapors in the conduit connecting said evaporator and still, a steam connection with said superheater for heating the same, and means for heating said evaporator from the exhaust steam of the superheater.

6. An apparatus for distilling liquids comprising a still, an evaporator, means for maintaining said still at distilling temperature, a preheating condenser having a conduit connecting said evaporator with said still, and a conduit connecting said still with said preheating condenser to preheat the vapor from said evaporator by the vapor from said still, a superheater between said preheating condenser and still for heating the vapors in the conduit connecting said evaporator and still, fractionating condensers and receivers connected with said preheating condenser, means for removing the uncondensed gases, means for maintaining a vacuum in the distilling apparatus and evaporator, a conduit connecting one of said receivers with said evaporator, a concentrator having a vapor connection with one of said condensers, and a conduit connecting other of

said receivers with said still and concentractor, valves in said last-mentioned conduit to direct the condensate into said still or concentrator, as desired.

7. The method of distilling liquids which comprises the steps of heating the liquid to be distilled by superheated steam which is out of direct contact with the liquid, condensing the vapors from the liquid, evaporating said condensed vapors by steam exhausted from the distillation of said liquid but said condensed vapors being out of direct contact with said steam, passing the steam resulting from the evaporation back into the liquid being distilled, preheating said last mentioned steam by said vapors resulting from said distillation and before said last mentioned steam reaches the liquid being distilled, and superheating the said preheated steam before the same reaches said liquid and by the first mentioned steam while the same is passing from the liquid being distilled to the condensed vapors being evaporated.

8. In an apparatus for distilling liquids, the combination with a still, of an evaporator, a preheating condenser, a steam coil for heating liquid in said still, conduits for directing vapors from said still through said preheating condenser and thereafter to said evaporator, means for heating the condensed vapors in said evaporator, a conduit for directing vapors from said evaporator through said preheater and thereafter to said still, said last mentioned vapors thereby being heated by said first mentioned vapors, and a superheater for superheating the vapors passing from said preheating condenser to said still, said superheater being heated by exhaust steam from said coil.

9. In an apparatus for distilling liquids, the combination with a still, of an evaporator, a preheating condenser, a steam coil for heating liquid in said still, conduits for directing vapors from said still through said preheating condenser and thereafter to said evaporator, means for heating the condensed vapors in said evaporator, a conduit for directing vapors from said evaporator through said preheater and thereafter to said still, said last mentioned vapors thereby being heated by said first mentioned vapors, and a superheater for superheating the vapors passing from said preheating condenser to said still, said superheater being heated by exhaust steam from said coil and said evaporator being heated by the exhaust steam from said superheater.

In testimony whereof I have signed my name to this specification on this 7th day of February, A. D. 1928.

OSCAR H. WURSTER.