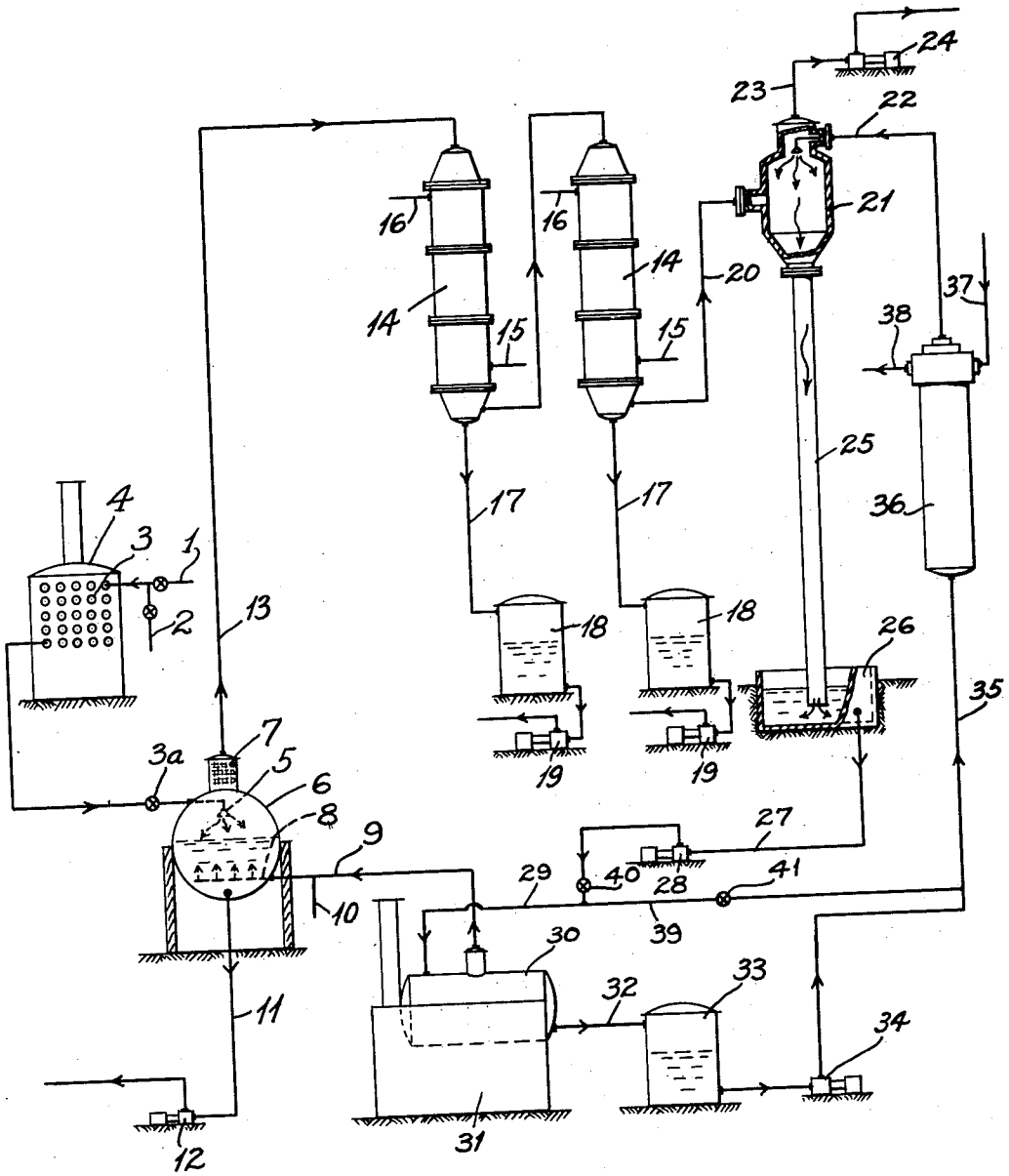
March 1, 1932. E. G. RAGATZ 1,847,625

HIGH VACUUM STEAM DISTILLATION

Filed March 7, 1928



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Patented Mar. 1, 1932 1,847,625

UNITED STATES PATENT OFFICE

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HIGH VACUUM STEAM DISTILLATION

Application filed March 7, 1928. Serial No. 259,691.

This invention relates to a process and apparatus for the distillation at high vacuum, that is, at low absolute pressure. It relates, particularly, to a process and apparatus of high vacuum steam distillation wherein the steam is condensed at relatively high temperatures and relatively low pressures, the pressure being insufficient of itself to condense the steam at said temperature. This is accomplished in the main by scrubbing the steam with a solvent to produce a water-solvent mixture whose vapor pressure is less than water and to produce a mixture whose vapor pressure is sufficiently low to cause condensation of steam at the pressures and temperatures employed.

It is well known that in ordinary vacuum distillation, the absolute pressure maintainable in the still is in part determined by the vapor pressure which exists at the terminal receiver immediately preceding the vacuum pump. Thus the minimum pressure maintainable in this receiver will determine the vacuum in the system. This pressure is evidently the vapor pressure of the condensate at the temperature in the terminal receiver. In steam distillation of materials to produce relatively high boiling point distillates, as in the distillation of lubricating oils, the final condensate which must be produced is water. When this distillation occurs at very low pressures in the neighborhood of a few millimeters of mercury, it is essential to provide means in the terminal receiver of condensing the steam at this pressure and to maintain the pressure of the water in the receiver by sufficiently cooling the condensate so as to produce a vapor pressure below the desired pressure. However, in order to maintain the vapor pressure of the steam condensate such as to maintain this very low vacuum, the water must be cooled to very low temperature. Thus at very low pressures in the neighborhood of 5 m. m. a temperature at the freezing point of water would be required. However, if the steam is condensed by use of ordinary cooling water, the average temperature of the condensate would be 100° F. and perhaps higher. This is determined by the available cooling water temperature and the proportion of steam condensed. Where water is obtained from cooling towers as is usual, temperatures of less than 100° F. are very costly to maintain. At a temperature of 100° F. the vapor pressure of water is 50 m. m.

It is the object of this invention to obviate the difficulties enumerated above. The object is to make possible high vacuum distillation with steam without excessive cooling of the condensate, and, in fact, to use only such cooling water as is available at the plant. In further discussion, the term "available cooling water temperature" will be defined as being that temperature which is obtainable by using a cooling water available at the plant where the process is operated when such cooling water is used in conventional methods either by direct contact with the steam or by intermediate heat exchange.

In operating the process of this invention, the steam resulting after the condensation of the oil vapor, which is at very low pressure, is scrubbed with a solvent and the solvent-water mixture is evaporated under a high vacuum. The steam which results from this distillation may be re-used in the process to produce a cyclic operation. By the re-use of the steam from the regenerating stills and boiler, a regeneration of the solvent-water mixture at substantially the distillation pressure of the apparatus is made possible. This results in a low regeneration temperature. Additionally, since this low temperature distillation produces a regeneration material of relatively low temperature, the amount of cooling which is required before it may be reintroduced into the condenser, to scrub the steam, is diminished. In many operations, especially where cooling water is a considerable item of expense, this produces a considerable saving.

The invention is directed to a high vacuum steam distillation whereby means is provided to condense the steam at available cooling water temperature by scrubbing it with a solvent which is cooled by available cooling water, and regeneration of the water-solvent mixture at a low absolute pressure, and reintroduction of the steam into the distilling system, thus to cause regeneration of the solvent

at substantially the pressure of the system. This invention is particularly directed to such a method and apparatus for high vacuum distillation below, approximately 25 m. m. whereby the residual steam is scrubbed to form a solvent mixture which will have a vapor pressure at, or below 25 m. m., the temperature of the condensate being obtained by means of available cooling water and the solvent mixture is regenerated at substantially the pressure of the system provided by the reintroduction of the regenerated steam into the system.

The process and apparatus will be better understood by reference to the accompanying drawing which illustrates the specific embodiment, illustrative, but not limiting the invention.

The figure on the accompanying drawing shows a schematic form of one embodiment of the apparatus for carrying out the process of the invention.

In the drawing, 1 is a charging line for liquid to be distilled, in this case lubricating oil stock. The oil is fed to 1 under desired pressure. Steam may be mixed with the oil if desired by injection through line 2. The oil is heated in coils 3 of furnace 4 and injected into still 6 through spray 5. The back-pressure on coils 3 is regulated by valve 3_a. Distillation of the oil occurs in still 6 with the aid of steam injected through spray 8. This steam, as will be explained later, consists of steam regenerated from still 30, supplemented by make-up steam injected through line 10. The unvaporized material is withdrawn through 11 by pump 12. The vaporized material passes through mist separator 7, which returns entrained liquid. The vapor passes through line 13 into a series of fractional condensers 14, cooled by oil, steam or water, as the case may be. The condensate is withdrawn through 17 into run-down tanks 18, from which liquid is withdrawn through 19. As will be understood by those skilled in the arts, by positioning the still 6 and condensers 14 at barometric height, the material will be discharged without the aid of pumps.

The uncondensed material consisting substantially of steam or fixed gases or light volatile oils passes from the last condenser 14 through line 20 into the barometric condenser 21 where it meets a stream of solvent liquid introduced through 22. The uncondensed material is withdrawn through 23 by the high vacuum pump 24. The solvent-water mixture passes down barometric leg 25 into the hot well 26, from which it is withdrawn through 27 by means of pump 28 and introduced into still 30 by means of line 29. Still 30 is heated by furnace 31. The generated steam is introduced through line 9 into the still 6, and steam is introduced into the system through 2 in addition to that through line 9. Steam may build up in the system and must withdraw. Line 10 may function to permit this withdrawal, as those skilled in the art will readily recognize. The regenerated solvent is withdrawn through line 32 into tank 33 and withdrawn by pump 34 through line 35, to be cooled in cooler 36. Cooler 36 is cooled by means of available cooling water introduced through 37 and exited through 38. If desired, a portion of the solvent-water mixture passing from pump 28 may be by-passed through 39 by partial regulation of the valves 40 and 41, to be re-cycled through the cooler and into the condenser.

As a specific example illustrating this process in its preferred embodiment, the following may be given:

Lubricating oil stock is introduced into coil 3, together with steam and heated to about 600° F. and then introduced into still 6 where additional steam is added. Still 6 is operated at about 20 m. m. absolute pressure and the ratio of the steam to the oil is such that the mixture passing 13 contains ¾ by volume of the steam, i. e. the partial pressure of the steam is 15 m. m. and the oil 5. The oil is condensed in condenser 14, and the residual steam passes to barometric condenser 21.

In the barometric condenser it is scrubbed by a solvent such as a salt solution, and the temperature and concentration and volume of the scrubbed liquid is such as to maintain about 100° F., and the requisite vapor pressure of the condensate so as to produce condensation of steam at pressure in the barometric condenser. This vapor pressure will depend on the back pressure of the system and is the pressure in the still minus the back pressure of the system. The requisite concentration can be readily determined by anyone skilled in the art since it is that concentration which would give a vapor pressure at 100° F., which will equal that necessary to maintain the requisite absolute pressure in the still 6. The mixture is withdrawn through 27 and introduced into still 30 where it is evaporated under vacuum, the steam generated being introduced into the still 6. The vacuum in still 3 would, therefore, be equal to the pressure in still 6 plus the back pressure on still 30. That is, still 30 will be operated at a sufficient pressure to pass the vapor into still 6. The concentrated material is passed to the cooler 36.

In operating with a salt solution care must be taken to concentrate the salt solution in 30 not above that concentration which would cause crystallization of the salt when cooled in cooler 36. Since the limit of temperature and concentration are fixed by the requisite concentration in condenser 21 and that in 36, it will be frequently found advisable to circulate a portion of the liquid from 26 directly into the cooler 36, through line 39. This will permit the maintenance of the req-

uisite concentration and volume of the solution in 21 and also permit of sufficient evaporation in 30.

While the specific example discloses the use of 20 m. m. it will be understood that absolute pressure may be lower as far as 1 or 2 m. m. or less, or above 25 m. m. as the case demands. The solvent may be either glycerine or any other liquid of relatively high boiling point and sufficient water solubility. Salt such as calcium chloride or other materials dissolved in water which have sufficient solubility are available. The principle here is the reduction of vapor pressure by a solute, as is well understood by those skilled in the art. This will enable anyone skilled in the art to choose the solute best suited for their conditions. The above description is not to be taken as limiting, but descriptive of the best modes of employing the invention, which I claim to be:

1. A method of high vacuum steam distillation which comprises steam distilling oil under high vacuum, condensing the oil vapors, scrubbing the steam with a solvent, maintaining the temperature and concentration of the solvent mixture thus formed to maintain the mixture at a vapor pressure less than the vapor pressure of water at the temperature of the mixture and evaporating the water from the solvent mixture under a pressure, substantially that of the said steam distillation of the oil.

2. Method of steam distilling oil under high vacuum which comprises vaporizing oil with steam under high vacuum, condensing the oil vapors thus generated without condensing the steam, scrubbing said steam vapors with a solvent, maintaining the pressure of the solvent mixture below the vapor pressure of water at the temperature obtainable by use of available cooling water, evaporating the water from the solvent mixture under a pressure, substantially that of the said steam distillation of the oil.

3. Method of high vacuum steam distillation comprising distilling oil with steam under high vacuum, condensing the resulting oil vapors without condensing the steam, scrubbing the steam with a solvent at a temperature not less than the available cooling water temperature and at an absolute pressure less than the vapor pressure of water at said cooling water temperature, removing the solvent water mixture, evaporating water from said solvent, re-introducing the regenerated steam into the oil undergoing distillation, cooling said solvent by heat exchange with available cooling water, and scrubbing further steam with said solvent in cyclic operation.

4. A method of high vacuum distillation of oil which comprises steam distilling oil at a low absolute pressure, fractionally condensing the oil but not condensing the steam, cooling the steam to a temperature of itself insufficient to condense the steam at the pressure maintained thereon by contacting said steam with a solvent and thus condensing the steam at the said pressure and temperature, removing the solvent water mixture, evaporating the water from the solvent, re-introducing the regenerated steam into the oil undergoing distillation, cooling the solvent and re-introducing the solvent into the steam to be condensed, in cyclic operation.

5. A method of high vacuum steam distillation which comprises distilling oil at a low absolute pressure with steam, condensing the steam at a low absolute pressure by scrubbing the same with a liquid, evaporating the water from the mixture thus formed, re-introducing the evaporated water into the oil undergoing distillation and re-introducing the regenerated liquid to condense further steam in cyclic operation.

6. An apparatus for high vacuum distillation comprising a steam still, a condenser, means for maintaining high vacuum in the steam still and in the condenser, means for introducing a scrubbing liquid into the condenser, an evaporator, means for introducing the scrubbing liquid into the evaporator, means for introducing the regenerated steam from the evaporator into the steam still, means for withdrawing the scrubbing liquid from the evaporator, a cooler, means for passing the scrubbing liquid from the evaporator through the cooler and to the condenser.

Signed at Los Angeles, in the county of Los Angeles and State of California, this 29th day of February, A. D. 1928.

EDWARD G. RAGATZ.