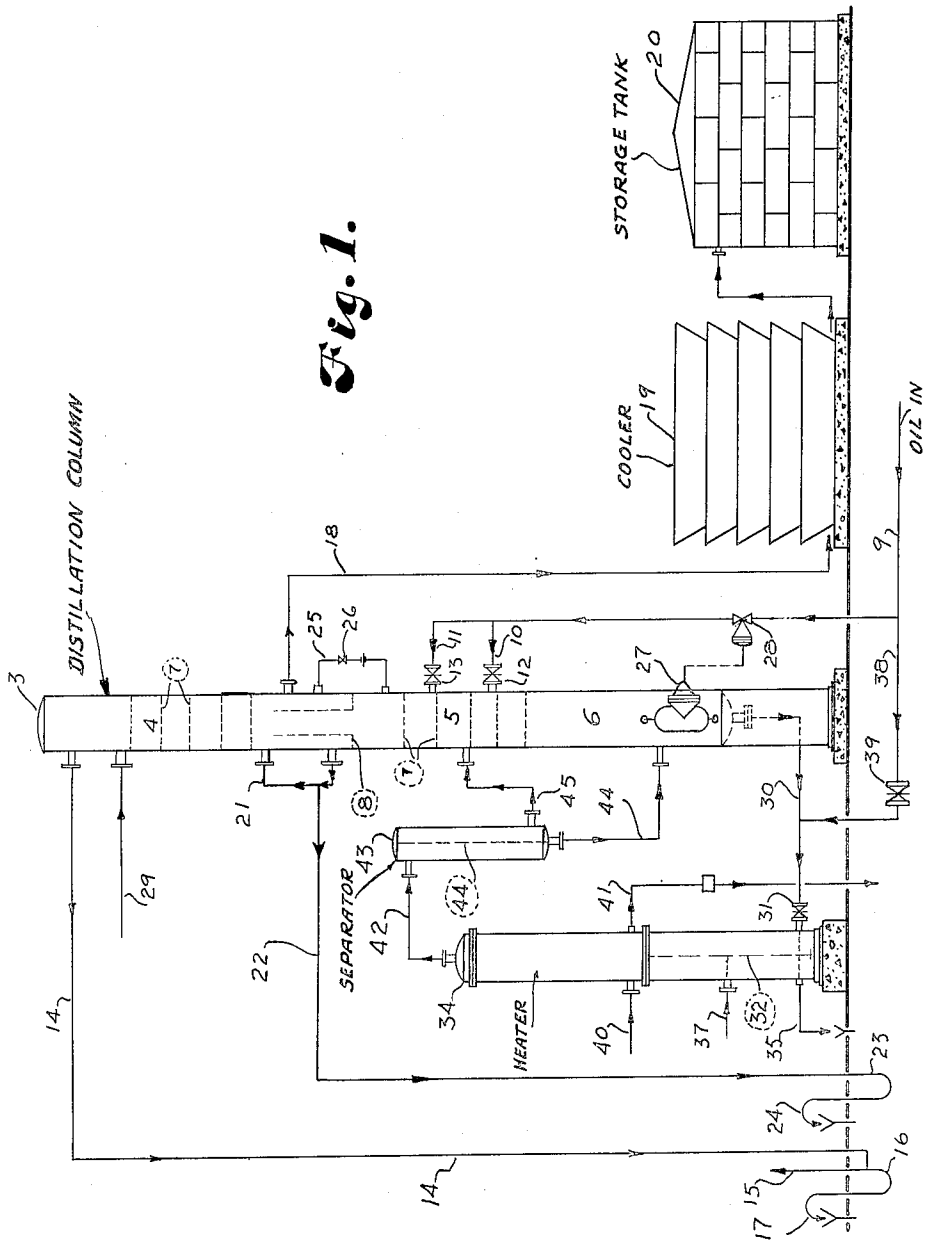
July 4, 1939. N. K. RECTOR 2,164,593

METHOD FOR DISTILLING OILS

Filed March 29, 1937 2 Sheets-Sheet 1

Fig. 1.



INVENTOR.
NORMAN K. RECTOR
BY Barry & Cyr
ATTORNEYS.

July 4, 1939. N. K. RECTOR 2,164,593

METHOD FOR DISTILLING OILS

Filed March 29, 1937 2 Sheets-Sheet 2

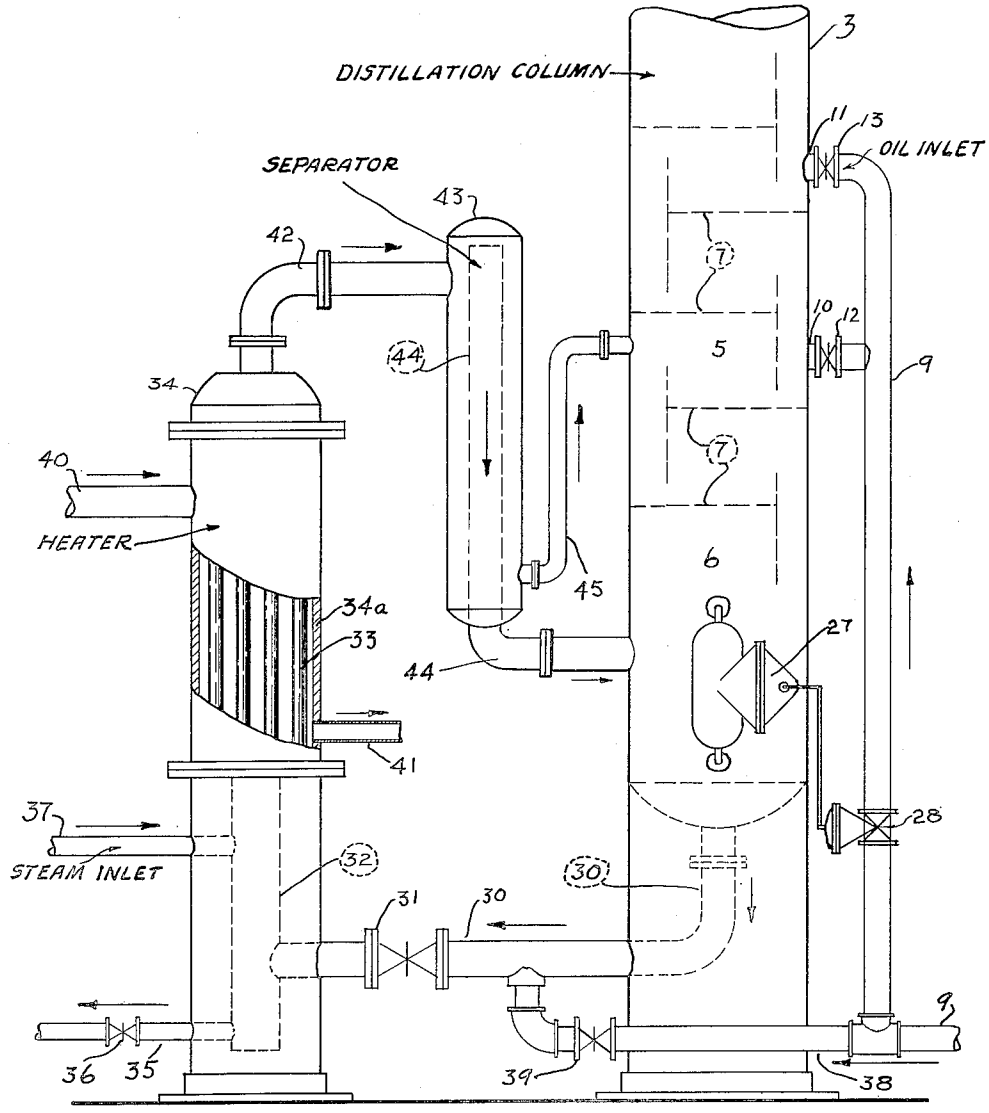


Fig. 2.

INVENTOR.
NORMAN K. RECTOR
BY Barry & Cyr
ATTORNEYS.

Patented July 4, 1939 2,164,593

UNITED STATES PATENT OFFICE

2,164,593

METHOD FOR DISTILLING OILS

Norman K. Rector, Tulsa, Okla., assignor to Petroleum Engineering, Inc., Tulsa, Okla.

Application March 29, 1937, Serial No. 133,721

8 Claims. (Cl. 196—85)

This invention relates to a method for distilling oils, and relates particularly to a distillation method for separating a minor portion of relatively non-vaporizable impurities from a major portion of substantially completely vaporizable hydrocarbon oil.

One object of this invention is to distill oil by circulating the oil in a cyclic path, heating the oil at one point in the path, vaporizing it another, and maintaining the cyclic flow without the aid of mechanical propelling means.

Another object is to utilize a gas as a medium for maintaining the cyclic flow of oil and to also lower the distilling temperature of the oil below its normal boiling range.

This invention is particularly adapted for use in connection with absorption processes, such as are used for the recovery of gasoline from natural gas or other gaseous or vaporous materials. In such processes, the absorption menstruum, which is circulated in contact with gases and vapors to recover gasoline therefrom, also picks up mechanical impurities and absorbs heavy materials such as gums and asphalts and becomes dirty after a period of use. These impurities, although minor in quantity, are undesirable and must be removed from the menstruum, if the efficiency thereof is to be maintained at a high level.

Absorption menstruums which have become dirty are usually cleaned by various distillation methods, and this invention provides an improved method and apparatus for this purpose, as will be readily apparent from the following description when read in conjunction with the accompanying drawings, in which:

Fig. 1 is a diagrammatic flow sheet of the process showing the general relationship of the various apparatus elements for practicing the process of this invention, and

Fig. 2 is a detail of the apparatus shown in Fig. 1.

Referring to the drawings, 3 designates a distillation column comprising a condensing section 4, a stripping section 5 and an accumulator section 6. Conventional bubble type trays 7 are mounted in condensing and stripping sections 4 and 5, respectively, of the column and condensing section 4 is fitted at its lower end with a receiving tray 8 for the collection of condensate formed in the condensing section 4 thereabove. A charge pipe 9 leads into stripping section 5 at spaced points, through branch pipes 10 and 11 which are fitted with valves 12 and 13 respectively. A vapor discharge pipe 14 leads from the top of column 3 to a vent pipe 15 at the lower end of which is a liquid trap 16 having a discharge pipe 17. A discharge pipe 18 leads from an upper level of receiving tray 8 through a cooler 19 to a storage tank 20. A pressure equalizing pipe 21 leads from a lower level of tray 8 back into the vapor space of column 3 above tray 8. A second discharge pipe 22 is connected to pipe 21 and leads therefrom to a trap 23 which discharges through a pipe 24. Pipe 22 connects to pipe 21 at a level somewhat below that at which pipe 18 leaves receiving tray 8. A pipe 25, in which is mounted a valve 26, leads from an upper level of tray 8, but below the outlet point of pipe 18, into stripping section 5. A liquid level controller 27 is mounted on column 3 in operative relationship with accumulator section 6 and is operatively connected to a control valve 28, which is mounted in pipe 9. A pipe 29 leads into condensing section 4 of column 3.

A pipe 30, in which is mounted a valve 31, leads from the lower portion of accumulator section 6 into the lower end of pipe 32 which is in communication at its upper end with the interiors of tubes 33 of a tube and shell type heater 34, the shell thereof being designated by the numeral 34a. A pipe 35, in which is mounted a valve 36, leads from the lower end of pipe 32. A pipe 37 leads into pipe 32 at a point above the point of entry therein of pipe 30. A branch pipe 38, in which is mounted a valve 39, leads from pipe 9 into pipe 30. An inlet pipe 40 and an outlet pipe 41 are connected to the shell 34a of heater 34 to permit the passage therethrough of heating fluid.

A pipe 42, in communication with the upper ends of tubes 33, leads therefrom into the upper portion of a separator 43. A pipe 44 extends from a point in the interior of separator 43 near the upper end thereof, downwardly therethrough and emerges from the lower end of separator 43 and is connected into column 3 at a point below the lowermost tray of stripping section 5. The upper end of pipe 44 is in open communication with the interior space of separator 43. A pipe 45 leads from the lower portion of separator 43 into column 3 at a point between some of the trays 7 of the stripping section 5, and above the point of entry into column 3 of pipe 44 but below the level of the open end of pipe 44 in separator 43.

In one specific case, the above described apparatus is utilized to practice the process of this invention, in the manner to be described below. In this case the oil to be distilled is absorption oil of a type commonly used in absorbing gasoline from natural gas or refinery vapors, and has normally a gravity of 38 to 42° A. P. I., a boiling

range of from about 350° F. to 550° F., and a mean boiling point of about 450° F. This oil has become contaminated with about 2 percent of impurities such as scale and rust, gummy and asphaltic materials, all of which are unvaporizable at the mean boiling temperature of the absorption oil. The oil, generally preheated to some extent, is fed through pipe 9, thence through either or both of pipes 10 and 11 into the stripping section 5 of column 3. The oil flows downward through stripping section 5 and flows over trays 7 making counter-current contact with heated oil vapors and steam flowing upwardly through stripping section 5. The oil vapors and steam have previously been heated and supplied to column 3 in a manner to be subsequently described, and have been heated to a temperature of about 350° F. which temperature, together with the boiling point lowering effected by the steam present, is sufficient to vaporize the major portion of the downflowing stream of charge oil. The mixture of oil vapors and steam finally resulting from the counter-current contact in stripping section 5, flow upward in column 3 from stripping section 5 into condensing section 4, where condensation of the oil vapors and steam takes place in a manner to be more fully described hereinafter.

The charge oil entering column 3 from pipes 9, 10 and 11, while generally preheated, is at a temperature lower than that of the oil vapors and steam with which it is contacted, and will produce some refluxing of these vapors with condensation of the heavier fractions thereof, thereby producing reflux fractionation of the vapors which, while not extensive, will be sufficient to insure the "knocking back" of any of the impurities which may be carried by the vapors.

The portion of the charge oil which remains unvaporized after contact with the oil vapors and steam in stripping section 5, and which contains the undesirable impurities, since they are unvaporizable at the temperature of the contact, will flow from the lower portion of stripping section 5 into accumulator 6, where a body of oil will be maintained at constant level by action of liquid level controller 27. The fluctuations in level of liquid in accumulator 6 will cause controller 27 to open or close valve 26 in pipe 9, as required, to regulate the rate of inflow of charging oil to the system in order to hold substantially constant the liquid level in accumulator 6.

Oil from accumulator 6 will flow therefrom by gravity flow through pipe 30 into the lower portion of pipe 32 and thence upward into the tubes 33 of heater 34. The level of liquid in accumulator 6 is maintained sufficiently high to raise the oil flowing through pipe 32 to about the lower extremities of tubes 33. Open steam, which, in this case, is exhaust steam at a temperature of from about 212° F. to 225° F., is introduced through pipe 37 into the oil flowing upward in pipe 32 and in mixing with the oil, produces a lift effect which serves to propel the oil the remaining distance through the tubes 33 and thence through pipe 42 into separator 43. Heating fluid is introduced through pipe 40 into the shell 34a of heater 34 and, by indirect heat exchange, heats the mixture of oil and steam passing through tubes 33 to a temperature sufficient to vaporize the major portion of the oil. The heating fluid, which in this particular example is high pressure steam, is discharged from shell 34a through pipe 41 to waste.

As a result of the heating applied in heater 34, the material entering separator 43 from pipe 42 will now consist of a mixture of steam, oil vapors and unvaporized oil, and will separate in separator 43 into a vapor stream comprising steam and oil vapors and a liquid stream consisting of unvaporized oil. The steam which has been mixed with the oil in pipe 32 will not only assist in lifting the oil through the heater 34 into separator 43, but will also lower the normal mean boiling point of the oil, thereby reducing the amount of heating required in heater 34 to vaporize the major portion of the oil in separator 43. The separated stream of oil vapors and steam will enter the open end of pipe 44 and will pass therethrough into column 3 below the trays 7 of stripping section 5. The unvaporized oil in separator 43 will be discharged from the lower portion thereof through pipe 45 into stripping section 5 at a point which is below the level of the open end of pipe 44 in separator 43. As a result of this relative arrangement, as soon as the level of oil in separator 43 rises above the level of the point of entry of pipe 45 into column 3, the oil will flow by gravity from separator 43 through pipe 45 into column 3 and will continue to flow as oil continues to enter separator 43 from pipe 42.

The unvaporized oil entering stripping section 5 from pipe 45 will mix on trays 7 with the charge oil entering from pipes 10 and 11 and the mixture will flow downwardly over trays 7 through stripping section 5 in counter-current to the stream of oil vapors and steam entering column 3 from pipe 44, thereby producing the initial stripping of the charge oil as mentioned above and also further stripping the unvaporized oil entering column 3 from pipe 45.

The oil vapors leaving stripping section 5 together with the steam, will flow upward in column 3 through condensing section 4 in counter-current contact with a down-flowing stream of water which is introduced in the upper portion of condensing section 4 through pipe 29. The water so introduced, will be preheated to a temperature just below its boiling point or approximately 212° F. As a result of such temperature, the water, in flowing downward over the trays of condensing section 4 in contact with the hotter mixture of oil vapors and steam, will itself be largely vaporized, but will condense, at the same time, all the oil, the initial boiling point of which is about 350° F., and therefore, considerably higher than the temperature of the water. At the same time, only a very small portion of the steam will be condensed. The condensed oil and the small amount of steam condensate thus produced, will collect in receiving tray 8 at the bottom of condensing section 4, and because of the difference in specific gravity of these two liquids, two separate layers will form in receiving tray 8. The condensed oil will be continuously withdrawn from the upper level of tray 8 through pipe 18 and cooler 19 into storage tank 20. The steam condensate will be withdrawn from a lower level of tray 8 through pipes 21 and 22 to trap 23 and thence through pipe 24 to waste.

Steam remaining uncondensed after contact with the water, together with that formed by evaporation of the condensing water, will flow from the upper portion of column 3 through pipe 14 and will be discharged from the system through vent pipe 15. Water produced by condensation of the steam in its passage to vent pipe

15 will be trapped in trap 16 and will discharge therefrom through pipe 17 to waste.

To assist in maintaining a constant quantity of oil in the system, pipe 25 provides means for permitting condensed oil to return from receiving tray 8 into the circulatory system and valve 26 in pipe 25 provides means for regulating the rate of back-flow of condensed oil so that only as much clean oil will leave the system through pipe 18 as enters it originally through pipe 9.

Circulation of oil from column 3 through heater 34 and back into column 3, and continuous distilling of the oil in the manner described while traveling through this circuit, will cause the various impurities, which are substantially unvaporizable at the distillation temperature, and which were originally contained in the charge oil, to be concentrated in accumulator section 6. Since these materials constitute only a relatively small fraction of the original charge, they may be permitted to circulate with the unvaporized oil and become concentrated therein for relatively long periods of time. Then, after extended periods of distillation in the manner described, the distillation operation is discontinued and the concentrated impurities are discharged from the system by opening valve 36 in pipe 35.

It will be evident from the foregoing that the cyclic flow of the oil throughout the distillation system is accomplished entirely without the aid of mechanical propelling means. Gravity flow is utilized to bring the oil from the high point of its cycle, which is the upper portion of separator 43 to the low point of the cycle which is the lower portion of pipe 32, and a steam lift is then utilized to lift the oil from its low point of flow back to the high point in separator 43, thus completing the cycle. The cost of equipment is thereby reduced. It will also be evident, that the gaseous medium used to assist the cyclic flow of the oil may also be utilized simultaneously to lower the normal boiling point of the oil, thereby reducing the heat required for distillation of the oil.

Instead of introducing the charge oil into column 3 directly through pipes 9, 10 and 11, the charge oil may be introduced at any other point in the cyclic path of the oil. As one example, charge oil may be introduced directly into pipe 30 through branch pipe 38 and valve 39. Similar connections may be made elsewhere throughout the cyclic path of the liquid oil.

While steam has been described as the preferred gaseous medium for both aiding the cyclic flow of oil and for lowering the boiling point of the oil to be distilled, other gaseous mediums may be used. However, any gaseous medium selected should be insoluble in the oil at the temperature at which they are in contact with each other. Where steam is used, it is desirable that the oil before being brought in contact therewith, should be heated to a temperature above the condensation point of the steam, in order not to adversely affect the lifting properties of the steam, which must be in the vapor state to accomplish this purpose.

The heating medium utilized for heating the mixture of oil and gas passing through tubes 33, may be high temperature steam, hot oil or other heating fluid. Instead of using a shell and tube type heater, as shown in the drawings, tubes 33 may be mounted in a furnace and the heat supplied to the fluid passing through the tubes by gases of combustion generated in the furnace. Heater 34, instead of being arranged in the manner shown in the drawings to provide for vertical lifting of the oil through the tubes and thence into separator 43, may be horizontally arranged and may form a part of pipe 42. In this case, the heater would be at about the point of maximum height in the cyclic path and oil lifted by gaseous medium to the entrance of the tubes of the heater, would flow therethrough directly into separator 43 and thence continue through the cycle, in the manner previously described.

The temperature to which the oil is heated in heater 34 will vary with the boiling range of the particular oil to be distilled, and will be influenced by the lowering in the boiling point of the oil effected by the action of the particular gaseous medium which is mixed with the oil.

The pressure to be maintained in the system is likewise variable. In the particular example above described, the pressure employed throughout the system is substantially atmospheric, but may be higher or lower and may be sub-atmospheric, if desired, particularly where it may be desirable to avoid overheating of the oils to be distilled, or to distill without cracking, oil which tends to disintegrate when heated to its normal boiling point.

Other methods and apparatus for condensing and separating the oil vapors and gaseous medium than that described, may be utilized and such other methods and apparatus are well known in the art and will be readily apparent to those skilled in the particular art to which this invention appertains.

It will also be understood that while the above description shows the invention as it is applied directly to the distillation of hydrocarbon oils, other liquids as well may be distilled in a similar manner and by similar means to remove therefrom small fractions of relatively high boiling impurities.

It should also be noted that while it is advantageous in the above-described apparatus to provide separator 43, in order that a primary separation of oil vapors and steam from unvaporized oil may be provided before these materials are again contacted in column 3, such separator is not essential to the process and apparatus of this invention. The heated mixture may be discharged directly from heater 34 into the stripping section 5 of column 3 and the final separation permitted to take place at that point without disadvantageously affecting the final results to be desired from the process.

What I claim and desire to secure by Letters Patent is:

1. The process of distilling oil containing a minor portion of impurities which are non-vaporizable at a temperature at which the major portion of the oil is substantially completely vaporizable, which comprises flowing said oil in a closed cycle from a distillation zone through a heating zone thence through a separating zone and back to said distillation zone, injecting steam into said oil at a point in said cycle outside said distillation zone and anterior to said heating zone, heating the mixture of oil and steam in passage through said heating zone to a temperature sufficient to vaporize said major portion of the oil without decomposition thereof but insufficient to vaporize said minor portion, vaporizing said major portion of said oil in said separating zone, therein separating the resulting oil vapors and steam from the unvaporized minor portion and continuously discharging these separated fractions into said dis-

tillation zone, the oil vapors and steam being introduced therein below the unvaporized minor portion, recycling said unvaporized minor portion through said cycle and adding fresh oil continuously to the oil flowing in said cycle.

2. The process of distilling oil containing a minor portion of impurities which are non-vaporizable at a temperature at which the major portion of the oil is substantially completely vaporizable, which comprises flowing said oil in a closed cycle from a distillation zone through a heating zone thence through a separating zone and back to said distillation zone, injecting a vaporizing gas into said oil at a point in said cycle outside said distillation zone and anterior to said heating zone, heating the mixture of oil and vaporizing gas in passage through said heating zone to a temperature sufficient to vaporize said major portion of the oil without decomposition thereof but insufficient to vaporize said minor portion, vaporizing said major portion of said oil in said separating zone, therein separating the resulting oil vapors and vaporizing gas from the unvaporized minor portion and continuously discharging these separated fractions into said distillation zone, the vapors and vaporizing gas being introduced therein below the unvaporized minor portion, recycling said unvaporized minor portion through said cycle and adding fresh oil continuously to the oil flowing in said cycle.

3. The process of distilling oil containing a minor portion of impurities which are substantially non-vaporizable at a temperature at which the major portion of the oil is substantially completely vaporizable, which comprises flowing said oil in a closed cycle from a distillation zone through a heating zone thence through a separating zone and back to said distillation zone, moving the oil through a part of said cycle by gravity flow and through the remainder of said cycle by injecting into said oil a gas which propels the oil and which also lowers the normal boiling point of said major portion of the oil, heating the mixture of oil and gas in passage through said heating zone to a temperature sufficient to vaporize said major portion of the oil without decomposition thereof but insufficient to vaporize said minor portion of the oil, vaporizing said major portion of said oil in said separating zone, therein separating oil vapors and vaporizing gas from the unvaporized minor portion and continuously discharging these separated fractions into said distillation zone, the vapors and vaporizing gas being introduced therein below the unvaporized minor portion, withdrawing said vapors and vaporizing gas continuously from the distillation zone, recycling said unvaporized minor portion of the oil through said cycle and continuously adding fresh oil to the oil flowing in said cycle.

4. A process of removing heavy impurities from absorbent oil comprising passing the oil containing the impurities downwardly successively through a stripping section and an accumulator section of a distillation zone, withdrawing oil from the accumulator section, injecting a vaporizing gas into the withdrawn oil to cause the oil to travel upwardly, passing the mixture of oil and gas through a heating zone and therein heating the oil to a temperature sufficient to vaporize the oil without decomposition thereof but insufficient to vaporize said impurities, afterwards passing the heated mixture into a separating zone and therein separating the heated mixture into a liquid oil fraction and a vapor fraction comprising oil vapors and said gas, separately withdrawing said fractions from said separating zone and introducing the liquid oil fraction into the stripping section, introducing said vapor fraction into the accumulator section, flowing the liquid oil fraction in the distillation zone counter-current to the vapor fraction, withdrawing oil vapors from the upper portion of the stripping section and condensing them, and withdrawing said impurities from the accumulator section.

5. A method of removing heavy impurities from an absorbent oil of the character used as a menstruum in separating condensable vapors from hydrocarbon gases comprising passing the oil contaminated with said impurities successively downwardly through a stripping section and an accumulator section of a distillation zone, withdrawing oil from the lower portion of the accumulator section, injecting a vaporizing gas in the withdrawn oil to cause it to travel upwardly, passing the mixture of oil and gas through a heating zone and therein heating it to a temperature sufficient to vaporize the oil without decomposition thereof while in the presence of the gas but insufficient to vaporize said impurities, passing the heated mixture into a separating zone and therein separating the mixture into a liquid oil fraction containing said impurities and a vapor fraction comprising oil vapors and said gas, withdrawing the liquid oil fraction from the separating zone and introducing it into the stripping section of the distillation zone, withdrawing said vapor fraction from the separating zone and introducing it into the accumulator section at a point below the stripping section, passing the vapor fraction upwardly through the stripping zone counter-current to the liquid oil fraction and to the charging stock, then passing the vapor fraction through a condensing section in the upper portion of the distillation zone and cooling it therein to a temperature sufficient to condense oil vapors but insufficient to condense said gas, discharging the gas from the upper portion of the condensing section, discharging condensed oil from the lower portion of the condensing section, and discharging impurities from the bottom portion of the accumulator section.

6. A method of treating oil containing a minor portion of impurities which are non-vaporizable at a temperature at which the major portion of the oil is substantially completely vaporizable, which comprises flowing said oil in a closed cycle from a distillation zone through a heating zone thence through a separating zone and back to said distillation zone, injecting steam into said oil at a point in said cycle outside said distillation zone and anterior to said heating bone, heating the mixture of oil and steam in passage through said heating zone to a temperature sufficient to vaporize said major portion of the oil without decomposition thereof but insufficient to vaporize said minor portion, vaporizing said major portion of said oil in said separating zone, therein separating the resulting oil vapors and steam from the unvaporized minor portion and continuously discharging these separated fractions into said distillation zone, the oil vapors and steam being introduced therein below the unvaporized minor portion, discharging said oil vapors and steam from the upper portion of the distillation zone and passing them into a condensing zone, cooling the oil vapors in the condensing zone to a temperature sufficiently low to condense the oil vapors, but insufficient to condense said steam, separately discharging the

steam and condensate respectively from the upper and lower portions of the condensing zone, and discharging said minor portion of impurities from the lower portion of the distillation zone.

7. The method of distilling oil which comprises, mixing said oil with low pressure steam, utilizing said low pressure steam to lift the oil through a heating zone, heating the mixture of oil and said low pressure steam in said heating zone by means of high pressure high temperature steam to an oil vaporizing temperature but below a decomposing temperature, passing the heated mixture to a separating zone, therein separating the heated mixture into a liquid oil fraction and a vapor fraction comprising oil vapors and said low pressure steam, separately withdrawing said fractions from said separating zone and introducing them into a stripping zone, therein flowing the liquid oil fraction counter-current to the vapor fraction to thereby vaporize additional quantities of said oil and separately withdrawing oil vapors and unvaporized oil from said stripping zone.

8. A method of removing impurities from absorption oil having a gravity of about 38 to 42 A. P. I. and a boiling range of about 350° F. to 550° F., comprising preheating the oil by passing it downwardly successively through the stripping section and an accumulator section of a distillation zone, withdrawing oil from the lower portion of the accumulator section and mixing it with steam, then heating the mixture to a temperature of about 350° F. and passing the same into a separating zone, separating the mixture in the separating zone into a vaporous fraction and a liquid fraction, passing said liquid fraction from the separating zone into the stripping section, passing said vaporous fraction into the accumulator section and causing it to rise counter-current to the charging stock and said liquid fraction whereby substantially all of the charging stock and liquid fraction are vaporized and discharged from the upper end portion of the stripping section, cooling the vapors passing from the upper portion of the stripping section and thereby condensing the vapors of the oil and separating the same from the steam, and discharging said impurities from the bottom portion of the accumulator section.

NORMAN K. RECTOR.