March 29, 1932. M. H. TUTTLE 1,851,550

DISTILLATION OF LUBRICATING STOCK

Filed Aug. 7, 1929 3 Sheets-Sheet 1

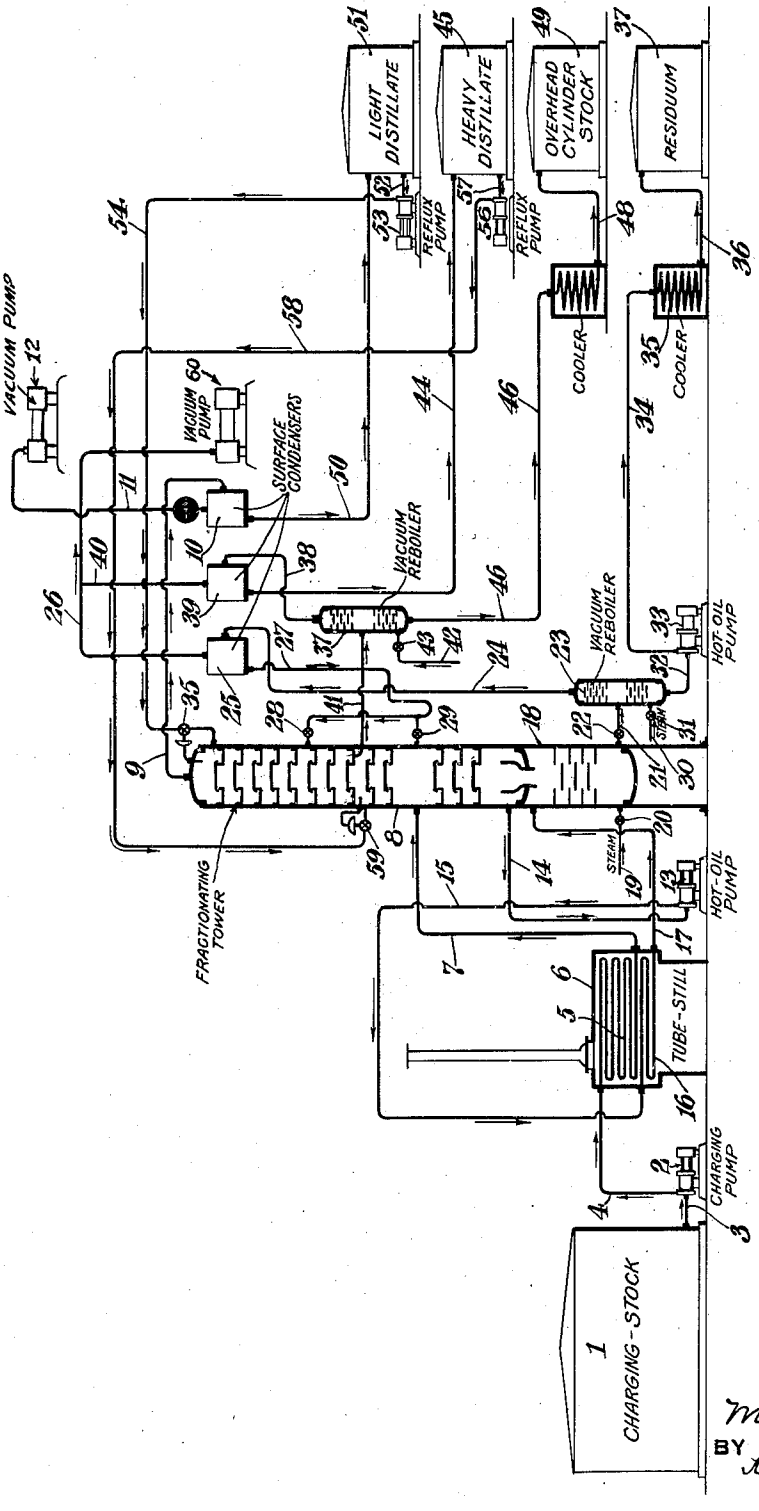

Fig. 1.

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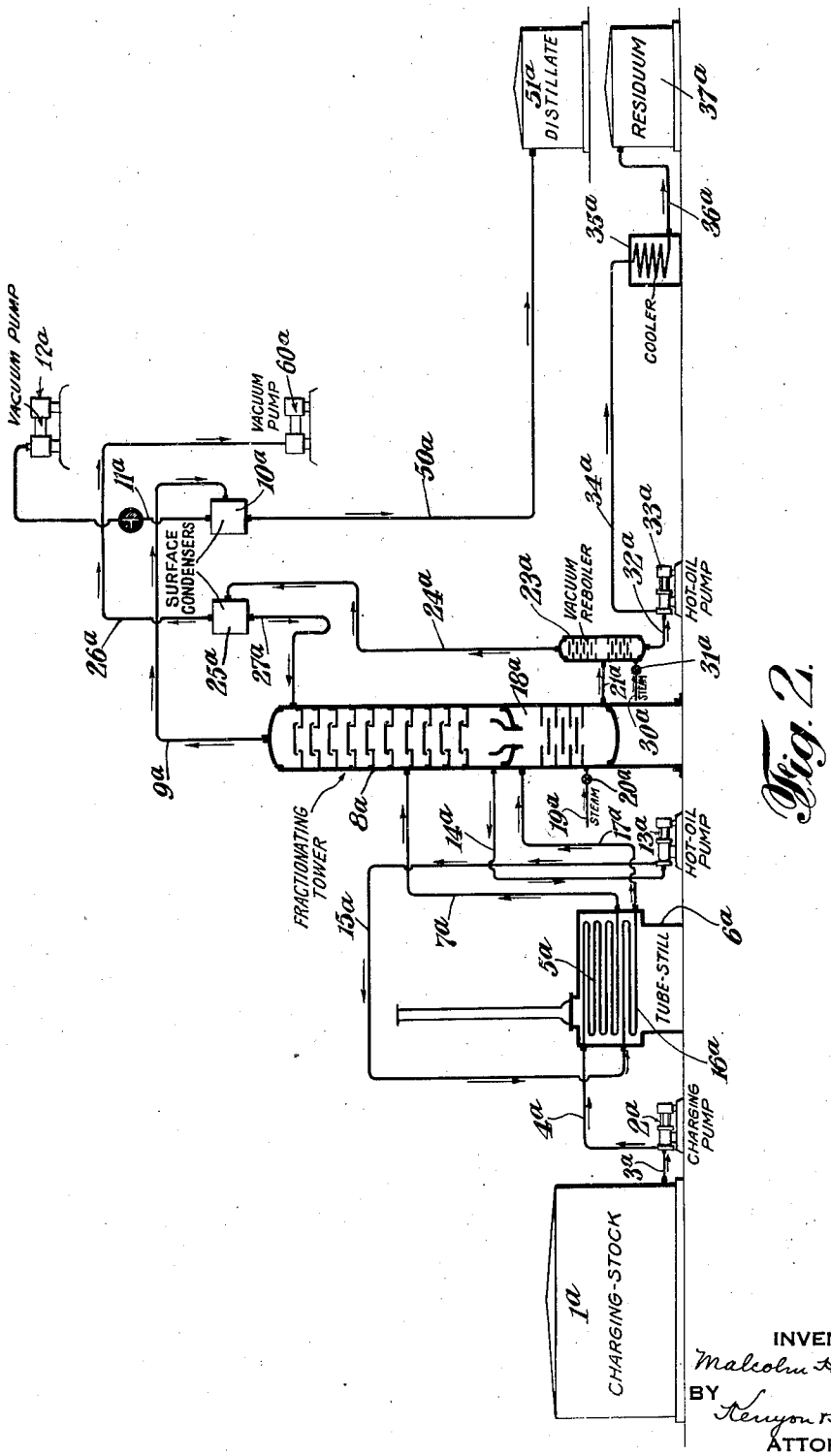

Fig. 2.

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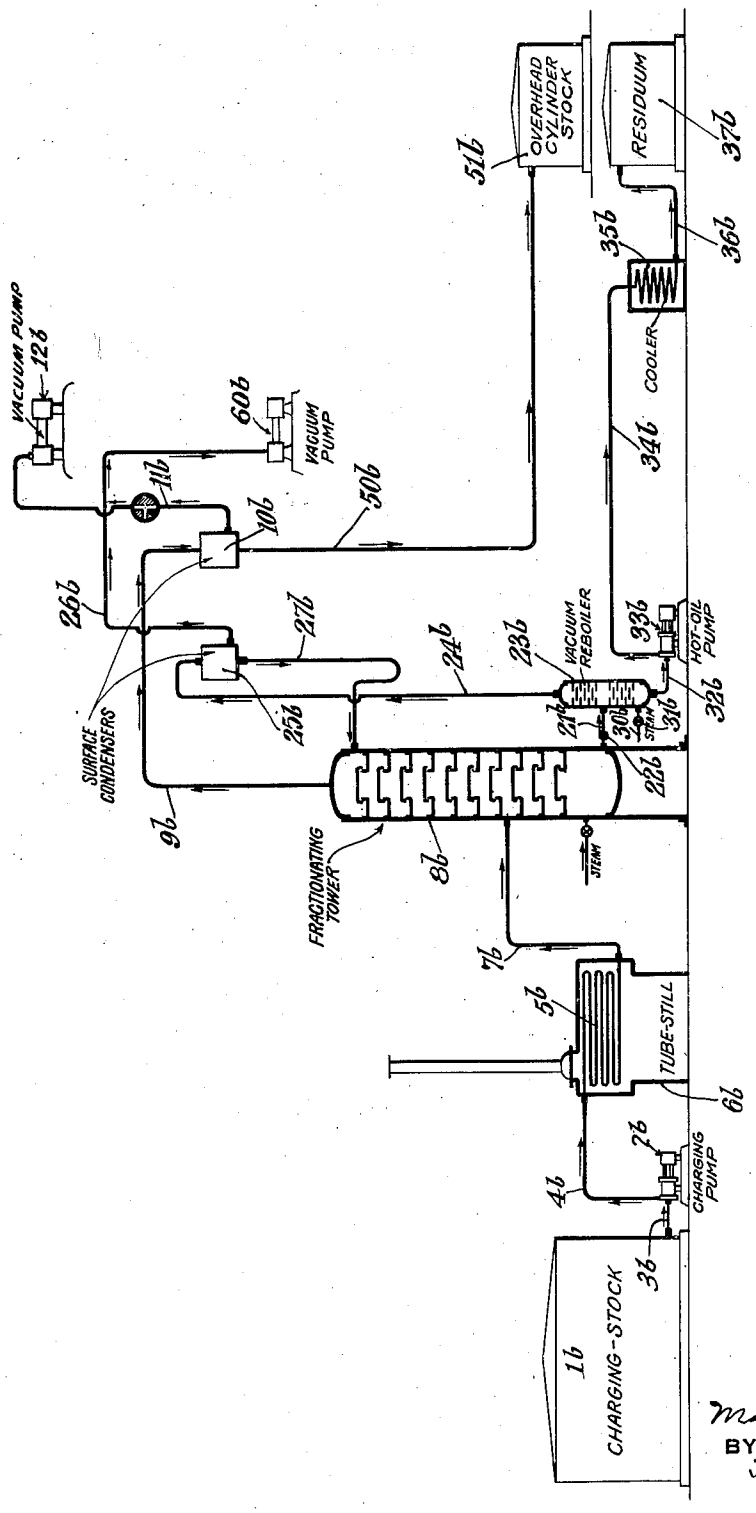

Fig. 3.

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DISTILLATION OF LUBRICATING STOCK

Application filed August 7, 1929. Serial No. 384,031.

This invention relates to the distillation of lubricating stock from paraffin or mixed base crude oil.

An object of this invention is an improved method of and apparatus for obtaining a large yield of overhead cylinder stock under conditions such that negligible decomposition occurs and the composition of the stock is controllable.

According to this invention, charging stock which may be paraffin or mixed base crude oil from which the low boiling compounds have been removed, is subjected to fractional distillation at low absolute pressure to separate the major portion of the lubricating stock and the unvaporized charging stock is subjected to reboiling at lower absolute pressure to effect further vaporization of lubricating stock which is condensed and utilized as reflux in the fractional distillation operation. The fractional distillation preferably is effected by means of successive heating operations and the utilization of a portion of the charging stock as recycle stock. Steam preferably is used to assist vaporization of the lubricating stock and preferably condensate occurring in an intermediate zone of the fractional distillation operation is subjected to re-distillation under reduced absolute pressure to separate the same into products of boiling points intermediate to those of the distillate from the fractionating operation and the unvaporized residuum.

In lubricating stock obtained according to this invention, the wax remains in amorphous form when chilled to −40° F. The fractions containing wax are separated from the charging stock in the first vaporizing operation at a temperature which is insufficient to crystalize the wax and only the fractions which contain substantially no wax are subjected to higher temperatures.

The distillation operation is carried on preferably at sub-atmospheric pressure and the temperatures to which the charging stock is subjected are such that negligible decomposition occurs. Fractionation is preferably effected by use of a bubble tower by which sharp fractionation is obtained, although a bubble tower is not in itself suited to low pressure distillation because of the pressure required to cause vapor to flow upwardly through the bubble decks, thus resulting in a higher pressure at the point of feed and points of removal of liquid fractions than is maintained at the point of removal of the vapors, thereby causing less vaporization than if the pressure throughout the tower were the same as at the point of vapor discharge. However, the subjection of the liquid fractions to vacuum reboiling and the use of the distillate thus obtained as reflux in the bubble tower results in the obtaining of maximum vaporization of the volatile constituents of such fractions and also in the obtaining of sharp fractionation. The pressure in the reboiler is the same, or lower than that maintained in the top of the bubble tower and this combination permits the final stripping from the charging stock of its lighter fractions at the lowest possible pressure. It also permits return to the bubble tower of liquid having a boiling point range low enough for fractionation in the bubble tower at the higher pressure.

Other objects, novel features and advantages of this invention will be apparent from the following specification and accompanying drawings, wherein:

Fig. 1 discloses an apparatus for effecting distillation in accordance with the invention.

Fig. 2 discloses a modified form of an apparatus, and

Fig. 3 discloses a further modification.

Referring now to Fig. 1, charging stock which may be paraffin or mixed base crude oil from which the low boiling compounds have previously been removed, is forced from the tank 1 by the pump 2 through the pipes 3 and 4 to the heating element 5 of a tube still 6 in which the stock is discharged through the pipe 7 into the bubble tower 8, at a temperature ranging from 600 to 700° F. Vaporization occurs within the tower 8 and the vapors pass upwardly through the bubble decks, while the non-vaporized portions pass downwardly through additional bubble decks to the base of the tower. The ascending vapors encounter reflux liquid supplied to the tower as will subsequently be described and the

fractionated vapor is conducted from the top of the tower through the pipe 9, the temperature of the vapor ranging from 300 to 400° F. The pipe 9 leads to a surface condenser 10, which is connected by pipe 11 with a vacuum pump 12. The vacuum pump 12 maintains the desired pressure in the bubble tower 8 and also removes the uncondensible gases formed in the distillation operation.

The non-vaporized portion of charging stock encounters ascending vapors supplied to the base of the bubble tower as will be subsequently described, these vapors being of higher temperature than the liquid stock. The liquid collecting in the base of the bubble tower is drawn by the pump 13 through the pipe 14 and is forced through the pipe 15 into the secondary heating element 16 of the tube still 6. From the tube still, the heated liquid is conducted by the pipe 17 and is discharged into the reboiling or vaporizing chamber 18 at a temperature between 675 and 775° F. The vaporizer 18 communicates with the bottom of the bubble tower 8 and the vapors produced in the vaporizer pass into the bubble tower and flow upwardly therethrough. These vapors volatilize lower boiling point constituents from the downwardly flowing liquid stream and the vapors thus formed pass into the upper section of the bubble tower where they are subjected to fractionation along with the vapors resulting from the first vaporizing operation. The condensate produced in the above operation collects in the bottom of the bubble tower 8 and is recycled through the secondary heating element 16. Steam may be introduced into the vaporizer 18 through the pipe 19 controlled by the valve 20 to decrease the partial pressure of the oil and thereby increase the amount of vaporization.

The unvaporized stock collects in the base of the vaporizer 18 and is conducted by pipe 21 controlled by valve 22 to a vacuum reboiler 23. The upper portion of the reboiler 23 is connected by a pipe 24 with a surface condenser 25, which in turn is connected by pipe 26 with the vacuum pump 60. A pipe 27 is provided to reflux condensate from the condenser 25 to the bubble tower 8 and has two points of connection with the tower, valves 28 and 29 being provided to control the flow into the tower. The unvaporized charging stock is supplied to the vacuum reboiler 23 at temperatures between 650 and 750° F. and the vapors are discharged through the pipe 24 at temperatures between 600 and 700° F. The temperature of the reflux condensate from the condenser 25 ranges from 400 to 500° F. Steam may be introduced into the reboiler 23 through the pipe 30 controlled by the valve 31 and the unvaporized stock in the reboiler is withdrawn through pipe 32 by the pump 33 and caused to flow through the pipe 34, cooler 35 and pipe 36 to the run-down tank 37.

A second vacuum reboiler 37 is connected through pipe 38, surface condenser 39 and pipe 40 with the vacuum pump 60. When reflux condensate from the condenser 25 is returned to the bubble tower through the valve 28, the heavier fractions of ascending vapor are condensed and removed through the pipe 41 to the reboiler 37 which is maintained at lower absolute pressure than exists in the bubble tower 8 at the inlet to the pipe 41. Steam may be admitted to the base of the vacuum reboiler 37 through a pipe 42 controlled by valve 43, thereby decreasing the partial pressure of the oil. The reduction of the partial pressure, together with the low absolute pressure maintained in the reboiler produces vaporization of the lighter fractions. Such vapors are conducted through the pipe 38 to the surface condenser 39 in which the non-condensible gases are removed and the liquid in the condenser is conducted by pipe 44 to the run-down tank 45. The unvaporized fractions are conducted from the vacuum reboiler through the pipe 46, cooler 47 and pipe 48 to the run-down tank 49. The temperature of the side-cut withdrawn from the bubble tower ranges from 400 to 500° F. and the vapor produced in the vacuum reboiler 37 ranges from 350 to 450° F. while the unvaporized fractions range from 350 to 450°F. The vacuum pump 12 removes the non-condensible gases from the bubble tower and also maintains the bubble tower under low absolute pressure. The pressure in the vacuum reboilers is maintained by the vacuum pump 60 as low at least as the pressure maintained at the top of the bubble tower, but at lower pressures than are maintained in the sections of the bubble tower with which they are connected.

The vapors conducted from the top of the bubble tower through the pipe 9 are condensed in the surface condenser 10 and the condensate is discharged through the pipe 50 into the run-down tank 51. The amount of liquid condensed in the bubble tower 8 and the degree of fractionation is controlled by returning reflux from the tank 51 to the top of the bubble tower. The reflux is drawn through the pipe 52 by the pump 53 and passed through the pipe 54 and temperature control valve 55 to the bubble tower.

Heavier liquid fractions may also be returned to the bubble tower by drawing liquid from the run-down tank 45 by the pump 56 through the pipe 57 and conveying such liquid through the pipe 58 and temperature control valve 59 at a point below the point of removal of the side fraction, thereby making possible to control the amount of the higher boiling point hydrocarbons in the side fractions.

With this form of apparatus, light dis-

tillate, heavy distillate and overhead cylinder stock are obtained. The conditions under which these products are obtained are such that negligible decomposition occurs and the composition of these products is controllable through the medium of the various reflux liquids. The successive heating operations make it possible to obtain fractions containing wax in which the wax remains in amorphous form when chilled to −40° F. The vapors produced by the second vaporization operation, by reason of their contact with the unvaporized remainder from the first vaporizing operation insure complete removal from the charging stock of the lower boiling constituents so that none of them are subjected to the second heating.

Referring to Fig. 2, charging stock is drawn from the tank 1*a* by the pump 2*a* through the pipe 3*a* and is conducted by the pipe 4*a* to the heating element 5*a* of the tube still 6*a* from which it is discharged by the pipe 7*a* into the bubble tower 8*a*. Vapor is conducted from the top of the bubble tower 8*a* by the pipe 9*a* to the surface condenser 10*a* which is connected by the pipe 11*a* with the vacuum pump 12*a*. Unvaporized charging stock collecting in the base of the bubble tower 8*a* is withdrawn by the pump 13*a* from the pipe 14*a* and supplied by the pipe 15*a* to the secondary heating element 16*a* of the tube still 6*a* and is discharged from the heating element through the pipe 17*a* into the vaporizer 18*a* which communicates with the base of the bubble tower 8*a*. A pipe 19*a* controlled by a valve 20*a* is provided for supplying steam to the evaporating chamber 18*a*. A pipe 21*a* leads from the base of the vaporizing chamber to a reboiler 23*a*, the top of which is connected by a pipe 24*a* with a surface condenser 25*a* which is connected by pipe 26*a* with the vacuum pump 60*a*. A pipe 27*a* leads from the surface condenser 25*a* to the top of the bubble tower 8*a* to provide reflux condensate. A pipe 50*a* leads from the surface condenser 10*a* to the run-down tank 51*a* in which the distillate is collected. A pipe 30*a* controlled by valve 31*a* is provided for supplying steam to the reboiler 23*a* to assist vaporization. The unvaporized oil in the reboiler 23*a* is withdrawn by the pipe 32*a* and is delivered by the pump 33*a* through the pipe 34*a* to the cooler 35*a* from which it is conducted by the pipe 36*a* to the tank 37*a*.

The operation of the system disclosed in Fig. 2 is generally similar to the operation of the system disclosed in Fig. 1 except that no side cut is taken and no reflux is used except that obtained from the surface condenser 25*a*. The unvaporized charging stock from the first vaporization operation is additionally heated to provide higher boiling point vapors which are discharged from the vaporizer 18*a* into the base of the bubble tower 8*a*. Likewise, the unvaporized stock from the second vaporization operation is subject to decreased pressure in the vacuum reboiler 23*a* to effect further vaporization and to supply reflux for use in the bubble tower 8*a*.

In the modification disclosed in Fig. 3, charging stock is drawn from the tank 1*b* by the pump 2*b* through the pipe 3*b* and is conducted by the pipe 4*b* to the heating element 5*b* of the tube still 6*b* from which it is discharged by the pipe 7*b* into the bubble tower 8*b* at a point approximately half way between the top and bottom of the tower. Vapor is conducted from the top of the bubble tower 8*b* by the pipe 9*b* to the surface condenser 10*b* which is connected by the pipe 11*b* with the vacuum pump 12*b*. Unvaporized charging stock collecting in the base of the bubble tower 8*b* is conducted through the pipe 21*b* controlled by the valve 22*b* to the vacuum reboiler 23*b*. A pipe 24*b* conducts vapor from the reboiler 23*b* to a surface condenser 25*b* which is connected by the pipe 26*b* with the vacuum pump 60*b*. A pipe 27*b* leads from the condenser 25*b* to the top of the bubble tower 8*b* to provide reflux condensate therefor. The pipe 50*b* leads from the condenser 10*b* to the run-down tank 51*b* in which the distillate is collected. A pipe 30*b* controlled by the valve 31*b* is provided for supplying steam to the reboiler 23*b* to assist vaporization. The unvaporized oil in the reboiler 23*b* is withdrawn by the pipe 32*b* and is delivered by the pump 33*b* through the pipe 34*b* to the cooler 35*b* from which it is conducted by the pipe 36*b* to the tank 37*b*.

In this apparatus, the residual oil from the vaporization operation is reboiled at lower absolute pressure to effect further vaporization and supply reflux for use in the bubble tower 8*b*. The pressure in the top of the bubble tower may be the same as that in the vacuum reboiler, but the pressure on the liquid stock in the vacuum reboiler is less than the pressure to which liquid in the base of the bubble tower is subjected.

A three-way valve is provided in each of the pipes 11, 11*a*, and 11*b* by means of which the bubble towers may be connected to the vacuum pumps, 12, 12*a* and 12*b*, respectively or may be put in communication with the atmosphere. This makes it possible to carry out the distillation either under sub-atmospheric or atmospheric pressure as is desired.

The absolute pressure in both the bubble tower and the reboilers may be varied to suit the lubricating stock handled and the desired viscosity of the lubricating distillate. Some lubricating stocks produce distillates which are difficult to dewax and in such cases it is advisable to use very low absolute pressures. With such stocks, 40 millimeters of mercury absolute pressure at the top of the bubble tower and from 2 to 10 millimeters of mercury absolute pressure at the re-boilers has been found satisfactory. While lower pres-

sure than 40 millimeters may be used at the top of the bubble tower, such pressure is not advisable because of the difficulty in condensing steam under such pressures. With a pressure of 40 millimeters absolute at the top of the bubble tower, steam used in the tower to assist in vaporization is condensed in the surface condenser 10 so that the vacuum pump 12 need handle only the non-condensable gases. With some lubricating stocks, it is possible to operate the bubble tower above 40 millimeters absolute and in some cases the bubble tower may be operated as high as atmospheric pressure. In such cases, the reboilers may be operated at absolute pressures of from 10 to 40 millimeters.

It is, of course, understood that other forms of apparatus may be used to carry out the process described and that various structural changes may be made in the apparatus disclosed without in any way departing from the spirit of the invention as defined in the appended claims.

I claim:

1. The method which comprises subjecting lubricating oil stock to fractional distillation at low absolute pressure, reboiling the unvaporized stock at lower absolute pressure, condensing the vapor from said reboiling operation, and utilizing the condensate thus obtained as reflux for the fractional distillation operation.

2. The method which comprises subjecting lubricating oil stock to fractional distillation at low absolute pressure, additionally heating the unvaporized portion of the lubricating oil stock and supplying the vapor thus obtained to the fractional distillation operation, reboiling the liquid remainder at lower absolute pressure, condensing the vapor from said reboiling operation, and utilizing the condensate thus obtained as reflux for the fractional distillation operation.

3. The method which comprises subjecting lubricating oil stock to fractional distillation at low pressure by successive heating operations and with recirculation of a portion of the stock during the second heating operation, reboiling the unvaporized stock at lower absolute pressure, condensing vapor from said reboiling operation and utilizing the condensate thus obtained as reflux for the fractional distillation operation.

4. The method which comprises effecting fractional distillation at low absolute pressure of lubricating oil stock by two successive heating operations and recirculation of a portion of the stock during the second heating operation, reboiling the unvaporized stock at lower absolute pressure, condensing vapor from said reboiling operation, utilizing the condensate thus obtained as reflux for the fractional distillation operation, subjecting a fraction produced in the fractional distillation operation to reboiling, and separately collecting the condensate and the unvaporized portion.

5. In a distillation system, a fractionator, means for supplying heated lubricating oil stock to said fractionator at an intermediate point, means for maintaining low absolute pressure in said fractionator, a reboiler, means to conduct liquid from said fractionator to said reboiler, means for maintaining the pressure in said reboiler at least as low as the lowest pressure in the fractionator, means for condensing vapor from said reboiler, and means for refluxing to said fractionator condensate thus obtained.

6. In a distillation system, a fractionator, a heater, means for supplying lubricating oil stock through said heater to said fractionator at on intermediate point, means for maintaining low absolute pressure in said fractionator, a reboiler, means to conduct liquid from said fractionator to said reboiler, means for maintaining the pressure in said reboiler at least as low as the lowest pressure in the fractionator, means for condensing vapor from said reboiler, means for refluxing to said fractionator condensate thus obtained, a vaporizer communicating with the lower portion of said fractionator, a second heater, and means for conducting liquid from said fractionator through said second heater to said vaporizer.

7. The method which comprises heating lubricating oil stock to effect vaporization of low boiling fractions, fractionating the vapor thus obtained under low absolute pressure, reboiling the unvaporized fractions and condensate from the fractionating operation at higher temperature, subjecting the vapor thus obtained to fractionation with the lower boiling vapors, reboiling the residual liquid at lower absolute pressure, condensing the vapor thus obtained, and utilizing the same as reflux in the fractionating operation.

8. The method which comprises heating lubricating oil stock to effect vaporization of low boiling fractions, fractionating the vapor thus obtained, reboiling the unvaporized fractions and condensate from the fractionating operation at higher temperature, subjecting the vapor thus obtained to intimate contact with said liquid fractions and condensate prior to reboiling and to subsequent fractionation with said low boiling vapors, reboiling the residual liquid at low absolute pressure, condensing the vapor thus obtained, and utilizing the same as reflux in the fractionating operation.

9. In combination, a heater, a fractionating column, a conduit leading from said heater to said fractionating column, a reboiler, a conduit leading from the fractionating column to the reboiler, means for maintaining an absolute pressure in the reboiler lower than the lowest pressure existing in the fractionating column, and means for con-

densing the vapors produced by the reboiler and returning the same to the fractionating column as reflux.

10. In combination, a heater, a fractionating column, means including said heater to deliver lubricating oil stock to the fractionating column at a temperature sufficient to cause vaporization, a plurality of reboilers, means for heating the residual oil from the frictionating column and charging it to the first reboiler, means for maintaining low absolute pressure on the fractionating column and said first reboiler, means for returning to the base of the fractionating column vapors produced in the first reboiler, means for discharging residual oil from the first reboiler to the second reboiler, means for maintaining lower absolute pressure in said second reboiler than in said first reboiler, means for removing and condensing vapors produced in the second reboiler, and means for returning such condensate to the fractionating column as reflux.

11. In combination, a heater, a fractionating column, a conduit leading from said heater to said fractionating column, a reboiler, means for conducting liquid fractions from the fractionating column to the reboiler, means for maintaining the pressure in said reboiler lower than the lowest pressure in the fractionating column, means for condensing the vapors produced in the reboiler, and means for returning the same to the fractionating column as reflux.

12. In a system for the distillation of lubricating oil stock a, heater, a fractionating column, a conduit leading from said heater to said fractionating column, a reboiler, means for conducting liquid from the fractionating column to the reboiler, condensers connected to said fractionating column and reboiler, means for maintaining the same absolute pressure at the outlet of each condenser, and means for returning condensed vapor as reflux to the fractionating column from one or both of said condensers.

In testimony whereof I have signed my name to this specification.

MALCOLM H. TUTTLE.