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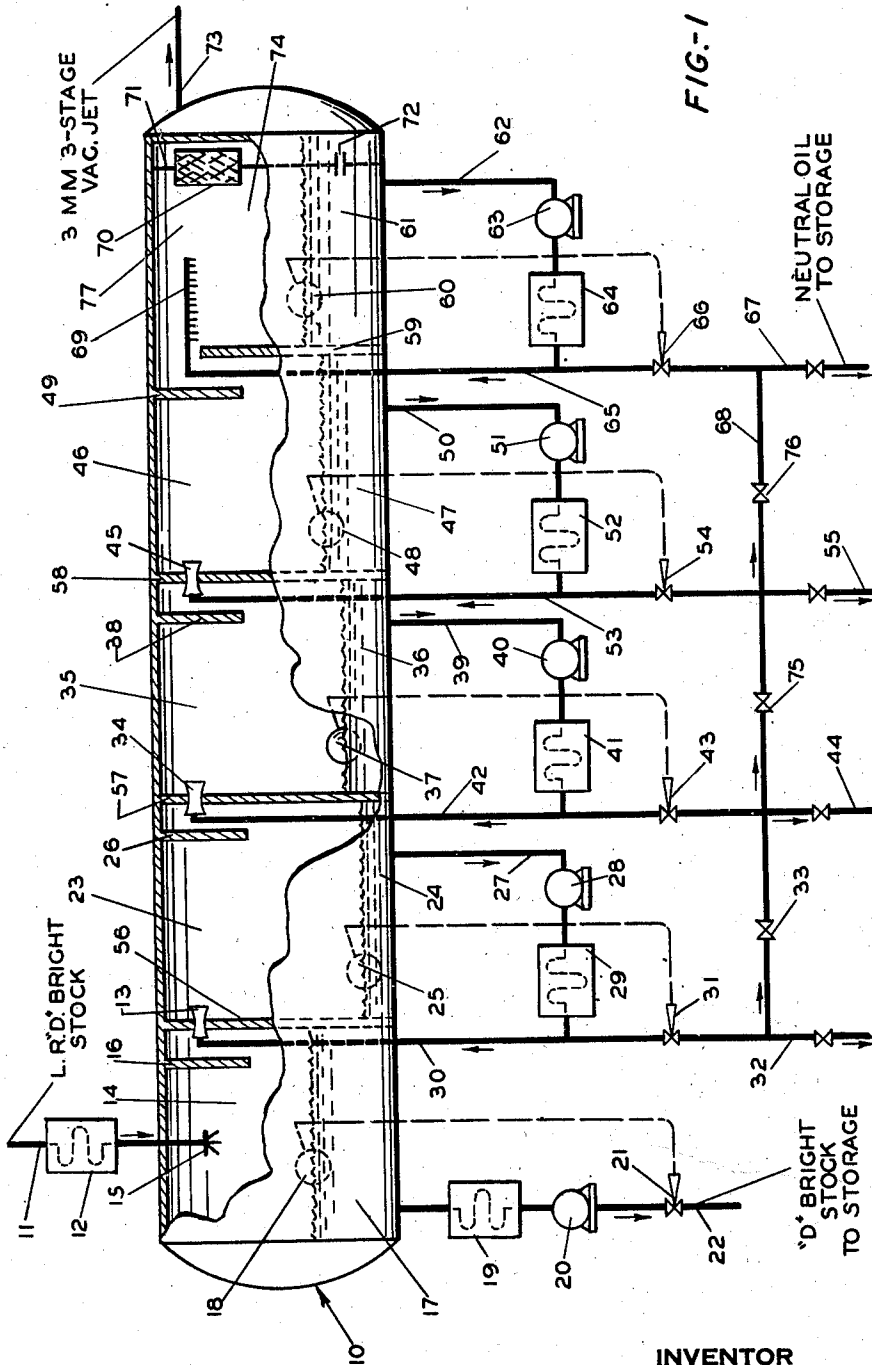
P. M. WADDILL

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REFINING OF LUBRICATING OILS

Filed Jan. 3, 1944

2 Sheets-Sheet 1



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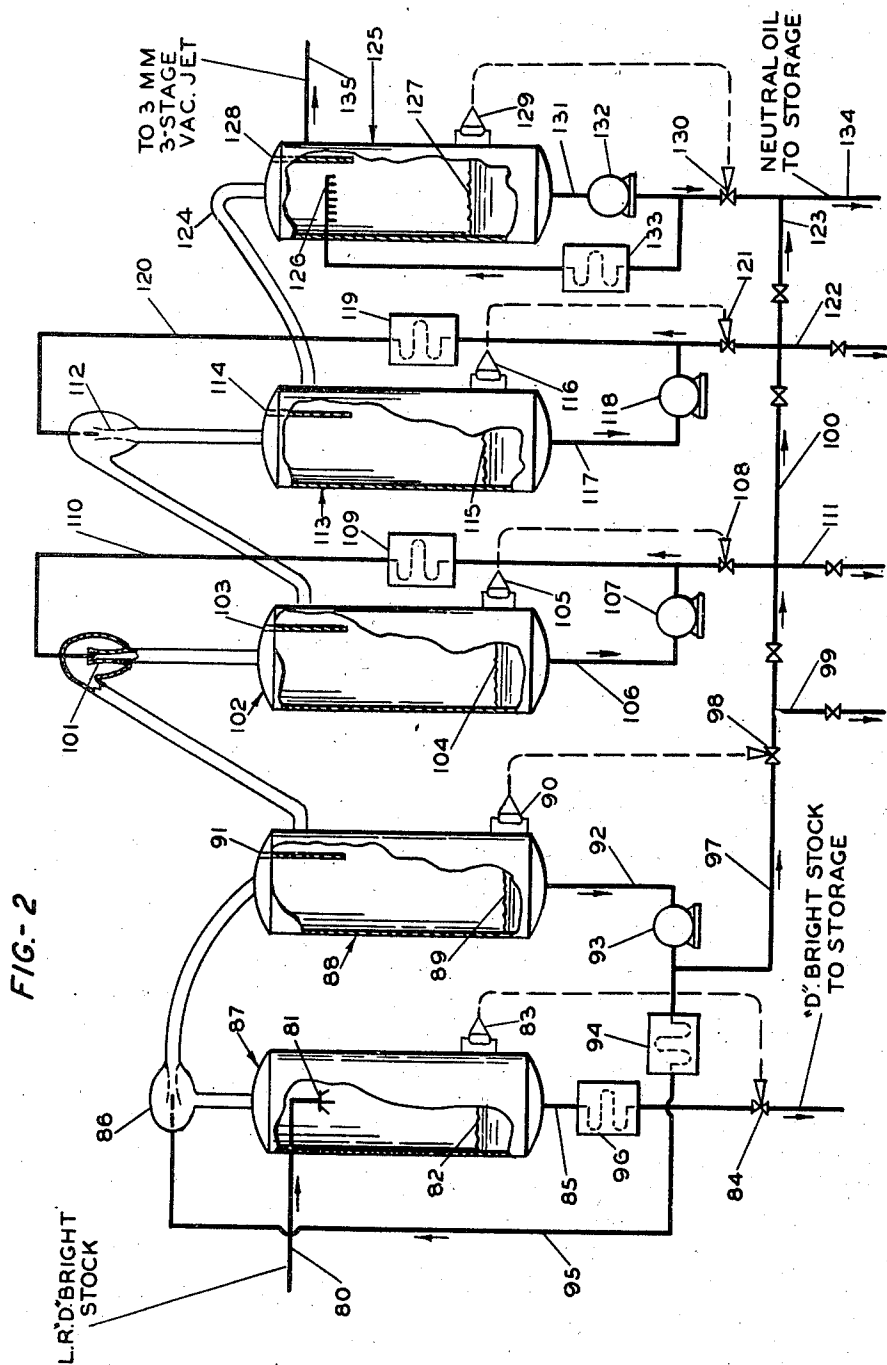
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REFINING OF LUBRICATING OILS

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3 Claims. (Cl. 196—77)

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This invention relates to a method for distilling oils and more particularly it relates to a method for the vacuum distillation of lubricating oils.

The conventional rectification or fractionation tower, in which vapor is fractionated by repeated countercurrent contacts with liquid reflux, usually imposes an appreciable back pressure on the source of the vapor. In order to maintain constant or uniform flow through a given fractionator a certain minimum inlet pressure must be maintained. This pressure, of course, depends upon many factors, such as the number of trays, the tower throughput, outlet pressure, and others. Appreciable reflux liquid is carried on each tray and a certain pressure frequently as high as one half pound or even more per square inch per tray is consumed in forcing the upward flow of the vapors being fractionated. Thus a 30 tray tower may consume approximately 1 atmosphere of pressure, and in view of this consideration, one atmosphere of vacuum on a fractionator outlet will be fully dissipated at the fractionator inlet. Thus, the futility of bubble tray fractionation in high vacuum systems is obvious.

Lubricating oil types of hydrocarbons have very high boiling points, so high, in fact, that such oils frequently cannot be distilled without decomposition. By the use of steam, the distillation temperature of some lubricating oils can be lowered sufficiently to avoid thermal decomposition. Large amounts of steam are required in such distillation hence steam costs are high. In addition, the steam distilled oils must be separated from the condensed steam and dried before marketing. Thus, vacuum distillation in some form offers many advantages over steam methods. Some refiners use combined steam-vacuum processes, and such methods offer definite advantage over the use of steam alone.

I have found a method of heavy oil distillation wherein vacuum alone is used, and my process has many advantages over processes in which steam is used.

One object of my invention is to provide a lubricating oil distillation method.

Another object of my invention is to provide a method of lubricating oil distillation by vacuum and in the absence of steam.

Still another object of my invention is to provide a process for the fractionation of lubricating

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oil stocks by vaporization under high vacuum in the absence of steam followed by fractional condensation of the vapors.

Yet another object of my invention is to provide a process for the separation of neutral oils from bright stocks by vacuum distillation wherein the color and color stability are markedly improved, which advantage is reflected in considerable saving in decolorizing clay costs.

Still other objects and advantages will be apparent to those skilled in the art from a careful study of the following disclosure, which in conjunction with the attached drawing, forms a part of this specification.

Figure 1 shows, diagrammatically, one form of apparatus in which the process of my invention may be practiced, and

Figure 2 shows, diagrammatically, another form of apparatus in which the process of my invention may be carried out.

Ordinary, commercially available vacuum producing steam jets do not give sufficient vacuum for best lubricating oil distillation. I have found that neutral oils condensed in the chambers of my apparatus make excellent circulating media, and when used with properly designed vacuum producing jets, very low vacua can be produced. In a preferred embodiment of my invention, I use the conventional 3-stage steam jet to obtain a vacuum of approximately 757 millimeters of mercury, that is, a residual absolute pressure of 3 millimeters of mercury, and supplement this with a series of properly designed oil operated jets wherein pressures of $\frac{1}{10}$ millimeter of mercury or lower can be obtained. I have found that by using pressures as low as $\frac{1}{10}$ to $\frac{1}{100}$ millimeter that neutral oil constituents can be distilled from bright stocks at a temperature sufficiently low as to avoid much of the thermal decomposition or color formation usually obtained when the distillation is obtained at higher pressures.

My process may be better understood by reference to Figure 1 in which numeral 10 represents a, preferably horizontally arranged, vacuum still apparatus. This cylindrical vessel may, however, be set upright, with the chamber or compartment having the greatest vacuum being disposed at the top of the vessel or at its bottom, as desired. This horizontally disposed vessel 10 may be divided into five compartments, as repre-

sented by numerals 14, 23, 35, 46 and 74, by partitions 56, 57, 58 and 59. Partitions 56, 57 and 58 carry Venturi jets 13, 34 and 45, respectively. These jets are the above referred to jets, which are operated by condensed oils. Each compartment is equipped with a liquid level controller, as represented by numerals 18, 25, 37, 48 and 60, which are intended to control the amount of liquid in the respective chambers, the liquid in the said chambers being identified by numerals 17, 24, 36, 47 and 61.

In the operation of the process, when using the apparatus represented in Figure 1, long residuum D bright stock from a bright stock still, not shown, passes through line 11, a heat transfer means 12 in which the oil stock is heated or cooled, as required, through a spray nozzle 15 into the compartment 14, which is the first of the series. A final vapor outlet 73 is connected to a 3-stage steam operated vacuum producing jet, not shown. This jet produces a vacuum of the order of 3 mm. absolute pressure which is communicated to the final chambers 74 and 46. The oil jet 45 operating to reduce the pressure in compartment 35 to approximately 1 mm. residual pressure discharges into the compartment 46. Similarly, oil jet 34, takes suction on compartment 23 and reduces the pressure therein to about $\frac{1}{2}$ mm., while the oil jet 13 reduces the pressure in compartment 14 to the very low pressure of about $\frac{1}{16}$ millimeter of mercury. The above mentioned bright stock feed at a temperature of approximately 500° to 700° F. is fed into said compartment 14 through spray nozzle 15. The oil at the temperature of 500° to 700° upon being sprayed into the high vacuum chamber 14 undergoes considerable vaporization, the lower boiling hydrocarbon constituents being vaporized. These lower boiling hydrocarbon constituents are the type of oily compounds normally contained in the so-called "neutral oils." While the "neutral oil" hydrocarbons vaporize under these conditions, the higher molecular weight, higher boiling hydrocarbons remain as a liquid and settle to the bottom of this chamber and remain as liquid 17. This liquid, termed "D" bright stock, is discharged through a line 22, cooled in a cooler 19, the flow being controlled by a pump 20 and a valve 21, the operation of the latter being controlled by the liquid level controller 18. The stock passes from line 22 to a storage vessel, not shown. This stock is of good color, good color stability and of generally excellent quality, since the oil has been maintained at a relatively low distillation temperature for a very short period of time.

Since the charge oil is sprayed by nozzle 15 at a high velocity a splash plate or baffle 16 is necessary to prevent liquid from being carried by oil jet 13 into the next compartment in the series.

As stated hereinbefore, the pressure maintained in compartment 14 is about $\frac{1}{16}$ millimeter of mercury, and the pressure in compartment 23 is about $\frac{1}{2}$ mm. This latter pressure is higher than the $\frac{1}{16}$ millimeter, and amounts to compression, thus the highest boiling constituents of the vapors entering compartment 23 are condensed by compression and temperature reduction since temperature therein is maintained at about 400° to 600° F. Condensate formed therein settles to form the liquid 24. This liquid is withdrawn by a pump 28 through a line 27, a cooler 29 and passes through a line 30 to operate the said oil jet 13 which assists in evacuation of chamber 14. The oil with the entrained vapors issuing from the Venturi-jet assembly 13 discharge into the

compartment 23, the circulating liquid oil settling as liquid along with additional condensate oil. A baffle plate 26 prevents the high velocity discharge from jet 13 from interfering with the operation of jet assembly 34. Excess liquid condensate not needed for the effective operation of Venturi-jet 13 operates the liquid level controller device 25 which in turn opens an outlet valve 31 and permits removal of condensate through a line 32 to individual condensate storage, not shown, or through a manifold line 58 containing valves 33, 75 and 76 and a line 67 to a composite neutral oil storage, not shown.

The oil vapors not condensed by compression from $\frac{1}{16}$ mm. to $\frac{1}{2}$ mm. in chamber 23 are further compressed to about 1 mm. pressure on passage through Venturi jet 34 into compartment 35. Compartment 35 operates in a manner similar to that of compartment 23, that is, by an increase of pressure and a decrease of temperature to, say, about 300° to 400° F. Condensate oil passes through a line 39 and is pumped by a pump 40 through an exchanger 41, a line 42 to the nozzle or jet of venturi 34. A baffle 38 prevents liquid oil being carried into the next successive chamber 46. Excess condensate oil not needed for the operation of the venturi 34 and accumulating in chamber 35 is removed therefrom through the operation of the liquid level controller 37 which actuates a valve 43, as the level of the oil 36 rises, the float 37 rises also and operates to open valve 43, and conversely when oil 36 has been pumped out to a predetermined low level the float 37 operates to close valve 43. The oil so removed may pass through a line 44 to an individual storage, not shown, or may pass through the composite neutral oil header line 68 containing valves 75 and 76 and line 67 to composite neutral oil storage, not shown.

In a similar manner compartment 46 operates. The pressure is raised therein to about 3 mm. mercury pressure by Venturi jet 45, while the temperature is lowered to from approximately 150° to 250° F., and the condensate thus produced is used for the operation of the jet by passage through a line 50, a pump 51, an exchanger 52 and a line 53 to the jet. Excess oil accumulating in this chamber is removed by the operation of liquid level controller mechanism 48 and an outlet valve 54. Excess condensate oil may pass to individual storage, not shown, through a line 55 or through a line 68, valve 76 and line 67 to composite neutral oil storage, not shown. A splash plate 49 prevents liquid oil from jet 45 from passing into the final spray compartment 74.

Oil vapors, uncondensed in compartment 46, pass between baffle plate 49 and partition plate 59 into spray compartment 74 in which most or substantially all of the remaining oil vapors are removed by absorption in oil issuing from sprays 69. Excess oil accumulating in this scrubbing chamber is removed through the operation of a valve 66, which is actuated by the float controller mechanism 60, the oil passing through a line 62, a pump 63, a heat exchanger 64, the said valve 66 and line 67 neutral oil storage, not shown, while the main volume of the oil is continuously circulated through line 62 and a line 65 which latter leads directly to the scrubbing sprays.

The temperature of oil spray 69 is adjusted by heat exchanger 64 to cause condensation of the desired neutral oil in chamber 77 and yet permit the undesired water vapor and light hy-

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hydrocarbon vapor traces to be ejected through line 73.

A partition plate 71 contains a mist remover 70 for removal of oil mist which fails to settle in chamber 74. A small opening 72 permits liquid accumulating in a small chamber 77 free access to the spray chamber from which it may be removed as explained hereinbefore.

In summarizing the above detailed operation it might be mentioned that in normal operation the amount of bright stock oil remaining in the first of the sequence of chambers is relatively large, while the several intermediate fractions accumulating in chambers 23, 35 and 46 are relatively small in volume. The output from the scrubbing compartment constitutes by far the major portion of the neutral oil production.

The several heat exchange means mentioned above may be used as heaters, or coolers, or some as heaters and the remaining as coolers, all depending upon the particular operation desired. For example, exchanger 12 may operate as a heater to impart to the feed stock sufficient sensible heat to evaporate all of a desired portion of the oil on passage through sprayer 15 into the vacuum compartment 14. In case the said feed stock comes from a still tower or other apparatus employing a relatively high temperature, the temperature of the oil may be such that heater 12 may not be needed, in which case it may be bypassed or said apparatus may serve to cool the feed oil in case temperature is higher than desired. Similarly, the several heat exchange means 29, 41, 52 and 64 may be operated in a manner as dictated by the product desired from each of the respective chambers. These heat exchange means are, of course, operated in cooperation with the degree of vacuum in said respective chambers, as will be understood by those skilled in the vacuum distillation art. For example, a light neutral oil may be produced in the scrubber chamber, while a neutral oil having a slightly greater viscosity will come from the next successive chamber 46. A still more viscous oil will come from chamber 35 while the most viscous of the oils vaporized in vaporizing chamber 14 will be produced in chamber 23. The relatively heavy bright stock will be removed from the vaporizer chamber 14. The apparatus may be so operated as to produce oils having substantially S. A. E. viscosities, for example, neutral oil being produced in spray chamber 74, S. A. E. 10 in chamber 46, S. A. E. 20 in chamber 35, and S. A. E. 30 in chamber 23. My process, as mentioned, is variable in nature, and with the proper design and construction of the apparatus many products of good quality can be made. It might be mentioned that the drawings are merely diagrammatic in form, as for example, the size and number of chambers. The chambers even need not be the same size, but may be made in approximate proportion to relative amount of each fraction to be removed. The amount or volume of oil carried in the sump portion of each chamber may be at least somewhat dependent upon the rate of recycle through the venturi discharging into the chamber.

Conditions of operation will obviously be different for processing different oil stocks, for example, the processing of a Pennsylvania bright stock oil will require greater vacuum and/or higher temperatures than will a Mid-Continent stock, and the latter will require more severe conditions than will a Coastal oil stock.

The apparatus shown in Figure 2 of the draw-

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ing may be used in the operation of my process in substantially the same manner of the apparatus illustrated in Figure 1. The primary difference between the two types of equipment is that according to Figure 2 the vacuum chambers are separate and independent vessels while in Figure 1 the chambers are portions or compartments of one large vessel.

In the operation of the process when using the apparatus illustrated in Figure 2, the long residuum bright stock enters the apparatus by way of a line 80 having been previously heated in a still or heater, not shown. At the proper temperature the charge oil enters a vessel 87 through a spray nozzle 81. Sufficient sensible heat contained in the oil causes the desired amount of distillation as the oil is sprayed into the vacuum of said vessel 87, for example, $\frac{1}{9}$ millimeter of mercury absolute pressure. The residual, unevaporated oil accumulates in the bottom of said distillation vessel, the level of which oil is represented by numeral 82, and is controlled by a liquid level control apparatus 83 which operates to open and close a valve 84 for discharge of the accumulated oil 82, for example, a "D" bright stock oil, as mentioned hereinbefore. This oil is preferably cooled in a cooler 96 on passage through a line 85 on its way to storage, not shown.

The distilled oil vapors originating in vessel 87 are removed therefrom by passage through a Venturi jet 86. This jet maintains the above mentioned $\frac{1}{9}$ mm. pressure in vessel 87 by discharge into a next successive vessel 88 in which a total pressure of about $\frac{1}{3}$ mm. may exist. At this greater pressure some condensation of vapors occurs yielding a condensed, liquid, neutral type oil, represented by numeral 89. This condensate oil is withdrawn through a line 92 by a pump 93, heated or cooled as required by a means 94 and transferred through a line 95 to Venturi jet 86 for its operation. The excess liquid accumulated in vessel 88 and not needed for the operation of venturi 86 is removed through line 92, a manifold line 97 through a valve 98, the operation of which is actuated by a float controller device 90 disposed in vessel 88. This excess oil or "make" may be removed from the system through an individual product line 99 or passed on through manifold lines 100, 123 and 134 to storage, not shown. A baffle plate 91 prevents liquid passing from vessel 88 through the vapor line to the next successive vacuum jet.

A next vessel, in sequence, 102, is operated by a venturi 101 maintaining a pressure of approximately 1 mm. which pressure is a threefold increase and causes condensation of another fraction of neutral oil from the vapors from the previous vessel 88. Condensate 104 accumulates, a portion being recirculated through a line 106, by a pump 107, and through a line 110 for the operation of the vacuum jet 101. The recirculated oil may be heated or cooled in an exchanger 109, as desired. The oil not needed for operation of the said jet may be withdrawn from the system through a neutral oil line 111, the flow being controlled by a float controller 105 and a valve 108; or may be passed through the manifold line 100, lines 123 and 134 to composite neutral oil storage, not shown, as desired. A baffle plate 103 prevents exit of liquid oil through the vapor line to the next successive vessel.

The vapors issuing from vessel 102 pass through another venturi 112 and are discharged therefrom into a vacuum vessel 113 at a higher pressure of

about 3 mm. and at this pressure additional liquid oil is condensed. This oil identified by numeral 115 is used for vacuum producing purposes by circulation through a line 117, by a pump 118, through a heat exchanger 119 and on through a line 120 to the jet 112. Excess oil not needed for vacuum producing purposes passes from line 117 through a valve 121 and a neutral oil line 122 to individual storage, not shown, or may be passed through valve 121, lines 123 and 134 to composite storage, also not shown. A float controller device 116 operates to control the opening and closing of valve 121. A splash plate 114 prevents liquid passing from vessel 113 through a vapor line 124 to a next successive vessel 125.

In the final vessel 125 is mounted a liquid spray device 126 for extracting oily constituents from the residual vapors entering from vapor line 124. The absorption oil or washing oil as it may be termed, is the extracted oil itself, the washing cycle being passage of the oil represented by numeral 127 through a line 131 by a pump 132, through an exchanger 133 on to the spray nozzles 126. The oil not needed for circulating is withdrawn from the vessel by operation of a float controller 129 which actuates a valve 130 permitting the said oil to pass through line 131, pump 132 and line 134 to a neutral oil storage which is not shown in the drawing. A baffle plate 128 prevents passage of mist, etc., through a final suction line 135 to a primary 3-stage steam vacuum jet apparatus, not shown.

The distillation temperatures held within the several vacuum chambers of Figure 2 may be substantially those given in connection with the discussion of Figure 1, or may be varied to meet the conditions presented by a given oil stock.

This second apparatus embodiment, as mentioned hereinbefore, operates substantially as does the apparatus of Figure 1. Exact operating temperatures and pressures will be determined at least in part by the type of feed stock to be processed, the grade of oils to be evaporated and subsequently condensed in each stage and other factors too numerous to mention, all of which are readily understood by one versed in the vacuum distillation art.

The oils produced herein are of good color and good color stability on account of the relatively low distillation temperatures possible and the low distillation temperatures are possible because of the use of the oil operated vacuum jets. The production of the oils with good color characteristics markedly decreases clay decolorizing costs, and these costs in conventional high temperature operations are high.

It might also be mentioned that the process steps may be easily operated and controlled since each vacuum jet operates independently of the remaining jets, each being operated according to the oil stock in process and the product desired.

By the use of my herein described vacuum distillation process, the residual viscous oil and the relatively less viscous condensate oils are relatively "bright" as regards moisture. Commercial practice, however, would probably dictate the installation of clay treating for best continuous operation. Conventional oil distillation operations usually present high clay treating costs, and since such costs are substantial, any appreciable reduction therein will be economically valuable and herein is one important advantage of my process.

For purposes of simplicity many valves, automatic controlling devices, etc., have been omit-

ted from the figures and from the descriptions. This apparatus is of standard design and readily obtainable from manufacturers, and the operation of which is understood by those skilled in the oil refining art.

This second embodiment of apparatus (Figure 2) may be used to produce a "D" bright stock and a series of neutral oils, or a bright stock and series of oils of standard S. A. E. viscosities, as well as is the apparatus of Figure 1. All product streams from either apparatus will be "bright" since they are produced under high vacuum and low temperatures such as 500°-700° F. for compartments 14 and 87 of Figures 1 and 2, respectively, temperatures of 400°-600° F. for the next successive compartment in each figure, while the third compartment of each may operate at temperatures as low as 300°-500° F. The final "washing" compartments operate at about 150°-250° F. It will be readily appreciated by those skilled in the art that these said temperatures are exceptionally low for lubricating oil distillation operations.

My process is not intended to be limited to the distillation of lubricating oil but may be applied to the distillation of any oil or oil stock wherein vacuum or vacuum and steam distillation are advantageous.

I claim:

1. A process for the subatmospheric distillation of lubricating oil stocks, comprising discharging the lubricating oil stock in a heated condition into the first of a series of serially connected containers the interior of which is maintained at sub-atmospheric pressure to vaporize a portion thereof, the liquid residue therein forming one product of the process, discharging the vapor portion into the next container to condense a portion thereof, causing discharge of the vapor into the second container by aspirating it by a portion of the condensate of that container, progressively introducing the vapor fraction in each container into the succeeding container by aspiration with a part of the liquid portion of the succeeding container, and withdrawing the liquid portions from each container as products of the process, the vapor portion discharged into each container being discharged into the space thereof above the collected liquid condensate therein.

2. A distillation apparatus of the type described comprising a plurality of containers forming isolated spaces, injector connections for connecting said containers in series, means for maintaining an all over subatmospheric condition in said spaces, means for discharging a heated lubricating oil stock into the first of said spaces and connections from each of several of said spaces to the injector discharging thereinto for delivering a portion of the condensate of the space to the injector, each injector discharging the vapors of one associated space into the other associated space at a higher pressure, and means for withdrawing the condensates injected in each container therefrom as products of the process, said injectors discharging into the spaces above the condensate bodies collected therein.

3. An apparatus of the character described comprising in combination a series of at least three chambers, said chambers being adapted to contain liquids and vapors, each of said chambers having a liquid outlet, means connecting said chambers in series for vapor transfer consisting of liquid actuated vapor injectors, each of said injectors providing a vapor outlet for the first of said chambers and a vapor inlet for the

second of said chambers to which it is connected, each of said injectors discharging into the vapor space of the associated second chamber, means to supply injector liquid under pressure directly from the second of said chambers connected together by said vapor injector to actuate each of said vapor injectors, whereby there is no entrainment of liquid from the first of said chambers and said injector liquid returns directly to the chamber from which it came.

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