Oct. 8, 1940. R. V. BECKNELL 2,217,356

MINERAL OIL DISTILLATION

Filed May 3, 1937 3 Sheets-Sheet 1

Fig. 1.

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MINERAL OIL DISTILLATION

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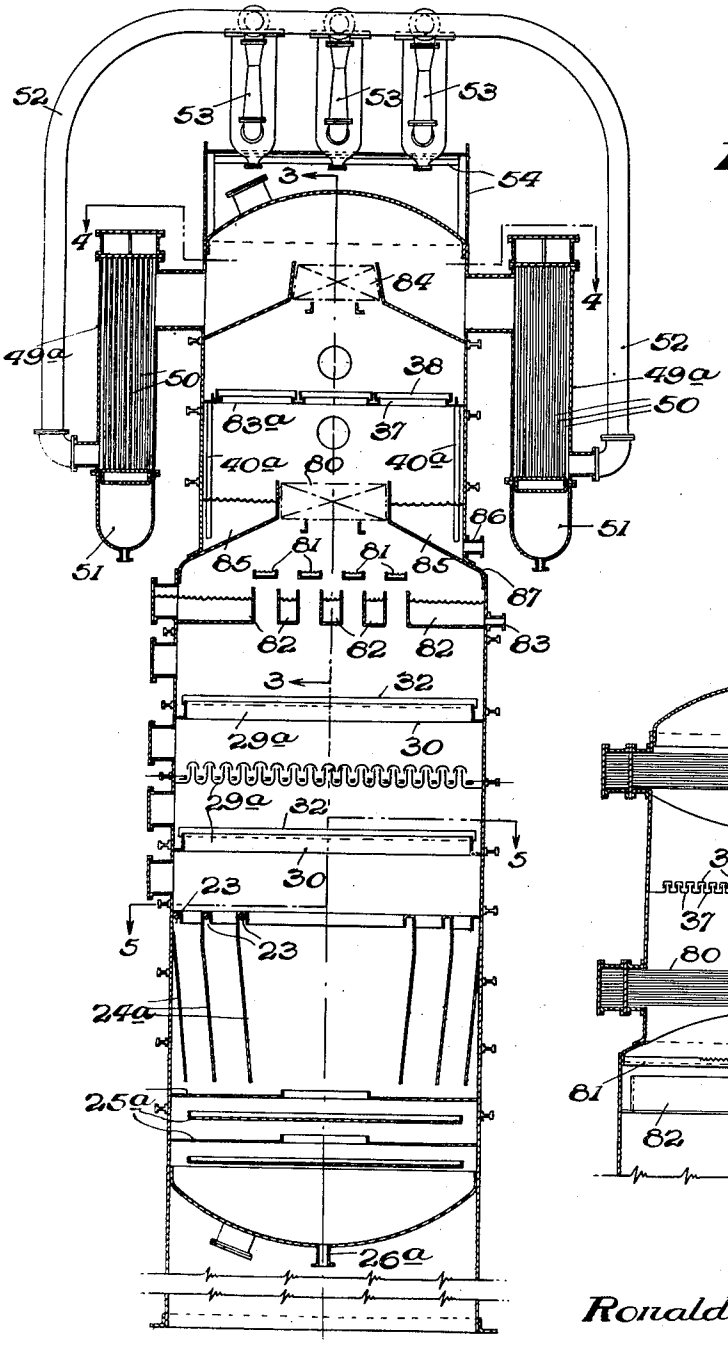


Fig. 2.

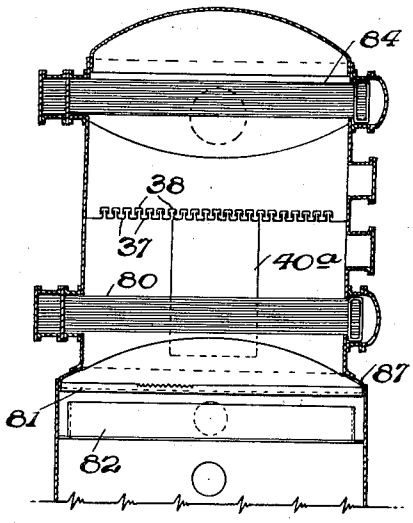


Fig. 3.

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MINERAL OIL DISTILLATION

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Fig. 4.

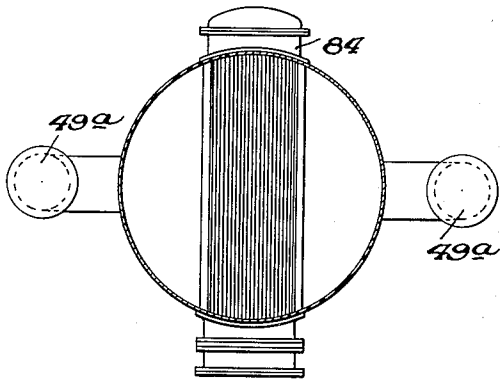


Fig. 6.

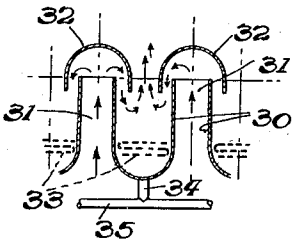


Fig. 5.

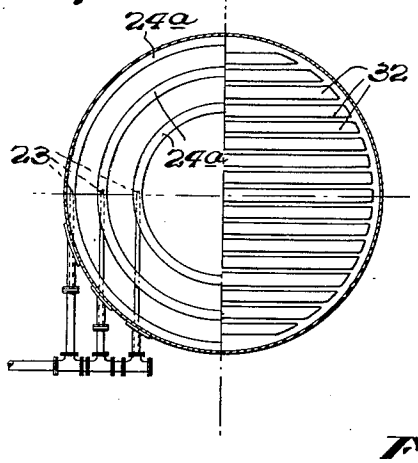


Fig. 7.

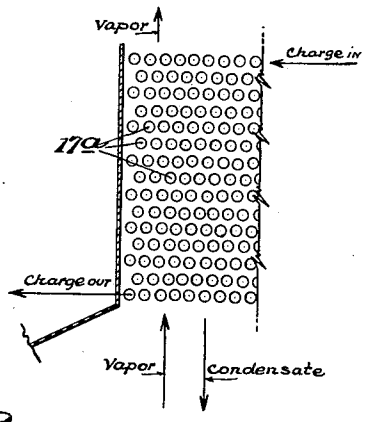
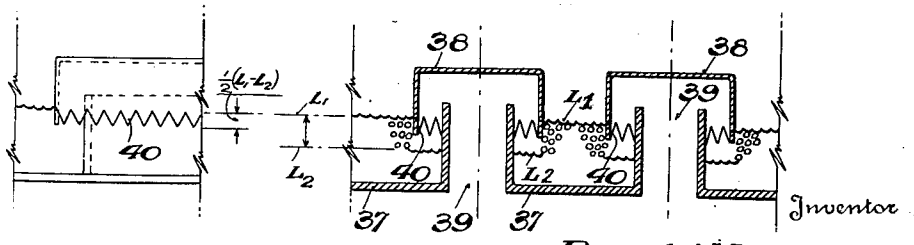


Fig. 8.



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Patented Oct. 8, 1940

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UNITED STATES PATENT OFFICE

2,217,356

MINERAL OIL DISTILLATION

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Application May 3, 1937, Serial No. 140,501

2 Claims. (Cl. 196—106)

This invention relates to mineral oil distillation; and it relates more particularly to systems for the manufacture of lubricating oil by distillation of suitable mineral oil charging stocks under very high vacuum, that is, under very low absolute pressure.

An important object of the present invention is to provide a high vacuum distillation system for the production of lubricating oils as overhead distillates, wherein the time during which the mineral oil material undergoing distillation is subjected to heat at the necessary temperatures shall be minimized, thus minimizing the amount of thermal decomposition or cracking that occurs in the distilling operation.

A further object of the invention is to accomplish this objective by providing for such a system component apparatus units which, in addition to cooperating in the general combination or system to minimize the time factor aforesaid, shall individually possess novel features of construction and operation which render them of general utility in oil refining and distillation, besides giving them special utility in an apparatus combination or system of the character above referred to.

Other objects and advantages of the invention will appear more fully as the description proceeds.

Although the employment of very high vacuum, e. g. absolute pressure of 25 mm. mercury absolute and much lower, enables the manufacture of lubricating oils as overhead distillates which can be marketed and used directly as finished lubricating oils, either without any further refining acid treatment and filtration or with only relatively slight refining, it is nevertheless a fact that, notwithstanding the much lower temperatures at which distillation can be effected under such very low absolute pressures, a certain amount of unavoidable cracking or decomposition occurs, in taking off the heavier lubricating cuts or fractions from the reduced charging stock if the stock is held for any substantial length of time at temperatures substantially higher than about 625° F. Since the amount of cracking undergone by mineral oil always depends not only upon the temperature to which it is heated but also upon the time it is subjected to that temperature, and since it is in some cases necessary to go to fairly high temperatures in order to get off certain desired lubricating values of the charging stock, the importance of so conducting the distillation as to minimize the time factor mentioned becomes paramount. However, there are many practical difficulties tending to complicate the problem of thus cutting down the time factor while at the same time enabling production of overhead lubricating oil distillates of the desired high grade and satisfactory character. Although these difficulties are encountered to a greater or less extent in the high vacuum distillation of the heavier fractions of any lubricating oil charging stock, they are particularly acute in the case of paraffin base stocks typified by Pennsylvania crudes. This is because, in order to vaporize the heavier lubricating oil fractions, such stocks must be heated, even when distilled under the very high vacua mentioned, to final distillation temperatures well above that at which substantial cracking occurs rather rapidly. Accordingly, while the present invention is of utility in high vacuum distillation of lubricating oil charging stocks generally, it has particular applicability to paraffin base charging stocks for the reasons just pointed out. Therefore, in the explanation of the principles of the invention hereinafter to be set forth, reference will be made more particularly, for the sake of a concrete and illustrative example, to a system particularly well adapted for high vacuum distillation of a lubricating oil charging stock derived from Pennsylvania or similar paraffin base crude; but it will be understood that this is merely typical of the various uses of which the system and its component units are capable and is in no way intended to limit the scope of the invention.

A desirable practical embodiment of the invention is illustrated in the accompanying drawings, wherein—

Fig. 1 illustrates more or less diagrammatically in the form of a general layout or flow sheet a typical high vacuum distillation system within the scope of the invention;

Fig. 2 is a sectional elevation of a modified form of fractionating tower or column which in some cases may be used to advantage in place of the particular form of tower illustrated in Fig. 1;

Fig. 3 is a vertical section of the upper part of the tower or column on the line 3—3 of Fig. 2;

Figs. 4 and 5 are horizontal sections on the lines 4—4 and 5—5, respectively, of Fig. 2;

Fig. 6 is a sectional detail, on an enlarged scale, of the construction of entrainment eliminator devices of a special type which may be advantageously employed in the towers shown in Figs. 1 and 2;

Fig. 7 is a detail of a desirable arrangement of condenser tubes; and

Fig. 8 is a detail of bubble tray construction which may be employed to advantage.

Referring first to Fig. 1 of the drawings, the system therein illustrated is particularly well adapted for high vacuum distillation of a paraffin base crude charging stock for production of overhead lubricating oil distillates. In this instance, the system is of the double flash type, comprising two separate flash and fractionating towers 10 and 11. The first is for the production of average lubricating oil distillates containing component fractions having viscosities up to as high as 150 seconds at 210° F., for example; while the second, which is considerably smaller, is for the production mainly of overhead bright stock having an average viscosity as high as 200 seconds at 210° F. All figures for viscosity given herein are to be understood as referring to viscosity determined by means of the Saybolt universal viscosimeter.

In this instance, it will be assumed that it is desired to make, in each tower, two side stream fractions of lubricating oil distillations containing wax, which are to be sufficiently fractionated to give the lower (higher viscosity) side stream product a high flash point. In tower 10, therefore, the fractionating section, consisting most desirably of a single fractionating or bubble tray or plate 12, is located between the points at which these two cuts (e. g., light distillate and heavy distillate) are removed from that tower, in order to give the desired fractionation between the two products therein made. Bubble tray 13 is similarly arranged in tower 11 for the same purpose. The two cuts made in the second tower may be an overhead bright stock cut and a relatively small cut of considerably lower average viscosity representing in part the overlap between vapor and liquid residue in the flashing effected in the first tower.

In the present example, it is further assumed that the charge to the distillation system is a residual product resulting from topping crude in a preceding atmospheric pressure topping operation in which the light ends, including gasoline, naphtha, kerosene and most of the gas oil have already been removed, but substantially without removal of lubricating oil compounds of the crude. In a typical instance, the topped crude thus charged into the system may represent 40 per cent of the original crude. Generally, the crude will have been treated with caustic soda before the topping operation, as by adding, say, 0.2 pint of 40° Baumé caustic soda solution per barrel of original crude. Since the water from this addition, as well as that originally contained in the crude oil, will have been practically all removed in the atmospheric topping unit, the topped crude charged into the high vacuum distillation unit or system shown in Fig. 1 will be practically dry but will of course contain soda. Where the crude employed is one containing no light ends, such as gasoline and kerosene, such crude may be charged directly into the system and the caustic soda introduced into the crude stream after it has been preheated to a temperature of approximately 300° F., the crude being then discharged into an atmospheric dehydrating tank or tower (not shown) where the water naturally contained in the crude, as well as the water from the soda solution, is evaporated before entering the first high vacuum fractionating tower of the system.

Assuming the charging stock to be a topped Pennsylvania crude, it may enter the system from the topping unit through line 14 at around 200° F., being pumped by the crude charging pump 15 through heat exchange in the two fractionating towers, including pressable wax distillate condenser 16 and heavy distillate condenser 17 in tower 10, and condenser 18 in tower 11, thereby being pre-heated to a temperature of, say, about 485° F. The pre-heated charging stock then goes to the pipe still heater indicated generally at 19, passing first through convection tube bank 20, where it is heated to approximately 595° (e. g.), and then through the radiant tube bank 21, where it is heated to a temperature at the heater outlet of, say, 745° F., at substantially which temperature it is discharged at 22 into the vaporizing section of tower 10, entering the tower in this instance through three inlet nozzles 23, as more clearly shown in Fig. 5. These nozzles are provided with restricted discharge openings whereby the hot charging stock is given a very high velocity flow (e. g., 50 to 100 feet per second) and is directed tangentially against a plurality of evaporating members 24 (three in this instance) which, as here shown, take the form of hollow inverted frusto-conical shells. The hot oil thus projected against these vaporizing members near the upper edges of their inner faces is caused to circulate rapidly over those faces and spread out in a thin film. The slight downward tapering of the shells causes the downward pull of gravity on the oil to be balanced to a certain extent by the centrifugal force due to the rapid whirl given the oil, thus tending to prolong the time required for it to flow from the top of each evaporating member to the bottom. In this operation, a considerable part of the oil undergoes evaporation, the unvaporized liquid residue dropping from the lower edges of members 24 to a series of evaporating pans or trays 25 of any suitable design, shown here as of the disc and doughnut type, and thence to the bottom of the tower. The unvaporized liquid residue, amounting to, say, 25 per cent of the charging stock, leaves the bottom of the tower immediately through line 26 and is discharged into accumulator tank 27, the upper part of which is connected by vent line 28 to tower 11, as shown.

The combination of evaporating members 24, 25, arranged as shown in the vaporizing section of tower 10 provides a very large evaporating surface favoring extremely rapid evolution of oil vapors under the low absolute pressure prevailing in this part of the tower, which may advantageously be in the neighborhood of 10 mm. of mercury. Also, by immediately leading unvaporized liquid residue from the bottom of the tower (typically at around 625° F.), no pool of hot liquid residue accumulates and lies in the bottom of the fractionating tower to increase the time factor of this residue in the tower and thus give rise to some cracking in said tower with resultant production of cracked vapors passing upwardly through the tower to contaminate overhead lubricating cuts obtained from this tower. Instead, by thus passing the unvaporized liquid residue immediately into the accumulator tank 27, any decomposition products formed by holding the hot liquid residue prior to its further treatment in the next flash stage and volatile at the temperatures involved are given off in said accumulator tank and therefore pass over

through vent line 28 into tower 11 where they will not be detrimental.

The oil vapors rising from the evaporating section 24—25 of tower 10 and having a temperature of, say, about 645° F., pass upward through entrainment separator devices 29, three in this instance, where all entrained liquid particles are removed and dropped back into the unvaporized liquid residue. The detailed construction of these entrainment separator devices may desirably be as shown in Figs. 2, 5 and 6, consisting essentially of a series of horizontal parallel troughs 30, suitably supported at their ends by the tower wall and separated by spaces or vapor slots 31. These slots are surmounted by inverted semi-cylindrical trough-like caps 32. As indicated by the arrows in Fig. 6, vapors passing upward at high velocity (e. g. 40 feet per second) through the slots or spaces 31 are compelled by caps 32 to reverse their direction and to pass downwardly between each cap and trough, which are so positioned relatively as to constrict the vapor passage somewhat at this point and thereby to increase the vapor flow velocity (e. g. to 60 feet per second). After leaving this constricted passage at such higher velocity, the vapors are forced to turn through a full 180° angle and then pass on up through the tower, while the entrained liquid particles are thrown directly downward and caught in the bottom of the troughs. In order to prevent or minimize splashing and re-entrainment of the trapped liquid, a body 33 of suitable trapping material that is foraminous, reticulate, or otherwise of such character as to permit ready passage of liquid therethrough, is provided in the lower part of each trough 30. For this purpose, a plurality of layers of screen wire or the like serve admirably, the assemblage being most desirably supported in the trough at some little distance above the bottom, as shown, to provide a shielded space for reception of the separated liquid. Liquid collecting in these protected spaces thus provided in the troughs 30 passes through outlets 34 into a common drain 35, by which the liquid is returned to some lower point in the tower (e. g., to the bottom).

This type of construction for the entrainment separators involves but a very low pressure drop, on the order of only 0.5 mm. mercury per separator device under conditions normally prevailing in the system, making a total drop of only 1.5 mm. for the entire entrainment separating section here illustrated.

In the form of the invention illustrated in Fig. 1, the vapors leaving the entrainment separating section pass next through the fractionating section provided by bubble tray 12, already referred to hereinabove, where a heavy wax-bearing lubricating distillate cut is fractionated from the lighter vapors. Reflux for this fractionating tray is provided by the fractionating condenser 17 previously referred to, which is located above the tray and cooled by incoming charging stock on its way to the pipe still heater, thus constituting a part of the heat exchange whereby said charging stock is preheated.

In order to enable this fractionating of the heavy distillate cut to be accomplished with the minimum pressure drop, and in order to provide high ratio of slot area to tower area, the bubble tray and bubble caps of the fractionating device 12, which may be of the general type more fully illustrated in Figs. 2 and 3, may advantageously be of the special construction shown in greater detail in Fig. 8. In a typical instance, the bubble tray may be designed to carry an effective liquid head of approximately 1 inch, causing a pressure drop of approximately 1.4 mm. Hg. The vapor velocity for this pressure drop can be as high as 80 feet per second, depending on the density of the oil vapor; and the liquid head of 1 inch assumed, diminishes or increases accordingly as this vapor velocity is reduced or increased.

In the construction shown in Fig. 8, the bubble tray comprises a series of terminally supported troughs 37 in spaced parallel arrangement, with which cooperate caps 38 in the form of inverted troughs respectively located above the spaces or slots 39 between troughs 37. In this construction, the customary "teeth" 40 provided at the lower edges of the cap troughs 38, instead of extending clear down to the desired nominal liquid level L_2 on the inside of the caps from the normal level L_1 on the outside of the caps, as is the usual practice, extend only half of this distance (which distance is here assumed to be 1 inch), as clearly shown in Fig. 8. In the ordinary type cap, the free unit slot area is only half the total area below the top of the slots, while in this design the free slot area is 75 per cent of the total area below the top of the slots, which increases the slot area per unit length or per cap by approximately 50 per cent.

The overflow from the bubble tray passes through down pipes 40a into a catch basin 41 located inside the tower (Fig. 1), which is equipped with an outside float type cage liquid level controller 42, the heavy distillate fraction or cut collected in this catch basin and having a temperature of, say, 540° F., being pumped away by pump 43 through cooling means 44 to storage. This heavy lubricating distillate fraction, in a typical instance, may have an average viscosity of 80 seconds at 210° F. and may comprise 25 per cent of the charging stock.

The uncondensed vapors leaving the bubble tray fractionator 12 pass upwardly through the heavy distillate fractional condenser 17 which, likewise, is designed for very low pressure drop (e. g., approximately 1 mm. mercury). A suitable arrangement of the tubes 17a of this condenser is shown in Fig. 7, where the tube bank is fifteen rows of tubes high and the tubes are arranged on a triangular pitch. There are no horizontal baffles of any kind in this condenser and at all times the liquid condensate is falling downward in a direction opposite to the rising vapor. In this way, a certain amount of reboiling of the condensate takes place in the condenser itself, which is desirable for present purposes, particularly because it prevents the condensate being cooled considerably below its boiling point and concomitant undesirable condensation, as well as absorption and retention therein, of lighter hydracarbons. The charge oil used to cool this condenser 17 enters the top layer of tubes of the unit and thence through the succeeding layers of tubes in series down through the unit, leaving at the bottom. Most advantageously, each layer of tubes is divided into two liquid passes, making a total of 30 passes on the cooling medium side of the particular condenser unit here illustrated. It is desirable to have as low a temperature differential between the cooling medium and vapor as is practical without using a condenser unit of excessive size. The purpose of this is to prevent cooling the condensate on each tube much below

its boiling point, thus favoring a certain amount of re-boiling of the condensate as it falls down to the tubes below.

The vapors leaving the heavy distillate condenser 17 at, say, 510° pass through a set of fractionating pans 45, which may be similar in character to the liquid catch plates 81—82 shown on a somewhat larger scale in Fig. 2; thence through the wax distillate condenser 16 at the top of the tower. As here shown, this condenser is a separate unit located on the exterior of the tower, but it may be located inside the tower if desired. Its construction and operation are virtually the same as those of the heavy distillate condenser 17. The wax distillate collecting in catch basin 46, having a temperature of, say, 420° F., for example, may be withdrawn by pump 47 equipped with liquid level controller 47a, and pumped through cooler 48 to storage. This cut which has a viscosity of 115 seconds at 100° F., for example, may constitute approximately 44 per cent of the charging stock.

The uncondensed gas oil vapors leaving the wax or light distillate condenser 16 at, say, about 355° F. in a typical instance, pass to gas oil condensing means 49, consisting most desirably of duplicate vertical type condensers mounted at the side of the tower as indicated at 49a in Fig. 2, the vapors flowing in a single pass downwardly parallel to the tubes 50 so that the vapor and condensate are cooled to as low a temperature as possible and as quickly as possible. The liquid gas oil condensate, which in a typical instance has a viscosity of around 52 seconds at 100° F. and amounts to perhaps 6 per cent of the charge, falls into a compartment 51 at the bottom of each condenser and is pumped away to storage through line 51a. These condensers are water-cooled.

The non-condensable gas leaving the gas oil condenser or condensers is withdrawn through large-diameter vapor line 52 by suitable vacuum-producing means, such as the 3-stage ejectors 53 which may be located on the framework and platform 54 at the top of the tower, in the manner indicated in Fig. 2. One of the chief advantages in locating the gas oil condensers on the side of the tower, as indicated at 49a (Fig. 2), is to leave the space at the top of the tower free for mounting the ejectors. By this method, it is possible to have the entire system supported by the tower itself, thus eliminating all separate structural steel supports for the different units. This is a substantial advantage, because towers or columns operated at high temperature and having a considerable height expand as much as two inches or more in being heated up from the cold, rendering necessary the installation of large expansion bends or spring mountings where the condensers and ejectors are mounted on a structural framework separate from the tower.

The ejectors maintain an absolute pressure of approximately 4 mm. Hg at the outlet of the gas oil condensers; and with all units designed for very low pressure drops, as described, an absolute pressure at the point of flash (22) as low as 10 mm. Hg is easily maintained under these assumed conditions.

The side stream wax-containing distillates produced by the operation described hereinabove are sufficiently closely fractionated to provide products of adequately high flash points and suitable for subsequent de-waxing by available solvent de-waxing processes, such as the benzol-acetone-toluol and benzol-methyl-ethyl-ketone processes. However, if these side stream wax-containing distillates are to be de-waxed by conventional pressing and centrifuging methods, it is advisable to provide for closer fractionation between the heavy distillate and the lighter distillate and also to practically reverse the volume proportions of these cuts, so that the heavy distillate cut shall amount to approximately 43 per cent of the charge oil and the light distillate approximately 26 per cent. The latter will then be a pressable wax distillate, properly speaking; while the heavy distillate, whose wax content will be largely amorphous, will nevertheless include relatively light fractions or ends carrying pressable wax. Removal of these lighter ends from the heavy distillate can be effected in various ways as, for example, in a high vacuum re-fractionating tower (not shown) provided with a sufficient number of bubble trays to accomplish the desired closer fractionation. In order to accomplish this fractionation the heavy distillate cut is heated in the re-fractionating tower to a sufficiently high temperature to re-vaporize all of the pressable wax distillate, the necessary heat being provided by means of a separate tubular heater. The re-fractionating tower may, for example, be constructed with four bubble trays above the point of the charge inlet and two bubble trays below the charge inlet. The tower should also be equipped with a re-boiler below the series of bubble trays; and it should also be provided with a reflux condenser for supplying reflux to the bubble trays. The overhead pressable wax distillate vapor leaving this re-fractionating tower may be returned to tower 10 at a point above the wax distillate drawoff (46) and below the fractionating pans 45. This auxiliary refractionating tower would operate, under these circumstances, at an absolute pressure approximating 11 mm. Hg at the point of vaporization below the bubble tray system; and since only the lighter ends of the heavy distillate have to be re-vaporized in order to substantially free the heavy distillate of its pressable wax distillate component, the outlet temperature of the separate tubular heater employed with this auxiliary re-fractionating tower is not high enough to cause troublesome decomposition.

The liquid residue from tower 10 collecting in accumulator tank 27 is pumped therefrom by pump 54a through the radiant tube bank 55 of pipe still heater 19, this bank being separate from the first mentioned radiant tube bank 21 and located in a separate firing chamber 56, which has its own firing means 57 controllable independently of firing means 58 of the other heater chamber; but the firing gases from both heater chambers exit through the common flue 59 in which the aforementioned convection tube bank 20 is located. In radiant tube bank 55, the residue pumped from accumulator tank 27 is very rapidly heated to a temperature of approximately 825° F. at which it is discharged into tower 11 at 60, where the second flash vaporization occurs at the very low absolute pressure (e. g. 6–8 mm. Hg) prevailing at this point in the tower. Because of the particular design and arrangement of the pipe still heater 19, it is possible to heat the aforesaid residue from 670° F., the approximate temperature at which it ordinarily enters tube bank 55, to 825° F., in a relatively very short period of time, only about 70 seconds in a typical instance, considerably less than half the time which would be required if the heater were designed in the conventional manner. In other

words, the time factor involved in this final heating of the charging stock residue is reduced to approximately 40 per cent of what it would be with the ordinary type of pipe still heater.

The reduced crude stock entering tower 11 at the approximate maximum temperature (i. e. heater outlet temperature) of 825° as aforesaid, is discharged into an evaporating section which is desirably similar to that of the fractionating tower 10, employing vaporizing surfaces 61, 62, of the same type as 24 and 25 employed in tower 10. The unvaporized liquid residue, or that portion of it representing the normal make of flux oil residue, leaving the base of the tower at about 760° in a typical instance, is passed by pump 63 through cooler 64 to storage, this flux oil residue usually amounting to around 10 per cent of the original charge oil. Instead of passing this residue through the cooler, it may be utilized in a heat exchange for recovery of much of its contained heat units. Most desirably, a portion of the total final residue leaving the tower 11 is diverted through line 65 by pump 66 and pumped back to accumulator tank 27 where it mixes with and substantially raises the temperature of the residue from tower 10 and passes therewith through tube bank 55 of the final heater. The volume of flux oil residue thus constantly being re-circulated, after the system is operating normally, may desirably be about one-half that of the residue from tower 10 with which it is mixed and whose temperature it thus quickly and easily raises before entry into final heater 56. This re-circulation accomplishes the further very desirable result of enabling a constant through-put to be maintained through the final heater and of providing a sufficiently high rate of flow to enable keeping the tube size in this final heater above a minimum of, say, 1.5 inches internal diameter. At diameters less than this, considerable trouble is likely to be experienced from coking and from deposition of soda contained in the charging stock. The pumping rate of the re-circulating pump 66 is controlled by an automatic liquid level control 67 located in the accumulator tank, so that a constant liquid level will be maintained in this tank. Without such re-circulation or re-cycling, it is difficult to maintain a constant rate of flow through the final heater because, although liquid residue normally flows from the fractionating tower 10 at a substantially constant rate, some fluctuation may occur occasionally due to slight changes in operating temperatures and pressures in tower 10 or rate of charge thereto. Consequently, in the absence of said re-cycling, and if the operation of the final heater charging pump 54a were controlled by the liquid level in accumulator 27, any change in rate of flow of residue from tower 10, unless immediately noticed by the plant operator, could cause a sudden stoppage of said final heater charging pump, with resultant damage to the final heater tubes. By providing the aforesaid recirculation or recycling of residue from tower 11, this difficulty is overcome and a method for maintaining a constant through-put through the final heater is provided.

The vapors flashed off in the vaporizing section of tower 11 have a temperature of around 770° (e. g.). They pass up through entrainment separators 68, bubble tray 13, fractional condenser 18 and fractionating pans 69, all of which are designed for very low pressure drops, being most desirably similar in construction and general function to entrainment separators 29, bubble tray 12, fractional condenser 17 and fractionating pans 45, employed in tower 10.

Heavy bright stock, containing wax, is removed as a side stream distillate just below bubble tray 13 at a temperature approximating 670° (e. g.), being pumped by pump 70 through cooler 71 to storage, the operation of pump 70 being automatically controlled as indicated at 72. In a typical instance, this bright stock amounts to around 10 to 11 per cent of the charge and has an average viscosity of 160 seconds at 210° F. It has a very high flash point, 600° F. open cup test and even higher in some cases. This is a very unusual product to be obtained by overhead distillation from a charging stock of the Pennsylvania type. It can be finished and made color stable by treatment with a comparatively small amount of clay. It is feasible, with this apparatus system, to produce from Pennsylvania crude, as an overhead distillate, heavy bright stocks having average viscosities as high as 190 to 200 (or more) seconds at 210° F. and flash points around 635° open cup. Such products have not been known in the art heretofore. Their boiling points are as much as 100° F. higher than bright stocks obtained as overhead distillates from non-wax-bearing crudes by high vacuum distillation.

In the particular arrangement here shown, a smaller percentage (e. g., between 2 and 3 per cent) of extra-heavy distillate, also containing wax, may be removed as a side stream above the heavy bright stock cut by pump 73. This product, fractionated out of the vapors leaving condenser 18 (at about 610° F.), by the action of fractionating pans 69 and fractional condenser 69a, is taken from the column at a point below condenser 18 at a temperature approximately 580° F. and is pumped through cooler 74 to storage, the operation of the pump being automatically controlled in the usual way as indicated at 75. This product may have, typically, a viscosity of about 90 seconds at 210° F. and an open cup flash point of approximately 550°. This product can be blended back with heavy bright stock or finished separately. It is to be noted that this product, representing in part a slight overlap between the flashed vapors and liquid residue in tower 10, is produced in relatively very small percentage as compared to the percentage that would normally be produced when operating at higher absolute pressures (including atmospheric), where such overlap would be very much higher.

Residual uncondensed vapors passing condenser 69a at about 400° are conducted to final condenser 76 where a so-called header distillate or "stink oil," usually amounting to around 2 per cent or less of the charge, is condensed. It consists almost entirely of partially decomposed products resulting from the very high temperature (825°) of the final heater discharge. It is pumped away by pump 76a.

Condenser 76 is connected by large-diameter vapor conduit means (like 52) to the vacuum producing means, a 3-stage ejector system in this instance.

Although the partial condenser 69a may be cooled by charge oil like the other partial condensers shown in the illustrated system, it is here shown as provided with an oil trim cooling system, consisting of accumulator tank 77, pump 78 and cooler 79. In this system, a fairly light gas oil is suitable to use as the cooling medium to be circulated. Water cooling of the partial

condenser 69a would give rise to difficulties due to vaporization in the tubes of the cooling unit, with deposition of silt and excessive tube corrosion.

All the condensers in the system described are advantageously designed for very low pressure drop, not more than about 1 mm. Hg absolute per unit as a maximum; and those in the final tower 11 are most desirably designed for only half that drop, or 0.5 mm. Hg.

It will be seen that because of the construction and arrangement of the vaporizing section, entrainment-separating section and fractionating section of each of the towers 10 and 11, the resistance to flow of vapors at high velocity upwardly through these towers is reduced to a practical minimum, thus also minimizing absolute pressure drop or difference between point of flashing and the tower outlet through which the final uncondensable vapors and fixed gases are withdrawn by the vacuum-producing equipment. In the typical operation assumed in the foregoing description, this pressure difference amounts to only 6 mm. Hg in tower 10, and 4 mm. in tower 11. Such drop need not exceed 10 mm. as a maximum in systems embodying the principles of the invention. The vacuum-producing equipment employed should in all cases be of such capacity that, in the normal operation of the system, said equipment shall be capable of withdrawing uncondensable vapors and gases from the vapor outlets of the towers with sufficient rapidity to maintain at said outlet an absolute pressure not substantially exceeding 6 mm. Hg, and most desirably not exceeding 4 mm.

Referring again to the pipe still heater 19, the use of a heater of this particular type, which embodies novel features of construction and arrangement, contributes substantially to the achievement of one of the important objects of the invention, namely, reducing the time to which the charging stock has to be subjected to the relatively high final heating temperature, besides effecting important economy in fuel. In the construction illustrated, that part of the unit containing the convection tube bank 20 and radiant tube bank 21 and fired by burner means 58 may be of conventional construction. The furnace gases from burner means 58, after being cooled to approximately 1200° F. by radiation to the radiant tube bank 21 pass down through the convection bank 20, being thereby cooled to about 550° F. The final heater 56, however, independently fired by burner means 57, comprises only a radiant bank of tubes 55. The furnace gases in this final heater are also cooled to approximately 1200° by radiation to tubes 55, and then pass through openings in the dividing wall between the two furnaces and join the flue gases of the first furnace, passing down through the common flue containing the aforesaid convection tube bank 20. In this way, notwithstanding the fact that the charge oil entering the final heater tubes 55 from accumulator 27 is at a temperature of 670°, thus rendering it impractical to provide sufficient heat transfer surface in a convection tube bank to cool the combustion gases to a temperature lower than approximately 740°, the heater construction and arrangement of the present invention, whereby said charge oil from accumulator 27 is heated to the high final temperature of 825° by passage solely through the radiant tube bank 55, makes it possible to cool the gases from the final heater 56 down to the same economical low temperature, approximately 550°, as the gases from the first heater are cooled. This not only results in a fuel saving of approximately 10 per cent in the final heater, but it has the further advantage that all of the heat transfer to the charge oil going to the final tower 11 is radiant heat, thus permitting the use of a minimum tube surface area. By reducing this surface area, the volume of oil contained in tubes 55 is correspondingly reduced, resulting in a tower time factor and thus reducing the amount of cracking that occurs. In other words, in the pipe still heater here illustrated, the charge oil going from accumulator 27 to tower 11 remains in the final heater for a period of time on the order of only about 70 seconds, in a typical instance; whereas if a heater of the conventional type were employed, the oil would have to remain in the heater tubes on the order of 175 seconds in order to achieve the same increase in temperature. Otherwise stated, the present arrangement enables a reduction of the time factor, in this stage of heating to the final flashing temperature, to approximately 40 per cent of what would otherwise be required. The importance of this in any process where the charge oil must be heated to a temperature at which cracking occurs with appreciable rapidity is obvious, and this importance is particularly pronounced where, as here, the final temperature of the charge is much above that at which it is generally recognized cracking of a relatively heavy lubricating charging stock begins to be appreciable.

From the foregoing, it will be seen that the invention provides a high vacuum distillation system of which the various component parts are all designed and particularly adapted to cooperate in reducing the period of time during which a charging stock must be subjected to the relatively high temperatures requisite for effecting adequate vaporization and recovery, as overhead distillates, of the lubricating values contained therein. Operation of the system is characterized by extremely rapid vaporization through systematic filming over vaporizing surfaces of large area, as well as high velocity flow of the resultant vapors through entrainment-separating and fractionating means of a type interposing minimum resistance to vapor flow with consequent minimum drop in vapor pressure, while at the same time adequate fractionation for production of the desired products as side stream cuts is accomplished. In addition, large reduction is accomplished in the time factor involved in heating to the final flashing temperature at which extensive cracking would necessarily occur were it not for thus cutting down the time factor in question. The practical importance of such a system in enabling the successful handling of charging stock by a procedure involving the employment of flash vaporizing temperatures, especially in the final flashing stage, so high as to be generally regarded as excessive and dangerous, is sufficiently obvious without further comment.

The distillation system as thus far described is especially well adapted, as already stated, for processing a paraffin base charging stock, such as a topped Pennsylvania crude, where it is desired to make two side stream fractions of wax distillate which are separated by more or less close fractionation as may be desired. In operating on a non-wax-bearing crude, on the other hand, it may often be desired to effect in the first flash-vaporizing stage (i. e., in the first tower of the system) only a fractionation between the gas oil cut and the side stream lube

distillates. Under these circumstances, certain important practical advantages are attained by using the modified tower construction illustrated in Fig. 2 in place of tower 10 in the system above described and illustrated in Fig. 1. The final tower of that system, tower 11, may also be replaced, if desired, by the modified type shown in Fig. 2.

Referring to Fig. 2, it will be seen that this modified type of tower is provided in its lower part with a vaporizing section consisting of coned evaporating members 24ª and evaporating trays or pans 25ª similar to the corresponding vaporizing surfaces 24, 25 of tower 10, nozzles 23 being arranged to direct high velocity jets of heated charge oil against the coned members 24ª to film the charge thereover for a very rapid vaporization at the low absolute pressure (e. g., 10 mm. Hg) prevailing in this section of the tower. Unvaporized liquid residue exits through outlet 26ª, while flashed off vapors travel upwardly through entrainment separator devices 29ª, similar in construction to separators 29 of tower 10. Leaving the last of this series of three entrainment separators 29ª, the vapors, instead of passing next through a bubble tray section as in tower 10, are subjected to fractional condensation by passage through the intertubular spaces of fractional condenser 80 to condense out a heavy lubricating distillate which collects on liquid catch plates or trays 81, 82 and is withdrawn as a side stream at 83 by an automatically controlled pump (not shown) in the usual manner. The vapors in passing from the entrainment separating section through the liquid catch plates 81, 82, undergo a certain amount of fractionation owing to the contact of upwardly flowing vapors with liquid overflowing the edges of the upper set of catch plates 81. The edges of these latter may be V-notched or serrated so that there may be a uniform distribution of liquid streams falling from these upper catch plates to the lower catch plates 82. The cooling medium employed in the tubes of the heavy distillate condenser 80 is charge oil that is to be preheated, and this condenser is operated at a sufficiently high temperature to fractionally condense a substantial part of the higher boiling vapors without danger of condensing any gas oil at this point in the tower, as will be further explained presently.

Vapors leaving the condenser 80 pass next through a fractionating section consisting in this instance of a single bubble tray indicated generally at 83ª, the detailed construction of which may be the same as that of bubble trays 12 and 13 hereinabove described. The reflux for this fractionating section is provided by light distillate condenser 84 in which charge oil to be preheated is also employed as the cooling medium. The liquid fraction collecting on the bubble tray flows therefrom through down pipes 40ª into the compartments 85 on either side of the condenser 80. Since the vapor below these compartments is somewhat hotter than the liquid, a certain amount of re-boiling takes place in these compartments, resulting in closer fractionation of this cut which is drawn off as a side stream at 86 by an automatically controlled pump (not shown).

Uncondensed vapors leaving the light distillate condenser 84 pass thence into the water cooled gas oil condensers 49ª, the resultant gas oil condensate collecting in 51 and being pumped away to storage, while residual uncondensable vapors and fixed gas are drawn through large diameter lines 52 by the 3-stage ejector equipment 53; all as hereinabove described in connection with the system shown in Fig. 1.

In processing a non-wax-bearing crude, as has been assumed in describing the modified tower construction of Figs. 2 and 3, where it would not be essential to make more than one lube distillate side stream cut and a gas oil overhead cut, it would be possible to operate with only one fractional condenser, such as the light distillate condenser 84. But in that case, all of the reflux heat from the tower would have to be removed by this one fractional condenser at a point where the vapor temperature is comparatively low, and it would be impossible, as a practical matter, to pre-heat the charge oil (used as cooling medium) to a temperature above approximately 350° F. in this one fractional condenser 84. In thus preheating the charge to 350°, an insufficient amount of heat would be removed and it would be necessary to use some other cooling medium in addition. However, if another condenser is placed below the fractionating section 83ª, as here shown, where the vapor temperature is considerably higher than it is above the fractionating section, the charge may be preheated to approximately 100° higher temperature by being utilized as the cooling medium in this second condenser, and all the necessary heat may be removed by the charge.

Since it is not desired to separate the two lubricating oil distillate cuts from each other by close fractionation when processing a non-wax-bearing crude as in the particular case here assumed (in practice they will ordinarily be pumped together), the average lubricating oil distillate produced will have a relatively broad boiling range, making it a simple matter to fractionally condense, by means of the condenser 80 below the fractionating section, a large proportion of the higher boiling vapors while avoiding condensation of gas oil at this point. Another advantage of this arrangement and mode of operation is that the volume of vapor passing through the bubble tray fractionating section is considerably reduced and the diameter of the tower can be reduced at this point, as indicated at 87 in Figs. 2 and 3, thus effecting substantial economy in cost of construction. By thus arranging the second fractional condenser below the fractionating section of the tower, the capacity of a given tower is greatly increased over that of one of otherwise similar construction and arrangement, this increase amounting to as much as 50 per cent in a typical instance.

Where the system of the invention is employed in processing a non-wax-bearing charging stock, it is usually not necessary to employ temperatures in either flashing stage nearly so high as when processing a wax-bearing stock. Nevertheless, even in such lower-temperature operations, employment of the system herein disclosed affords marked advantages in the way of economy and increased yield of desired products of high quality.

Although of general utility, the system herein disclosed is especially well adapted to be employed as a primary high vacuum distillation unit for production of high grade lubricating oil distillates which are to be re-distilled in a re-run unit (not shown), generally in the presence of a neutralizing agent (e. g. caustic soda), for direct production of finished lubricating oils as overhead distillates that are sweet and stable without having to be acid treated or filtered.

When the system is operated in conjunction with such a re-run unit, the small percentage of final residuum resulting from the re-run operation may advantageously be pumped from the re-run unit through line 88 into accumulator tank 27 at a constant rate proportional to its rate of production in the re-run unit. It then passes, in mixture with the rest of the charge accumulated in tank 27, through the final heater and is re-flashed in tower 11. Since the greater part of this residue from the re-run unit is as heavy as the bright stock overhead distillate produced in tower 11, the yield of this bright stock product is thus increased accordingly. This increase may amount to as much as 2.5 per cent of the crude, depending upon the type of charging stock. Moreover, this continuous re-cycling of the re-run residue through the final flashing stage of the primary system has the further advantage that it obviates the necessity for accumulating the re-run residue over a period of time until enough is available to re-flash it in large bulk through a primary distillation system such as that herein disclosed. Such practice involves loss of time; and it also introduces difficulties from an operating standpoint, particularly because of the character of the residue where soda is used in the re-run operation. In that case, the re-run residue, besides containing the unused excess of soda, also contains the non-volatile components of the products of reaction of soda with unstabilizing and odor-producing impurities present in the charging stock supplied to the re-run units; all of which makes the processing of such a re-run residue, by itself, a somewhat troublesome problem. This trouble is avoided by constantly feeding it back in small amount into the primary system, as indicated at 88, so that it is extensively diluted, constituting only about 1 per cent (e. g.) of the charge.

Although, in the foregoing detailed description of desirable practical embodiments of the invention, reference has been made at various points to specific dimensions, operating temperatures, pressures, pressure drops, etc., this is to be understood as only for the purpose of illustrating typical good practice within the scope of the invention and as affording concrete illustrative examples as an aid to a better understanding of its underlying principles, and that the invention is not restricted to such details. Moreover, various features of apparatus construction and arrangement herein disclosed are believed to be novel per se, as well as in combination with other units of a multi-flash system, whether or not employed in a system operating under vacuum of any degree. The invention is therefore not to be understood as limited in all respects to vacuum distillation systems.

Certain subject-matter disclosed in this application is also disclosed in the copending application of John E. Schulze and Ronald V. Becknell, Serial No. 140,500, filed concurrently herewith.

What is claimed is:

1. A high vacuum distillation system for the manufacture of mineral lubricating oils as overhead distillates which comprises, in combination, a flash and fractionating tower provided with vaporizing surfaces, and also having an entrainment-separating section and a fractionating section which includes a bubble tray, said entrainment-separating and fractionating sections being so constructed and arranged that the resistance they offer to vapor flow in the normal operation of the tower results in a pressure drop therethrough not exceeding 10 mm. Hg, means for delivering heated charging stock to said vaporizing surfaces, means for withdrawing fractionated products from said tower in side stream, an accumulator outside said tower, means for continuously transferring from the base of said tower to said accumulator unvaporized liquid oil residue as rapidly as it separates in said tower, a second flash and fractionating tower having entrainment-separating and fractionating sections, means for withdrawing liquid oil residue from said accumulator for further heating said residue and for delivering it to said second tower, means for withdrawing fractionated products from said second tower in side stream, and vacuum-producing means connected to the vapor outlet of the fractionating section of each tower and capable of maintaining at such outlet an absolute pressure as low as 4 mm. Hg.

2. A fractionating tower for high vacuum distillation of mineral oil comprising, in combination, a vaporizing section; an entrainment-separating section comprising cooperating trough and cap members arranged to abruptly reverse the flow direction of vapors ascending from said vaporizing surfaces, thereby to throw entrained liquid into said trough members, and means for draining separated liquid from said troughs; and a fractionating section, including bubble tray means, located above said entrainment-separating section, said tower having a vapor outlet above said fractionating section, and said entrainment-separating and fractionating sections being so constructed and arranged that the pressure drop between said outlet and said vaporizing section does not exceed 10 mm. Hg.

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