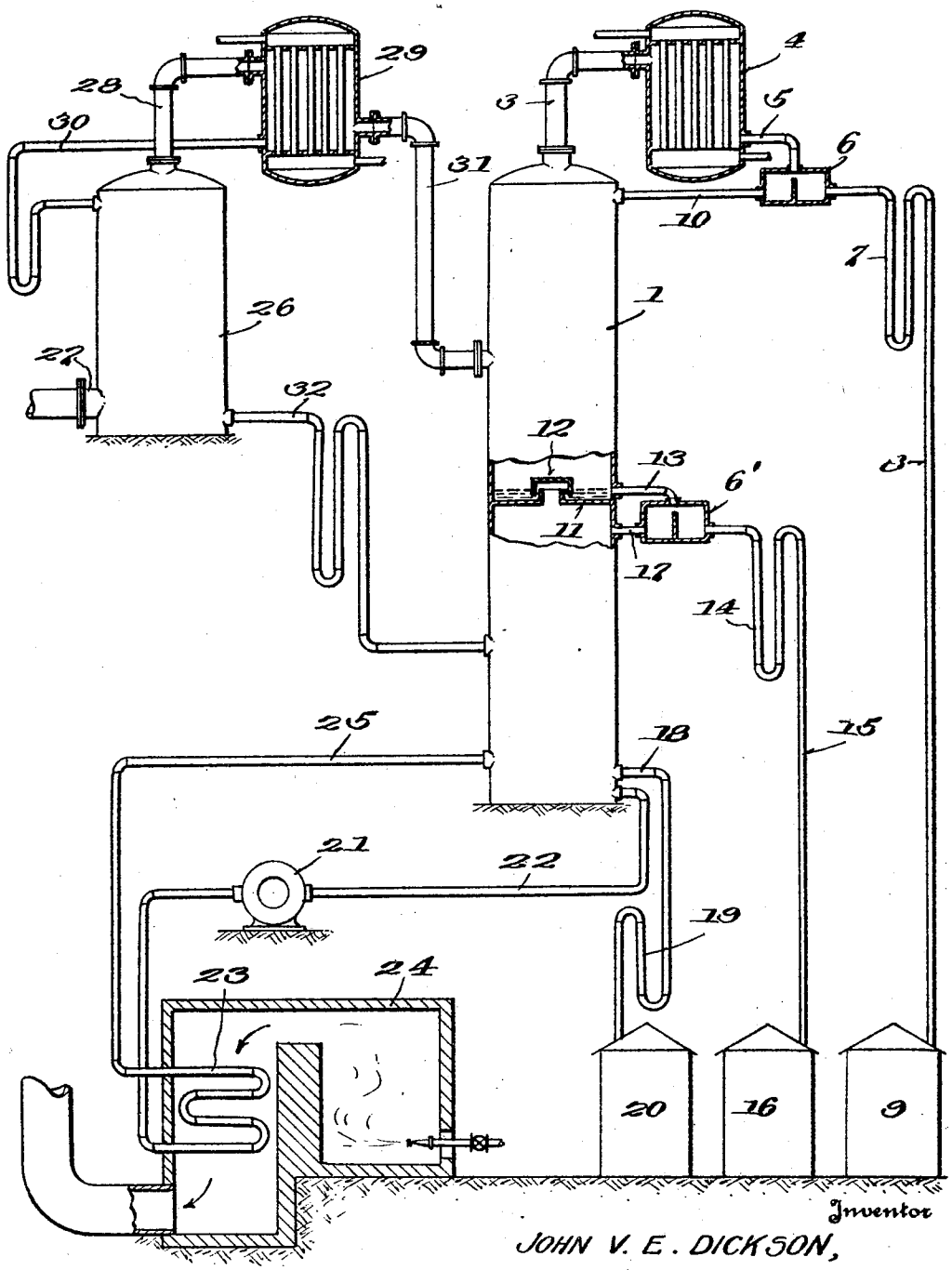
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DISTILLATION OF MINERAL OILS

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DISTILLATION OF MINERAL OILS

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This invention relates to distillation of mineral oils; and it has to do more particularly with processes of and apparatus for distilling crude petroleum for the production therefrom, directly and without redistillation, of cleanly separated fractions. This application is a division of my pending application Serial No. 8318 filed February 11, 1925.

In distilling oil to separate it into fractions, the oil commonly heated by passing it through a series of pipes or other heating means, set in a furnace, and then discharged into a separating chamber in which the vapors formed by heating are separated by gravity from the part of the mixture which remains liquid. The vapors then pass on and are condensed, while the liquid is collected separately. This, however, does not make a good separation of the oil into two fractions, the vapors carrying with them a part of the heavier material which it is preferable to retain in the liquid, and the latter retaining lighter material which for proper separation should pass off with the vapor. Moreover, it is often desirable to obtain two or more distinct fractions of different boiling ranges, from the vaporized portion, besides the residue which has not been vaporized.

In order to obtain the desired results it has usually been necessary to resort to a redistillation either of the total condensate or of some of the fractions obtained by a rough fractional condensation. In order to avoid redistillation, a variety of fractionating condensers, dephlegmators, towers and columns have been used, but in most cases these have not led to completely satisfactory results. The apparatus and methods used fail to give the best fractionating results for various reasons but in general they have the common fault of condensing each fraction in contact with a stream of vapors containing all the constituents having lower boiling points than the fraction in question. For instance, in some cases the vapor mixture is passed successively through a series of condensers or dephlegmators, in each one of which one fraction is condensed and removed. But being condensed in the presence of the lower-boiling constituents, it necessarily carries away with it some of these and its quality as a close-boiling fraction is thereby impaired.

The principal object of the present invention is to overcome these difficulties and to render commercially feasible the distillation from mineral oil, directly and without redistillation, of three groups of products separated from each other with the greatest accuracy or sharpness consistent with simple design and economical operation. These three groups may be designated generally as follows: first, a low-boiling portion comprising the gasolines; second, a relatively high-boiling portion comprising chiefly gas oil; and third, a portion comprising products whose boiling points are intermediate the first two groups mentioned and consisting mainly of the kerosenes, but including also in some cases certain products lighter than and not strictly classifiable as a kerosene but of too high an initial boiling point to be properly termed a gasoline. A more specific object of the invention is to get a relatively sharp cut between the kerosene (of which one or more may be produced) and the gasolines.

Generally described, the process of the invention comprises heating crude oil or other mineral oil to a temperature sufficiently high to vaporize the three groups of products before mentioned, namely, gasolines, gas oil, and products intermediate these. The vapors of these products are then subjected to a preliminary fractionation in apparatus of any suitable type in order to obtain, on the one hand, vapors consisting mainly of constituents boiling lower than kerosene and containing very little constituents, if any, boiling higher than kerosene; and, on the other hand, a liquid consisting largely of gas oil, and in any event consisting mainly of constituents boiling higher than kerosene, but also containing a variable minor percentage of constituents belonging properly in the final gasoline and kerosene cuts which it is desired to obtain. The vapors and liquid thus separated as the result of the preliminary fractionation are not in equilibrium relation to each other, but on the contrary are separated by a distinct gap not character-

izing the relation of a liquid and its equilibrium vapor.

The vapors and liquid resulting from the aforesaid preliminary fractionation are now introduced, respectively, into a secondary counterflow rectifying column at different levels therein, the selection of these levels being most desirably such that each of the so-introduced products is approximately in equilibrium with the liquid and vapor present in the column at the level where that product is introduced. This is an optimum condition, however, and not an absolutely rigid requirement of the process. By properly controlling the reflux to the secondary column, it is possible under these conditions of operation to withdraw kerosene from a point or points in said secondary column intermediate the levels at which the preliminary-separated vapors and liquid are respectively introduced thereinto.

By way of further explanation of the principles underlying the invention, an example illustrating a practical way in which the invention may be applied to the distillation of a crude oil will now be given, reference being made to the accompanying drawing which illustrates an apparatus system suitable for use in carrying out the new process. Said drawing, which is to be understood as being more or less diagrammatic in character, illustrates such a system of apparatus in side elevation.

The crude oil to be distilled is first brought to a temperature sufficiently high to insure vaporization of the desired constituents thereof at substantially ordinary atmospheric pressure. This may be accomplished in various ways. One convenient way of heating the oil is to force it through the coils of a pipe still, not shown, in which it attains a temperature sufficiently high to vaporize desired constituents thereof. The crude oil thus superheated in the pipe still is discharged through pipe or inlet 27 to the bottom of the dephlegmator or other suitable countercurrent fractionating apparatus 26. Vapors of the desired constituents of the crude are flashed off and are then subjected to a preliminary fractionation in the countercurrent fractionating apparatus 26 to obtain a liquid product and a vapor product, the vapors consisting mainly of constituents lighter than kerosene, and the liquid product consisting mainly of constituents heavier than kerosene. The uncondensed vapors pass out through the vapor outlet pipe 28 to a partial condenser 29 where the higher-boiling portion thereof is condensed to provide reflux which returns to the top of the dephlegmator or column 26 through the trapped return pipe 30. The vapors not condensed in the partial condenser 29 pass on through the vapor line 31 and enter the upper part of a secondary fractionating column 1. The liquid from the bottom of the preliminary fractionating column or dephlegmator 26 flows through the trapped pipe 32 and enters the lower part of the secondary fractionating column 1. The relative amounts of the vapor fraction and the liquid fraction produced as a result of the preliminary fractionation which goes on in the dephlegmator 26, according to the well-known principles of repeated evaporation and condensation, depend upon the rate of the supply of cooling medium to the partial condenser 29. This should be regulated to the point where substantially all of the gasoline constituents pass on as vapor, carrying with them some of the kerosene constituents. The operation of the preliminary fractionating column or dephlegmator 26 should be so regulated and controlled that the overhead or vapor product leaving through pipe 31 contains substantially no material boiling above a predetermined temperature, say 500° F. in the present example. The remainder of the kerosene constituents and all higher-boiling material will pass through the connecting pipe 32 to the lower part of the column 1.

The aforesaid vapor from the top and the liquid from the bottom of preliminary fractionating column or dephlegmator 26 are now further treated in the countercurrent fractionating apparatus 1 in order to produce the desired sharply-cut final product. In the upper part of the fractionating column 1, which contains rings or other known packing material, or plates with bubble-caps, or any suitable means for bringing vapor and liquid into intimate contact, the material contained in the vapors entering through pipe 31 and boiling above the desired end point of the gasoline that is to be made, is removed from the vapors by countercurrent scrubbing and flows downwardly through said tower or column. On the other hand, the light material boiling below 500° F. which is contained in the liquid entering column 1 through pipe 32, is vaporized and passes upwardly through said column. Such gasoline constituents as are present in this vapor pass for the most part to the top of the column without being liquefied, while most of the constituents of said vapor boiling below 500° F. but above the desired end point of the gasoline collect as liquid on a plate or plates in column 1 intermediate the points at which pipes 31 and 32 enter said column, together with the products boiling between 450° and 500° F. that were contained in the vapors entering said column through pipe 31. Therefore one or more sharply cut products, intermediate gasoline on the one hand and gas oil on the other, and containing but a small proportion of the already small proportion of light material in the vapor coming from the lower part of the column, can be withdrawn as a liquid from the secondary fractionating tower or column 1. Such

an intermediate product, assumed in this instance to be a kerosene, collects as a liquid on reservoir plate 11 located at an intermediate point in the column 1, as shown, most desirably about midway between inlets 31 and 32. This liquid is prevented from descending through plate 11 by the bell 12 which at the same time permits vapors to rise through it. This liquid is withdrawn through pipe 13 which leads to the divided-flow box 6'. This divided-flow box may be of any suitable type and its function is to divide the stream of condensate which flows from plate 11 into two streams in any suitable determined proportion, one stream flowing through the seal 14 and the outflow pipe 15 to the tank 16, the other stream flowing through the return pipe 17 into the column 1 at a point a little below the plate 11 to take part in the fractionating.

It will be noted that the end point of the material leaving the primary column 26 as vapor can be very definitely controlled, whereas the initial boiling point of the liquid material leaving the bottom of the primary column can not be controlled to a similar extent as a practical matter, said liquid material being mostly gas oil but containing constituents boiling all the way down into the gasoline range. But since substantially all the gasoline constituents at points in the secondary column 1, above the point at which this liquid product of the preliminary fractionation is introduced, will be in vapor form as already pointed out, a kerosene cut practically free of gasoline may be drawn off in liquid form.

The liquid coming from the base of the primary column or dephlegmator 26 may be assumed to be at a temperature approximating 500° to 525° F. In order to remove the kerosene and even all the gasoline from this liquid by ordinary distillation methods, it would be necessary to raise the temperature of the liquid to about 620° F. which, of course, would cause more or less heavy material to boil off in addition to the light products sought. According to the present method, however, this heavier material is separated by countercurrent scrubbing and made to pass downwardly in a manner similar to that by which the heavier material is cut out of the vapors in the upper part of column 1.

The heavy material collects in the lower portion of the column 1 and the fractionation here exhausts this material of practically all kerosene constituents. The heat necessary to maintain proper operation of column 1, in addition to that entering at 31 and 32, is supplied to the bottom as follows: The level of liquid in the bottom of column 1 is held constant by having a large enough outlet pipe 18 at a definite distance from the bottom of the column 1 so that the residual fraction, stripped gas oil, continually overflows through the outlet pipe 18 and seal 19 to the tank 20. The pump 21 draws oil through the feed pipe 22 which is lower than the outlet pipe 18 and passes it through the heating coil 23 in the furnace 24 and back through the pipe 25 into the column 1. By reason of the preliminary fractionation effected in column 26 the heating coil 23 is required to handle very much less material than would otherwise be necessary and hence a substantial saving in operation and equipment is made possible.

The light vapors leaving the top of column 1 pass by vapor pipe 3 to a total condenser 4 where they are condensed and run as liquid through inflow pipe 5 to a divided-flow box 6 which functions to divide the stream of condensate into two streams in any suitable determined proportion. One of these streams will flow by seal 7 and outflow pipe 8 to tank 9 and will consist of gasoline of a boiling range which will depend on the ratio between the two streams of divided condensate and upon the particular kind and quantity of the contact-producing media in the upper part of the column 1. The other stream of divided condensate flows through the backflow pipe 10 into the upper part of the column 1 to maintain the proper countercurrent fractionating conditions therein.

The operation of the apparatus can be controlled by regulating the temperature in various parts of the apparatus and by regulating the amount of reflux returned to column 1.

It is to be understood that the invention in its broader aspects is not restricted to the details of operation given above, and that the specific embodiments of the process described in explaining the underlying principles involved are merely illustrative and are not intended to be limiting.

What is claimed is:

1. In the distillation of mineral oil for production therefrom of a gasoline fraction, a gas oil fraction, and an intermediate fraction, the process which comprises heating mineral oil to a temperature sufficiently high to vaporize desired constituents thereof, effecting a preliminary fractionation of said vapors to obtain on the one hand vapors consisting mainly of constituents lower boiling than kerosene, and on the other hand liquid consisting mainly of constituents higher boiling than kerosene, introducing the thus separated vapors and liquid, respectively, into a countercurrent fractionating column at different points, condensing vapor products issuing from the upper part of said column and reflecting to the upper part of said column a part of the resultant condensate, and withdrawing from a point in said column intermediate those at which said separated vapors and liquid were introduced a liquid product of boiling range intermediate those of gasoline and gas oil, respectively.

2. The process of distilling mineral oil as defined in claim 1, further characterized by

the fact that the preliminarily separated vapors and liquid are introduced into said column at points where they are, respectively, in approximate equilibrium with the vapor and liquid in the column at those points.

3. In the distillation of mineral oil for production of cleanly separated gasoline, kerosene, and gas oil fractions therefrom, the process which comprises superheating mineral oil, flashing off vapors therefrom, effecting a preliminary fractionation of said vapors to obtain on the one hand vapors consisting for the most part of constituents lighter than kerosene, and on the other hand liquid consisting for the most part of constituents heavier than kerosene, introducing the thus separated vapors and liquid, respectively into a fractionating column at different points, refluxing to the upper part of said column a part of the vapor products leaving the top thereof, and withdrawing a kerosene product in liquid form at a point in said column intermediate those at which said separated liquid and vapor were introduced.

4. In the distillation of mineral oil for production therefrom of a relatively low boiling fraction, an intermediate fraction, and a relatively high boiling fraction, the process which comprises heating mineral oil sufficiently high to vaporize desired constituents thereof, effecting a preliminary fractionation of said vapors to obtain on the one hand vapors consisting mainly of constituents lower boiling than said intermediate fraction, and on the other hand liquid consisting mainly of constituents higher boiling than said intermediate fraction, introducing the thus separated vapors and liquid, respectively, into a countercurrent fractionating column at different points, condensing vapor products issuing from the upper part of said column and refluxing to the upper part of said column a part of the resultant condensate, and withdrawing said intermediate fraction from a point in said column intermediate those at which said separated vapors and liquid were respectively introduced.

5. A distilling process which consists in vaporizing mineral oil, fractionating the oil vapors to obtain therefrom two separate fractions, one of which is a liquid containing most of the high-boiling portion of said vapors, and the other of which is in vapor form and includes most of the low-boiling portion, further fractionating the vapor fraction by introducing the same into a fractionating column at a point in the upper part thereof, further fractionating the liquid fraction by introducing the same into said fractionating column at a point in the lower part thereof, leading off low-boiling vapors from the top of the column, withdrawing a high-boiling liquid from the base of the column, and withdrawing an intermediate-boiling liquid from said column at a point between the points at which the aforesaid vapor fraction and liquid fraction are respectively introduced.

6. In a column for fractionating mixed fluids, means for withdrawing from the bottom of said column a part at least of the liquid formed by condensation therein, means for heating the withdrawn liquid, and means for passing said withdrawn liquid through said heating means and back into said column, in combination with means for fractionating the original mixed fluids into a liquid fraction and a vapor fraction previous to their entry into said column, and means for introducing said liquid fraction and said vapor fraction individually into said column at two different points.

7. In a column for fractionating mixed fluids, means for withdrawing from the bottom of said column a part at least of the liquid formed by condensation therein, means for heating the withdrawn liquid, and means for passing said withdrawn liquid through said heating means and back into said column, in combination with means for fractionating the original mixed fluids into a liquid fraction and a vapor fraction previous to their entry into said column, means for introducing said liquid fraction and said vapor fraction individually into said column at two different points, and means for withdrawing a liquid from said column at a point intermediate those at which said separated liquid and vapor fractions were introduced.

8. In the process of continuously fractionating mixed fluids the step which comprises withdrawing from the bottom of said fractionating means a part at least of the liquid formed therein by condensation, and passing the withdrawn liquid continuously through heating means and thence at an increased temperature back into said fractionating means while separating said mixed fluids into a liquid and a vapor previous to their entry into said fractionating means, introducing said previously separated liquid and vapor individually into said fractionating means at different points, and withdrawing condensate from said fractionating means at a point intermediate the points of entry of said previously separated liquid and vapor.

9. The process of continuously fractionating mineral oil which comprises heating mineral oil to form a mixture of liquid and vapor, separating said liquid and vapor, fractionating the vapor by introducing the same into a fractionating column at a point in the upper part thereof, fractionating said liquid by introducing the same into said fractionating column at a point in the lower part thereof, condensing vapor products issuing from the upper part of said column and refluxing to the upper part of said column a part of the resultant condensate, withdrawing a high-boiling liquid from the lower part of said

column, passing a part of said withdrawn liquid continuously through heating means and thence at an increased temperature back into said fractionating column, and withdrawing an intermediate-boiling liquid from said column at a point intermediate those at which the separated liquid and vapor were respectively introduced.

In testimony whereof I hereunto affix my signature.

JOHN V. E. DICKSON.