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J. E. BELL

FRACTIONAL DISTILLATION OF PETROLEUM

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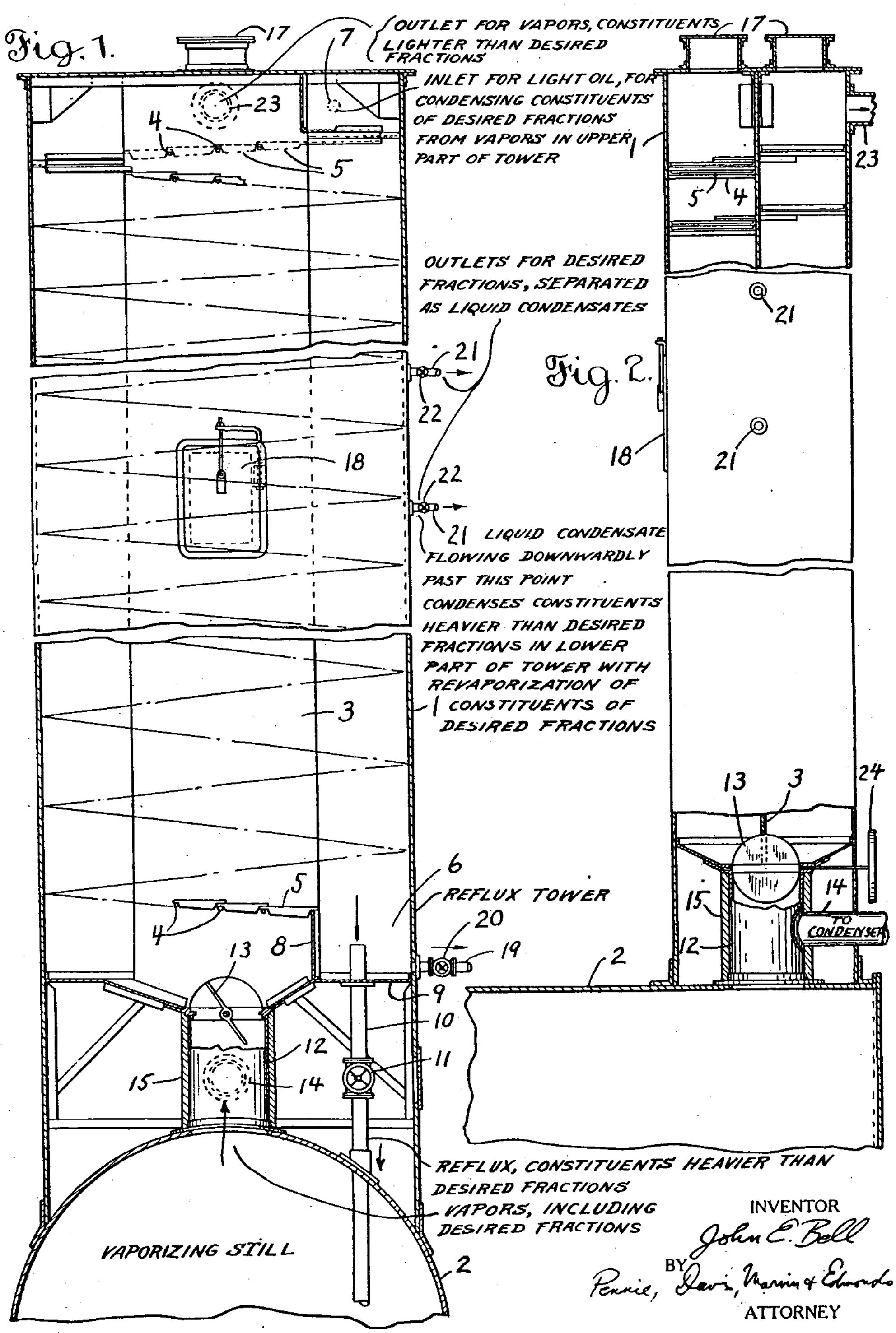

INVENTOR
John E. Bell
BY Pennie, Davis, Marvin & Edmonds
ATTORNEY

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JOHN E. BELL, OF BROOKLYN, NEW YORK, ASSIGNOR TO SINCLAIR REFINING COMPANY, OF CHICAGO, ILLINOIS, A CORPORATION OF MAINE.

FRACTIONAL DISTILLATION OF PETROLEUM.

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This invention relates to an improved method of fractional distillation of petroleum oils and more particularly to a fractional distillation process in which fractions of intermediate boiling point are separated from the oil.

According to the present invention, the oil from which an intermediate fraction or cut is to be recovered is subjected to distillation, with refluxing of the vapors, and there is introduced into the top of the reflux tower a light distillate which will be re-evaporated in the upper portion of the tower and which will condense the heavier vapors arising in the tower; and this operation is so regulated that a fraction or cut of substantially constant composition is obtained and drawn off at an intermediate point in the tower.

The invention is of more or less general application to the fractional distillation of petroleum oils where a heavier or intermediate fraction or cut of the oil is desired. The invention is of special value, however, in the separation of lubricating oil cuts or fractions from heavier petroleum oils or distillates, where such lubricating oils are desired relatively free from constituents of higher and lower boiling points.

In the practice of the invention, a petroleum oil containing the desired fraction is subjected to distillation until vapors of the desired fraction are produced. These vapors, together with heavier vapors, are subjected to a regulated refluxing operation, with introduction of lighter distillate into the top of the reflux tower. This lighter distillate will serve to condense the vapors of the desired fraction, and the condensed vapors will flow downwardly in the tower and will in turn condense heavier vapors and themselves be revaporized. This condensing and vaporizing operation will take place throughout the height of the reflux tower so that the heaviest vapors will be returned as liquid to the still, while the lightest vapors will be removed in vapor form from the top of the tower, and the vapors of the desired distillate or distillates, in excess of the amounts required for alternate condensation and reevaporation in the tower, will be removed at an intermediate point, or at intermediate points, of the tower. Vapors of the desired distillate or distillates will extend upwardly into the tower above the point at which the distillate is withdrawn and will serve to vaporize the lighter constituents and will themselves be condensed. The desired distillate in the form of liquid will also flow downwardly in the tower below the point or points from which it is withdrawn and will serve to condense the heavier vapors and will itself be revaporized. The heavier vapors from the still will thus be condensed in the lower portion of the tower, while the vapors of the desired distillate that enter the tower from the still can be withdrawn from an intermediate portion of the tower in the manner described.

The invention will be further described in connection with the accompanying drawings illustrating a suitable apparatus in which the invention can be practised.

In the accompanying drawings, Fig. 1 shows, in a somewhat conventional and diagrammatic manner, a still and reflux tower with parts broken away, and Fig. 2 is a similar view taken at right angles to that of Fig. 1.

The apparatus illustrated comprises a still shell 2, having a reflux tower 1 mounted thereon. This reflux tower is of the construction illustrated and described in my copending application Serial No. 703,338 filed April 1, 1924. The reflux tower is of a general helical construction with an oblong or oval cross section. It has a central plate 3 dividing the two sides of the tower from each other and a series of trays or pans 5 supported at the sides of the tower on cross rods 4, and suitably secured at the turns of the tower as by welding to the shell 1. These trays or pans 5 form a path of flow for the oil which is of a general helical contour and the successive turns of the helix form between them a helical passage for the upward flow of vapors.

The liquid flowing down over the helical surfaces collects in the reservoir 6 at the bottom of the tower and overflows through the pipe 10 to the still 2. The pipe 10 has a regulating valve 11 therein for shutting off the connection when desired. The reflux reservoir 6 has a side plate 8 and a bottom plate 9 forming two of the walls thereof. An outlet 19 with valve 20 therein permits withdrawal of liquid from the reflux reservoir where this is desired.

The tower is provided with an inlet pipe 7 for the introduction of feed oil in the form of light distillate and is provided with an outlet pipe 23 for the escape of light vapors. Manholes 17 at the top of the tower and side manholes 18 on the side permit access to the interior of the tower when desired.

The tower is connected with the still by the vapor pipe 12 which has a butterfly valve 13 at its upper end for disconnecting the tower when desired, this butterfly valve being operated by a handle 24, and a side connecting pipe 14 having a valve therein (not shown) for permitting direct connection from the still to a condenser. A layer of heat insulation 15 is shown around the vapor outlet pipe 12 and it will be understood that similar insulation (not shown) may protect the outside of the tower as a whole.

At one or more intermediate portions of the tower outlet pipes 21 are provided having valves 22 therein. These pipes connect with depressions formed in one or more of the trays or pans 5 to permit the drawing off of liquid therefrom.

While the process of the invention can be practised in reflux towers of different types and constructions, yet a helical tower such as that illustrated is of particular value, because of the intimacy of contact which it enables to be secured between the vapors and liquid. The helical construction of the tower gives a prolonged path of contact of the vapors and liquid. The vapors and liquid come into direct contact with each other throughout their entire path, and in addition the liquid and vapors are in heat interchanging relation through the walls of the pans forming the helix. This heat interchange through the walls of the helical surface is particularly effective because the vapors are a complete turn removed from the liquid of the turn above and an increased temperature difference accordingly exists between the liquid and vapors. The vapors accordingly are cooled both by the liquid with which they come into direct contact and with the cooler liquid in the next higher turn of the helix, while the liquid in turn is heated by the vapors which come in direct contact therewith and in addition is heated by the vapors of higher temperature in the next lower turn of the helix. In this way a close fractionation is made possible since the liquid constituents of lower boiling point will tend to be vaporized first from the down-flowing liquid and the heavier vapors will tend to be condensed to liquid.

The still used in carrying out the process may be of different types and constructions, but an ordinary shell still is suitable. Only the upper portion of the shell of such a still is illustrated in the drawing but it will be understood that such a still may in practice be of relatively large size and capacity, for example, 14 feet in diameter and 40 feet long. With such a still the reflux tower may for example have a height of around 30 feet and may have cross-sectional dimensions of about 6 x 12 feet. In the production of a lubricating oil cut or fraction, from crude oil, the still may be charged with crude oil or with reduced crude and the light constituents first removed therefrom. The helical tower permits a close fractionation to be obtained and a sharp cut between the fractions. All of the constituents going into a 450 end point gasoline may thus be first removed from the crude oil and obtained as a gasoline distillate. The distillation may then be continued and a gas oil distillate obtained, and the operation continued to such a point that lubricating oil vapors are formed in the still and driven up into the reflux tower.

When the distillation has progressed to such an extent that the lubricating oil is thus being vaporized, a light distillate is introduced into the top of the tower through the pipe 7. Such a light distillate, such as a distillate which has previously been taken out as an overhead distillate from the crude oil, will be heated by contact with the vapors rising into the upper part of the tower, and will be re-evaporated in the upper portion of the tower. This heating and re-evaporation of the light distillate will in turn condense the heavier vapors with which the light distillate comes in contact and these condensed vapors will flow down as liquid through the tower where they will be re-heated by contact with the heavier up-rising vapors and will condense and return them toward the bottom of the tower. The vapors forming the desired lubricating oil fraction, as they rise into the upper portions of the tower, will be cooled and condensed to liquid by contact with the cooler liquid flowing down through the tower, while the lighter liquid constituents will be vaporized from the condensed vapors until, at some intermediate point in the tower, the down-flowing liquid will be of the composition desired in the lubricating fraction or cut. This oil is then drawn off from this intermediate portion of the tower, for example, at one or another of the outlet pipes 21 in the tower construction shown.

All the reflux or lubricating oil fraction flowing downwardly is not thus removed as it is necessary for a portion to continue on down over the lower pans in the tower and in re-evaporating to throw down and condense the still heavier vapors leaving the still. These re-evaporated portions of the desired lubricating oil cut, re-evaporated in the lower portion of the tower below the lubricating oil outlet, will again pass upwardly in the tower and will be condensed in the upper part of the tower, above the outlet from which a part of the lubricating oil is withdrawn, and these condensed constituents will again flow back toward the still.

A part of the desired lubricating oil fraction is thus kept in circulation in the tower, being vaporized in the lower portion of the tower and serving to condense and throw back the heavier constituents, and the vapors being then condensed in the upper portion of the tower and serving to vaporize the still lighter constituents. The condensate or reflux returning from the tower to the still will accordingly be substantially free from lubricating oil constituents, or will be relatively much lower in such constituents, while the vapors rising from the still into the reflux tower will be relatively richer in vapors of the desired lubricating oil fraction. The additional vapors thus entering the tower can be removed as the lubricating oil fraction or cut in the manner above described. That is, an approximately constant amount of the lubricating oil vapors and liquid is kept in circulation in the tower, to maintain a condition of equilibrium therein, while the additional lubricating oil fraction entering the tower with other vapors, and particularly with heavier vapors, is removed from the tower in the manner above described. The portion of the lubricating oil fraction remaining in the tower, or a relatively fixed amount of such lubricating stock vapors and liquid, serves to keep up the operation of the reflux tower, vaporizing and maintaining in vapor form, at a point above the lubricating oil outlet, the vapors of lower boiling point than the desired lubricating oil cut and condensing and returning to the still the heavier vapors, heavier than the desired lubricating oil cut. A condition of equilibrium or of approximate equilibrium is thus maintained in the tower, withdrawing off of the intermediate lubricating oil fraction at an intermediate portion of the tower, condensing and returning to the still the heavier vapors, and vaporizing and removing as distillate the lighter vapors introduced as distillate into the top of the tower. The lubricating oil vapors are too heavy to go overhead as distillate and too light to run back into the still as reflux so that the excess of lubricating oil coming from the still, over and above that required to maintain the operation of the tower, is withdrawn at an intermediate point as the desired lubricating oil cut or fraction.

By feeding into the top of the tower a lighter distillate than the desired lubricating oil distillate, such as gasoline, this lighter distillate will all be vaporized in the upper portion of the tower so that no lighter constituents will be present in the lubricating oil drawn off. The light distillate introduced at the top of the tower is also free from heavier constituents, heavier than those desired in the lubricating oil, so that this light distillate will not introduce undesirable heavier constituents into the lubricating oil.

The lubricating fraction of crude oil is obtained from the vapors containing crystalline wax. Such vapors come over from the still with the vapors carrying the amorphous wax. The vapors containing the amorphous wax, however, are heavier and are first condensed in the lower part of the reflux tower.

In order to obtain the vaporization of the cut carrying the amorphous wax, the temperature required in the still will usually be sufficient to crack some of the heavier constituents, since some cracking will take place after a temperature of around 650° F. has been obtained. This cracking of the oil during the recovery of the lubricating oil cut or fraction results in the formation of a small amount of light materials, lighter than the desired lubricating oil fraction, which lighter materials will go into either gasoline or light gas oil and will pass off from the top of the tower.

With the continuation of the distillation, accordingly, the lighter constituents will be driven off with the overhead distillate, the desired lubricating oil fraction will be obtained as a heart cut or intermediate cut, from the intermediate portion of the reflux tower, and the heavier vapors, such as those containing the amorphous wax and which are not desired in the lubricating oil, are condensed and returned to the still and are subjected to cracking therein, with the production of lighter constituents which are removed in the manner described.

The distilling operation can thus be continued, with the removal of the lubricating oil fraction, and refluxing and return and cracking of the heavier constituents of the oil, until the greater portion of lubricating oil has been removed, although the extent to which the cracking of the heavier oil is carried can be regulated and limited and the operation discontinued before all of the lubricating oil has been recovered.

Where crude oil is used as the charge for the still, the still may be first charged partly full of the crude oil and heated to drive off the lighter distillate and additional feed may then be introduced into the top of the reflux tower. If this feed is introduced after all or approximately all of the lighter vapors have been driven out of the still charge, and if the rate of feed of crude oil is properly regulated, practically all of the lighter constituents of the crude oil can be recovered as an overhead distillate so that only the heavier oil will enter the still. The still charge of heavier oil will in this way increase until the still has been filled to the point which is predetermined as the maximum level. If the gasoline and gas oil have been removed in this way, the still will then be full of heavier oil from which the lubricating cut can be obtained in the manner described.

Instead of taking out both the gasoline and gas oil distillates in the manner just described, only the gasoline distillate may be thus removed. The still may thus be filled partly full of crude oil, the fires started and vapors driven up into the tower and the lighter vapors passed over to the condenser until all of the gasoline is driven off. With continued rise in temperature, the feed of crude oil to the top of the tower will be started and this oil will be fed in slowly at first, with regulation of the feed to maintain a vapor temperature, which will insure the removal of all of the gasoline as distillate. As the still gets hotter the rate of feed will increase and the reflux from the tower will gradually fill the still. The still may then be heated to distill off the gas oil and the distillation continued until the gas oil is removed and until lubricating oil vapors are formed and enter the reflux tower. The feed of light distillate into the top of the tower is then started and with continued heating of the still and vaporization of lubricating oil, the cold distillate fed into the top of the still will serve to condense and throw down the heavier vapors and prevent the lubricating oil vapors from escaping at the top of the still while the vapors heavier than those desired in the lubricating oil will be condensed in the lower portion of the still, in the manner described, and returned to the still. The small proportion of light materials formed from the cracking of the amorphous wax and heavier constituents will pass off with the re-vaporized distillate from the top of the tower. The heavier vapors from the still will be refluxed and returned to the still. From an intermediate point, the lubricating oil cut will be removed from the tower, this intermediate point being such that the lubricating oil vapors rising above such point will in condensing, vaporize the lighter constituents flowing downwardly as liquid in the upper portion of the tower, and the intermediate point being also such that a part of the lubricating oil, flowing downwardly below such intermediate point, will condense and throw down the heavier vapors which are not desired in the lubricating oil fraction.

It will thus be seen that the obtaining of the desired lubricating oil fraction involves the introduction of light distillate into the top of the tower, lighter than the desired lubricating oil, and the introduction of lubricating oil vapors into the bottom of the tower, together with vapors heavier than the lubricating oil vapors; and that the operation is so regulated that the heavier vapors are condensed in the lower part of the tower, while the lighter distillate is re-evaporated and removed from the upper portion of the tower, and the lubricating oil itself, in excess of that required to maintain the intermediate zone of lubricating oil vapors and liquid, is drawn off at an intermediate point from the tower. In this way, after the condition of equilibrium has once been established in the tower, all or practically all of the lubricating oil vapors leaving the still and entering the tower can be removed from the tower as the lubricating oil cut, these vapors being too heavy to go overhead and too light to run back into the still, and the cooling effect of the distillate introduced at the top of the tower neutralizing the heating effect of the still so as to maintain the equilibrium conditions desired for taking out the desired fraction or cut from the tower. The light vapors leaving the top of the tower contain the full amount of the light vapors that were pumped into the tower as a cooling medium, as well as such additional amount of light vapors as are formed in the still by the cracking of part of the still charge. In effect, this light distillate may also be considered to circulate in the tower, much as the lubricating oil itself circulates. That is, the distillate is introduced as liquid into the top of the tower and is re-evaporated and removed as vapor from the top of the tower so that the distillate thus introduced does not flow downwardly to the portion of the tower from which the lubricating oil cut is being removed. The light distillate thus introduced and vaporized, nevertheless, exerts a desired cooling and condensing effect and condenses and returns the lubricating oil vapors which rise into the upper portion of the tower, so that the condensed lubricating oil can be in part drawn off from an intermediate point or points of the tower, and in part will serve to cool and condense the heavier vapors in the lower part of the tower.

The invention is also applicable to the obtaining of other intermediate fractions from crude oil or crude oil distillates. In order to obtain such an intermediate distillate, the oil containing such intermediate distillate together with heavier constituents is subjected to a distillation and refluxing operation similar to that above described and light distillate, lighter than the desired fraction, is introduced into the top of the reflux tower. This light distillate will serve to cool and condense the vapors of the desired distillate, while the light distillate itself will be revaporized in the upper portion of the tower. The condensed vapors of the desired distillate will flow downwardly in the tower, and, in the lower portion of the tower, will be revaporized and will serve to cool and condense the heavier vapors, which will thus be returned to the still while the desired distillate can be recovered as an intermediate or heart cut from an intermediate portion of the tower.

The operation is so regulated that the light distillate fed into the top of the tower will, on being re-evaporated, supply a sufficient cooling effect to cause the heavier vapors to be returned to the still, leaving the desired fraction to be drawn off at an intermediate point. This desired fraction will be too heavy to go overhead with a light distillate and will be too light to be returned to the still. It will accordingly circulate in the central portion of the tower, being condensed by the lighter distillate in the upper portion, and being re-evaporated by the heavier vapors in the lower portion, while the additional amount of vapors of the desired fraction which enter the bottom of the reflux tower from the still, in excess of the amount required for the operation of the tower, can be withdrawn from an intermediate point or points, in the manner above described.

While I have described the invention as applied to the production of a single distillate or cut from an intermediate point of the reflux tower, yet the invention is also applicable for obtaining a plurality of fractions or cuts from various intermediate points in the height of the reflux tower. In general, the lightest cut or fraction will be heavier than the light distillate entering at the top of the tower while the heaviest fraction will be lighter than the heaviest vapors rising from the still. Each of these intermediate fractions will be made up of condensed vapors, condensed in the portion of the tower above the point from which the distillate is withdrawn. The condensed vapors, however, as they flow downward in the tower, will be subjected to the heating action of the upflowing vapors, and the lighter constituents will be reevaporated further up in the tower than the heavier constituents, while the heavier constituents will be condensed lower down in the tower than the lighter vapors. With a helical tower of the construction and operation illustrated and described, a close fractionation can be obtained, and a plurality of intermediate cuts or fractions can be withdrawn from intermediate points in the tower.

It will thus be seen that the invention is of more or less general application for obtaining intermediate fractions or cuts of the oil undergoing distillation and that these intermediate cuts or fractions are obtained without admixture with heavier constituents inasmuch as the heavier constituents are condensed in the lower portions of the reflux tower, lower than that from which the cut or fraction is withdrawn. The fractions are also relatively free from lighter constituents, since the lighter constituents are re-evaporated in the upper portions of the tower above the point from which the particular cut or distillate in question is withdrawn.

It will further be seen that the invention involves a circulation of vapors within the tower to maintain conditions of equilibrium therein such that the desired fraction or fractions can be withdrawn. This circulation includes a circulation of light distillate which is introduced in liquid form, re-evaporated in the upper portion of the tower, and withdrawn as vapors. It also involves a circulation of the intermediate fraction or fractions which are to be withdrawn, these fractions being vaporized in the lower part of the tower and being condensed in the upper portion of the tower, and being withdrawn from an intermediate portion. The still from which the distillation is taking place will be freed from the vapors of the desired distillate or distillates and from all lighter constituents, while the heavier constituents will be refluxed and returned and may be subjected to cracking with the production of additional amounts of the lighter distillates.

I claim:

1. The improvement in the fractional distillation of crude petroleum oils, which comprises subjecting the oil to a vaporizing operation and removing the gasoline and gas oil constituents therefrom, subsequently continuing the vaporization to vaporize lubricating oil constituents together with heavier constituents, introducing the vapors from the said continued vaporization into the lower end of a reflux tower, independently introducing a hydrocarbon oil lighter than the lubricating oil fraction into the upper end of the reflux tower, whereby the vapors of the lubricating oil rising into the upper part of the tower are condensed therein, causing the condensed lubricating oil constituents to condense heavier vapors in the lower part of the tower with re-evaporation of the lubricating oil constituents therein, and drawing off a part of the liquid lubricating oil fraction from an intermediate part of the tower.

2. The improvement in the fractional distillation of petroleum oils for the recovery of lubricating oils therefrom, which comprises subjecting petroleum oil from which the gasoline and gas oil constituents have been removed to a vaporizing operation, passing the resulting vapors including the lubricating oil constituents together with heavier vapors into the lower end of the reflux tower, independently introducing into the upper end of the reflux tower an hydrocarbon oil lighter than the lightest component of said vapors which will be vaporized and which will thereby condense the vapors of the lubricating oil in the upper part of the tower, causing the condensed lubricating oil to condense heavier vapors in the lower part of the tower with re-vaporization of the lubricating oil, maintaining an intermediate zone in the tower in which lubricating oil vapors are flowing upwardly and condensed lubricating oil is flowing downwardly and withdrawing a part of the condensed lubricating oil from the said intermediate zone.

3. The improvement in the fractional distillation of petroleum oils for the recovery of lubricating oil therefrom, which comprises subjecting heavier oils containing crystalline and amorphous wax to a vaporiz-

ing operation, passing vapors containing such constituents and substantially no constituents lighter than the lubricating oil fraction from the vaporizing operation into the lower end of a reflux tower, independently introducing an hydrocarbon oil lighter than the lubricating oil fraction into the upper end of the reflux tower to condense the vapors of the lubricating oil rising to the upper portion of the tower, causing the condensed lubricating oil to flow downwardly condensing the vapors containing the amorphous wax in the lower portion of the tower, maintaining an intermediate zone in the tower below which the heavier vapors containing the amorphous wax are condensed and above which the light oil introduced into the upper end of the tower is vaporized, and drawing off the lubricating oil containing the crystalline wax from the said intermediate zone of the reflux tower.

4. The improvement in the fractional distillation of hydrocarbon oils, which comprises subjecting an oil containing substantially no components lighter than the desired fractions to a vaporizing operation, passing the vapors therefrom into the lower end of a reflux tower, independently introducing into the upper end of the reflux tower a hydrocarbon oil lighter than the desired fractions, and withdrawing a plurality of fractions from different intermediate portions of the said reflux tower.

5. The improvement in the fractional distillation of hydrocarbon oils, which comprises subjecting an oil containing substantially no components lighter than the desired fraction to a vaporizing operation, passing vapors of the oil from the vaporizing operation to the lower end of a reflux tower, independently introducing an hydrocarbon oil lighter than the desired fraction into the upper end of the reflux tower, maintaining the liquid constituents within the reflux tower in the form of a continuous downwardly flowing film in direct contact on its upper surface with the vapors rising through the tower and in indirect heat exchanging relation on its lower surface with the said vapors, and withdrawing the desired fraction as liquid from said downwardly flowing film at an intermediate point in the reflux tower.

6. The improvement in the fractional distillation of hydrocarbon oils, which comprises supplying oil containing a fraction to be separated and substantially no components lighter than said fraction to a vaporizing operation wherein the said fraction is vaporized together with heavier constituents, subjecting the vapors therefrom to a refluxing operation in direct contact with liquid constituents of said fraction whereby heavier constituents are condensed from the vapors and the liquid constituents of said fractions are vaporized, independently introducing an hydrocarbon oil lighter than the said fraction into a refluxing zone and subjecting the vapors escaping from the first refluxing operation to a continued refluxing operation in direct contact with said lighter hydrocarbon oil, whereby the constituents of said fraction are condensed from the vapors, supplying part of this condensate to the first refluxing operation and withdrawing part of this condensate as the fraction to be separated.

7. The improvement in the fractional distillation of hydrocarbon oils, which comprises supplying oil containing a fraction to be separated and substantially no components lighter than the fraction to be separated to a vaporizing zone and subjecting the oil to a vaporizing operation therein, passing vapors of the oil from the vaporizing zone through a continued refluxing zone, independently supplying oil lighter than the said fraction to the end of the refluxing zone away from the vaporizing zone, and taking off the fraction to be separated from an intermediate point in the refluxing zone.

In testimony whereof I affix my signature.

JOHN E. BELL.