

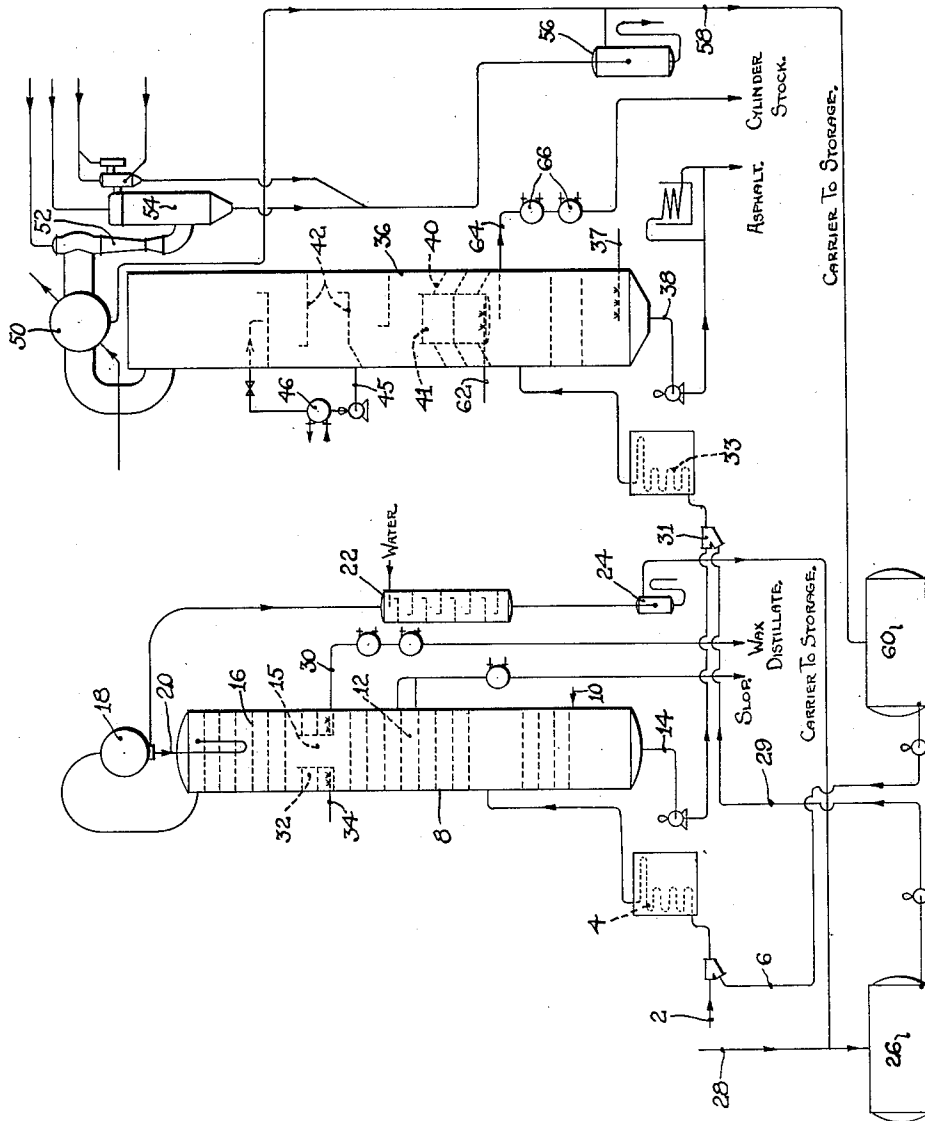
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METHOD OF SEPARATING HEAVY PETROLEUM MIXTURES

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METHOD OF SEPARATING HEAVY
PETROLEUM MIXTURES

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The present invention relates to methods of separating heavy petroleum mixtures, and is an improvement in the process described in my Patent No. 1,905,156, dated April 25, 1933.

My patent describes a process of separating lubricating stock and asphalt from an uncracked bottoms mixture thereof by flash vaporization in the presence of a diluent which is preferably an oil fraction previously obtained from the same crude oil and of materially lower boiling point than the oils to be vaporized. The bottoms mixed with the diluent are heated in a confined stream to prevent any gradual or progressive vaporization, and are then released into a vaporizing zone of low pressure in which all of the oils vaporize as a unit or blanket cut from the asphalt.

In order to obtain the lubricating stock as a separate fraction, it is necessary to subject the vapors to fractional condensation. As pointed out in the patent, however, it may be impracticable, and is in fact unnecessary, to attempt close rectification. It is desirable to avoid the use of a large number of decks which would diminish the vacuum in the vaporizing zone. Accordingly, simple condensation is preferably relied upon. This condensation may be controlled so that the lubricating stock is obtained in substantially pure form uncontaminated by any of the diluent. However, a small amount of the lubricating stock may be left with the diluent when the latter is condensed and the resulting contamination of the diluent may prevent satisfactory use thereof. To separate the diluent from the small quantity of lubricating stock mixed with it would be difficult by ordinary distillation methods, owing to the disproportion in volume of the two materials.

It is one object of the present invention to provide a method of separating the oil vapors to reduce contamination of the diluent to a minimum, and it is another object of the invention to provide a simple and convenient method of restoring the diluent to substantially its original form for re-use in the process, or for other purposes.

With these objects in view, the present invention comprises the method hereinafter described and particularly defined in the claims.

The accompanying drawing is a diagram of the preferred apparatus for practicing the present invention.

The invention is hereinafter described as embodied in a process for separating the components of a bottoms oil mixture from which constituents of the crude lighter than wax distillate have been previously removed.

The feed stock comprising a mixture of wax

distillate (light lubricating stock), cylinder stock (heavy lubricating stock) and asphalt is introduced through a pipe 2 to a still 4. Prior to entrance to the heater, the feed stock is mixed with a quantity of diluent or carrier which, according to my patent, is a material vaporizable under conditions at which vaporization of the cylinder stock is negligible. Such a material is preferably a kerosene having a narrow range of boiling points, about 450–550° F. at atmospheric pressure. The diluent introduced into the feed pipe 2 through the pipe 6 is a material that has been used in the final step of the process and is contaminated by the presence of a small quantity of cylinder stock, as will hereinafter be described. The entire mixture is heated to a temperature preferably between 700° and 725° F. and is then discharged into a column 8 which is preferably maintained under atmospheric pressure. The material entering the column undergoes flash vaporization which is enhanced by the introduction of steam near the bottom of the column, as indicated at 10. The oil vapors are rectified in the usual manner by reflux in the section 12 to effect a separation between the wax distillate and the cylinder stock. The residue withdrawn from the bottom of the column through the pipe 14 comprises a mixture of cylinder stock with asphalt.

The mixed vapors of the wax distillate and diluent pass upwardly through a vapor pipe 15 and are rectified in a section 16. The diluent, which is subject to close rectification, is condensed in an overhead condenser 18, and a portion thereof is returned through the pipe 20 as reflux to the section 16. The remainder of the diluent, together with any uncondensed vapors thereof, is passed through a scrubber 22 wherein the condensation is completed. The condensed diluent and water pass to a decanter 24 from which the completely rectified diluent is removed and carried to a clean storage tank 26. A pipe 28 enters the clean storage tank from a previous step in the process wherein the diluent is originally obtained as a light fraction such as kerosene, preferably having a narrow range of initial to end boiling point, as 450–550° F.

In the column 8, wax distillate is removed as an intermediate product through a pipe 30 which leads from a side stripping section 32 into which steam is introduced at 34 for removal of any remnant of diluent. Inasmuch as this column operates at atmospheric pressure and by the use of steam, a sufficient number of decks can be used to effect as complete a separation as de-

sired between the wax distillate and the diluent.

The residue from the column 8 comprises a mixture of non-volatile asphaltic residuum with valuable heavy lubricating components designated as cylinder stock. This residue is subjected to the process described in my patent, to vaporize the cylinder stock from the asphalt. For this purpose, the residue is pumped through the line 14, in which it is mixed with clean diluent pumped from the clean storage tank 26 through a line 29 to a coupling 31. The entire mixture passes through a pipe still 33 in which it is heated to a temperature sufficient to vaporize all the oils as a unit from the asphalt. This temperature is below the cracking point of any of the materials and preferably about 700° F. The heated mixture is introduced into a final column 36 maintained under a moderate vacuum, preferably an absolute pressure of 75 to 100 mm. of mercury. Upon introduction into the column, the cylinder stock and the diluent vaporize as a unit and the asphaltic residue continues downwardly to the bottom of the column, being subjected to a countercurrent of steam introduced at 37. The asphalt is withdrawn as residue from the bottom of the column through a pipe 38.

The mixed oil vapors pass through entrainment catchers 40, preferably arranged annularly around a stripping section 41 of the type described in my copending application Serial No. 622,209, filed July 13, 1932. The oil vapors continue upwardly through a series of cascade decks 42, illustrated as being four in number. The decks extend inwardly from opposite sides of the column to provide a tortuous path for the flow of vapors. The lowermost deck discharges liquid directly into the stripping section 41. Condensate is withdrawn from one of the lower decks by a pipe 45 and is pumped through a cooler 46 and discharged on the uppermost deck 42 from which it cascades downwardly over the other decks.

The liquid recirculated through the cooler 46 comprises substantially pure cylinder stock in mixture with a smaller quantity of diluent. A constant amount of liquid is continuously recirculated and the condensate in excess of the amount recirculated overflows into the stripping section. The rising vapors comprising the mixture of cylinder stock and kerosene are therefore contacted with cooled liquid of high boiling point and as a result, nearly all of the cylinder stock is condensed. In operation, the vapors passed between the decks 42 are at a temperature of approximately 650°. These vapors consist of a mixture of cylinder stock which, by itself, would condense at approximately that temperature under the vacuum employed and kerosene vapors which, by themselves, would condense at approximately 250°. The recirculated condensate is cooled in the cooler 46 to an intermediate temperature of about 400°, at which temperature its vapor pressure is negligible. The recirculated condensate absorbs heat from the vapor mixture and effectively condenses nearly all of the cylinder stock. At the same time, it condenses only a small quantity of kerosene, the condensing point of which is about 150° below the minimum temperature of the recirculated liquid.

The diluent vapors containing a small remnant of cylinder stock are passed to the overhead condenser 50 and thence into a vacuum jet 52 and a scrubber 54, the condensate being finally passed to a decanter 56, from which it runs

through a pipe 58 to a storage tank 60. This storage tank contains nearly pure diluent contaminated by a small quantity of cylinder stock uncondensed in the passage of the vapors through the contact decks 42. It is this material which is pumped through the line 6 for mixture with the material entering the heater 4 of the column 8.

The liquid passing to the lowermost deck 42 of the column 36 overflows into the stripping section 41 wherein it is subjected to a countercurrent flow of steam introduced at 62. This steam effectively strips from the liquid any slight amount of kerosene that may be condensed in the decks 42. The completely distilled cylinder stock is withdrawn from the stripping section by a pipe 64 and is passed through coolers 66 to storage.

It will be seen that although a close rectification is not attempted in the column 36, the ultimate products are nevertheless completely separated. Any remnant of diluent which finds its way into the cylinder stock because of incomplete fractionation is effectively removed in the stripping section 41. On the other hand, any contamination of the diluent by a small quantity of cylinder stock is of no consequence since the diluent is recycled to the column 8 in which rectification can be carried out to any degree of precision since the column is operated under atmospheric pressure and the back pressure of the rectifying is unimportant.

It will be seen that the present invention provides for the complete separation of heavy materials designated herein as wax distillate, cylinder stock, and asphaltic residuum, without cracking of any of the materials in any stage of the process. The invention also provides for continuous recovery and re-use of the diluent. The diluent returned to the clean storage tank 26 is of the same composition as that originally introduced through the pipe 28. Hence, the diluent may be withdrawn from the clean storage tank and used for any commercial purpose.

Having thus described the invention, what is claimed is:

1. The method of distilling oils which consists in adding a contaminated diluent, formed in a subsequent stage of the process, to a bottoms mixture containing light and heavy lubricating stocks and non-volatile asphaltic residuum, subjecting the mixture to a primary fractional distilling operation to obtain relatively clean diluent as an overhead product, light lubricating stock as a side product and a mixture of heavy lubricating stock and asphalt as residue, mixing relatively clean diluent formed as aforesaid with the residue, heating the mixture, introducing the heated mixture into a vaporizing zone to vaporize the lubricating stock and the diluent as a unit, fractionally condensing the vapors into lubricating stock and contaminated diluent, and recycling the latter to the primary distilling stage to remove therefrom any remnant of lubricating stock.

2. The method of distilling oils which consists in adding a contaminated diluent, formed in a subsequent stage of the process, to a bottoms mixture containing light and heavy lubricating stocks and non-volatile asphaltic residuum, subjecting the mixture to a primary fractional distilling operation to obtain relatively clean diluent as an overhead product, light lubricating stock as a side product and a mixture of heavy lubricating stock and asphalt as residue, mixing relatively clean diluent formed as aforesaid with the

residue, heating the mixture, introducing the heated mixture into a vaporizing zone to vaporize the lubricating stock and the diluent as a unit, maintaining the vaporizing zone under vacuum, 5 fractionally condensing the vapors into lubricating stock and contaminated diluent, and recycling the latter to the primary distilling stage to remove therefrom any remnant of lubricating stock.

3. The method of distilling oils which consists 10 in adding a contaminated diluent, formed in a subsequent stage of the process, to a bottoms mixture containing light and heavy lubricating stocks and non-volatile asphaltic residuum, subjecting the mixture to a primary fractional distilling operation to obtain relatively clean diluent as an 15 overhead product, light lubricating stock as a side

product and a mixture of heavy lubricating stock and asphalt as residue, mixing relatively clean diluent formed as aforesaid with the residue, heating the mixture, introducing the heated mixture into a vaporizing zone to vaporize the lubricating stock and the diluent as a unit, passing the vapors through a condensing zone, withdrawing a part of the condensate from the condensing zone, cooling the withdrawn condensate and returning it to the condensing zone as the condensing medium, thereby condensing lubricating stock as a 10 product, separately condensing diluent contaminated by a small proportion of lubricating stock, and recycling the contaminated diluent to the primary distilling operation. 15

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