May 30, 1933. G. B. COUBROUGH 1,911,993

METHOD FOR DISTILLING HEAVY OILS

Filed Jan. 21, 1930

Fig. 1

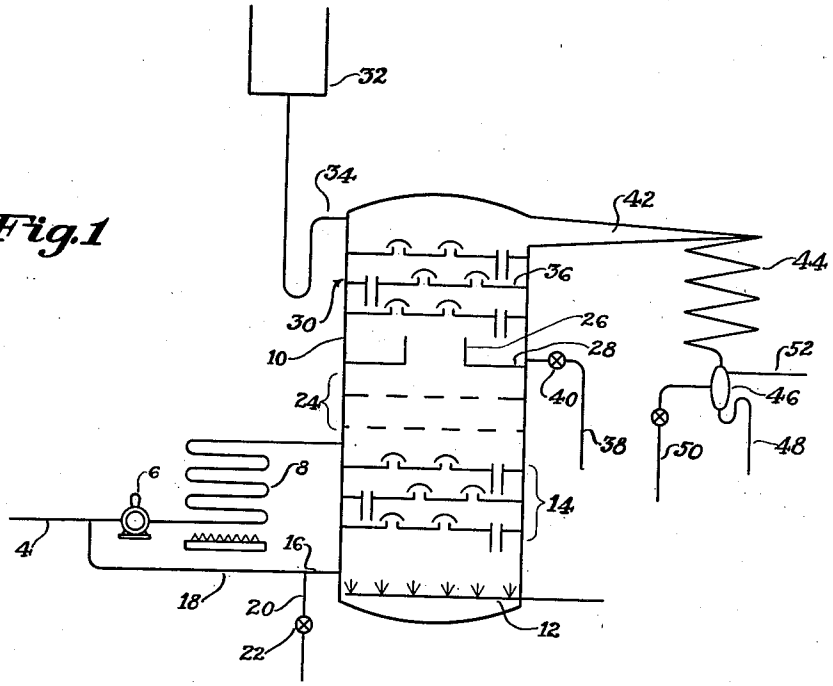
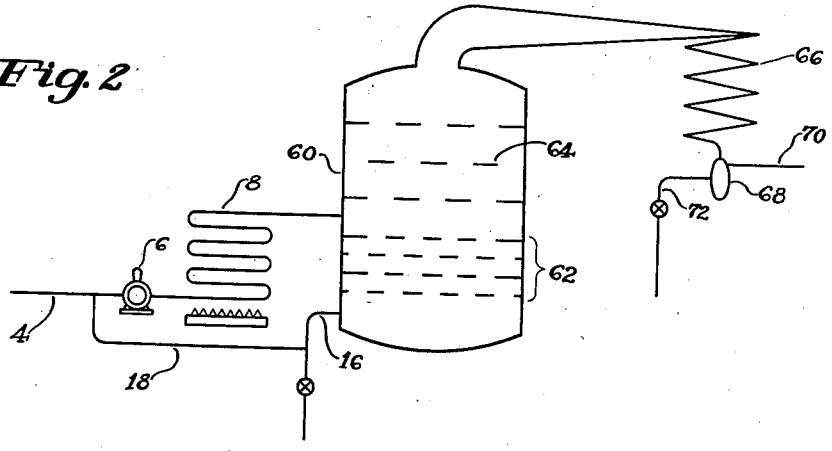


Fig. 2



Witness

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UNITED STATES PATENT OFFICE

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METHOD FOR DISTILLING HEAVY OILS

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The present invention relates to methods and apparatus for distilling heavy oils.

The recovery of heavy oils such as wax distillates from petroleum bottoms is usually attended with difficulties, principally due to the high temperatures which must be employed for distillation and which tend to cause decomposition or cracking. Batch methods of distillation are objectionable in that the temperature increases as vaporization proceeds. Flash vaporization, as practised under existing methods, is not entirely satisfactory because the entire heat of vaporization of the distillate oils is taken from heat stored in the residual oil so that upon vaporization of the desired components, the temperature falls to a point below which vaporization cannot continue. In either case, vaporization of the valuable constituents comes to an end before the available quantity of product is obtained.

The object of the present invention is to provide a method and apparatus for distilling heavy oils from bottoms to obtain the maximum recovery of the valuable constituents and at temperatures not sufficiently high to effect undesirable decomposition.

With this object in view, the present invention makes use of the method which consists in first heating the bottoms from which it is desired to recover one of more oils as a product and introducing the heated bottoms into a column where vaporization is facilitated by vacuum, or by a counter-current of steam. It has always been considered that the most favorable composition of the original mixture for large recovery of distillate comprises a large percentage of the lighter desired oils and only a small percentage of residual product, for the reason that the boiling point of the mixture is not greatly higher than the boiling point of the overhead product alone. I have discovered, however, that the conditions which have heretofore been considered most favorable are in fact the least conducive to complete recovery of the distillate oils and that for maximum effectiveness of distillation, the original mixture should contain only a small proportion of the oils desired as distillate and a relatively large proportion of materials having a substantially lower vapor pressure. When the bottoms which are to be divided contain a relatively small percentage of lighter oils desired as distillate in accordance with the present invention, the remaining relatively unvaporizable components act as a large heat reservoir to maintain the temperature of the mixture against excessive depression upon abstraction of the heat required to vaporize the entire body of distillate oils. An important feature of the invention, therefore, comprises adding to the original bottoms a quantity of relatively unvaporizable heat holding material prior to heating of the mixture and introduction thereof into the vaporizer column, the quantity of heat holding material added being sufficient to maintain the temperature against depression below the boiling point upon vaporization of the lighter oils.

This important feature of the present invention is of particular value in recovering heavy lubricating stock (heavy wax distillate) from bottoms which comprise a mixture of the lubricating stock with asphalt. The asphalt, although itself a non-volatile substance, tends to hold the lubricating stock in such stable equilibrium therewith that recovery of all of the lubricating stock including the heaviest and most valuable end components is by ordinary methods extremely difficult. According to the present invention, such bottoms prior to heating are mixed with a sufficient quantity of heat holding material to make the lubricating stock only a small proportion of the total mixture. Preferably the heat holding material is additional asphalt which forms a part of the residual product and which does not require to be ultimately separated from the asphalt already contained in the bottoms. By this process, the lubricating stock is substantially completely vaporized in a single operation and the residual product comprises nearly pure asphalt holding practically none of the lubricating stock in mixture therewith.

The invention although described above as being applicable to the recovery of heavy lubricating stock from an asphaltic residue is not limitd in its use to such products but may be employed for separating oils from each other whether or not asphalt may be present. Thus the process may be employed for distilling off a light wax distillate, leaving a residue of heavy lubricating stock either with or without asphalt in mixture therewith.

Another feature of the invention comprises a method and apparatus whereby the effect of addition of heat holding material to the bottoms is brought about by recirculation of the residual product through the heater. Thus in the case of separation of heavy wax distillate from asphalt, a predetermined percentage of the asphaltic residue is pumped back to be mixed directly with the bottoms passing to the heater. This feature of the invention effects substantial economies in operation in that the sensible heat of the residue is available for heating the original mixture.

The vaporization step may be carried out under dry vacuum or with steam. In dry vacuum distillation, the addition of the heat holding material acts to raise the boiling point of the mixture, the rise in boiling point, however, being more than offset by the maintenance of temperature afforded by the increased capacity for storing heat. But the use of steam offers the additional advantage that the boiling point of the mixture is not greatly increased by the addition of the heat storing material, and therefore the maximum advantage of the augmented heat storage capacity is obtained. In distilling extremely heavy oils, the use of a vacuum together with steam is to be preferred because the advantages of both temperature methods are availed of, and the oils are well protected against decomposition.

Other features of the invention consist of certain novel features of construction and modes of operation hereinafter described and particularly defined in the claims.

In the accompanying drawing, Fig. 1 is a diagrammatic illustration of the preferred form of apparatus for practising the invention to distill heavy oils with steam and Fig. 2 is a diagram of the preferred form of apparatus for practising the invention under dry vacuum.

The invention is herein illustrated and described as embodied in a method and apparatus for recovering heavy lubricating stock (heavy or amorphous wax distillate) from bottoms which comprise a mixture of the lubricating stock with asphalt.

Referring to Fig. 1 which shows an apparatus for practising the invention with steam, the bottoms are pumped through a supply line 4 by a pump 6 to a heater 8 where they are heated to the maximum safe temperature without decomposition. The heated oil is admitted into a mediate point of a vaporizing column 10 into which the steam is introduced through a perforated steam pipe 12 at the bottom. The heated mixture flows downwardly over boiling cap decks 14 where it is subjected to the ascending steam. In this zone, the lubricating stock is removed from the asphalt, the latter being collected in the bottom of the column and drawn off by a pipe 16. The pipe 16 connects by a pipe 18 with the inlet side of the pump 6 so that a part of the asphalt is recirculated through the pump and heater to be again introduced into the column. The remainder of the asphalt is drawn off as a product through the pipe 20, the amount of recirculated asphalt being controlled by a valve 22 in the offtake pipe.

Above the point of admission of the oil into the column, there are provided a series of entrainment catching decks 24 to remove particles of asphalt which may be carried upwardly by the ascending current of steam and vapor. The mixture of steam and vapor continues through a center pipe 26 which extends upwardly from a collecting deck 28. Inasmuch as it is undesirable to condense the steam-vapor mixture because of the tendency to form emulsions with the wax, the wax distillate is liquefied in the upper section 30 of the column by means of a counter-current of a light non-emulsifiable oil introduced into the top of the column. The non-emulsifiable oil preferably comprises gas oil which is contained in a supply tank 32 and admitted to the column through a pipe 34. A plurality of boiling cap decks 36 are provided in the upper part of the tower. As described in the co-pending application of Coubrough, Serial No. 355,871 filed April 17, 1929, the gas oil displaces the heavy wax distillate from the ascending steam-vapor mixture permitting the wax distillate to collect on the deck 28 from which it is drawn off through a pipe 38 controlled by a valve 40. The mixture of steam and gas oil passes out of the column through a vapor pipe 42 to be condensed in a condenser 44. The condenser discharges into a bottle 46 from which the water and gas oil are drawn off through pipes 48 and 50. If a vacuum is to be employed in combination with the steam, the bottle connects with a vacuum line 52 leading to a suitable vacuum pump. Even with the heaviest oils, the vacuum need not be too high to render the use of boiling cap decks impracticable.

In operation, a sufficient amount of asphalt is recirculated through the pipe 18 to make a large preponderance of asphalt in the supply entering the heater. The return

of asphalt may be adjusted to make the asphalt comprise as much as 90% of the entering mixture. The proportions, having been once established, may be easily maintained by proper regulation of the rate of withdrawal of the asphaltic residue through the pipe 20.

The bottoms are heated in the heater to the maximum safe temperature without decomposition and a sufficient quantity of steam is employed to insure complete vaporization of the lubricating stock. By the present invention, substantially all of the wax distillate is vaporized in a single passage through the counter-current zone 14, the recirculated asphalt containing practically none of the valuable oil constituents. The separation has been found to be so complete that care must be taken to maintain the asphalt in the pipes 18 and 20 at a temperature above the melting point, otherwise an actual solidification of pure asphalt may occur. The recovery of the residue as an asphaltic product capable of solidification upon cooling is indicative of the complete removal of the valuable oil constituents.

The recirculation of the residue comprises in effect the addition of heat storing material to the bottoms. Upon admission of the mixture into the column, vaporization of the oils takes place, owing to the contact of the heated liquid with the steam, and because of the reduction in pressure if a vacuum is employed. Inasmuch as the only heat available for the latent heat of vaporization of the distillate components is the sensible heat of the mixture, a drop in temperature immediately follows. It is to be noted that the steam is not employed to contribute heat to the mixture; its purpose is to facilitate vaporization of the volatile oils, and it may in fact be itself heated by the mixture as it rises through the vaporizing zone.

It will be seen that according to ordinary methods of heavy oil recovery, the temperature drops rapidly until it reaches a point no longer effective to vaporize the distillate components. According to the present invention, however, the presence of a large heat reservoir of substantially less volatile material than the distillate maintains the temperature against depression below the boiling point of the mixture until complete vaporization of the distillate has been effected.

The invention as above described is employed for the recovery of a heavy though volatile oil product from a substantially non-volatile residue. The invention may also be employed to effect a separation between two volatile products, for example, to effect a sharp cut between a light lubricating stock to be obtained as a distillate and a heavy lubricating stock to fall into the residue. In order to prevent depression of the temperature upon vaporization of the lighter oils, the residue is recirculated through the pipe 18. This process is useful both on oils which contain asphalt and those which have no asphaltic content.

In Fig. 2 is shown an apparatus for practising the present invention under dry vacuum. The supply line 4, the pump 6, the heater 8, the residue draw-off pipe 16 and the recirculating pipe 18 are as in Fig. 1. The oil heated in the heater 8 is admitted to the vacuum column 60 at a mediate point and the oil flows downwardly over decks 62 which are preferably of the staggered or overlapping type in order not to seal against the high vacuum. The upper part of the column is provided with entrainment catching decks 64. The vapors pass into a condenser 66 from which the condensate is discharged into a bottle 68 connected with a vacuum line 70. The distillate is drawn off directly by a pipe 72.

The method of distillation under dry vacuum is similar to the steam distillation method described above in that the addition to the bottoms of a material having a high heat storage capacity makes for maintenance of temperature in the column even though substantially complete vaporization of the distillate is carried out.

Although the specific embodiments of the invention are described as relying on the recirculation of the residual product for creating the heat storage reservoir for the mixture, it will be understood that it is not essential to employ the residual material of the process for this purpose although such operation is desirable because of the economy of operation. The heat storage material may be added to the original supply and later recovered from the residue. The use of asphalt as a heat storage medium is to be preferred because of its extremely low volatility, whereby in the specific example given above of recovering heavy wax distillate from the asphalt, no problem of fractionation or rectification is presented.

Having thus described the invention, what is claimed is:

1. A method of recovering oils from petroleum bottoms containing asphalt which consists in adding to the original bottoms a quantity of asphalt having high heat storage capacity, heating the mixture, continuously admitting the heated mixture into a vaporizing zone in counter-current with steam to vaporize the oils by the heat stored in the mixture, the quantity of added asphalt being sufficient to maintain the temperature against depression to a point below which vaporization cannot continue, until substantially complete vaporization of the oils has been effected.

2. A method of separating heavy oils from bottoms containing asphalt which consists in adding to the bottoms a quantity of asphalt to act as a heat storage medium, heating the mixture, and admitting the heated mixture into a vaporizing zone in counter-current with steam, the quantity of asphalt added being sufficient to supply the heat of vaporization of the oils without excessive depression of temperature.

3. A method of recovering oils from petroleum bottoms containing asphalt which consists in adding to the original bottoms a quantity of asphalt having high heat storage capacity, heating the mixture, continuously admitting the heated mixture into a vaporizing zone maintained under vacuum to vaporize the oils by the heat stored in the mixture, the quantity of added asphalt being sufficient to maintain the temperature against depression to a point below which vaporization cannot continue, until substantially complete vaporization of the oils has been effected.

4. A method of separating heavy oils from bottoms containing asphalt which consists in adding to the bottoms a quantity of asphalt to act as a heat storage medium, heating the mixture, and admitting the heated mixture into a vaporizing zone maintained under vacuum, the quantity of asphalt added being sufficient to supply the heat of vaporization of the oils without excessive depression of temperature.

5. A method of separating heavy oils from bottoms containing asphalt which consists in adding to the bottoms a quantity of asphalt to act as a heat storage medium, heating the mixture, admitting the heated mixture into a vaporizing zone maintained under vacuum, the quantity of asphalt added being sufficient to supply the heat of vaporization of the oils without excessive depression of temperature, withdrawing asphalt from the vaporizing zone, and recirculating a part of the asphalt withdrawn to serve as the heat storage medium to be mixed with the original bottoms.

6. A method of separating heavy oils from bottoms containing asphalt which consists in adding to the bottoms a quantity of asphalt to act as a heat storage medium, heating the mixture, admitting the heated mixture into a vaporizing zone in counter-current with steam, the quantity of asphalt added being sufficient to supply the heat of vaporization of the oils without excessive depression of temperature, withdrawing asphalt from the vaporizing zone, and recirculating a part of the asphalt withdrawn to serve as the heat storage medium to be mixed with the original bottoms.

In testimony whereof I have signed my name to this specification.

GEORGE B. COUBROUGH.