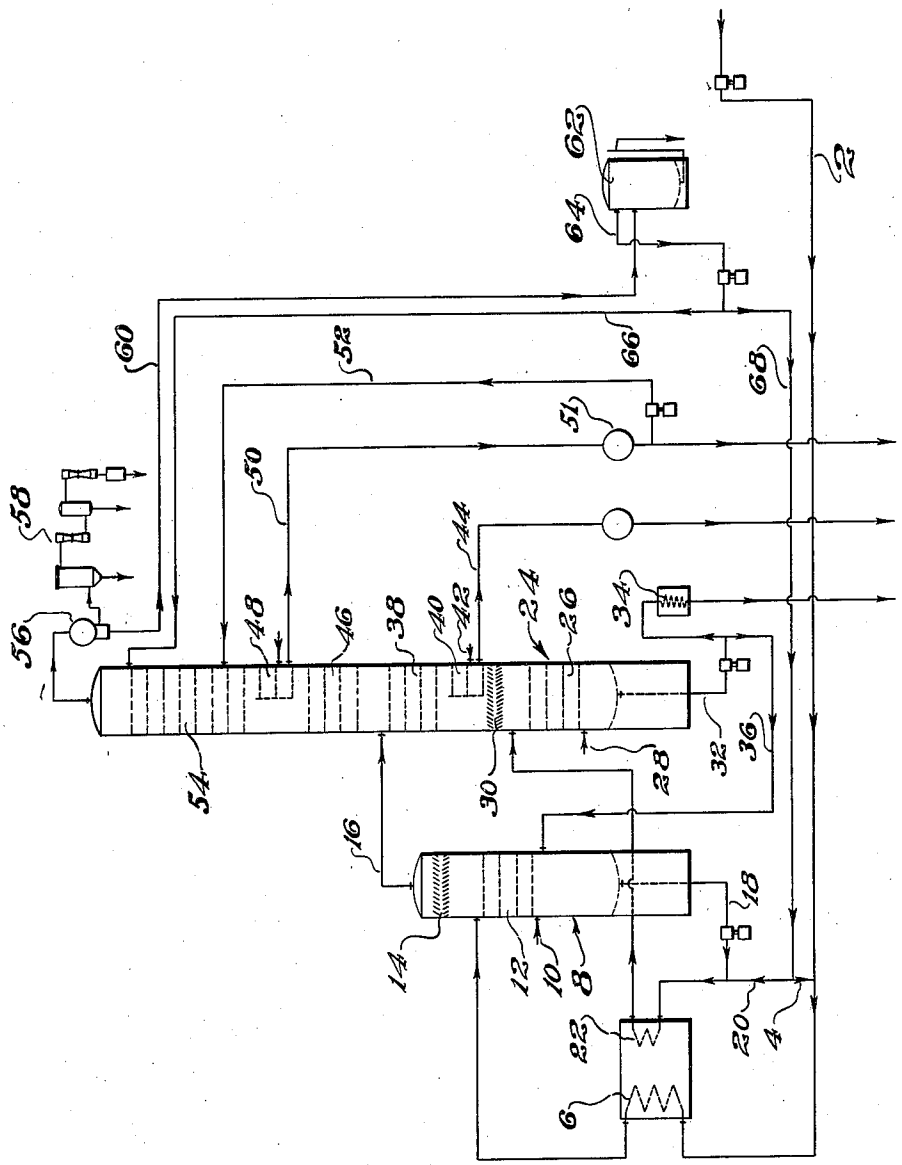
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METHOD FOR DISTILLING RESIDUES

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METHOD FOR DISTILLING RESIDUES

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The present invention relates to methods for distilling petroleum residues and more particularly to the distillation of residues containing a small percentage of asphalt.

The method described in the Coubrough Patent No. 1,963,888 granted June 19, 1934, involves the addition of both asphalt and a low boiling diluent to a residual mixture consisting primarily of heavy lubricating stock and asphalt, followed by flash vaporization to separate the oils from the asphalt. The essential function of the diluent is to permit vaporization of the oils at a temperature below the cracking point and the function of the added asphalt is to afford a heat carrier for the flash vaporization. The method is entirely feasible, particularly in the handling of oils of low asphalt content, but in many instances, the addition or recirculation of sufficient asphalt for supplying the heat of vaporization of the oils involves some practical difficulties.

It is the principal object of the present invention to distill residues for recovery of lubricating stocks and residuum without the necessity for adding or circulating a large quantity of residual material.

With this and other objects in view, as will hereinafter appear, the present invention contemplates a double flash distillation whereby a reduced crude is subjected to flash vaporization in a column followed by removal of unflashed material, addition thereto of diluent, and finally subjection of the mixture to a second flash vaporization. With crudes of intermediate asphalt content, substantially complete de-asphaltization of the crude can be effected without recirculation of asphalt, while with oils of very low asphalt content, the amount of recirculated asphalt may be much less than in the process of the above-mentioned Coubrough patent.

The accompanying drawing is a diagram of the preferred form of apparatus for practising the invention.

The method is herein illustrated and described as applied to the distillation of an Oklahoma City crude containing less than 2% of asphalt. The crude is reduced by any suitable fractionation to 25% reduced crude, consisting primarily of oil components designated as lubricating oil stock, with a small amount of asphalt (about 7 to 8% of the reduced crude). It will be noted that in this example, the asphalt content is comparable to the 10% asphalt content of the specific starting material mentioned in the Coubrough patent.

The reduced crude is fed by a line 2 and is mixed with a diluent at 4. The diluent is preferably a low boiling fraction such as gas oil or kerosene. One part of diluent may be used for one to three parts of feed. The mixture is heated in a pipe still 6 to a temperature of about 750–770° F., and discharged into a vacuum column 8 maintained under an absolute pressure of about 50–100 mm. of mercury. Vapors are formed by flash vaporization, that is, by the contained heat of the mixture upon release into the region of low pressure. The vapors comprise the diluent and some of the lubricating oil stock. Vaporization of the oils is enhanced by steam introduced at 10, a set of stripping decks 12 being interposed between the steam inlet and the charging point. Suitable entrainment separators 14 are provided in the upper part of the column.

The vapors, comprising substantially all of the diluent and a considerable part (about 85% in the example chosen) of the lubricating oil stock, are removed from the column by a pipe 16 and are passed into a second column to be referred to. The residue remaining unvaporized in the column 8 comprises the asphalt of the original crude, together with the remainder of the lubricating oil stock. No attempt is made to reduce to asphalt in the column 8, but by the vaporization of some lubricating oil stock, the percentage asphalt content of the unvaporized residue is greatly increased; in the example chosen, the residue contains about one part of asphalt to two parts of the heaviest lubricating components.

The residue is withdrawn by a pipe 18 and mixed with additional diluent of the same character introduced through a pipe 20. The mixture is heated in a second heating section 22 to a temperature of about 750–770° F., and introduced into a final vacuum column 24, in which an absolute pressure of about 50–100 mm. of mercury is maintained in the vaporizing zone. This column has a steam stripping section 26 below which steam is introduced at 28. Above the charging point, the column is provided with entrainment separators 30. By virtue of the flash vaporization, the oils comprising both the heavy lubricating oil stock and the diluent are substantially completely vaporized from the asphalt and the vapors progress upwardly through the column for fractionation. The asphalt is withdrawn at 32 and a portion is cooled in the cooler 34 and taken off as a product. With oils of very low asphalt content, as the Oklahoma City crude above referred to, a part of the asphalt may be recirculated by a line 36 to the base of

the column 8 for mixture with the residue of that column. It will be seen that in that case, the material charged to the heater 22 and the column 24 has added thereto both a diluent and asphaltic residue after the manner of the above-mentioned Coubrough patent. By virtue of the preliminary reduction with diluent in the column 8, however, that amount of asphalt thus recirculated may be comparatively small. Such recirculation is in fact necessary only with crudes of low asphalt content, when substantially complete separation of oils from asphalt is required. With crudes of somewhat higher asphalt content, the line 36 may be omitted.

The diluent and lubricating oil stock vapors pass above the entrainment catchers 30, and enter a fractionating section 38 where they are subjected to descending reflux. Below the section 38 is a restripping section 40, into which liquid reflux descends, to be stripped by steam introduced at 42. The heavy lubricating oil stock is withdrawn from the stripping section as a product at 44. As stated in the Coubrough Patent No. 1,905,156, when substantially complete de-asphaltization is desired, provision should be made for preventing passage of reflux into the vaporizing zone, and accordingly the bottom of the stripping section 40 is imperforate.

The vapors from the column 8 enter by the pipe 16 into the column 24 immediately above the fractionating section 38. Above the point of vapor admission, the column is provided with a fractionating section 46 and a restripping section 48. A light lubricating oil stock is withdrawn as a product from the section 48 by a pipe 50 and cooled in a cooler 51, a portion thereof being returned by a pipe 52 to a higher point of the column as reflux. Above the section 48 is a fractionating section 54 in which the diluent is finally separated from the lubricating stock. The diluent is condensed in a condenser 56 which connects with the usual final condensing and vacuum jet system indicated generally at 58. Condensed diluent and water (from condensation of steam) run from the condenser 56 by a pipe 60 to a vessel 62 from which the diluent is decanted at 64, a part of the diluent being pumped by a line 66 into the top of the column as reflux and the remainder being passed by a line 68 which joints the diluent feed pipes 4 and 20 for mixture with the feeds to the heating sections 6 and 22, respectively.

The restripping section 48 is employed when it is desired to separate the lubricating oil stock into two products. If all of the lubricating oil stock is to be recovered as a single product, the section 48 and the lines 50 and 52 may be omitted, the entire side product being taken off at 44. For the character and quantity of the diluent to be used, the general disclosure of the Coubrough Patent No. 1,905,156 may be followed. The diluent should be non-adjacent to the bottoms, or as defined in a more precise manner in said patent, it should be of a character to be completely vaporizable under conditions of temperature and pressure at which vaporization of the bottoms would be negligible. The quantity of added diluent may approximate the total quantity of residue at 18.

One of the principal advantages of the present invention is the further reduction of reduced crudes of relatively low asphalt content in the first column 8 to such a degree that the primary residue contains a sufficient percentage of asphalt for supplying heat of vaporization in the secondary vaporization step carried out in the column 24. Accordingly, the amount of asphalt recirculated may be considerably less than required in the process of the Coubrough Patent No. 1,963,888. Thus the practical difficulties of handling the asphalt are materially reduced, and asphalt of even higher quality may be obtained. With crudes of somewhat higher asphalt content, such as an average Mid-Continent crude, recirculation of asphalt may be avoided altogether, and the line 36 may be omitted.

The process is of the greatest value when substantially complete de-asphaltization of the crude is desired, to obtain all of the heaviest and most valuable lubricating components as distillates and asphalt of exceptional hardness as residue. Such de-asphaltization may be accomplished without the necessity of heating in any stage to a point at which appreciable decomposition of the oils might occur. If, however, it is desired to obtain a softer asphalt as a product, this may be done even more easily, by simply reducing the temperatures or by operation under less vacuum.

It will be understood that the invention is not limited to the particular examples herein given, nor to separation of any particular oils from the asphalt. In the Oklahoma City crude above referred to, the oil fractions are designated as light and heavy lubricating oil stock, but the invention is equally applicable to the separation of any high boiling oils from the asphalt. Such high boiling oils may in any case be generally designated as lubricating stock, and are so referred to in the appended claims.

Having thus described the invention, what is claimed is:

1. The method of separating oils and asphalt from petroleum bottoms consisting essentially of lubricating stock and not substantially more than 10% of asphalt which consists in adding to the bottoms a diluent miscible therewith and of materially lower boiling point than the bottoms, subjecting the mixture to flash vaporization by continuously heating the mixture and introducing it into a vaporizing zone to vaporize the diluent and the greater part of the lubricating stock and to leave a residue containing asphalt in much higher proportion than in the original mixture, adding diluent to said residue, continuously heating the mixture of residue and diluent and introducing it to a second vaporizing zone under vacuum for flash vaporization of the diluent and the remaining lubricating stock from the asphalt, the temperature of each heating operation being sufficient to effect maximum vaporization without cracking, condensing diluent and lubricating stock while preventing return of condensate to either vaporizing zone and withdrawing asphaltic residue from the second vaporizing zone.

2. The method of separating oils and asphalt from petroleum bottoms consisting essentially of lubricating stock and not substantially more than 10% of asphalt which consists in adding to the bottoms a diluent miscible therewith and of materially lower boiling point than the bottoms, subjecting the mixture to flash vaporization by continuously heating the mixture and introducing it into a vaporizing zone to vaporize the diluent and the greater part of the lubricating stock and to leave a residue containing asphalt in much higher proportion than in the original mixture, adding diluent and further asphalt to said residue, continuously heating the mixture of res-

idue and diluent and introducing it to a second vaporizing zone under vacuum for flash vaporization of the diluent and the remaining lubricating stock from the asphalt, the temperature of each heating operation being sufficient to effect maximum vaporization without cracking, condensing diluent and lubricating stock while preventing return of condensate to either vaporizing zone and withdrawing asphaltic residue from the second vaporizing zone.

3. The method of separating oils and asphalt from petroleum bottoms consisting essentially of lubricating stock and not substantially more than 10% of asphalt which consists in adding to the bottoms a diluent miscible therewith and of materially lower boiling point than the bottoms, subjecting the mixture to flash vaporization by continuously heating the mixture and introducing it into a vaporizing zone to vaporize the diluent and the greater part of the lubricating stock and to leave a residue containing asphalt in much higher proportion than in the original mixture, adding diluent to said residue, continuously heating the mixture of residue and diluent and introducing it into a second vaporizing zone under vacuum for flash vaporization of the diluent and the remaining lubricating stock from the asphalt, passing the vapors from both vaporizing zones through a common fractionating zone, separately condensing diluent and lubricating stock while preventing return of condensate to either vaporizing zone and withdrawing asphaltic residue from the second vaporizing zone.

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