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PROCESS OF SEPARATING CONSTITUENTS OF MINERAL OILS

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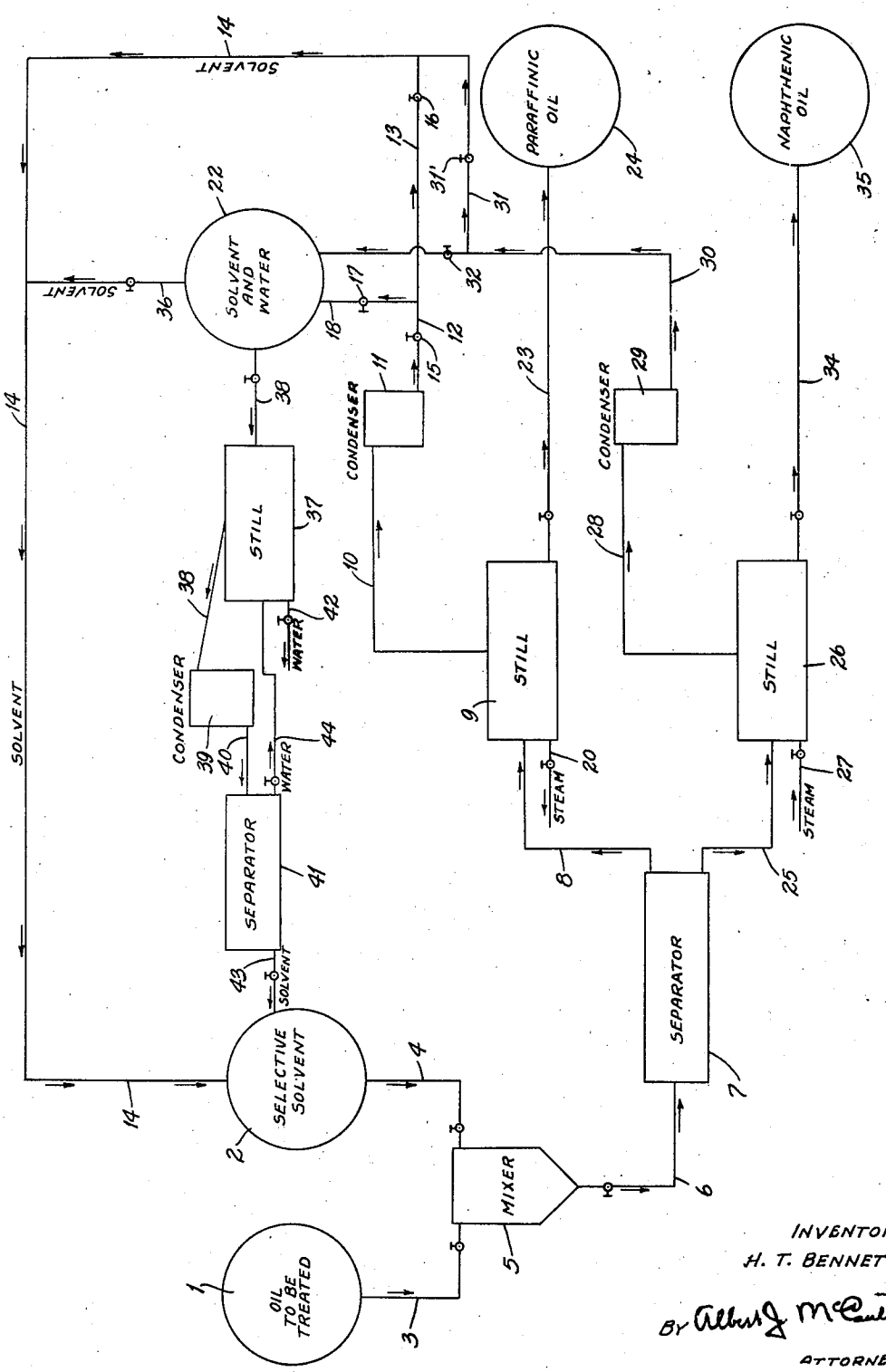

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PROCESS OF SEPARATING CONSTITUENTS OF MINERAL OILS

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5 Claims. (Cl. 196—13)

This invention relates to processes of separating constituents of mineral oils, one of the objects being to provide a method of recovering approximately all of the solvent from fractions of a mineral oil which have been separated by treatment with a selective solvent. This application is a continuation in part of an application Serial No. 519,698, filed by me on March 2, 1931.

Mineral oils, including petroleum oils, and especially the petroleum lubricating oils, contain different constituents which may be separated from each other by treating the oil with a suitable selective solvent, and thereafter separating the selected fraction from the other fraction of the oil.

Numerous advantages may be obtained by the use of such solvents to improve the quality of the oil. The paraffinic wax may be removed to improve the pour point of the oil, or the naphthenic portion may be separated from the more valuable paraffinic portion to produce a high viscosity index lubricating oil which is non-sludging and has a low carbon content.

In the commercial treatment of these oils with a selective solvent, large quantities of relatively expensive solvents are usually employed. These solvents should be recovered from the oil fractions and reused for subsequent extractions. Since the recovered solvent is used repeatedly, a very small loss of solvent during each treatment will eventually result in a considerable loss of the solvent.

In the preferred form of the invention, a very large percentage of the solvent can be economically recovered from the oil fractions with a minimum decomposition of the solvent by distilling these fractions at relatively low temperatures and treating the same with steam. However, a quantity of solvent, depending upon the nature of the solvent, is contained in the condensed steam. The amount of solvent contained in the condensed steam is usually relatively small in proportion to the volume of water present.

An object of this invention, therefore, is to provide an economical and efficient process and apparatus for extracting mineral oil with a solvent, wherein the solvent may be effectively separated from the steam employed in removing the solvent from the oil fractions.

A further object is to provide a system wherein a highly efficient selective solvent may be reused for repeated extractions of oils with practically no loss of solvent.

With the foregoing and other objects in view, the invention comprises the novel method, construction, combination and arrangements of parts hereinafter more specifically described and shown in the accompanying drawing, which illustrates one form of the invention. However, it is to be understood that the invention comprehends changes, variations and modifications which come within the scope of the claims hereunto appended.

In the preferred form of the invention, BB' dichlorethyl ether is employed as the selective solvent, but other halogenated ethers may be used, and some of the advantages of the present invention may be obtained in using various other solvents including nitrobenzene, phenol, aniline, cresol, furfural, dichlorethylene, trichlorethylene, etc.

The drawing is a diagrammatical view of one form of an apparatus embodying the features of this invention.

The oil to be treated may be confined in a container 1 and the solvent in a container 2. The oil and solvent may be transmitted through pipes 3 and 4 to a mixer 5 wherein the oil and solvent may be intimately mixed. The pipes 3 and 4 are provided with suitable valves to regulate the delivery of oil and solvent. The solvent and oil, after being agitated in the mixer 5, may be transmitted through pipe 6 to a separator 7 where the oil is separated into fractions more paraffinic and more naphthenic than the original oil.

The more paraffinic fraction may be withdrawn through pipe 8 to still 9 wherein a substantial portion of the solvent is readily distilled from the oil preferably under a pressure less than atmospheric pressure. The solvent, which is usually free of water at this time, is transmitted through vapor line 10, condenser 11 and pipes 12, 13 and 14 to the solvent container 2, valves 15 and 16 being open and valve 17 being closed.

After a large portion of the solvent has been separated from the oil in the vacuum still 9, steam may be injected through pipe 20 to remove the last portion of the solvent from the oil. The pipe 20 is provided with a valve to control the delivery of steam to the still 9.

At this time, while steam is injected into the still 9, the valves 15 and 17 are open and valve 16 closed so that the solvent and condensed steam may pass from the condenser 11, and through pipes 12 and 18 to the separator or set-

tler 22. The solvent and water is thus diverted from the return pipe 14.

After the solvent has been removed from the more paraffinic fraction of the oil, this fraction is discharged through pipe 23 to a tank 24.

The more naphthenic fraction of the oil is transmitted through pipe 25 to a vacuum still 26 wherein the solvent may be separated from the oil in a manner similar to the separation which occurs in still 9. The readily removable portion of the solvent, which is not subjected to the steam, may be transmitted through vapor line 28, condenser 29, pipes 30, 31 and 14 to the solvent container 2, valve 32 being closed and valve 31' being open at this time. Thereafter, steam is introduced through a pipe 27 provided with a suitable regulating valve.

When steam is injected into the still 26, a valve 32 is opened and the valve 31' closed, so that the solvent and condensed steam are transmitted to the container 22.

The more naphthenic fraction of the oil in the still 26 may be transmitted through pipe 34 to tank 35.

The free solvent which settles by gravity in the container 22, may be passed through pipes 36 and 14 to the solvent tank 2. The water containing dissolved or suspended solvent remaining in container 22 may be transmitted through a pipe 38 to a still 37. The solvent with about 10% to 20% of the water in the form of steam passes from the still 37, through vapor line 38, to a condenser 39, the condensate being transmitted through pipe 40 to a separator 41. The still 37 is provided with a pipe 42 for the discharge of the water remaining in the still after the distilling operation therein has been completed.

The solvent, which is separated by gravity from the small quantity of water in separator 41, is returned through a pipe 43, to the solvent container 2. The water in the separator 41 is discharged through a pipe 44, which preferably leads back to the still 37, to provide for a removal of the solvent contained in this water.

It will now be understood that almost all of the solvent may be transmitted in an endless course from the solvent container 2, through the separating station including the separator 7, the solvent recovery station including stills 9 and 26 for the separation of the solvent from the oil, then through return conductors leading back to said solvent container.

These return conductors include the return pipe 14 which receives the free solvent from the pipe 13 as well as solvent which is diverted through the separating chamber 22. Said return conductors also include the pipe 43 which returns the smaller quantity of solvent obtained by distilling a portion of the water discharged from said separating chamber 22.

As an illustration of one form of this invention, I will now describe a specific process wherein a chlorinated solvent, such as BB' dichlorethyl ether, may be employed to provide a high viscosity index oil. It is understood that the scope of the invention is not limited to the following specific description of details.

About one part of a mid-continent lubricating oil stock may be agitated with about two parts BB' dichlorethyl ether in the mixer 5 at a temperature of about 80° F. to 105° F. While at substantially the same temperature the mixture of oil and BB' dichlorethyl ether are transmitted to the separator 7 where the naphthenic and paraffinic fractions gradually separate from each other, the upper layer of material discharged through pipe 8 being more paraffinic than the original oil, while the lower layer, discharged through pipe 25, is more naphthenic than the original oil.

The temperature in the vacuum stills 9 and 26 may be about 280° F., during the first part of the run, and it is desirable to thereafter increase this temperature to about 330° F., while introducing live steam through pipes 20 and 27 to remove the last portion of the solvent.

During the first part of the run in the stills 9 and 26 the recovered BB' dichlorethyl ether may be transmitted directly to solvent container 2, and thereafter, when steam is introduced into the stills 9 and 26, the BB' dichlorethyl ether and condensed steam is preferably transmitted to the separator 22. The free BB' dichlorethyl ether which settles in the separator 22, is returned to the solvent container 2 through pipes 36 and 14. The water remaining in the settler 22 may contain about 1% solvent, and this water is transmitted to the still 37. About 10% to 20% of this water is distilled, with the result of removing practically all of the dichlorethyl ether, the resultant condensate being transmitted to the separator 41, wherein substantially all of the condensed BB' dichlorethyl ether may then be returned through pipe 43 to the solvent container 2. The water in separator 41 and still 42 may be discharged through pipes 44 and 42. However, the pipe 44 preferably leads from said separator 41 to the still 37.

To simplify the illustration, I have shown a single separating chamber 7, but it is to be understood that the paraffinic fraction of the oil may be subjected to successive mixing and separating operations, using additional quantities of fresh solvent; or the naphthenic solution resulting from one separating operation may be used in preliminary mixing and separating operations, so as to remove some of the naphthenic portions from the original oil before it is subjected to the treatment with fresh solvent.

In actual practice, I prefer to subject the fresh oil to a preliminary extraction, using the napthenic solution of another extracting operation to remove some of the naphthenic constituents of the original oil. Thereafter, the oil from which these naphthenic constituents have been removed, is mixed with fresh solvent and permitted to settle, so as to remove additional naphthenic constituents from the desired paraffinic oil. As a specific illustration of the results obtained from such operations, I will refer to the extraction of a mid-continent lubricating oil, having a gravity of 26.4, a flash point of 405° F., a viscosity of 255 at 100° F., and a viscosity index of 75. This oil was mixed with the naphthenic solution resulting from a previous extraction, and after separating into a layer more paraffinic than the original oil and a layer more naphthenic than said original oil, the paraffinic layer was removed and mixed with fresh BB' dichlorethyl ether, using 2½ parts of the ether to 1 part of the oil. After being allowed to settle, the paraffinic portion was removed and distilled to recover solvent therefrom. The resultant paraffinic lubricating oil had a gravity of 30.8, a flash point of 425° F., a viscosity of 198 at 100° F., and a viscosity index of 100.

The original oil was treated with sulphuric acid and clay before subjecting it to the ex-

tracting process. The paraffinic and naphthenic products may be subjected to a light treatment with sulphuric acid, using about ¼ to ½ pound of the acid for each barrel of the oil, and thereafter contacted with a suitable adsorbent, or absorbent material, such as fuller's earth, or other suitable clay. When treated in this manner, both products are valuable lubricating oils, the greatest value being in the high viscosity index paraffinic oil, having a high resistance to sludging and carbon formation, but the low viscosity index naphthenic oil is quite satisfactory for use in machines that do not require a high viscosity index lubricant.

It is, of course, understood that suitable valves and pumps will be employed to efficiently control the transmission of the several fluids from one station to another, and a complete commercial system which may be a countercurrent system will include numerous other details not shown in the accompanying drawing. However, this drawing is merely a diagrammatical illustration of one form of the invention, and in view of the foregoing disclosure, such details will be apparent to those skilled in the art. Furthermore, the invention extends to various modifications within the scope of the terms employed in the following claims.

I claim:

1. The process of treating oils having different constituents which comprises mixing the oil with a halogenated ether, separating the mixture into fractions, separately distilling one of said fractions with steam to separate the halogenated ether from said fraction, condensing said steam, distilling a relatively small quantity of said condensed steam to recover approximately all of the solvent contained in the condensed steam, and separating the last mentioned solvent from the condensed steam.

2. The process of treating oils having high and low viscosity index constituents which comprises treating an oil with dichlorethyl ether to effect a separation of the oil into fractions having a higher viscosity index and a lower viscosity index than the original oil, separately distilling said fractions and treating them with steam to recover dichlorethyl ether from said fractions, condensing said steam, and distilling said condensed steam to recover dichlorethyl ether contained therein.

3. The process of treating oils having high and low viscosity index constituents which comprises treating an oil with dichlorethyl ether to effect a separation of the oil into fractions having a higher viscosity index and a lower viscosity index than the original oil, separately distilling said fractions and treating them with steam to separate the dichlorethyl ether from said fractions, condensing said steam, distilling about 10 to 20 percent of said condensed steam to remove approximately all of the dichlorethyl ether contained in said condensed steam, and separating the last mentioned dichlorethyl ether from said condensed steam.

4. The process of treating oils having high and low viscosity index constituents which comprises treating an oil with dichlorethyl ether to effect a separation of the oil into fractions having a higher viscosity index and a lower viscosity index than the original oil, separately distilling said fractions under a vacuum, in the absence of steam, to recover a large percentage of said dichlorethyl ether from said fractions, thereafter injecting steam into the liquid undergoing said vacuum distillation to continue the removal of dichlorethyl ether, condensing said steam, and distilling about 10 to 20 percent of the condensed steam to remove dichlorethyl ether therefrom.

5. The process of treating lubricating oil stocks having high and low viscosity index constituents which comprises adding beta beta' dichlorethyl ether to the oil to be treated, agitating the resultant mixture of oil and solvent at a temperature from about 80° to 110° F., allowing the mixture to settle into a fraction having a higher viscosity index than the original oil and a fraction having a lower viscosity index, separating said fractions, separately distilling said fractions under a vacuum to recover a large percentage of said dichlorethyl ether, and thereafter removing substantially all of the remaining dichlorethyl ether by introducing steam into the material undergoing said vacuum distillation, condensing said steam, removing the free dichlorethyl ether from the condensed steam, and distilling said condensed steam to recover substantially all of the dichlorethyl ether contained therein.

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