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OIL SEPARATION

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This invention relates to a process of separating components of a mixed base oil.

The use of solvents such as acetone alone or with other solvents, ethyl alcohol, ethyl acetate, and mixtures of alcohols with benzol, for separating components of a mixed base oil, has been attended with serious faults and difficulties, including lack of selectivity, miscibility of the solvent with water, high volatility involving fire hazard and substantial losses, low solubility of oil in the solvent, unstability, and toxicity. In the case of mixed solvents it has been difficult to maintain the mixture at proper concentration.

Due partly to the conditions just recited, no satisfactory process has heretofore been developed for separating mixed base oils (including paraffinic and naphthenic components such as found in the Mid-Continent field), into its paraffinic and naphthenic components.

The principal objects of this invention therefore are to produce paraffinic oils from a mixed base oil.

One phase of the invention consists in the use of ethylene dichloride for separating paraffinic hydrocarbons from a body of oil.

A further object of the invention is to adjust the conditions of a separating process for overcoming obstacles to the use of ethylene dichloride as a solvent.

Other objects of the invention will be apparent in the following description of the improved process as applied to the treatment of Mid-Continent oil stocks.

In one aspect, the invention consists in mixing ethylene dichloride with a body of a mixed base oil including paraffins, at relatively high temperatures, chilling the mixture sufficiently to bring about separation of a substantial proportion of the paraffins from the other components to form layers of liquid, and segregating the paraffin layer from the other layers.

The invention will be described in detail in the specific aspect of employment in the treatment of a mixed base oil composed principally of paraffins and naphthenes, to obtain a lubricating oil having a relatively high paraffin number, it being understood that the principles involved may be adapted to the treatment of other stocks for obtaining analogous results.

A body of the mixed base oil is introduced into a body of ethylene dichloride, which will be referred to as the solvent, in the proportion of 20 per cent of oil to 80 per cent of the solvent, both liquids being at a temperature above 40° Fahrenheit. The mixture is agitated sufficiently, for example by stirring, to assure thorough mixing.

The solution is then cooled to approximately 5° Fahrenheit and held at the low temperature for such time as to assure separation of the several layers and preferably for approximately four hours.

Care is taken to assure equal cooling of all portions of the solution, for example by providing a chilling bath at the temperature to which the solution is to be reduced, or by agitation to prevent excessive cooling of portions of the solution contacting the walls of the container or cooling coils. Occasional agitation of the solution during the cooling step is preferred whatever method of cooling may be employed, to promote equal rates of cooling in all portions of the solution, and retard reactions prior to attainment of the desired temperature substantially throughout the solution.

One to four hours may be required to reduce the temperature of the solution to the desired value. The solution is then allowed to stand for approximately four hours, the temperature being maintained at the desired value, to bring about separation of hydrocarbon components of the mixed base oil.

The liquid body, after standing at the low temperature until the precipitation of oil has occurred and is substantially complete, comprises two distinct layers. The upper layer consists of a solution of the solvent in paraffinic oil. The lower layer consists of a solution of naphthenic oil in the solvent.

The upper layer, including precipitated wax and paraffinic oil is then reduced, for example in a steam still, the bottoms reduced with fire and steam, and then treated by usual processes. The solvent is recovered substantially quantitatively and may be reused. The paraffinic hydrocarbons produced have higher A. P. I. gravity and lower viscosity, but markedly lower viscosity ratio, than the original oil. The paraffin number of the separated and recovered paraffinic oil is also substantially higher than the paraffin number of the original mixed base oil.

The lower layer of the refrigerated mixture is likewise reduced and treated, and the ethylene dichloride constituent thereof recovered for reuse. The oil constituent has substantially lower A. P. I. gravity than the original oil, much higher viscosity, a very materially higher viscosity ratio, and a low paraffin number.

In one example of details of a run made according to the process described above, a mixed base

oil stock may have a viscosity of about 120 at 210° F., a viscosity ratio of 18.5, and a paraffin number 79.5. The paraffinic oil obtained by the process will have substantially the same viscosity, say 120 at 210° F., a viscosity ratio of about 14, and a paraffin number of 96.5.

The oil obtained from the bottom layer of the solution in the run being described, will have a viscosity of about 160 at 210° F., a viscosity ratio of about 36, and a paraffin number of 49.

The control of the viscosity ratio depends partly on the percentage of the total oil found in the top layer of the refrigerated mixture, which should be at about 35 per cent of the oil in the mixture in the particular case cited, the occurrence of higher percentages in this layer resulting in higher viscosity ratios. The promotion of equal cooling of all portions of the mixture, by agitation, and avoidance of cooling any portions substantially below the predetermined separating temperature, also has important influence on control of the viscosity ratio, and that the more accurately these conditions are regulated, the lower the viscosity ratio will be.

The extent of precipitation of oil under refrigeration, and the quality of the paraffinic oil produced, tend to vary with the concentration of the original solution and the separating temperature. Good results may be obtained with solutions having oil content varying from 15 to 30 per cent cooled to a temperature of 30° F., or below. The percentage of oil and the separating temperature should be low to obtain a paraffinic oil having the highest paraffin number. The preferred solution includes 15 to 20 per cent of the mixed base oil, and the preferred separating temperature is 5° F.

Refrigeration is controlled to prevent cooling of the solution or any portion thereof substantially below the determined low temperature. In the treatment of the mixed base oil containing approximately 35 per cent of paraffinic oils, limiting of cooling to approximately zero, and, more definitely, about minus 5° F., will prevent development of adverse effects. If the temperature is carried too low, a certain amount of the naphthenic oil in the lower layer is thrown out of solution and is finally found in the upper layer, thus partially defeating the purpose of the process.

In the treatment of other mixed base oils, the temperature would be controlled under the same principles, for example by determining the point at which the solution of oil in the solvent would be disturbed, and limiting refrigeration accordingly.

The solvent occurs in the top layer to the extent of only 25 to 50 per cent, when a mixed base oil containing approximately 35 per cent of paraffinic components is treated as above described. While there is some difference in the specific gravity of the two oils after they are separated from the base oil, the separation occurs because the top layer contains a great deal less solvent than the lower layer. In other words, oil of a paraffinic nature and the solvent under discussion seem to be compatible at the temperatures used only within quite narrow limits, and such a solution contains a rather small percentage of the solvent. Any excess solvent in the solution then is to be found in the mixture with the naphthenic oil which constitutes the lower layer. For this reason, only 15% to 30% of oil is used in the base solution so as to have a large excess of ethylene dichloride present in the lower layer, thus diluting the naphthenic oil with considerable quantities of the solvent and so reducing the power of the naphthenic oil to dissolve the paraffinic oil.

The several steps and phases of the process may be combined into a continuous process. For example, the following procedure may be used:

A solution may be prepared including oil and solvent in such proportions as may be indicated due to the percentage composition of the oil, for example, a solution of approximately 20 percent of oil and 80 percent of ethylene dichloride, at any temperature above 40° F., and preferably between 80° F. and 100° F.

Thorough mixing is brought about either by agitation in batch or by feeding the oil and solvent through a proportioning device and mixing in coils to secure thorough solution. The mixture of oil and solvent is then passed through refrigerating coils or chilled by other means to the desired temperature, the preferred temperature being approximately 5° F., and passed into a centrifuge. The solution of solvent in paraffinic oil having a specific gravity which is much less than the solution of naphthenic oil in the solvent, will separate readily in the centrifuge after which the two solutions are reduced, the solvent and oils being recovered and retreated as desired.

The ethylene dichloride used as a solvent and reagent boils at 182° F., a high enough point to make recovery of the solvent simple. The solvent is extremely stable, non-toxic, and has explosive mixture limits much narrower than most other solvents which might be used for the purpose. The amount of oil soluble in the solvent is satisfactory, as 20 to 30 per cent of oil in the solution can be worked with easily, to give good selectivity.

The invention has been described as applied to the treatment of a particular type of oil, and preferred conditions have been given for the treatment of said type of oil, in order to disclose a practical application of the process, it being understood that temperatures, percentages of oil in a solvent, and like conditions may be varied from the examples given, for treating other types of oil stock, under different conditions, without departing from the spirit of the invention.

The particular type of oil for which the preferred conditions of treatment have been stated is one containing approximately 35% of oil of a paraffinic character, and the steps named are adapted to control the percentage of the total oil in the solution which will appear in the upper layer, to about 35 per cent of the total oil. This control is effected by limiting the total oil treated in a given solution, and/or by control of the temperature of separation. The ratio of oil to solvent in the original solution is thus governed by the percentages of paraffine and naphthene in the oil. An excess of the ethylene dichloride will in any case be used to dissolve the oil at temperatures above 40° F.

Tests made with base oil containing 35 per cent of paraffinic oil, indicate that when the proportion of total oil to solvent materially exceeds 20 per cent, and the separating temperature is close to 32 degrees Fahrenheit, the viscosity, viscosity ratio, and paraffinic number of the separated portion of the solution that contains paraffinic oil, vary only slightly from these properties of the base oil. Lowering of the temperature of such a mixture to 20° F. will lower the viscosity and viscosity ratio and increase the paraffine number of the paraffinic portion of the solution.

On the other hand, lowering of the proportion of total oil to solvent to 20 per cent, and chilling to about 32° F., noticeably reduces the viscosity

ratio of the paraffinic product and increases its paraffine number, while chilling a solution containing 20 per cent of total oil to 5 degrees Fahrenheit further reduces viscosity and viscosity ratio, and further increases the paraffine number of the paraffinic product.

What I claim and desire to secure by Letters Patent is:

1. The process of oil separation consisting of adding a mixed base oil including paraffinic and naphthenic components to a body of ethylene dichloride in substantially the proportions of 15 to 30 per cent of oil to 85 to 70 per cent of the ethylene dichloride, maintaining the mixture above 40° Fahrenheit for sufficient time to effect maximum dissolution of the components in the ethylene dichloride, reducing the temperature of the mixture to approximately 5 degrees above zero Fahrenheit for effecting precipitation of a substantial proportion of the naphthenic component, and separating the components.

2. The process of oil separation consisting of mixing ethylene dichloride with a body of mixed base hydrocarbon oil containing paraffinic and naphthenic fractions, at a temperature above 40 degrees Fahrenheit to dissolve the oil in the ethylene dichloride, subjecting the mixture to refrigeration sufficiently to reduce the temperature thereof below 35 degrees Fahrenheit and above 5 degrees below zero for effecting separation of the paraffinic fraction, allowing the mixture to stand for effecting separation of the fractions into layers including a layer comprising a solution of naphthenic hydrocarbons in ethylene dichloride and a layer comprising a solution of ethylene dichloride in paraffinic hydrocarbons, and segregating the layers.

3. The process of oil separation consisting of mixing ethylene dichloride with a body of mixed base hydrocarbon oil containing paraffinic and naphthenic fractions at a temperature above 40 degrees Fahrenheit to dissolve the oil in the ethylene dichloride, subjecting the mixture to refrigeration sufficient to reduce the temperature thereof below 35 degrees Fahrenheit and above 5 degrees below zero for effecting separation of the paraffinic fraction, allowing the mixture to stand for effecting separation of the fractions into layers including a layer comprising a solution of naphthenic hydrocarbons in ethylene dichloride and a layer comprising a solution of ethylene dichloride in paraffinic hydrocarbons, segregating the layers, and retreating the ethylene dichloride solution for recovering hydrocarbons therefrom.

4. The process of oil separation including mixing a quantity of a mixed base oil containing approximately 35 per cent of paraffinic oil with a body of ethylene dichloride at a temperature above 40° F. to form a solution, chilling the solution to a temperature below 35° F. to separate the solution into an upper layer containing paraffinic oil and a lower layer containing naphthenic oil, and limiting the quantity of base oil added to the solvent for controlling the proportion of the base oil included in said upper layer.

5. In the art of refining mineral oils, the process which comprises separating an oil containing paraffinic and naphthenic hydrocarbons into fractions respectively richer in paraffinic and naphthenic compounds by extracting said oil with ethylene dichloride.

6. In the art of refining mineral oils, the process which comprises adding ethylene dichloride to an oil containing paraffinic and naphthenic hydrocarbons at such temperature as to effect solution, cooling the solution to form a two layer system, one layer being richer in paraffinic compounds and the other layer being richer in naphthenic compounds, and separating the upper layer from the lower layer.

7. In the art of refining mineral oils, the process which comprises bringing a mineral oil containing paraffinic and naphthenic hydrocarbons into contact with ethylene dichloride thereby to effect solution of a portion richer in naphthenic hydrocarbons in the ethylene dichloride, separating the solution so formed comprising one fraction from the remainder of the oil comprising a second fraction and recovering the ethylene dichloride, thereby to obtain a fraction of the oil richer in naphthenic hydrocarbons and another fraction richer in paraffinic hydrocarbons.

8. In the art of refining mineral oils, the process which comprises adding ethylene dichloride to a liquid oil containing paraffinic and naphthenic hydrocarbons at temperatures above 40° F., cooling the solution to a temperature sufficient to form two layers, separating the layers, separating the ethylene dichloride and paraffin wax from the upper layer, and separating the ethylene dichloride from the lower layer to produce one fraction richer in paraffinic and a second fraction richer in naphthenic hydrocarbons than said liquid oil.

9. In the art of refining mineral oils, the process which comprises adding a predominate proportion of ethylene dichloride to a mixed base oil containing paraffinic and naphthenic hydrocarbons at a temperature to effect solution, cooling the solution to form a two layer system of naphthenic hydrocarbons in ethylene dichloride and ethylene dichloride in paraffinic hydrocarbons, separating the layers and separating the ethylene dichloride.

10. In the art of refining a mixed base mineral oil containing paraffinic and naphthenic hydrocarbons, the steps of adding ethylene dichloride to the mixed base oil at a temperature to effect solution, cooling the solution to cause separation into layers, one layer containing predominately ethylene dichloride and naphthenic hydrocarbons in solution; another layer containing predominately paraffinic hydrocarbons and a lesser amount of ethylene dichloride, and separating the layers, the amount of mixed base oil used being so limited in quantity in proportion to the volume of ethylene dichloride as to cause the naphthenic hydrocarbon layer to contain a substantial excess of ethylene dichloride.

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