

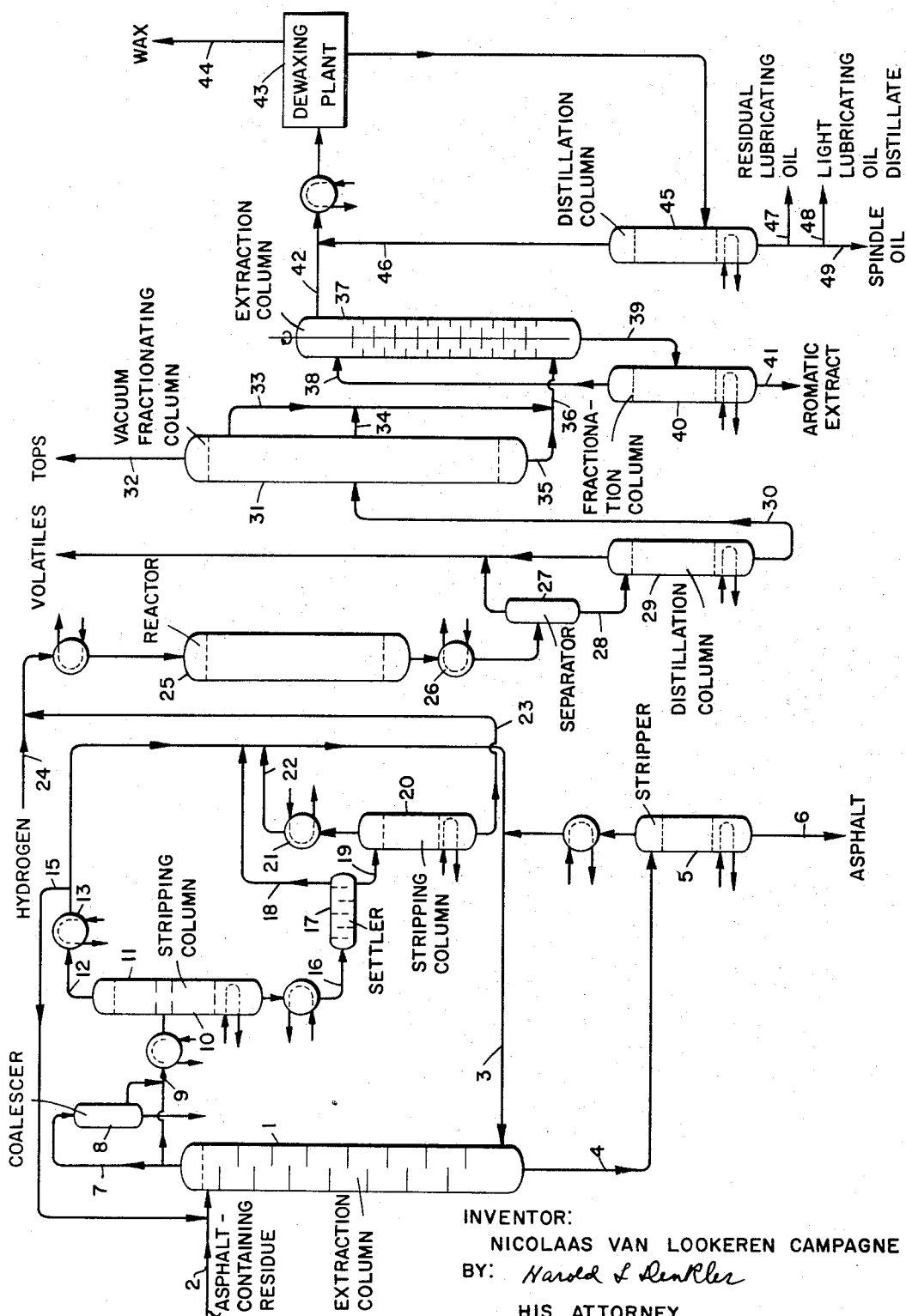
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LUBRICATING OIL BY HYDROTREATING PENTANE-ALCOHOL-DEASPHALTED
SHORT RESIDUE

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1

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LUBRICATING OIL BY HYDROTREATING PENTANE-ALCOHOL-DEASPHALTED SHORT RESIDUE

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This invention relates to a process for the preparation of lubricating oils from asphalt-containing residual petroleum fractions.

In the conventional preparation of lubricating oil from asphalt-containing residues, the residue is first subjected to a fractional distillation in order to obtain light and medium lubricating oils. Subsequently, the remaining heavy residue, the so-called "short residue," is de-asphalted by means of a light hydrocarbon such as propane. In general, after a usual dewaxing operation to lower the pour point and an aromatic extraction operation to improve the viscosity index, the de-asphalted residue is suitable for use as a base lubricating oil. By "base lubricating oil," it is meant an oil which, as such or upon blending with other oil and/or admixture of additives, is ready for sale as a finished lubricating oil.

A disadvantage of the conventional process, however, is that the yield of valuable lubricating oil components from a propane treatment is proportionally low, a relatively large part of the residue, in many cases 60% by volume or more, being removed as an asphaltic residue. These high losses are generally necessary since de-asphalting to a lower yield of "asphalt" would lead to a lubricating oil which does not meet the requirements of present specifications, especially with regard to the Ramsbottom Carbon Test and the viscosity level.

It has now been found that a considerably higher yield of lubricating oil from asphalt-containing residual petroleum fractions is obtained by contacting the residue with a selective solvent mixture consisting essentially of a combination of one or more C_3^+ hydrocarbons and one or more alcohols. With this hydrocarbon-alcohol mixture two non-miscible, liquid phases are formed namely, an asphaltic raffinate phase and an oleaginous extract phase, which can easily be separated. The raffinate phase yields an asphalt-like product with a high content of asphaltenes. After the complete or partial removal of the hydrocarbon-alcohol mixture from the extract phase a de-asphalted oil, or oil solution, is obtained which, after a catalytic treatment with hydrogen at elevated temperature and pressure, yields a viscous base lubricating oil with an excellent viscosity index.

By " C_3^+ hydrocarbons" it is meant hydrocarbons and mixtures thereof having three or more carbon atoms in the molecule.

The process of the invention comprises extracting asphalt-containing petroleum residues with a C_3^+ -hydrocarbon-alcohol mixture, separating the resulting liquid asphaltic raffinate phase from the oleaginous extract phase, separating the extract phase into a hydrocarbon-alcohol mixture and a de-asphalted oil, and subjecting the de-asphalted oil to a catalytic treatment with hydrogen at elevated temperature and pressure.

In the present process the relatively large amount of potential lubricating oil components which is removed with the asphalt in the usual treatment with propane is retained by extracting the residue with a C_3^+ -hydrocarbon-alcohol mixture. The raffinate is mainly asphaltenes and resins and contains little potential lubricating oil components. The extract, which contains the added components, is a complex mixture of relatively high-molec-

2

ular weight hydrocarbons, partly having a naphthenic and polyaromatic structure and as a rule having relatively high sulfur and nitrogen content. As a result of the presence of the additional components, the extract—unlike residues de-asphalted with propane—is not suitable as such to be further refined to lubricating oil by extraction and dewaxing. However, the extract is a suitable lubricating oil stock if it is subjected to a catalytic treatment with hydrogen at elevated temperature and pressure. During this hydrogen treatment, the complex mixture of relatively high-molecular weight naphthenic and polyaromatic compounds are converted into lubricating oil components. In the hydrogen treatment, removal of sulfur and nitrogen is effected and ring opening of polyaromatic and naphthenic structures occurs, which results in hydrocarbons with good viscosity properties. Another advantage of the hydrogen treatment is that upgrading of the other oil components occurs at the same time.

In connection with the foregoing it should be noted that with treatment of asphalt-containing residues with C_3^+ hydrocarbons only, such as pentane, a higher yield of the extract may be also obtained. It has been found, however, that in such a case, the additional components are considerably less suitable to be converted into lubricating oil components by means of a catalytic hydrogen treatment. With the present solvent combination, in which the C_3^+ hydrocarbons act as a solvent and the alcohols as an anti-solvent, a very special separation of the residue is effected. The solvent combination according to the invention has, on the one hand, a solvency for oil which is equal to, or greater than, that of aliphatic hydrocarbons, while on the other hand, it has less solvency for asphaltenes and thus is able to extract deeper into the residue. Also, the separation which is effected in the complex mixture of higher-molecular weight compounds present in the residue is quite different from that obtained with hydrocarbons or alcohols only. The hydrocarbon-alcohol mixture causes, for instance, the polar sulfur and nitrogen compounds, which are potential lubricating oil components—with the exception of the highest-molecular weight representatives—to be completely or substantially extracted, whereas the wax content in the extract is relatively low. According to the present invention much higher yields of lubricating oil can therefore be obtained from a given residue; in many cases this yield is twice as high as that obtained by the conventional method of de-asphalting with propane.

In addition to a considerable increase in yield of lubricating oil components the present process yields at least two other important advantages.

It is possible to carry out the extraction with the present hydrocarbon-alcohol mixture at normal temperature and pressure as opposed to, for example, propane de-asphalting. In addition, distillation costs can be reduced to a considerable degree by carrying out the extraction with a hydrocarbon-alcohol mixture of which the hydrocarbon, or hydrocarbon mixture, has a lower boiling point, or lower boiling range than the alcohol, or alcohol mixture. In this event, only the lower-boiling hydrocarbon component has to be evaporated from the extract phase. Separation between the oil and the alcohol then occurs, an alcohol phase being formed spontaneously. The alcohol layer is easily separated from the oil and is, after being mixed with the light hydrocarbon distilled off, recycled to the process. The oil, as such or after any remaining solvent has been stripped off, is subjected to the catalytic hydrogen treatment.

Another advantage of the present solvent combination is that as a result of both the choice of hydrocarbons and alcohols and the choice of the hydrocarbon to alcohol ratio the desired yield of oil components from asphalt-

containing residues of different natures can be obtained in a simple way.

With respect to the composition of the hydrocarbon-alcohol mixture, the C_3^+ hydrocarbon component preferably consists of one or more aliphatic hydrocarbons with 4–10 carbon atoms in the molecule. Instead of pure hydrocarbons or mixtures thereof technical mixtures, such as hydrocarbon oil fractions—for example, light naphtha fractions with boiling ranges of between 10 and 150° C.—may be used. There is no objection to these mixtures containing minor quantities of other hydrocarbons as long as the mixture as such retains the character of a C_3^+ hydrocarbon. For the sake of simplicity the invention will be described below using pentane as the solvent.

The alcohol component in the solvent combination preferably consists of one or more aliphatic alcohols with 1–4 carbon atoms in the molecule. There is no objection to these alcohols containing other alcohols and/or water as long as the mixture as such retains the character of aliphatic alcohols with 1–4 carbon atoms, preferably not more than 2% by volume of water should be present in the extraction solvent.

The amount of alcohol in the solvent mixture can vary over a broad range and in general is about 15% to about 80% by volume. The amount of alcohol to be used in a given situation depends upon the nature of the alcohol and on the hydrocarbon. With, for example, a pentane/alcohol mixture, the concentration of alcohol is desirably about 15–35% for methanol and 55–80% for butanol.

Very good results were obtained with an extraction solvent consisting of a combination of pentane, methyl alcohol and isopropanol. Especially suitable mixtures consist of 50–75 parts by volume of pentane, 15–35 parts by volume of methyl alcohol and 10–20 parts by volume of isopropanol.

As a rule the extraction is carried out with 2–20 parts by volume and preferably 4–10 parts by volume of hydrocarbon/alcohol mixture per part by volume of asphalt-containing residue. The solvent ratio used in a given situation is determined by, among other factors, the type of residue and by the nature and content of the asphalt components. Relatively high solvent ratios, which—for example—may be useful in bringing about a rapid phase separation, may be used without difficulty, considering the low cost of recovering the solvents from the extract phase.

The yield of oil components usually shows a relatively large increase if the percentage by volume of the hydrocarbon component in the hydrocarbon-alcohol mixture is increased; to a lesser extent this is also the case if a higher ratio by volume of the hydrocarbon-alcohol mixture on residue is chosen. Higher oil yields may also be obtained by performing the extraction at higher temperatures.

In the present process, a raffinate phase is obtained which is liquid at room temperature. This results from the fact that the hydrocarbon-alcohol mixture spreads over both the extract phase and the raffinate phase. As a rule the raffinate phase contains about 35–70 percent by volume of hydrocarbon-alcohol mixture and at this dilution the asphalt phase is sufficiently liquid to ensure rapid separation. If desired, the raffinate phase can be worked to recover additional oil components. Thus, for example, low boiling, normally liquid aliphatic hydrocarbons or solvent mixture can be mixed with the raffinate to precipitate asphaltenes which can be removed in hydrocyclones or other separation devices. After removal of the hydrocarbon-alcohol components, asphalt is obtained in the form of a hard, brittle product, which, owing to its high asphaltene content, is of better quality than propane asphalt and may be applied, for instance, in the preparation of synthetic asphaltic bitumens.

To promote rapid and homogeneous mixing of the asphalt-containing residue and the extraction solvent, it is recommended to lower the viscosity of the residue beforehand, which may be effected, for instance, by dissolving a certain quantity of pentane and/or one or more other low-

viscous hydrocarbons therein. However, the quantity of pentane, etc. added should preferably not be so large as to bring about precipitation of asphaltenes in the residue. Very suitable is, for example, a dilution of 20–35% by volume of pentane calculated on residue. When the residue is thus diluted beforehand, a correspondingly smaller quantity of pentane or the like may be used, if desired, in the solvent combination. The viscosity may also be lowered by introducing the residue into the extraction solvent at a somewhat elevated temperature, for instance, between 80 and 90° C.

The extraction of asphalt-containing residues with the aid of the present hydrocarbon-alcohol mixtures can be effected both batchwise and continuously. It is preferably carried out, however, in a continuously operating, multi-stage countercurrent extraction system, one of the devices which may be considered for this purpose being a column installed vertically and provided with stationary baffles or a column in which a shaft carrying discs has been rotatably mounted, as is described, for instance, in British patent specification 690,730.

Both residues obtained by direct distillation of crude oils and those originating from thermal or catalytic cracking processes may be used as starting materials. It is recommended, however, to start from residues having paraffin-base crude oils as their origin. Further, preference is given to so-called "short residues," i.e. residues not containing light or medium lubricating oil components.

By subjecting the de-asphalted oil to a catalytic treatment with hydrogen at elevated temperature and pressure, polyaromatic and heteroatomic compounds are converted into valuable lubricating oil components with good viscosity properties. This conversion is attended with removal of sulfur and nitrogen as well as ring opening. Further, at elevated reaction temperatures in particular, a certain quantity of lower-molecular hydrocarbons is formed as a result of hydrocracking. The above results in a drop in viscosity which is greater as a higher hydrogenation temperature is chosen. This drop in viscosity is counter-balanced, however, by the relatively high viscosity of the de-asphalted oil. The yield of residual lubricating oil which is obtained from de-asphalted oil treated with hydrogen after removal of light and medium lubricating oil components, usually amounts to at least 1.5 times the quantity obtained according to the conventional mode of preparation, its viscosity being equal to, or higher than, that of the conventional oil.

The hydrogen treatment is carried out at elevated temperature and pressure with the aid of a hydrogenation catalyst. Such catalysts are well known and are available commercially and in general contain as a hydrogenation component one or more elements from the left-hand column of the sixth group (chromium, molybdenum, tungsten) and the eighth group of the Periodic System. These elements are preferably present as the sulfides and as a rule are supported on a carrier.

Carriers, for instance, can be natural or synthetic alumina and/or silica containing materials. Carriers containing silica with alumina can be advantageous, because of their tendency to open polyaromatic and naphthenic ring structures. Silica-alumina cracking catalysts containing about 60–90% silica are suitable carriers. Examples of suitable catalysts are combinations of nickel sulfide and/or cobalt sulfide with molybdenum sulfide and/or tungsten sulfide. The atomic ratio of the metals of the eighth group to those of the left-hand column of the sixth group is, as a rule, between 1:20 and 1:1. The reaction temperature is usually between 370° and 430° C.; temperatures between 380° and 420° C. being preferred, however. Above 420° C. hydrocracking will occur to a considerable extent, as a result of which the yield of lubricating oil is adversely affected. The hydrogen treatment is usually carried out at a pressure of at least 100 atm. and preferably above 150 atm. In general pressures above about 200 atm. are not used.

The hydrogen treatment preferably is effected under so-called "trickle" conditions. According to the "trickle" technique, which is described, for instance, in British patent specification 657,521, the oil, which may be partly liquid and partly in vapor form, passes downwardly over a solid catalyst bed in the presence of hydrogen or a hydrogen-containing gas; the oil flowing in a thin film over the catalyst particles.

Instead of pure hydrogen a hydrogen-containing gas, for instance, a mixture of hydrogen and light hydrocarbons, may be used. When hydrogen is used in excess of that consumed in the treatment, it is advantageous to recycle the excess hydrogen, generally after removal of undesired compounds, if necessary. The gases should preferably contain more than 50% by volume of hydrogen. Very suitable are, for instance, hydrogen-containing gases obtained from the catalytic reforming of gasoline fractions. The space velocity usually is 0.5-5 kg. and preferably 1-3 kg. of de-asphalted oil per hour per liter of catalyst. As a rule the hydrogen:oil ratio is between 100 and 1000 normal liters and preferably between 200 and 500 normal liters of hydrogen per kg. of oil.

In certain cases it may be desirable to subject the lubricating oil to another hydrogen treatment. In this procedure the same type of catalyst may be used as in the first hydrogen treatment, the difference being, however, that the catalyst is supported on an alumina carrier containing substantially no silica. This treatment, which should be looked upon as so-called hydrofining, may be performed at lower temperatures, for example, between 250° C. and 360° C. and preferably between 300° and 350° C. and at the same, or lower, hydrogen pressures, for instance, between 30 and 60 atm., and using 50-200 normal liters of hydrogen per kg. of oil. This second hydrogen treatment may serve, for example, to increase the stability of the oil and/or to improve its color.

After the hydrogen treatment (or treatments), the lubricating oil is usually subjected to a fractional distillation in vacuo to separate tops and to obtain light and/or medium lubricating oil distillates and the residual lubricating oil. The fractions obtained may subsequently be extracted according to the conventional method to remove aromatic compounds, etc., after which, if desired, the raffinates are dewaxed.

It should be noted that the dewaxing procedure according to the present process may be performed, if desired, before the hydrogen treatment, i.e. immediately after the de-asphalting process. This is advantageous, for example, in those cases in which the same type of hydrocarbon-alcohol mixture is used for dewaxing and de-asphalting.

The process of the invention will be described with reference to the drawing which is a schematic flow diagram of a preferred embodiment of the process.

The starting material is a residual oil fraction obtained by distillation of an asphaltic crude oil originating from the Middle East. This residual oil fraction was subjected to fractional distillation in order to obtain a light and a medium lubricating oil fraction.

The remaining asphalt-containing residue, having a sulfur content of 5.22% w. and a kinematic viscosity of 1284 cs. at 210° F., is diluted with a mixture of 20% w. pentane and 2% methyl alcohol in order to lower viscosity. The diluted residue is introduced to extraction column 1 via line 2. The extraction column is provided with baffles and is equivalent to 6 theoretical stages. The asphalt-containing residue is charged at the ratio of 100 parts by weight per hour. The extraction solvent is continuously introduced near the bottom of the column, via line 3, at a rate of 478 parts by weight/hour. The total composition of the extraction solvent, including the quantity added to the residue to lower the viscosity is 300 parts by weight of pentane, 125 parts by weight of methyl alcohol and 75 parts by weight of isopropanol.

The viscous raffinate phase removed from the bottom of the column via line 4 and containing 44 parts by weight of asphalt and 48 parts by weight of solvent mixture, is introduced continuously into stripping column 5 where, at a temperature of 240° C., the volatile components are removed with the aid of nitrogen. The asphalt withdrawn via line 6 has a Ring and Ball temperature of 102° C. and a penetration of <0.1 mm. at 25° C. (ASTM D-5-61).

At the top of the extraction column, the oil-containing extract phase is removed continuously via line 7 in the form of a clear liquid having a dark-green color. The extract phase is first passed through coalescer 8 filled with polypropylene wool so as to remove any entrained droplets of asphalt-containing raffinate phase and is then charged into stripping column 11 via line 9 and heat exchanger 10. Pentane, which is evaporated, is removed via line 12 and, after condensation in cooler 13, is recycled to the process through lines 14 and 15.

The bottom temperature of stripping column 11 is 70° C. and the top temperature 33° C. The heterogeneous mixture of de-asphalted oil and alcohols flowing from the bottom of stripping column 11 is cooled to 20° C. and introduced into settling vessel 17 via line 16. At the top of this settler, the separated alcohol mixture is removed and recycled to the process via line 18. The alcohol contains 2% w. of oil. The de-asphalted oil separated in settling vessel 17 is passed via line 19 to stripping column 20 where, at a bottom temperature of 120° C., remaining extraction solvent is removed with the aid of nitrogen and is recycled to the process via cooler 21 and line 22. In this way, 56 parts by weight of de-asphalted oil per hour is obtained, the properties of which have been listed in column 1 of Table I.

The de-asphalted oil is then subjected to a catalytic hydrogen treatment by charging it (via line 23) together with hydrogen (via line 24) into the top of reactor 25, which has been provided with a fixed catalyst bed, at a pressure of 175 atm. and a temperature of 410° C. The catalyst is sulfided nickel-molybdenum on an alumina carrier. The oil is passed through the reactor at a space velocity of 1 kg. of oil per liter of catalyst per hour, a hydrogen:oil ratio of 500 normal liters of hydrogen per kg. of oil being used. The effluent from reactor 25 passes through cooler 26 and is introduced into separator 27 for the separation of gaseous components, such as hydrogen and hydrogen sulfide. Oil from the separator is passed via line 28 to distillation column 29 to remove other volatile components. Bottoms from column 29 are passed via line 30 to vacuum fractionating column 31. Properties of the oil after the hydrogen treatment are included in column 2 of Table I.

Properties of oil treated with hydrogen at 375° C. under otherwise identical conditions are given in column 3 of Table I.

TABLE I

	1	2	3
	De-asphalted oil	Oil treated with H ₂ at 410° C.	Oil treated with H ₂ at 375° C.
Specific gravity, d 70/4	0.9454	0.8653	0.8863
Kinetic viscosity, 140° F. cs.	1495	36.8	243
VI	60		
Sulfur content, percent w.	4.24	0.22	0.73
Nitrogen content, percent w.	0.25	0.036	0.114
Asphaltene content, percent w.	<0.01	0	0
Ramsbottom carbon value	5.7	0.5	1.4

The oils thus obtained are further separately refined into high viscosity index lubricating oils by subjecting them successively to vacuum fractionation, extraction and dewaxing. During the vacuum fractionation (column 31) a tops fraction (via line 32), a spindle oil fraction (via

line 33) and a light lubricating oil fraction (via line 34) are separated. The bottom product (via line 35) as well

pane, this amounts to an increase in yield of lubricating oil of 82% w. and 84% w., respectively.

TABLE II

Products obtained by subjecting the asphalt-containing residue successively to de-asphalting, hydrogenation and conventional working-up	Yields and properties of experiment with hydrogen treatment at 410° C. (yields calculated on 100 p.b.w. of asphalt-containing residue)		Yields and properties of experiment with hydrogen treatment at 375° C. (yields calculated on 100 p.b.w. of asphalt-containing residue)		Yield and properties of residual lubricating oil according to conventional process, starting from same asphalt containing residue	
	Yields, Percent w.	Properties	Yields, Percent w.	Properties	Yield, Percent w.	Properties
Residual lubricating oil.....	23.0	Kin. visc. cs., 140° F., 165. VI, 95. Pour point, ° C., -12. Flash point, ° C., 270. Ramsbottom carbon test, 0.5.	28.5	Kin. visc. cs., 140° F., 230. VI, 95. Pour point, ° C., -10. Flash point, ° C., 270. Ramsbottom carbon test, 0.9.	15.5	Kin. visc. cs., 140° F., 160. VI, 95. Pour point, ° C., -10. Flash point, ° C., 270. Ramsbottom carbon test, 0.5.
Light lubricating oil distillate..	3.4	Kin. visc. cs., 140° F., 90. VI, 95. Pour point, ° C., -10. Flash point, ° C., >190.				
Spindle oil.....	1.8	Kin. visc. cs., 140° F., 45. VI, 95. Pour point, ° C., -10. Flash point, ° C., >190.				
Tops.....	8.6		1.3			
Gases (ex-hydrogen treatment).	5.5		4.5			
Aromatic extract.....	10.1		18.9			
Paraffin wax.....	6.1		5.3			
Asphalt.....	44.0		44.0			
Total of products and by-products obtained starting from 100 p.b.w. of asphalt-containing residue and 2.5 p.b.w. of hydrogen.	102.5		102.5		15.5	

as the lubricating oil distillate fractions are separately extracted in 4 stages in countercurrent extraction with furfural and subsequently dewaxed in order to obtain oils with a viscosity index of 95 or higher and a pour point of -10° C. or lower. The extraction is performed continuously in extraction column 37 provided with a rotating shaft with discs, a furfural: oil weight ratio of 3.8 being used. The oil fractions collected via lines 33, 34 and 35 are consecutively passed (via line 36) to column 37 near the bottom and are passed counter-currently to the furfural introduced near the top of the extraction column via line 38. The extract phase leaving the extraction column via line 39 is separated in fractionation column 40 into an aromatic extract, which is carried off via line 41, and furfural, which is recirculated to the extraction column via line 38.

After having been diluted with a mixture of equal parts of methylethylketone and toluene and after having been cooled down to -19° C., the raffinates leaving extraction column 37 via line 42 are introduced into dewaxing plant 43, a diluent:oil volume ratio of 4 being used. The wax obtained is carried off via line 44, and the filter oil is charged into distillation column 45. Methylethylketone/toluene is obtained from the top of column 45 and is recycled to the dewaxing plant via line 46. At the bottom of column 45, residual lubricating oil, light lubricating oil distillate and spindle oil are obtained via lines 47, 48, and 49, respectively. The yields of oils, calculated on 100 parts by weight of asphalt-containing residue +2.5 parts by weight of hydrogen, as well as the properties of these oils, have been listed in Table II.

For the sake of comparison, the same asphalt-containing residue was worked up into lubricating oil according to the conventional method by propane de-asphalting, followed by extraction with furfural and dewaxing. The yield of lubricating oil having a viscosity index of 95 and a pour point of -10° C. amounted to 15.5% w.

It is to be noted that by applying the process according to the invention, a considerably higher yield of lubricating oil is obtained, namely, 28.2% w. when the de-asphalted oil is subjected to a treatment with hydrogen at 410° C., and 28.5% w. in the experiment with a hydrogen treatment at 375° C. Calculated on the lubricating oil yield according to the conventional procedure using pro-

The hydrogen treatment used in this example is effected under "trickle" conditions, that is, conditions in which the oil flows downwardly over a fixed catalyst bed in the presence of a hydrogen-containing gas phase. The hydro-treatment may also be carried out such that the necessary hydrogen is completely or substantially dissolved in the de-asphalted oil beforehand. In this case it is quite possible to pass the oil, together with the dissolved hydrogen, upwards through the fixed catalyst bed. If desired, the solvent power may be increased by applying the oil diluted with a solvent having a greater solvent power for hydrogen than the oil. Suitable solvents are, for example, aliphatic hydrocarbons with 5-12 carbon atoms in the molecule or mixtures containing these hydrocarbons, such as naphtha and gasoline fractions.

I claim as my invention:

1. A process for extracting lubricating oils and potential lubricating oils from asphalt-containing residues which comprises contacting the residues with a solvent mixture consisting essentially of a C₄-C₇ aliphatic hydrocarbon and a C₁-C₄ alcohol, the concentration of alcohol in the solvent mixture being dependent upon the alcohol carbon number and varying between the range of 15-35 percent by volume for methanol and the range of 55-80 percent by volume for butanol, said hydrocarbon having a boiling point lower than said alcohol, separating an asphaltic raffinate phase from an oleaginous extract phase, evaporating solvent hydrocarbon from the extract phase which results in the formation of a liquid alcohol phase and a de-asphalted oil phase, and recovering de-asphalted oil.

2. The process according to claim 1 wherein the extraction is carried out with about 2 to 20 parts by volume of solvent per part by volume of asphalt-containing residue.

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