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**PRODUCTION OF LUBRICATING OILS INCLUDING HYDROFINING AN EXTRACT**

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**ABSTRACT OF THE DISCLOSURE**

Lube oils of improved viscosity index are produced in good yield by subjecting a lube oil charge stock to solvent extraction, hydrorefining the aromatic-rich extract, recombining the hydrorefined extract with the feed or the raffinate and then dewaxing.

This invention relates to the production of lubricating oils. More particularly it is concerned with the conversion of low quality lubricating oil stocks into lubricating oils of high viscosity index in good yields.

It has been customary in the past to increase the viscosity index of lubricating oils particularly crankcase oils by the addition thereto of viscosity index improvers. The high-viscosity index oils so produced were then satisfactory for use in automobile engines. However, increased demands for wide temperature range automatic transmission fluids and wide-range multigrade motor oils (such as 5W-30 and 10W-40), which normally would require increased amounts of viscosity index improvers, have created a need for higher VI base oils in order to minimize the quantity of VI improver used and thus improve product shear stability. There is thus now a great need for lubricating base oils which have a relatively high viscosity index. While this may not be too difficult when high quality lubricating oil stocks such as Pennsylvania oils are used as a starting material, there are not sufficient oils of such high quality available to meet the greatly increased demand for high viscosity index lubricating oils of the lower viscosity grades such as SAE 10 and SAE 20 and it is now becoming necessary not only to convert low grade lubricating oil stocks, that is stocks having a viscosity index of less than 80 or even less than 75 to produce oils having a viscosity index of at least about 110 but also to effect this improvement without suffering a substantial loss in yield.

Various processes are available for the refining of lubricating oil charge stocks. Conventionally, the lube oil stock obtained either by vacuum distillation or by deasphalting a vacuum residuum can be improved by solvent extraction to increase the viscosity index and by solvent dewaxing to lower the pour point. Acid treating may be used to improve the color, the stability and the resistance of the oil to oxidation and clay contacting is used to further improve the color and to neutralize the oil after acid treatment. More recently severe catalytic hydrogenation of the oil has been proposed as a substitute for solvent refining to increase the viscosity index of the oil. However the resulting product oil is not stable towards ultra-violet light and with certain feedstocks, relatively low yields are obtained.

It is an object of this invention to produce lubricating oils of high viscosity index from low-quality lubricating oil charge stocks. Another object is to produce lubricating oils of high viscosity index from low quality charge stocks in yields not obtainable by the processes of the prior art. Still another object is to produce lubricating oils which are stable to ultraviolet light. These and other

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objects will be obvious to those skilled in the art from the following disclosure.

According to our invention, there is provided a process for the production of lubricating oils which comprises subjecting a lubricating oil charge stock to solvent refining to produce a raffinate rich in paraffins and naphthenes and an extract rich in aromatics, contacting the extract with a hydrorefining catalyst under hydrorefining conditions and combining at least a portion of the hydrorefined extract with said raffinate or recycling it to the solvent refining step.

Examples of the lubricating oil charge stock contemplated by our process are petroleum fractions having a viscosity between 50 and 500 SUS at 210° F. Although the invention is applicable to any lubricating oil charge stock, it is particularly suitable for the treatment of medium-quality charge stocks, that is, those charge stocks having a viscosity index not greater than about 90 and more particularly for the treatment of low-quality charge stocks, that is, those having a viscosity index below about 80 to produce improved lubricating oils having a viscosity index at least 20 units higher than that of the charge stock.

Lubricating oil stocks are ordinarily obtained from the distillation of crude petroleum. The stock may be obtained as overhead from a vacuum distillation or may be obtained from the residue from vacuum distillation by deasphalting the residue by contact with, for example, a deasphalting agent such as propane, butane and the like and mixtures thereof. According to our process, the lubricating oil fraction is subjected to solvent extraction using a solvent having an affinity for aromatic hydrocarbons. Particularly suitable solvents include furfural, phenol, dichloroethyl ether and N-methyl-2-pyrrolidone. Advantageously, the extraction is carried out using a countercurrent flow technique, the solvent being introduced at the top of an extraction tower and the oil near the bottom with the tower being maintained at a temperature between about 150 and 250° F. The solvent to oil ratio may range between about 1 to 6 by volume. Oil is recovered from the top of the tower as raffinate and is advantageously further processed by heating and steam stripping to remove residual solvent. Solvent and extract are removed from the bottom of the extraction tower and are separated by distillation.

The extract is then subjected to catalytic hydrorefining at a temperature between about 600 and 900° F., a pressure between about 800 and 5000 p.s.i.g., a space velocity of about 0.1 to 5.0 volumes of oil per volume of catalyst per hour with a hydrogen rate of between about 1500 and 20,000 standard cubic feet per barrel of charge. Preferably, the temperature is maintained within the range of 650-830° F., the pressure between 1300 and 3,000 p.s.i.g., the space velocity between 0.15 and 1.5, (v./v./hr.) and the hydrogen rate between 3,000 and 10,000 s.c.f.b The gas used for the hydrogenation need not necessarily be pure hydrogen. Hydrogen having a purity of at least 65 percent and preferably at least 75 percent may be used.

The catalysts used in the hydrorefining step generally comprise a hydrogenating component carried on a support. The principal ingredient of the hydrogenating component is a Group VIII metal or mixtures of Group VIII metals or compounds thereof such as the oxides or sulfides. Examples of Group VIII metals which may be used in the hydrogenating component are nickel, cobalt and iron or mixtures thereof. The iron group metal should be present in an amount between about 2 and 40 percent preferably 3-15 percent based on the total weight of the catalyst composite. In conjunction with the iron group metal, a Group VI metal such as molybdenum or

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tungsten may be used. In such case the Group VI metal may be present in an amount between about 5 and 40 percent based on the weight of the composite, a preferred range being from 10 to 25 percent.

The hydrogenating component is carried on a base comprising a refractory inorganic oxide material such as alumina, silica, magnesia, zirconia, titania, crystalline aluminosilicates and the like and mixtures thereof. When the lubricating oil extract introduced into the hydrorefining zone is derived from a deasphalted residuum and it is desired to convert the extract into a lubricating oil of the SAE 10 or SAE 20 grade then advantageously the catalyst has an acidic support such as a crystalline aluminosilicate of the zeolite Y type of reduced alkali metal content, e.g., less than 1.0 weight percent composited with a mixture of an amorphous inorganic oxide material such as 70-90% silica and 10-30% alumina. If the extract has been obtained from a wax distillate, then advantageously the catalyst support has little if any cracking activity.

If the catalyst has cracking activity which on occasion it may be desired to suppress, then a small amount of ammonia or carbon monoxide may be added to the hydrogen. A suitable amount depending on the extent to which the cracking is to be suppressed will range between about 0.1 and 2.0 percent ammonia or carbon monoxide basis reactor feed gas. The catalyst may be used in the form of a slurry, a fluidized bed or a fixed bed. When used in the form of a fixed bed, the oil and hydrogen flow may be either upward or downward or the flow of hydrogen may be countercurrent to the downward flow of oil. In broad terms, the catalysts covered by the scope of this invention are those which have suitable activity for carrying out the reactions described herein. Included are those containing from 2 to 10 percent cobalt or nickel and 10 to 30 percent molybdenum or tungsten. Particularly suitable catalysts are those containing about 6 percent nickel and 20 percent tungsten or about 5 percent nickel or cobalt and about 12-15% molybdenum.

In one embodiment of the invention the hydrorefined extract after being subjected to a separation treatment for the removal of hydrogen and light distillates is recycled to the solvent refining zone into which it is introduced with fresh feed. In another embodiment of the invention the hydrorefined extract, after separation of the lighter materials, is combined with the raffinate from the solvent extraction zone and the combined stream is subjected to solvent dewaxing.

To improve its pour point, the oil is contacted with a dewaxing agent such as a mixture of equal parts of a ketone, for example, acetone or methylethyl ketone and an aromatic compound such as benzene or toluene, in a ratio of about 3 to 4 parts by volume of solvent per volume of oil. The mixture is cooled to a temperature of about 0 to -20° F. depending upon the desired pour point and the waxy components are removed from the chilled mixture by filtering or by centrifuging. The dewaxed liquid is then subjected to flash distillation and stripping to remove the solvent.

It is also within the contemplation of the invention that the hydrorefining may be effected in multiple stages. For example, if a two-stage process is employed, the extract is subjected in the first stage to a prehydrogenation adapted particularly to saturate aromatic compounds and the hydrogenated product is then subjected to catalytic hydrocracking in the second stage. When this particular embodiment is practiced, the first stage catalyst comprises a conventional hydrogenation component on a support such as alumina which has minimum cracking activity and the second stage utilizes a hydrocracking catalyst comprising a hydrogenation component on a support which is acidic in nature and is adapted to effect considerable conversion of the product from the prehydrogenation zone into lower molecular weight compounds. Such a second stage catalyst may comprise 0.1-5 weight per-

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cent platinum or palladium and 0.1-5% rhenium on a support such as low alkali metal content zeolite in admixture with one or more amorphous inorganic oxides such as a mixture of silica and alumina containing 70-90 weight percent silica and 10-30 weight percent aluminum.

It is also within the contemplation of the invention that the raffinate, in addition to solvent dewaxing, may be subjected to a hydrofinishing treatment which is a hydrogenation under quite mild conditions, generally, a temperature between about 400 and 650° F. and a pressure between about 400 and 1500 p.s.i.g. The catalyst used for the hydrofinishing treatment may be the same as the catalyst used for the prehydrogenation of the extract or a catalyst similar thereto. The hydrofinishing treatment is used particularly when the hydrorefined extract is combined with the raffinate rather than recycled to the solvent extraction step. The hydrofinishing treatment may precede or may follow the solvent dewaxing step. However, regardless of the sequence of the solvent dewaxing and the hydrofinishing, the hydrorefined extract is combined with the raffinate prior to the solvent dewaxing step.

The following examples are submitted for illustrative purposes only.

#### EXAMPLE I

In this example the charge stock is a distillate oil having the following characteristics:

TABLE I

Viscosity SUS/210° F. ....	55.7
Viscosity index .....	77
Pour point, ° F. ....	95

The charge is brought into contact with a catalyst composed of 6% nickel and 21% tungsten on a support composed of 22% decationized hydrogen form zeolite Y, 65% silica and 13% alumina, at a temperature of 680° F., a pressure of 1800 p.s.i.g., a space velocity of 1.0 v./v./hr. and a hydrogen rate of 6000 s.c.f.b. After separation of the lighter materials from the lube oil fraction, the lube oil cut is dewaxed by mixing it with a 50-50 mixture of methyl ethyl ketone and toluene, cooling the mixture to a temperature of -10° F., filtering the mixture and distilling off the solvents. The dewaxed oil is then solvent extracted with N-methyl-2-pyrrolidone at a solvent-to-oil ratio of 2:1 and a temperature of 180° F. to obtain a product having a viscosity index of 107. The characteristics of the intermediate and final products are set forth below in Table 2:

TABLE 2

	Hydrogenated feed	Dewaxed oil	Solvent refined dewaxed oil
Viscosity, SUS/210° F. ....	44.5	45.8	46.5
Viscosity Index .....	114	100	107
Pour point, ° F. ....	100	5	5
Yield, volume percent basis feed ...	84	63	55

The above examples represent a conventional procedure in which a lubricating oil fraction is subjected to severe hydrogenation, the hydrogenation product is dewaxed and a dewaxed oil is solvent extracted to obtain a product oil of the desired viscosity index. The solvent dewaxed oil, prior to solvent refining, is unstable to ultraviolet radiation whereas the solvent refined oil is stabilized in this respect.

#### EXAMPLE II

This example represents one embodiment of the process of the present invention. The charge stock in this example is the same as that used in Example I. However, the procedure in this example differs from that of Example I in that in this case the charge stock is first subjected to a solvent extraction treatment using N-methyl-2-pyrrolidone at a solvent-to-oil ratio of 2:1 and a temperature of 180° F. The extract is then sub-

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jected to hydrotreating treatment under the same conditions as the total charge in Example I. The hydrotreated extract is then combined with the raffinate and the combined stream is solvent dewaxed with a 50-50 mixture of methyl ethyl ketone and toluene at a temperature of  $-15^{\circ}$  F. Data on the intermediate products and the final product are set forth in Table 3 below in which the various columns are designated as follows:

- (1) Raffinate
- (2) Extract
- (3) Hydrotreated extract
- (4) Raffinate + hydrotreated extract
- (5) Dewaxed oil

TABLE 3

	1	2	3	4	5
Viscosity, SUS/210° F.....	51.6	71.0	39	44.7	46
Viscosity Index.....	110	11	100	114	107
Pour point, ° F.....	100	-----	80	90	0
Yield, volume percent basis feed.....	58	42	84	92	69

The product from Example II shows good stability toward ultraviolet light. It is also to be noted that although the product oils from Examples I and II have substantially the same characteristics, the product of Example II is obtained in a yield 25% greater than that of Example I.

## EXAMPLE III

This example is a repeat of Example II with the exception that the hydrogenated extract instead of being combined with the raffinate is recycled to the solvent extraction zone. By processing the raffinate as in Example II a product oil similar to that set forth in Column 5 of Table 3 is obtained with the exception that the product oil is obtained in a yield of 73% basis feed.

The above examples are for illustrative purposes only. Various other modifications of the invention as hereinbefore set forth may be made without departing from the spirit and scope thereof and only such limitations should be imposed as are indicated in the following claims.

We claim:

1. A process for the production of a lubricating oil of improved viscosity index which comprises passing a lubricating-oil reactant stream through a solvent extraction zone, separating from said reactant stream an aromatic

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rich extract then passing said reactant stream through a dewaxing zone to decrease the pour point thereof, subjecting said aromatic rich extract to hydrotreating by contacting same with a catalyst comprising an iron group metal or compound thereof supported on a base having cracking activity comprising a crystalline aluminosilicate of reduced alkali metal content at a temperature between  $650$  and  $830^{\circ}$  F. in the presence of added hydrogen and combining the hydrotreated extract with the reactant stream prior to its introduction into said dewaxing zone.

2. The process of claim 1 in which the hydrotreated extract is combined with the reactant stream prior to the introduction of said reactant stream into the solvent extraction zone.

3. The process of claim 1 in which the hydrotreated extract is combined with said reactant stream after said stream leaves said solvent extraction zone and prior to its introduction into the solvent dewaxing zone.

4. The process of claim 1 in which after solvent extraction the reactant stream is subjected to hydrofinishing.

5. The process of claim 4 in which the hydrofinishing precedes the dewaxing.

6. The process of claim 4 in which the hydrofinishing follows the dewaxing.

7. The process of claim 1 in which the hydrotreating is effected in two catalyst stages.

8. The process of claim 7 in which the first catalyst stage is principally an aromatic saturation stage.

9. The process of claim 1 in which the iron group metal is nickel or cobalt.

10. The process of claim 1 in which the lubricating oil reactant stream charged to the solvent extraction zone is a deasphalted residuum.

11. The process of claim 1 in which the solvent is N-methyl-2-pyrrolidone.

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