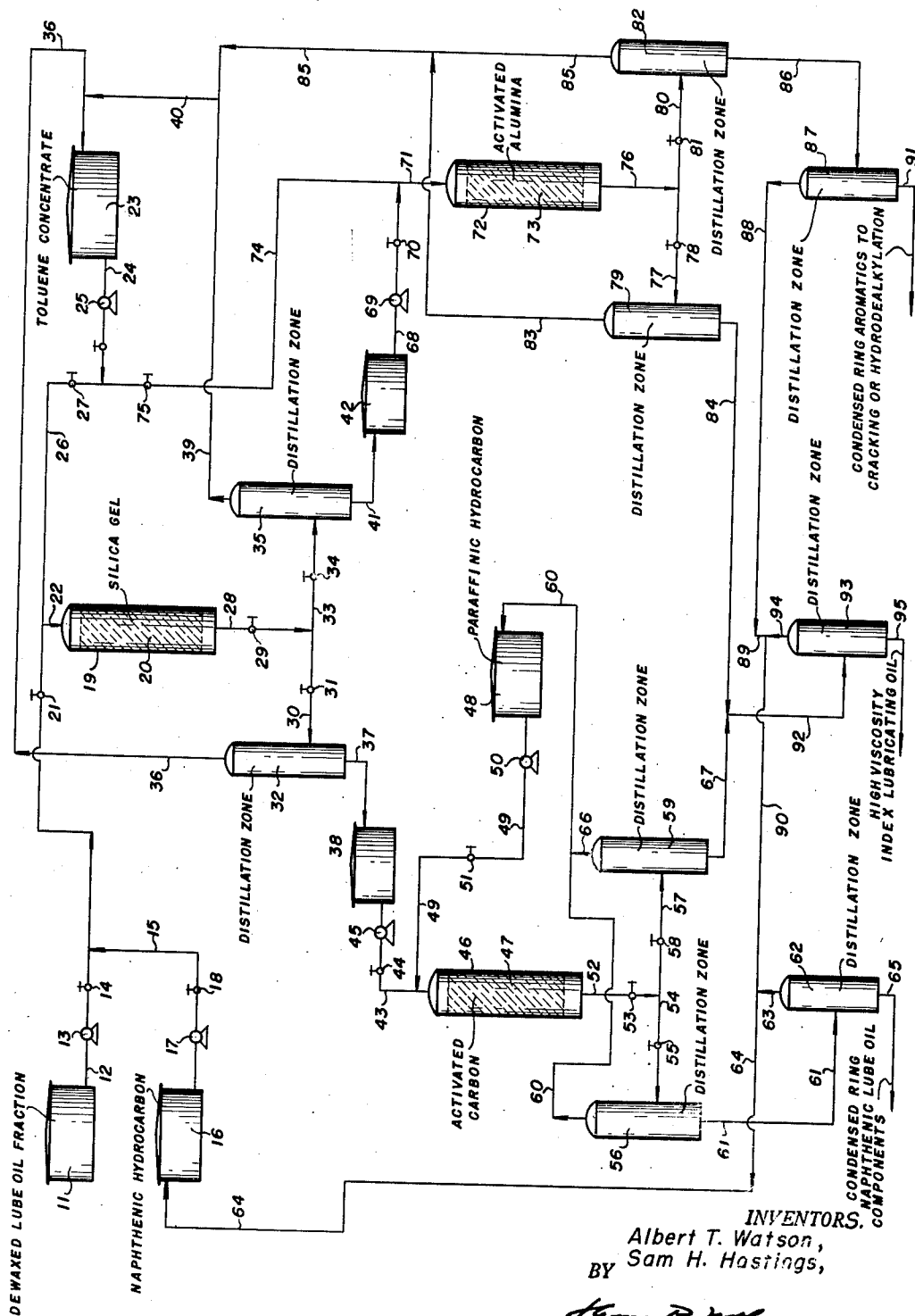


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PRODUCTION OF HIGH VISCOSITY INDEX
LUBRICATING OILS

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The present invention is directed to a method of producing high viscosity index lubricating oils. More particularly, the invention is directed to a method by which deleterious lubricating oil components are removed from a lubricating oil fraction containing them. In its more specific aspects, the invention is directed to a three-stage adsorptive separation process in which a lubricating oil fraction containing beneficial and detrimental components is separated selectively into the beneficial components and the detrimental components and the former recovered.

The invention may be described briefly as involving a method for producing high viscosity index lubricating oil from a lubricating oil fraction containing condensed ring naphthenic and condensed ring aromatic hydrocarbons in admixture with paraffinic and single ring naphthenic and single ring aromatic hydrocarbons in which the condensed ring naphthenic and the condensed ring aromatic hydrocarbons are removed and a lubricating oil containing substantially only paraffinic and single ring naphthenic and single ring aromatic hydrocarbons is recovered.

The invention involves also a method for producing high viscosity index lubricating oil in which a lubricating oil fraction containing paraffinic, single and condensed ring naphthenic and single and condensed ring aromatic hydrocarbons is separated into a first fraction containing predominantly saturated lubricating oil components and a second fraction containing predominantly aromatic lubricating oil components. The first fraction is separated into a third fraction containing condensed ring naphthenic lubricating oil components and a fourth fraction containing paraffinic and single ring naphthenic lubricating oil components. The second fraction is separated into a fifth fraction containing single ring aromatic lubricating oil fractions and a sixth fraction containing condensed ring aromatic lubricating oil components. The fourth and fifth fractions are combined and there is recovered from the combined fourth and fifth fractions a lubricating oil of high viscosity index, higher than that producible in conventional operations.

The invention may be practiced such that the lubricating oil fraction is separated into a predominantly saturated lubricating oil component and a predominantly aromatic lubricating oil component by charging the lubricating oil fraction and an aromatic hydrocarbon fraction alternately to a bed of silica gel, such that the predominantly aromatic-containing components in

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the lubricating oil are adsorbed preferentially with the predominantly saturated lubricating oil components being non-adsorbed. On charging of the aromatic hydrocarbon fraction, which has a boiling point lower than the boiling point of the lubricating oil fraction, the aromatic hydrocarbon fraction is adsorbed and the aromatic lubricating oil components are desorbed allowing them to be recovered.

The predominantly saturated lubricating oil component which will include paraffins, condensed and single ring naphthenes, is then charged to a bed of activated carbon and the feed thereto alternated with a paraffinic hydrocarbon solvent having a boiling point lower than the boiling point of the lubricating oil fraction and the boiling point of the aromatic hydrocarbon fraction. By alternately charging the predominantly saturated lubricating oil component and the paraffinic hydrocarbon solvent to the bed of activated carbon the single ring naphthenic and the paraffinic hydrocarbons are selectively adsorbed and the condensed ring naphthenic hydrocarbons are substantially unadsorbed, allowing a separation to be made and two fractions to be recovered from the bed of carbon alternately, one fraction containing condensed naphthenic hydrocarbon components and the other fraction containing paraffinic and single ring naphthenic lubricating oil components.

The predominantly aromatic hydrocarbon fraction recovered from the bed of silica gel is charged to a bed of alumina gel and the feed thereto alternated with an aromatic hydrocarbon fraction having a boiling point lower than the boiling point of the lubricating oil fraction whereby the predominantly aromatic lubricating oil component is separated into a fraction containing single ring aromatic lubricating oil components and another fraction containing condensed ring lubricating oil components, the alumina gel adsorbing selectively the condensed ring aromatic lubricating oil components which are desorbed by the aromatic hydrocarbon fraction which, in turn, is adsorbed by the alumina gel resulting in the alternate recovery from the bed of fractions containing single ring aromatic lubricating oil components in one instance and in another instance condensed ring aromatic lubricating oil components. The fractions which contain the single ring aromatic components and the paraffinic and single ring naphthenic lubricating oil components are combined and subjected to a recovery operation, such as a distil-

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lation operation to allow recovery of a finished lubricating oil fraction consisting substantially of single ring aromatic and single ring naphthenic hydrocarbons and paraffinic hydrocarbons.

The silica gel employed in the practice of the present invention will be the silica gel of commerce having a mesh in the range from about 14 to about 250 mesh but which may range as high as 350 mesh. Silica gel is a well known article of commerce and further description thereof need not be given here.

The activated carbon or activated charcoal, as it is sometimes termed, may be obtained from a large number of well known sources. For example, the activated carbon or charcoal may be prepared from bagasse, kelp, fruit pits, coconuts, farm products, such as corn cobs, cereals, waste cellulosic material, blood, petroleum acid sludges, petroleum residues and many other materials too numerous to mention here. The activated carbon or charcoal is used in a sense that it has been activated and will adsorb selectively hydrocarbons by types. Methods of activating carbon or charcoal are well known and since these methods do not form part of the invention they will not be described here. The activated carbon or charcoal will have mesh sizes comparable to that specified for silica gel.

The alumina gel will suitably be activated alumina and may be prepared by any of the methods well known to the art, such as, for example, by heating alpha alumina trihydrate to drive off combined water, or by drying and heating the gel formed by precipitation of $AlCl_3$ with ammonia or alkali. See also "Encyclopedia of chemical technology" edited by R. E. Kirk and Donald F. Othmer, the Interscience Encyclopedia Inc., New York, 1947, vol. I, pp. 642-3. The alumina gel will have mesh sizes comparable to that of the silica gel and the activated charcoal.

The invention will suitably be practiced at temperatures in the range from about 60° up to about 120° F. with preferred temperatures in the range from about 80° to about 100° F.

The several porous adsorbents including silica gel, activated carbon and alumina gel may suitably be used in beds and particularly in elongated vertical beds. It is desirable to provide the beds having a length to diameter ratio in the range from about 1:1 to about 200:1 with length to diameter ratios in the range from about 3 to about 100:1 being preferred. Good results may be obtained with length to diameter ratios of about 14 to 28:1. It may also be desirable to provide suitable internal baffling equipment, such as a vertical and horizontal baffles, in the columns to prevent channeling and also to allow contact between the streams flowing through the bed and the porous adsorbent; in fact, the beds may be suitably partitioned to provide in one particular bed a plurality of small beds having high ratios of length to diameter.

In view of the viscous nature of the lubricating oil fraction which might make the process of the present invention unattractive due to slow flow through the beds and in view of the upper temperature limit of 120° F. in which satisfactory separation is effected, it is desirable to admix the lubricating oil fraction with a naphthenic solvent or fraction boiling in the range from about 100° to about 360° F. This naphthenic solvent may contain hydrocarbons having 10 carbon atoms in the molecule. The naphthenic hydrocarbon solvent, it will be clear, boils below the boiling point of the lubricating oil fraction.

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The aromatic hydrocarbon fraction which is charged alternately to the beds of silica gel and alumina gel is suitably a toluene concentrate boiling in the range from about 90° to about 320° F. and containing approximately 50 to 100% of toluene but may be a xylene-containing fraction, if desired. When the aromatic hydrocarbon fraction is a toluene concentrate it will have a boiling point lower than the naphthenic hydrocarbon fraction.

The paraffinic hydrocarbon fraction which is charged alternately to the bed of activated carbon suitably has a boiling point in the range from 80° to 310° F. and should boil below the boiling point of the naphthenic hydrocarbon fraction.

The present invention will be further illustrated by reference to the drawing in which the single figure is a flow diagram of a preferred mode of operation.

Referring now to the drawing numeral 11 designates a charge tank which contains, for example, a dewaxed lubricating oil fraction which is introduced into the system by line 12 containing pump 13 and valve 14. By operating pump 13 and opening valve 14 the lube fraction is pumped through line 12. Line 12 is joined by line 15 which connects to tank 16 which contains a naphthenic hydrocarbon fraction which is introduced by line 15 containing pump 17 and valve 18 to adjust the viscosity of the lube fraction in line 12. Ordinarily the ratio of lube fraction to naphthenic hydrocarbon fraction will be about 1:2 but other ratios may be employed depending on the viscosity of the lube fraction. It should be sufficient to say that a sufficient amount of the naphthenic hydrocarbon fraction should be introduced by line 15 into line 12 to provide a viscosity of the admixture in the range from about 30 to 200 SSU which will allow the mixture to flow readily through adsorption column 19 which contains a bed of silica gel 20 indicated generally by the shaded portion.

The admixture of lube fraction and the naphthenic hydrocarbon flows through line 12 controlled by valve 21 into branch line 22 and thence downwardly through the bed of silica gel resulting in the selective adsorption of the predominantly aromatic components in the lube fraction. After the capacity of the silica gel for the aromatics present in the lube fraction is used, flow through lines 12 and 15 is discontinued by shutting down pumps 13 and 17 and closing valves 14 and 18, valve 21 being also closed. At this point in the operation, an aromatic hydrocarbon fraction, such as a toluene concentrate, is introduced into the system from tank 23 and through line 24 containing pump 25. Line 24 connects into line 26 which also connects into line 22. The valve 27 in line 26 is in the open position and allows flow from tank 23 through lines 24, 26 and 22. The toluene concentrate serves to desorb the aromatic lubricating oil components adsorbed on the silica gel bed 20 with the aromatics in the toluene concentrate being adsorbed selectively. These operations are alternated such that there is recovered from the adsorption zone 19 by line 28 controlled by valve 29 a first fraction and a second fraction. The first fraction contains the substantially saturated lubricating oil components which are discharged by line 30 controlled by valve 31 into a distillation zone 32, while the second fraction is alternately discharged by line 33 controlled by valve 34 into distillation tower 35. The second fraction contains the aromatic lubricating

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oil components in admixture with the naphthenic hydrocarbon and the toluene concentrate while the saturated lubricating oil components introduced into distillation tower 32 contains naphthenic hydrocarbon and toluene concentrate. In distillation zone 32 which is a conventional distillation apparatus and which is contemplated will be provided with all auxiliary equipment necessary for such operation, the predominantly saturated fraction is distilled to recover therefrom the toluene concentrate which is withdrawn as an overhead fraction through line 36 and routed thereby back into tank 23, the saturated lubricating oil components in admixture with the naphthenic hydrocarbon fraction being discharged by line 37 into a stock tank 38 from which this fraction is withdrawn for further processing as will be described.

The predominantly aromatic lubricating oil fraction introduced into distillation zone 35 by line 33 is distilled to recover as an overhead fraction toluene concentrate which is withdrawn by line 39 and introduced back into tank 23 by way of branch line 40 which connects into line 36. It is understood, of course, that distillation zone 35, like distillation zone 32, is provided with all auxiliary equipment necessary for such distillation operation. The predominantly aromatic lubricating oil components in admixture with naphthenic hydrocarbon fractions are withdrawn from zone 35 by line 41 into a stock tank 42 from whence the aromatic lubricating oil components may be withdrawn for further processing as will be described.

The predominantly saturated lubricating oil components in tank 38 are alternately charged by line 43 containing valve 44 and pump 45 into a second adsorption zone 46 which contains a bed of activated carbon 47 indicated generally by the shaded portion. The flow of the saturated lubricating oil components through adsorption zone 46 is alternated with flow of a paraffinic hydrocarbon which is withdrawn from tank 48 and introduced into zone 46 by line 49 containing pump 50 and controlled by valve 51. The substantially saturated lubricating oil components in tank 38 are charged to the zone 46 until the capacity of the activated carbon therein for adsorbing the paraffins and the single ring naphthenes which are more strongly held on the activated carbon than the condensed ring naphthenes has been utilized. When the capacity of the activated carbon has been utilized for adsorbing the paraffins and the single ring naphthenes, flow into adsorption zone 46 from tank 38 is interrupted by shutting down pump 45 and closing valve 44; then flow from tank 48 is initiated by starting up pump 50 and opening up valve 51. As a result the condensed naphthenes which have a very low viscosity index and are not as strongly adsorbed as the more paraffinic materials appear in the effluent from zone 46 prior to the better quality lubricating oil fractions which are more paraffinic in nature. As a result of alternating the flow of the fraction from tank 38 with the paraffinic hydrocarbons in tank 48, there are withdrawn from zone 46 by line 52 controlled by valve 53 third and fourth fractions which are introduced, respectively, by line 54 controlled by valve 55 into distillation zone 56 and by line 57 controlled by valve 58 into distillation zone 59. The fraction in line 54 is the third fraction which contains condensed ring lubricating oil naphthenic hydrocarbon fraction and the paraffinic hydrocarbon fraction while the fraction in line 57 is the fourth fraction which contains paraffinic

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and single ring naphthenic lubricating oil components as well as naphthenic hydrocarbon and paraffinic hydrocarbon fractions.

It is to be understood that distillation zones 56 and 59 are similar to distillation zones 32 and 35 and are provided with all auxiliary equipment needed for all such operations.

By operating distillation zone 56 the paraffinic hydrocarbon fraction being of a lower boiling point than the naphthenic boiling fraction is withdrawn overhead from zone 56 by line 60 and is discharged thereby back into tank 48 for reuse in the process. A fraction containing condensed ring lubricating oil components and naphthenic hydrocarbon fraction is discharged by line 61 into a distillation zone 62 where a separation is made between the condensed ring naphthenic lubricating oil components and the naphthenic hydrocarbon fraction, the latter being recovered by line 63 as an overhead fraction and returned by line 64 to tank 48 for reuse in the process as has been described, the condensed ring naphthenic lubricating oil components being discharged from the system by line 65 for further use as may be desired.

The paraffinic lubricating oil fraction containing the single ring naphthenic lubricating oil components in admixture with naphthenic and paraffinic hydrocarbon fractions is distilled in zone 59 to remove as an overhead fraction the paraffinic hydrocarbon fraction by line 66 which connects into line 60 and returns the paraffinic hydrocarbon fraction to tank 48 while the paraffinic and single ring aromatic lubricating oil components are withdrawn from zone 59 by line 67 for further processing as will be described.

The predominantly aromatic hydrocarbon effluent from adsorption zone 19 which is contained in tank 4 and is an admixture of aromatic lubricating oil components and naphthenic solvent is withdrawn by line 68 containing pump 69 and valve 70 and routed into line 71 which introduces the admixture into a third adsorption zone 72 which is provided with a bed 73 of alumina gel indicated generally by the shaded portion. Alternately to the charge of the aromatic lubricating oil material contained in tank 42 there is charged to the adsorption zone 72 the toluene concentrate contained in tank 23 which is routed to zone 72 by line 74 which connects into line 74 controlled by valve 75 which, in turn, connects into line 71. By alternately charging the toluene concentrate and the aromatic lubricating oil components in tank 42, it is possible to separate the aromatic lubricating oil components into a fraction containing condensed ring aromatic lubricating oil components and a fraction containing single ring lubricating oil components. The alumina gel in bed 73 will selectively adsorb the condensed ring aromatic lubricating oil components which will allow a separation. By charging alternately to the charge of the aromatic lubricating oil components the toluene concentrate, it is possible to desorb the adsorbed material from the alumina gel and to obtain fifth and sixth fractions from the adsorption zone 72. Thus the effluent discharged by line 76 may be separated into a fifth fraction which is introduced by line 77 controlled by valve 78 into a distillation zone 79. The fifth fraction will contain the single ring aromatic lubricating oil components, toluene concentrate and naphthenic hydrocarbon solvent. A sixth fraction which contains condensed ring aromatic lubricating oil components, toluene concentrate and a naphthenic

fraction is routed by line 80 controlled by valve 81 into a distillation zone 82. Distillation zones 79 and 82 are like the other distillation zones in that they are provided with all auxiliary equipment necessary for such operations. In distillation zone 79 conditions are adjusted to remove the toluene concentrate as an overhead fraction by line 83 for recycling to tank 23 as will be described further. A fraction containing single ring aromatic lubricating oil components and naphthenic hydrocarbon fraction is withdrawn from distillation zone 79 by line 84 for further processing as will be described.

The condensed ring lubricating oil components introduced into zone 82 by line 80 is also distilled by adjustment of conditions in zone 82 to allow recovery of the toluene concentrate as an overhead fraction by line 85. As will be noted, line 83 ties into line 85. Line 85 connects into line 40 which, in turn, connects to line 36 allowing the return of the toluene concentrate to tank 23. The condensed ring aromatic lubricating oil components and naphthenic hydrocarbon fraction is withdrawn from zone 82 by line 86 which introduces this material into a distillation zone 87 which is like the other distillation zones and is employed to separate the naphthenic hydrocarbon fraction from the condensed ring aromatic lubricating oil components. The conditions are adjusted in zone 87 to remove the naphthenic hydrocarbon fraction by line 88 which connects into line 89 and thence with line 90 which allows the naphthenic hydrocarbon fraction to be returned by line 64 to tank 16 for reuse in the process.

The condensed ring aromatic lubricating oil components are withdrawn from the system by line 91 and thereafter used as a cracking stock or as a feed stock to a hydro-dealkylation unit.

The fractions withdrawn from zones 59 and 79, which are the fourth and fifth fractions, by lines 67 and 84 are admixed and introduced by line 92 into still another distillation zone 93 which is like the other distillation zones which have been described. Conditions are adjusted in zone 93 to remove as overhead fraction the naphthenic hydrocarbon solvent by line 94 which connects into line 90 allowing the naphthenic hydrocarbon fraction to be routed by line 64 to tank 16. The finished lubricating oil of high viscosity index is withdrawn from zone 93 by line 95. This lubricating oil consists substantially of only single ring aromatic hydrocarbons and single ring naphthenic hydrocarbons and paraffinic hydrocarbons, the condensed ring naphthenic and aromatic hydrocarbons having been discarded as has been described.

A feature of the present invention is employing porous adsorbents which will separate among the various types of hydrocarbons in a lubricating oil fraction. Another feature of the invention is employing hydrocarbon fractions for use as diluents and desorbing agents which have divergent boiling points such that the viscosity of the lubricating oil fraction is reduced and such that the adsorbents are regenerated by alternately flowing the aromatic hydrocarbons fraction and the paraffinic hydrocarbon fraction through the beds of porous adsorbent as has been described. By virtue of the divergent or different boiling points, it is possible to make a separation among the various hydrocarbons separated as to types as provided in our invention.

Suitable feed stocks for the practice of the present invention are, for example, lubricating oil

fractions, such as distillates or residua, from Panhandle crudes from the Panhandle field in Texas and from Texas Coastal crudes. The fractions are preferably deasphalted and dewaxed prior to passing them to the adsorption system.

The following table shows the volume percent yield and the V. I. characteristics of hydrocarbon fractions obtainable from a dewaxed Panhandle Medium Motor Oil Distillate.

Table

	Vol. Percent	V. I.
Total Fraction.....	100	91.4
Separate fractions:		
Paraffinic and naphthenic.....	60	124
Condensed naphthenic.....	10	47
Single ring aromatic.....	8	95
Condensed ring aromatic.....	22	38 to -81

It will be seen from these data that by blending together the paraffinic and naphthenic fraction, which has a viscosity index of 124, and the single ring aromatic fraction having a viscosity index of 95, a lubricating oil of extremely high viscosity index may be obtained in good yield. By the conventional process of phenol extraction, it is not possible to raise the V. I. of such a distillate appreciably above 100, regardless of the severity of the extraction. For example, a V. I. of about 105 will be obtained when phenol extracting such a stock to about 60% yield.

The nature and objects of the present invention having been completely described and illustrated, what we wish to claim as new and useful and to secure by Letters Patent is:

1. A method for producing a high viscosity index lubricating oil which comprises separating by adsorption on silica gel a lubricating oil fraction containing paraffinic, single and condensed ring naphthenic and single and condensed ring aromatic components into a first fraction containing predominantly saturated lubricating oil components and a second fraction containing predominantly aromatic lubricating oil components, separating by adsorption on activated carbon the first fraction into a third fraction containing condensed ring naphthenic lubricating oil components and a fourth fraction containing paraffinic and single ring naphthenic lubricating oil components, separating by adsorption on activated alumina the second fraction into a fifth fraction containing single ring aromatic lubricating oil components and a sixth fraction containing condensed ring aromatic lubricating oil components, combining said fourth and fifth fractions and recovering from said combined fourth and fifth fractions a lubricating oil of high viscosity index.

2. A method for producing a high viscosity lubricating oil which comprises alternately charging to a bed of silica gel a lubricating oil fraction containing paraffinic, single and condensed ring naphthenic, and single and condensed ring aromatic hydrocarbons and an aromatic hydrocarbon fraction of a boiling point lower than that of the lubricating oil fraction, alternately recovering from the bed of silica gel a first fraction containing predominantly saturated lubricating oil components and a second fraction containing predominantly aromatic lubricating oil components, alternately charging to a bed of activated carbon the first fraction and a paraffinic hydrocarbon fraction of a boiling point lower than that of the aromatic hydrocarbon fraction,

alternately recovering from the bed of activated carbon a third fraction containing condensed ring naphthenic lubricating oil components and a fourth fraction containing paraffinic and single ring naphthenic lubricating oil components, alternately charging to a bed of alumina gel the second fraction and an aromatic hydrocarbon fraction having a boiling point lower than the lubricating oil fraction, alternately recovering from the bed of alumina gel a fifth fraction containing single ring aromatic lubricating oil components and a sixth fraction containing condensed ring aromatic lubricating oil components, combining said fourth and fifth fractions, and recovering from said combined fourth and fifth fractions a lubricating oil of high viscosity index.

3. A method in accordance with claim 2 in which the aromatic hydrocarbon fractions alternately charged to the bed of silica gel and alumina gel are identical.

4. A method for producing a high viscosity lubricating oil which comprises alternately charging to a bed of silica gel a lubricating oil fraction containing paraffinic, single and condensed ring naphthenic, and single and condensed ring aromatic hydrocarbons in admixture with a naphthenic hydrocarbon solvent of a boiling point lower than the lubricating oil fraction and an aromatic hydrocarbon fraction of a boiling point lower than that of the lubricating oil fraction and lower than that of the naphthenic hydrocarbon fraction, alternately recovering from the bed of silica gel a first fraction containing predominantly saturated lubricating oil components and a second fraction containing predominantly aromatic lubricating oil components, alternately charging to a bed of activated carbon the first

fraction and a paraffinic hydrocarbon fraction of a boiling point lower than that of the aromatic hydrocarbon fraction, alternately recovering from the bed of activated carbon a third fraction containing condensed ring naphthenic lubricating oil components and a fourth fraction containing paraffinic and single ring naphthenic lubricating oil components, alternately charging to a bed of alumina gel the second fraction and an aromatic hydrocarbon fraction having a boiling point lower than the lubricating oil fraction, alternately recovering from the bed of alumina gel a fifth fraction containing single ring aromatic lubricating oil components and a sixth fraction containing condensed ring aromatic lubricating oil components, combining said fourth and fifth fractions and recovering from said combined fourth and fifth fractions a lubricating oil of high viscosity index.

5. A method in accordance with claim 4 in which the naphthenic hydrocarbon fraction boils in the range between 100° and 360° F., the aromatic hydrocarbon fraction is a toluene concentrate boiling in the range between 90° and 320° F. and the paraffinic hydrocarbon boils in the range between 80° and 310° F.

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