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[54] LUBRICATING COMPOSITION
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[56] References Cited

UNITED STATES PATENTS		
2,525,788	10/1950	Fontana et al. 252/59
2,697,694	12/1954	Shalit et al. 252/59
2,779,753	1/1957	Garabrant et al. 252/59 X

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[57] ABSTRACT

A lubricating oil composition having good shear stability is obtained by blending together a mineral lubricating oil and a viscosity index improving amount of a polymer of a normal alpha olefin having from four to 16 carbon atoms per molecule. The polymer is obtained by polymerizing the alpha olefin or alpha olefin mixture in the liquid phase in the presence of aluminum chloride and a nonpolymerizing hydrocarbon diluent at a temperature between about -40° and +70° F. for a time sufficient to produce a polymer having a viscosity of about 40 to about 3000 centistokes at 210° F. The alpha olefin is introduced into the polymerization system at a rate of about 0.6 to about 60 moles of olefin per mole of aluminum chloride per hour. The addition of the olefin is continued until 2 to 200 moles of olefin per mole of aluminum chloride has been added.

10 Claims, No Drawings

LUBRICATING COMPOSITION

This invention relates to a lubricating oil composition and more particularly to a mineral lubricating oil composition having improved viscosity index characteristics.

The viscosity-temperature relationship of a lubricating oil is one of the more important characteristics of an oil in that it is this relationship which is indicative of the relative change in viscosity of an oil at high and low temperatures. As might be expected, the viscosity of most mineral lubricating oils changes rapidly with a change in their temperature. In general, mineral oils tend to become thinner as their temperature increases and thicker as their temperature decreases. The change in viscosity with temperature is greater with some oils than with others. While some change is tolerable, excessive change is undesirable. In motor oils of the type known as crankcase oils, it is desirable for the oil to have a viscosity which is sufficiently high at an elevated temperature to provide adequate lubrication and to prevent excessive oil consumption. On the other hand, the motor oil should have a viscosity which is sufficiently low at ambient temperature to provide ease of engine starting. Increasing the high-temperature viscosity of an oil while decreasing its low-temperature viscosity can be accomplished only by improving the viscosity-temperature relationship or, stated differently, by raising the viscosity index of the oil.

In the past, the viscosity index of a mineral lubricating oil has been improved by subjecting the base oil from which the lubricating oil composition is made to more drastic refining methods and/or by adding a viscosity index improver to the lubricating oil. For example, the viscosity index of some oils has been improved by subjecting the base oil to solvent extraction and treatment with aluminum chloride. Aluminum chloride refined or solvent extracted paraffinic oils, such as the Pennsylvania oils, have provided excellent base oils for many lubricating compositions having improved viscosity indices. Likewise, drastically refined Mid-Continent and Gulf Coastal oils have been widely used as base oils in forming lubricants having high-viscosity indices.

In addition to these refining methods, lubricating oils of improved viscosity indices have been obtained by hydrogenating various charge stocks derived from Pennsylvania, Mid-Continent, West Coast, Middle-East crudes, etc. It is known, for example, that improved viscosity-temperature relationships can be obtained when some lubricating oil stocks are either hydrofinished or hydrotreated. Hydrofinishing and hydrotreating some lubricating oil stocks, for example, has resulted in excellent base stocks for multigrade lubricants, i.e., lubricants suitable for use under a wide range of temperatures. Regardless of the treatment to which the various charge stocks are subjected, the viscosity index of the lubricating oil can be improved only up to a certain maximum by refining techniques alone inasmuch as further treatment has only an additional negligible effect on viscosity index characteristics. Further improvement can be effected by adding various types of viscosity index improving additives to the lubricating oil. In improving the viscosity index of lubricating oils by additives, recourse has been made to the introduction of various high molecular weight polymers such as polyisobutylene, polymerized esters of the acrylic acid series and the like. In most instances, the highest increase in viscosity index is obtained with polymers of the greatest molecular weight. While high molecular weight polymers have been generally satisfactory in improving the viscosity-temperature relationship of the oil, these polymers have not been entirely satisfactory inasmuch as their viscosity index improving characteristic in many instances is lost or substantially reduced when the oil in which they are incorporated is subjected to vigorous agitation and high shear rates and stresses.

We have found that a lubricating composition having a high-viscosity index and improved shear stability is obtained by incorporating in a mineral lubricating oil a minor amount, sufficient to improve the viscosity index of said oil, of a polymer of a normal alpha olefin having about four to about 16 carbon atoms per molecule or a polymer of a mixture of

normal alpha olefins where the average carbon number of the mixture is about four to about 16, said polymer being obtained by the process which comprises polymerizing said olefin in the liquid phase in the presence of aluminum chloride and a non-polymerizing hydrocarbon diluent, said olefin being introduced into the aluminum chloride-nonpolymerizing hydrocarbon reaction mass at a rate of about 0.6 to about 60 moles of said olefin per mole of aluminum chloride per hour and in an amount of about 2 to about 200 moles of olefin per mole of aluminum chloride, at a temperature of about -40° to about $+70^{\circ}$ F. for a time sufficient to produce a polymer having a viscosity of about 40 to about 3,000 centistokes at 210° F. We have found that a lubricating composition comprising a major amount of a mineral lubricating oil and a minor amount of a polymer obtained as described above in addition to being shear stable is also thermally stable.

In accordance with the invention, normal alpha olefins having about four to about 16 carbon atoms per molecule or a blend of normal alpha olefins where the average carbon number of the blend is between about four and about 16 are polymerized in high yield by a process which comprises contacting the olefin or blend of olefins in the liquid phase with aluminum chloride catalyst in a nonpolymerizing hydrocarbon solvent at a temperature of about -40° to about $+70^{\circ}$ F. under selected and correlated reaction conditions to give a polymer having a viscosity of about 40 to about 3,000 centistokes at 210° F. While we recognize that alpha olefins have been polymerized previously in the presence of aluminum chloride or aluminum bromide, a catalyst promoting agent and a non-polymerizing hydrocarbon diluent, we have discovered that, by carrying out the polymerization reaction under selected and correlated reaction conditions, shear stable polymers having viscosities of about 40 to about 3,000 centistokes at 210° F. are obtained and that these polymers when added to a mineral lubricating oil in amounts sufficient to improve the viscosity index of the oil produce a lubricating composition having good shear and thermal stability. The polymers obtained in accordance with the present invention not only improve the viscosity index of most mineral lubricating oils but also impart exceptional multiviscosity characteristics to certain hydrotreated mineral lubricating base oils, said multiviscosity characteristics heretofore having been obtained by utilizing compounds or compositions comprising constituents other than hydrocarbons, e.g., polymerized esters of the acrylic acid series.

The polymers obtained in accordance with the present invention can be incorporated in the mineral lubricating oil in any convenient manner. Thus, the polymers as such, can be added directly to the lubricating oil or they can be added in the form of concentrated solutions in a solvent such as a light lubricating oil in order to facilitate blending. If desired, such concentrated solutions also can contain other compatible addition agents designed to improve one or more properties of the oil. In the case of blended lubricating oils, the herein-disclosed polymers can be added to one of the component oils prior to blending. Some stirring, possibly with mild heating may be desirable to facilitate more rapid formation of a homogeneous mixture, but this is not absolutely essential.

Exemplary of the alpha olefins which are used in preparing the polymers according to the invention are butene-1, pentene-1, hexene-1, heptene-1, octene-1, nonene-1, decene-1, undecene-1, dodecene-1, tridecene-1, tetradecene-1, pentadecene-1, hexadecene-1 and mixtures thereof.

In preparing lubricating compositions of the present invention, the polymers are employed in concentrations in the range of about 2 to about 30 percent preferably about 5 to about 25 percent by volume based on the volume of the final lubricating composition, but other proportions can be used provided that the polymer is soluble in the oil to the extent to which it is used. The optimum amount of polymer employed may vary depending upon the characteristics of the particular polymer employed and upon the characteristics of the base oil to which the polymer is to be added. Ordinarily, a polymer

having a high viscosity will be used in amounts less than a polymer having a low viscosity. In any event, the polymer is added to the lubricating oil in an amount sufficient to improve the viscosity index of the oil. An especially shear stable lubricating composition having a high-viscosity index has been obtained by admixing about 8 to 10 percent by volume of a decene-1 polymer having a viscosity of about 380.7 cs. (1770 SUS) at 210° F. with about 90 to 92 percent by volume of a hydrotreated mineral lubricating oil.

In preparing the polymers utilized in the composition of the present invention, the alpha olefin or alpha olefin mixture is introduced into a reaction vessel containing aluminum chloride catalyst and a nonpolymerizing hydrocarbon diluent. Prior to introducing the alpha olefin into the reaction vessel, the mixture of aluminum chloride and nonpolymerizing hydrocarbon diluent is preferably contacted with hydrogen chloride for a short time, usually not more than about 1 to 2 hours, preferably about 15 to 30 minutes. Such contacting can be conducted merely by bubbling gaseous hydrogen chloride through the mixture of aluminum chloride and nonpolymerizing hydrocarbon diluent. The diluent is preferably a paraffin hydrocarbon such as butane. However, other nonpolymerizing hydrocarbon diluents can be used. The amount of the diluent may be the same that is normally used in other olefin polymerization reactions. For example, we may use the nonpolymerizing hydrocarbon diluent in amounts of about 1 to about 15 moles per mole of olefin. Larger or smaller amounts may be employed as desired.

The polymerization reaction is carried out generally with agitation at a temperature of about -40° to about +70° F., preferably at a temperature of about -20° to about +40° F. The pressure should be just sufficient to keep the alpha olefin in the liquid phase. Higher pressures have no advantage. While the rate at which the alpha olefin is introduced into the reaction vessel can vary over wide limits, we prefer to add the olefin to the reaction mixture at a rate of about 0.6 to about 60 moles of olefin per mole of aluminum chloride per hour, preferably about 2 to about 20 moles of alpha olefin per mole of aluminum chloride per hour. The amount of alpha olefin added comprises about 2 to about 200 moles of alpha olefin per mole of aluminum chloride, preferably about 10 to about 150 moles of alpha olefin per mole of aluminum chloride. The reaction time is sufficient to produce a polymer having a viscosity of about 40 to about 3,000 centistokes at 210° F. In most instances, polymerization to this extent is complete in about 1 to about 24 hours, and usually in about 3 to about 12 hours. In a preferred embodiment of the invention, a shear stable lubricating composition is obtained by incorporating in a mineral lubricating oil a minor amount, sufficient to improve the viscosity index of the oil, of a polymer of decene-1, said polymer being obtained by the process which comprises polymerizing said decene-1 in the liquid phase in the presence of aluminum chloride and butane as a diluent, said decene-1 being introduced into the aluminum chloride-butane reaction mass at a rate of about 2 to about 12 moles of decene-1 per mole of aluminum chloride per hour and in an amount of about 10 to about 100 moles of decene-1 per mole of aluminum chloride, at a temperature of about 25° to about 35° F. for a time sufficient to produce a polymer having a viscosity of about 40 to about 3,000 centistokes at 210° F. Inasmuch as the polymerization reaction is exothermic, means must be provided for removing heat during the polymerization. In a preferred embodiment the heat of reaction is removed by a heat exchanger through which a coolant circulates at about 10° to about 20° F. below the boiling point of the diluent. If desired, evaporative cooling can be effected by vaporization of the diluent from the reaction mass. At the conclusion of the polymerization reaction, the contents of the reaction vessel are emptied into a quantity of water, i.e., about 0.5 to about 20 times the volume of the reaction mass to decompose the catalyst. The polymer is separated from the water and then dried. The dried polymer is subjected to a topping distillation to 700° F. The polymer thus obtained has a viscosity within the range of 40 to 3,000 centistokes at 210° F.

The lubricating oil to which the polymerized alpha olefin is added according to the invention is advantageously a highly refined oil derived from a paraffinic, naphthenic or asphalt base oil. Thus, the lubricating oil can be one which has been obtained by conventional aluminum chloride refining and/or by solvent extraction. Aluminum chloride refined or solvent extracted paraffinic base oil, such as Pennsylvania oil, provides an excellent base oil for the composition of the invention. However, drastically refined Mid-Continent and Gulf Coastal oil may also be used. Hydrofinished and hydrotreated mineral oils, because of their improved stability over untreated oils and also their improved viscosity index characteristics are especially preferred lubricating oil bases for preparing multigrade lubricating compositions of the invention. Hydrotreating is distinguished from hydrofinishing in that the latter involves the use of milder hydrogenation conditions. In this regard, we prefer to employ an oil which has been hydrotreated. While hydrotreated oils are excellent base oils from which multigrade lubricants can be prepared according to the invention, we can also obtain multigrade lubricating oils using a hydrofinished or conventionally refined base oil.

Especially preferred hydrotreated oils are obtained when a deasphalted residuum is treated with hydrogen at a temperature within the approximate range of about 650° to about 850° F.; a pressure within the range of 1,500 to about 10,000 p.s.i.g.; a space velocity between about 0.1 and about 5 volumes of charge stock per volume of catalyst per hour; and a hydrogen recycle rate between about 2,000 and about 7,500 standard cubic feet per barrel of charge. A preferred set of operating conditions for hydrotreating include a temperature of 700° to 800° F., a pressure of 2,000 to 5,000 p.s.i.g., a space velocity of 0.25 to 2 and a gas circulation rate of 2,500 to 5,000 standard cubic feet of hydrogen per barrel of charge.

The viscosity index improving characteristics and shear stability of lubricating compositions containing the polymers of normal alpha olefins according to the invention will hereinafter be illustrated using various lubricating oil bases. It will be noted that many of the illustrative lubricating compositions satisfy the SAE viscosity requirements of multigrade lubricants.

The preparation of the polymers of C₆ to C₁₆ normal alpha olefins utilized in compositions of the invention will be illustrated by the following specific examples.

EXAMPLE I

Into a 3-liter, three-necked flask equipped with a stirrer, a condenser and a gas inlet tube was placed 1,465 ml. (15.1 moles) of butane and 11.0 grams (0.08 mole) of aluminum chloride. Hydrogen chloride gas as a promoter was bubbled through the stirred system for approximately 15 minutes. Decene-1 (1,284 ml. [6.78 moles]) was added to the reaction vessel over an 8-hour period. The reaction ratios were as follows:

Moles of decene-1 per mole of AlCl ₃	84.8
Moles of butane per mole of decene-1	2.23
Moles of decene-1 per mole of AlCl ₃ per hour	10.6

The temperature of the reactants was maintained at 30° F. After all the decene-1 had been added, the contents of the flask were poured into a vessel containing 1 liter of heptane and 2 liters of water. The butane was removed from the reaction mass by vaporization. After all the butane was removed, the heptane-polymer mixture was washed three times with 1.5 liters of water. The washed product was dried over anhydrous sodium sulfate and then fractionated by distillation. The yield of polymer having an initial boiling point of 700° F. was 90 percent based on the volume of the decene-1 charged. The polymer thus obtained had a viscosity of 380.7 cs. (1770 SUS) at 210° F. and a viscosity index of 115.

This decene-1 polymer was blended with various hydrotreated base oils. Inspections of the physical properties of these blends are shown in table I.

TABLE I

Composition.....	Percent by volume of—						
	A	B	C	D	E	F	G
Hydrotreated base oil:							
1.....	100				90		
2.....		100				90	
3.....			100				91.75
Decene-1 polymer.....				100	10	10	8.25
Inspections:							
Viscosity at 210° F., cs.....	5.60	6.10	6.22	380.7	10.79	11.33	9.79
Viscosity index, D567.....	138	132	135	115	142	140	139
Pour point, ° F.....	0	0	5	5	0	0	+10

When 90.0 volume percent of a hydrotreated base oil designated as hydrotreated base oil 1 was blended with 10 volume percent of the decene-1 polymer, the resulting lubricating composition (Composition E) met the viscosity specifications of a multigrade 10W/30 lubricating oil, having a viscosity of 10.79 cs. at 210° F. and a viscosity index of 142.

When 90.0 volume percent of a hydrotreated base oil designated as hydrotreated base oil 2 was blended with 10 volume percent of the decene-1 polymer, the resulting lubricating composition (Composition F) met the viscosity specifications of a multigrade 10W/30 lubricating oil having a viscosity of 11.33 cs. at 210° F. and a viscosity index of 140.

When 91.75 volume percent of a hydrotreated base oil designated as hydrotreated base oil 3 was blended with 8.25 volume percent of the decene-1 polymer, the resulting lubricating composition (Composition G) met the viscosity specifications of a multigrade 10W/30 lubricating oil having a viscosity of 9.79 cs. at 210° F. and a viscosity index of 139.

The excellent sonic shear stability of the oils prepared in example 1 is illustrated using the blend prepared by adding 8.25 volume percent of the decene-1 polymer to the hydrotreated base oil designated as hydrotreated base oil 3 (Composition G). In order to illustrate the shear stability of the polymers of the invention, the polymer-base oil blends are subjected to a proposed ASTM test for determining the shear stability of polymer containing oils. This test is described in ASTM Standards on Petroleum Products, Volume 1, 1961, page 1,160. According to this test, the material to be evaluated is subjected to irradiation in a sonic oscillator for a fixed period of time and the change in viscosity determined. The results obtained are presented in table II.

TABLE II

Composition: Percent by Volume	C	G
Hydrotreated Base Oil 3	100	91.75
Decene-1 Polymer	—	8.25
Inspections		
Viscosity at 100° F., cs.	34.55	59.25
Viscosity at 210° F., cs.	6.22	9.79
Viscosity at 0° F. (extrapolated), cs.	1,100	1,850
Viscosity Index, D567	135	139
Pour Point: ° F.	5	10
Sonic Shear Test (10 min.)		
100° F. Vis Loss: % of original viscosity	0.2	0.3
210° F. Vis Loss: % of original viscosity	0.3	0.4
Viscosity Index Loss: % of original viscosity index	0.0	0.1

An additional hydrotreated base oil as well as a hydrofinished light neutral distillate oil were blended with the decene-1 of example 1. For comparison, these oils were also blended with a conventional "Acryloid" viscosity improving agent. The results obtained are presented in table III.

TABLE III

Composition.....	Percent by volume of—						
	H	I	J	K	L	M	N
Hydrotreated base oil 4.....	100		90	97	96		
Hydrofinished light neutral distillate oil.....		100				85	95

TABLE III

Composition.....	Percent by volume of—						
	H	I	J	K	L	M	N
Decene-1 polymer.....			10			15	
"Acryloid" VI improver.....				3	4		5
Inspections, Viscosity at 210° F., cs.:							
Before sonic shear stability test.....	5.95	4.25	9.62	7.81	8.56	10.19	7.92
After sonic shear stability test.....	5.95	4.25	9.58	7.23	7.70	10.11	6.86
Viscosity index, D567:							
Before sonic shear stability test.....	130	99	136	146	149	138	152
After sonic shear stability test.....	129	99	136	142	143	138	146
Percent loss in thickening power *			1	31	33	1	29

▪ Loss in thickening power =
$$V_1 - V \over V_1 - V_0$$
where
V₁ = blend viscosity at 210° F. before shear test.
V = blend viscosity at 210° F. after shear test.
V₀ = viscosity of base oil at 210° F. before shear test.

$$\text{* Loss in thickening power} = \frac{V_1 - V}{V_1 - V_0}$$

where

V_1 = blend viscosity at 210° F. before shear test.

V = blend viscosity at 210° F. after shear test.

V_0 = viscosity of base oil at 210° F. before shear test.

The improved shear stability obtained with the decene-1 polymer in accordance with the present invention as compared with a conventional "Acryloid" viscosity index improver is clearly shown by the illustrative data in table III. It will be noted that after sonic irradiation, compositions of the invention, i.e., Compositions J and M, retained their high-viscosity indices with very little loss in thickening power. Contrariwise, the hydrotreated and hydrofinished base oils containing the "Acryloid" viscosity index improver did not retain their high-viscosity index and lost from 29 to 33 percent of their thickening power. The data thus obtained in the sonic shear stability test indicate that the alpha olefin polymers are surprisingly more stable than conventional "Acryloid" viscosity index improvers even when subjected to vigorous agitation and high shear rates and stresses.

The thermal stability of a lubricating composition of the invention is demonstrated by the data in table IV. In order to illustrate the thermal stability of polymers utilized in compositions of the invention, the blends were subjected to a modified London Heat Test. According to this test, the material to be evaluated is heated to 160° F. for 48 hours and the change in color is determined by ASTM D1500.

These tests were conducted with a hydrofinished light neutral distillate oil and the decene-1 polymer of example 1. For comparison, an additional composition was prepared utilizing a conventional "Acryloid" viscosity improving agent. In these tests, the lubricating composition was subjected to a temperature of 500° F. for 24 hours under an atmosphere of nitrogen.

The improved thermal stability obtained with an olefin polymer in accordance with the present invention as compared with a conventional "Acryloid" viscosity index improver is clearly shown by the illustrative data in table IV. It will be noted that the oil with the decene-1 polymer did not show any viscosity loss or increase in acidity (neutralization number) after exposure to 500° F. for 24 hours in a nitrogen atmosphere. The oil which contained the "Acryloid" viscosity index improver lost 12 percent of its thickening power and showed an increase in neutralization number after exposure to the same conditions.

TABLE IV

Composition.....	Percent by volume of—		
	I	M	N
Hydrofinished light neutral distillate oil.....	100	85	95
Decene-1 polymer.....		15	
"Acryloid" VI Improver.....			5
Physical characteristics:			
Viscosity at 210° F., cs.:			
Before heating to 500° F.....	4.25	10.19	7.92
After heating to 500° F.....	4.30	10.19	7.47
Viscosity index, D567:			
Before heating to 500° F.....	99	138	152
After heating to 500° F.....	100	137	150
Neutralization value, ASTM D974:			
Before heating to 500° F.....	0.07	0.05	0.04
After heating to 500° F.....	0.03	0.05	0.11

TABLE IV

Composition.....	Percent by volume of--		
	I	M	N
Color, ASTM D1500:			
Before heating to 500° F.....	<1.0	<1.0	<1.0
After heating to 500° F.....	<1.5	<1.5	<1.5
Percent loss in thickening power ^a		0	12

$$^a \text{ Loss in thickening power} = \frac{V_1 - V}{V_1 - V_0}$$

where

V₁=blend viscosity at 210° F. before thermal test.

V=blend viscosity at 210° F. after thermal test.

V₀=viscosity of base oil at 210° F. before thermal test.

EXAMPLES 2 to 11

In order to illustrate the invention, additional polymers were prepared with hexene-1, octene-1, decene-1, dodecene-1, tetradecene-1 and hexadecene-1 as the monomers. The conditions were similar to those used in example 1. The hydrocarbon diluent was butane. The catalyst was aluminum chloride. Prior to adding the alpha olefin, hydrogen chloride was bubbled through the mixture of diluent and catalyst for one-half hour. The temperature of the reactants was maintained at 30° F. The other reaction conditions and properties of the polymers are set forth in table V.

TABLE V.—PREPARATION OF POLYMERS

Alpha olefin monomer	Catalyst ^a	Diluent	Polymer preparation						Reaction ratios			Polymer properties, viscosity at—	
			Temperature, ° F.	Moles monomer	Moles catalyst	Moles diluent	Addition time, hours		A	B	C	100° F., cs.	210° F., cs.
Example:													
2.....	Hexene-1.....	AlCl ₃ +HCl	Butane	30	2.32	.13	16.1	4	17.8	6.94	4.45	4,795	206
3.....	do.....	Same.....	do.....	30	2.32	.13	16.1	2	17.8	6.94	8.9	3,023	148
4.....	Octene-1.....	do.....	do.....	30	11.0	.06	16.1	12	133.3	1.46	15.3	1,540	100
5.....	do.....	do.....	do.....	30	3.19	.08	19.2	8	39.9	6.02	5.0	2,450	163
6.....	Decene-1.....	do.....	do.....	30	1.50	.08	14.8	8	18.8	9.87	2.3	8,200	463
7.....	do.....	do.....	do.....	30	1.12	.08	4.8	6	14.0	13.21	2.3	3,459	229
8.....	Dodecene-1.....	do.....	do.....	30	1.55	.08	15.1	8	19.4	9.74	2.4	7,844	531
9.....	do.....	do.....	do.....	30	1.62	.07	16.1	4	23.2	9.94	5.8	1,000	79
10.....	Tetradecene-1.....	do.....	do.....	30	1.36	.08	13.1	8	17.0	9.63	2.1	2,034	161
11.....	Hexadecene-1.....	do.....	do.....	30	1.35	.08	15.1	8	16.9	11.19	2.1	1,869	157

^a HCl bubbled through diluent-catalyst mix for ½ hour prior to monomer addition; A=Moles of alpha olefin per mole of AlCl₃; B=Moles of butane per mole of olefin; C=Moles of alpha olefin per mole of AlCl₃ per hour.

When 15 percent by volume of polymers prepared from C₆ to C₁₆ alpha olefins was added to the hydrofinished light neutral distillate oil the resultant blend had viscosities in the range required for 10W/30 multiviscosity oils. The result obtained when blending 15 volume percent of the polymers with the base oil are shown in table VI.

TABLE VI

Composition.....	Percent by Volume of —						
	I	O	P	M ¹	Q	R	S
Hydrofinished light neutral distillate oil.....	100	85	85	85	85	8	85
Polymer.....		15	15	15	15	15	15
Polymer type: carbon No. of alpha-olefin used.....	C ₆	C ₈	C ₁₀	C ₁₂	C ₁₄	C ₁₆	
Polymer viscosity at 210° F., cs.....	380	380	380	380	380	380	60

TABLE VII.—RESPONSE OF LIGHT NEUTRAL DISTILLATE TO VARIOUS C₁₀ POLYMERS

Composition	Percent by volume of—							
	I	T	U	V	W	X	Y	Z
Light neutral distillate.....	100				90	90	85	85
Heavy C ₁₀ polymer.....		100			10			
Medium C ₁₀ polymer.....			100			10	15	
Light C ₁₀ polymer.....				100				15
Inspections; Viscosity; cs. at:								
0° F.....					2,175	1,641	2,000	1,300
100° F.....	22.84	4,400	5,500	436.7	77.77	45.40	63.83	34.26
210° F.....	4.25	2,464	350	42.52	12.70	7.69	10.28	5.83
Viscosity index, D567.....	99	122	122	125	141	135	137	123
Meets specifications.....					10W/30	10W/20	10W/30	10W/20
After 10 minutes sonic shear viscosity; cs. at:								
0° F.....					2,170			
100° F.....					70.49			
210° F.....					11.5			
Viscosity index, D567.....					140			

Inspections;							
Viscosity at 210° F., cs.....	4.25	10.2	10.2	10.2	10.2	10.2	10.2
Viscosity index, D567.....	99	133	135	138	139	141	142
Pour point, ° F.....	0	+5	+5	0	+10	+20	+40
Sonic shear test (10 minutes) 210° F. viscosity loss, cs.....							
Viscosity index loss.....							

¹ Other properties of Composition M are shown in Tables III and IV.

10 All the blended oils shown in table VI are shear stable, i.e., less than 1 percent loss in viscosity or viscosity index. The blended oils prepared from the polymers where the alpha olefin monomer contained six and eight carbons while having improved viscosity index characteristics were marginal oils with respect to the viscosity index. The blended oils from the polymers where the alpha olefin monomer contained 12 to 16 carbons had high pour points which, however, can be reduced by the use of a pour point depressant. The blended oils containing the decene-1 polymer, when both the viscosity index and pour point characteristics are considered, gave the most acceptable 10W/30 multiviscosity oil. It is evident that polymers of the invention made from C₆ to C₁₆ alpha olefins can be used to produce 10W/30 multiviscosity oil but best results are obtained with the polymer from decene-1.

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While polymers of normal alpha olefins having from about six to 16 carbon atoms per molecule have been described above with particular reference to their ability to improve the viscosity index of hydrofinished and hydrotreated mineral lubricating oils, it is to be understood that the polymers can also be utilized in improving the viscosity index of other mineral lubricating oils. For example, the polymers can be added to lubricating oils that have been derived from paraffinic, naphthenic or mixed base crude petroleum oils, and that have been subjected to solvent or sulfuric acid treatment, aluminum chloride treatment and other refining treatments. The improved characteristics of other hydrotreated and non-hydrotreated base oils with other polymers of the invention are illustrated in tables VII to XII.

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TABLE VIII.—EFFECT OF POLYMER ON VISCOSITY OF
BASE OILS

C₁₀ Polymer:
Viscosity: cs. at 100° F. 8600
Viscosity: cs. at 210° F. 542
Viscosity index, D567 120

Base oil description	Polymer, volume percent	Viscosity, cs.			Vis- cosity index, D567	Meets specifi- cations
		0 ° F. ¹	100 ° F.	210 ° F.		
70 Paralex.....	0	14.87	3.22		88	
	8.0	413	21.91	5.81	143	5W/20
	18.0	1,828	67.89	11.60	143	10W/30
Hydrotreated oil.....	0	24.43	4.91		140	
	1.4	783	27.71	5.41	144	
	2.9	804	30.29	5.90	145	5W/20
	11.5	1,500	59.16	10.42	145	10W/30

¹ Extrapolated values.

TABLE IX.—EFFECT OF POLYMER ON VISCOSITY OF
BASE OILS

C₁₀ polymer:
Viscosity: cs. at 100° F. 8,200
Viscosity: cs. at 210° F. 463
Viscosity index, D567 122

Base oil description	Polymer, volume percent	Viscosity, cs.			Vis- cosity index, D567	Meets specifi- cations
		0 ° F. ¹	100 ° F.	210 ° F.		
60 Texas distillate.....	0	228	9.675	2.34	49	
	4.9	434	17.22	3.77	117	
	9.8	565	27.55	5.88	150	5W/20
	14.8	912	42.3	8.25	157	10W/20
Light neutral distillate.....	0	825	21.8	4.12	97	
	13.3	2,150	63.85	10.28	137	10W/30
Medium neu- tral distillate..	0	3,475	50.6	7.00	104	
	9.0	6,080	96.2	12.27	123	20W/30
	15.0	8,680	142.2	17.14	125	20W/60
500 Texas oil....	0	109.8	8.655		30	
	6.7	180.6	13.93		78	
	9.5	223.1	17.26		90	
100 Texas oil...	0	21.5	3.61		19	
	4.8	1,800	34.7	5.55	107	
	9.7	2,100	54.3	8.55	131	10W/20
	14.5	3,475	76.05	10.90	129	20W/30
100 Paratex oil..	0	22.9	3.94		54	
	4.9	1,740	35.0	5.68	112	
	9.9	2,170	54.0	8.47	130	10W/20
100 paraffine oil.	0	20.9	3.80		64	
	4.9	1,260	29.9	5.30	121	
	9.9	1,780	45.85	7.36	127.5	10W/20
	14.8	2,715	74.2	11.35	135	20W/30
Hydrotreated oil.....	0	1,348	38.30	6.54	130	10W/20
	6.2	1,910	56.03	9.07	136	10W/20

¹ Extrapolated values.

TABLE X

C₁₀ polymer:
Viscosity: cs. at 100° F. 5950
Viscosity: cs. at 210° F. 380.7
Viscosity index, D567 115

Base oil description	Polymer, volume percent	Viscosity, cs.			Vis- cosity index, D567	Meets specifi- cation
		0 ° F. ¹	100 ° F.	210 ° F.		
Hydrotreated oil.....	0	869	29.5	5.60	138	
	10.0	1,760	64.09	10.79	142	10W/30
	15.0	2,455	85.17	13.58	140	10W/40
	0	1,195	34.4	6.10	132	10W/20
	10.0	2,170	69.54	11.33	140	10W/30
	15.0	3,105	97.78	14.87	138	20W/40
	0	1,110	34.55	6.22	135	10W/20
	8.25	1,850	59.25	9.79	139	10W/30

¹ Extrapolated values.

TABLE XI

C₁₀ polymer:
Viscosity: cs. at 100° F. 4969
Viscosity: cs. at 210° F. 317
Viscosity index, D567 122

Base oil description	Polymer, volume percent	Viscosity, cs.			Vis- cosity index, D567	Meets specifi- cations
		0 ° F. ¹	100 ° F.	210 ° F.		
Hydrotreated oil.....	0	1,089	33.92	5.97	130	10W/20
	10	2,100	62.97	9.98	136	10W/30

¹ Extrapolated values.

TABLE XII.—EFFECT OF POLYMER ON VISCOSITY OF
BASE OIL

C₈ polymer:
Viscosity: cs. at 100° F. 1,215
Viscosity: cs. at 210° F. 82.7
Viscosity index, D567 119

Base oil description	Polymer, volume percent	Viscosity, cs.			Vis- cosity index, D567	Meets specifi- cations
		0 ° F. ¹	100 ° F.	210 ° F.		
Hydrotreated oil.....	0	1,695	43.05	7.02	132	10W/20
	5.0	1,780	48.25	7.865	132	10W/20
	10.0	2,000	55.6	8.77	132	10W/20
	15.0	2,560	65.8	10.16	134	10W/30

¹ Extrapolated values.

The lubricating oil compositions of this invention can contain other addition agents normally added to lubricating oils for a specific purpose such as an oiliness agent, an extreme pressure agent, an antioxidant, a corrosion inhibitor, a foam suppressant, a dye, a sludge inhibitor, a pour point depressant and the like.

While our invention has been described with reference to various specific examples and embodiments it will be understood that the invention is not limited to such examples and embodiments and may be variously practiced within the scope of the claims hereinafter made.

We claim:

1. A lubricating composition comprising a major amount of a mineral lubricating oil and a minor amount, sufficient to improve the viscosity index of said oil, of a polymer of a normal alpha olefin having about four to about 16 carbon atoms per molecule, said polymer being obtained by the process which comprises polymerizing said olefin in the liquid phase in the presence of aluminum chloride and a nonpolymerizing hydrocarbon diluent, said olefin being introduced into the aluminum chloride-nonpolymerizing hydrocarbon reaction mass at a rate of about 0.6 to about 60 moles of said olefin per mole of aluminum chloride per hour and in an amount of about 2 to about 200 moles of olefin per mole of aluminum chloride, at a temperature of about -40° to about +70° F. for a time sufficient to produce a polymer having a viscosity of about 40 to about 3,000 centistokes at 210° F.

2. The lubricating composition of claim 1 wherein said minor amount is about 2 to about 30 percent by volume of the composition.

3. The lubricating composition of claim 1 wherein said olefin utilized in obtaining said polymer is hexene-1.

4. The lubricating composition of claim 1 wherein said olefin utilized in obtaining said polymer is octene-1.

5. The lubricating composition of claim 1 wherein said olefin utilized in obtaining said polymer is decene-1.

6. The lubricating composition of claim 1 wherein said olefin utilized in obtaining said polymer is dodecene-1.

7. The lubricating composition of claim 1 wherein said olefin utilized in obtaining said polymer is tetradecene-1.

8. The lubricating composition of claim 1 wherein said olefin utilized in obtaining said polymer is hexadecene-1.

9. The lubricating composition of claim 1 wherein said olefin utilized in obtaining said polymer is an alpha olefin mixture where the average carbon number of said mixture is about four to about 16.

10. A lubricating composition comprising a major amount of mineral lubricating oil and a minor amount, sufficient to improve the viscosity index of said oil, of a polymer of decene-1, said polymer being obtained by the process which comprises polymerizing said decene-1 in the liquid phase in the presence of aluminum chloride and butane as a diluent, said decene-1 being introduced into the aluminum chloride-butane reaction

mass at a rate of about 2 to about 12 moles of decene-1 per mole of aluminum chloride per hour and in an amount of about 10 to about 100 moles of decene-1 per mole of aluminum chloride, at a temperature of about 25° to about 35° F. for a time sufficient to produce a polymer having a viscosity of about 40 to about 3,000 centistokes at 210° F.

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