

[54] MINERAL OIL COMPOSITIONS
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[51] Int. Cl. C10m 1/32
[58] Field of Search..... 252/51.5 A; 260/80.73

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Assistant Examiner—A. P. Demers
Attorney, Agent, or Firm—T. H. Whaley; C. G. Reis

[57] ABSTRACT

A mineral oil composition of improved viscosity, pour depressing and detergent-dispersant properties comprising as a major component a mineral oil of lubricating viscosity and a viscosity index improving amount of an interpolymer of dialkylaminoalkyl methacrylate, styrene or alkyl substituted styrene, C₁₀-C₁₄ alkyl methacrylate and C₁₆-C₂₀ alkyl methacrylate.

5 Claims, No Drawings

MINERAL OIL COMPOSITIONS

BACKGROUND OF INVENTION

Polymeric additives derived from acrylic and methacrylic acid are extensively used in mineral lubricating oil compositions, particularly automatic transmission fluids, to impart desirable viscosity-temperature characteristics to the compositions. These additives are designed to modify lubricating oil so that changes in viscosity occurring with variations in temperature are kept as small as possible. Lubricating oils containing such polymeric additives essentially maintain their viscosity at the higher temperatures normally encountered in engine and transmission operations while at the same time maintaining desirable low viscosity fluidity at engine starting temperatures. The ability of a hydrocarbon oil to accommodate increased temperatures with a minimum decrease in viscosity is indicated by its Viscosity Index (VI). The greater this ability, the higher the Viscosity Index. Because of the aforementioned properties, these polymeric additives have been conveniently termed both "thickeners" and "viscosity index improvers."

Some of these prior polymeric additives are desirably multi-functional additives in that in addition to the VI improving properties, they also function as detergent-dispersants and pour depressants. Multi-purpose additives are particularly desirable for use in engine oils and automatic transmission fluids since the modern day demands on these oils and fluids are so great that large quantities of additives are required therein to meet the specifications. These increasing quantities pose the danger of reaching a point of being so large as to negatively effect the primary mission of the oil or fluid. Therefore, materials which have several additive functions are much in demand since they meet engine and transmission requirements with less total additives.

However, one of the principal failings of these prior multi-purpose Viscosity Index, detergent-dispersant, and pour improving additives is that they are inadequate in sufficiently maintaining desired fluidity at very low temperatures, e.g., of the order of -40°F .

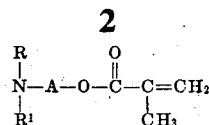
SUMMARY OF INVENTION

We have discovered and this constitutes our invention a mineral oil composition containing as a major component a mineral lubricating oil and between about 0.1 and 10 wt. % of an interpolpolymer of dialkylaminoalkyl methacrylate, styrene or alkyl substituted styrene, $\text{C}_{10}\text{-C}_{14}$ alkyl methacrylate and $\text{C}_{16}\text{-C}_{20}$ alkyl methacrylate, said interpolpolymer not only imparting multi-properties of Viscosity Index improvement, pour depressancy and detergent-dispersancy thereto but also substantially improving the low temperature fluidity of said compositions.

DETAILED DESCRIPTION OF THE INVENTION

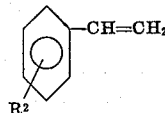
More specifically, our invention pertains to a hydrocarbon oil composition comprising a major amount, i.e., at least about 75 wt. %, of a mineral lubricating oil containing between about 0.1 and 10 wt. % of an interpolpolymer, said interpolpolymer consisting essentially of a tetrapolymer composed of the following monomers:

1. A dialkylaminoalkyl methacrylate characterized by the formula:



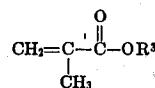
where R and R^1 are alkyl of from 1 to 2 carbons and A is a divalent saturated aliphatic hydrocarbon (alkaned-yl).

2. A styrene compound of the formula:



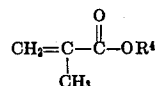
where R^2 is hydrogen or alkyl of from 1 to 10 carbons.

3. A $\text{C}_{10}\text{-C}_{14}$ alkyl methacrylate of the formula:



where R^3 is alkyl of from 10 to 14 carbons and

4. A $\text{C}_{16}\text{-C}_{20}$ alkyl methacrylate of the formula:



where R^4 is alkyl of from 16 to 20 carbons, said tetrapolymer having an intrinsic viscosity in benzene at 77°F . of between about 0.22 and 2.87, preferably between 0.68 and 1.2, said tetrapolymer consisting of between about 4 and 10 wt. % of said dialkylaminoalkyl methacrylate, between about 15 and 25 wt. % of said styrene compound, between about 40 and 60 wt. % of said $\text{C}_{10}\text{-C}_{14}$ alkyl methacrylate and between about 20 and 30 wt. % of said $\text{C}_{16}\text{-C}_{20}$ alkyl methacrylate.

The interpolpolymer ingredient is prepared by standard polymerization techniques such as forming a monomeric mixture consisting of between about 4 and 10 wt. % of the dialkylaminoalkyl methacrylate, between about 15 and 25 wt. % of the styrene compound, between about 40 and 60 wt. % of the $\text{C}_{10}\text{-C}_{14}$ alkyl methacrylate and between about 20 and 30 wt. % $\text{C}_{16}\text{-C}_{20}$ alkyl methacrylate in a liquid medium, advantageously a mineral oil of lubricating oil viscosity, preferably of a viscosity between about 110 and 220 SUS at 100°F ., said liquid medium desirably constituting between about 25 and 35 wt. % of the polymerization mixture. The resultant polymerization mixture is purged with an inert gas such as prepurified nitrogen and then heated to a temperature of between about 65° and 85°C ., followed by the addition of between about 0.4 and 0.15 wt. % of a polymerization catalyst such as azobisisobutyronitrile or benzoyl peroxide, and between about 0.08 and 0.2 wt. % of a chain stopper such as lauryl mercaptan. During the polymerization, the reaction is normally monitored by taking periodic samples from the reaction mixture and measuring the refractive index on said samples. The polymerization reaction mixture is normally continued until the refractive index stabilizes at between ± 0.0002 and 0.0009 units. Under the most preferred conditions and as a finishing step, additional

mineral oil diluent in an amount of between 65 and 75 wt. % of the polymerization mixture and polymerization catalyst in an amount between about 0.1 and 0.15 wt. % of the polymerization mixture are introduced and the temperature is maintained in the range of between about 95° and 105°C. for between about 2 and 3 hours.

The final polymerization product is normally between about 38 and 42 wt. % mineral lubricating oil concentrate of the aforescribed tetrapolymer. In the finished formulations the interpolymer concentrate is diluted with additional mineral oil or introduced into formulations containing additional mineral oil so that the final interpolymer content in the finished formulations is as heretofore described.

In the preparation of the aforescribed interpolymer specific examples of the dialkylaminoalkyl methacrylate contemplated herein are N,N-dimethylaminoethyl methacrylate, N,N-diethylaminoethyl methacrylate, N,N-dimethylaminopropyl methacrylate, and N,N-diethylaminopropyl methacrylate.

Specific examples of the styrene compounds are styrene, vinyl toluene, p-tertiary butyl styrene and p-tertiary octyl styrene.

Examples of the C₁₀-C₁₄ alkyl methacrylates contemplated herein are decyl methacrylate, undecyl methacrylate, dodecyl methacrylate, tridecyl methacrylate, tetradecyl methacrylate and mixtures of the alkyl methacrylates in the defined alkyl carbon atom range. Mixtures are formed in the alkyl methacrylate manufacture when commercial alcohols are employed in the monomer manufacture since many commercial alcohols employed are in actuality a mixture of adjacent and closely adjacent homologs with one alcohol chain length predominating, thus producing in fact a mixture of methacrylate products.

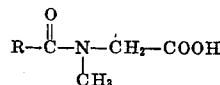
Specific examples of the C₁₆-C₂₀ alkyl methacrylates contemplated herein are hexadecyl methacrylate, heptadecyl methacrylate, octadecyl methacrylate, nonadecyl methacrylate, eicosyl methacrylate and mixtures thereof.

The mineral hydrocarbon lubricating oils contemplated herein for use in the finished lubricating oil compositions and in the preparation of the interpolymer are derived from a wide variety of hydrocarbon base oil materials such as naphthenic base, paraffinic base and mixed based mineral oils, e.g., having an SUS viscosity at 100°F. of between about 35 and 1,000.

When the finished lubricant compositions are to be employed as automatic transmission fluids, the interpolymer content is desirably between about 0.5 and 5 wt. % and the mineral lubricating oil base is desirably present in an amount of between about 90 and 97 wt. %, advantageously having an SUS viscosity of between about 40 and 150 SUS at 100°F., preferably between about 50 and 125, the remainder of the transmission fluid compositions being composed of standard additives normally found therein. A preferred base oil for automatic transmission fluid compositions comprises approximately 70 to 95 wt. % of a refined distillate oil and 5 to 30 wt. % of a refined residual fraction which imparts desired high flash point and lubricity to the oil. A preferred residual fraction comprises a paraffin base residuum which has been propane deasphalted and subjected to centrifuged dewaxing which has an SUS viscosity at 210°F. below about 250. A particularly ef-

fective base oil mixture comprises 65 vol. % of a furfural refined, acid treated, clay contacted solvent dewaxed paraffin base distillate having an SUS viscosity at 100°F. of 100, a viscosity index of about 100, a flash above 380°F. and a pour point below about +10°F., 22 vol. % of an acid treated naphthenic base distillate having an SUS viscosity of 100°F. of 60, a flash above 300°F. and a pour below -40°F., and 13 vol. % of a paraffin base residuum which has been propane deasphalted, centrifuged dewaxed and clay contacted and which has an SUS viscosity at 210°F. of about 160, a flash above 530°F. and a pour of +5°F.

As heretofore stated, the compositions contemplated herein which are suitable as transmission fluids contain additional additives such as supplementary detergent-dispersants, antirust-corrosion inhibitors, antioxidants and friction modifiers. Examples of such supplementary additives are set forth in U.S. Pat. No. 3,640,872: for example, detergent-dispersants such as the alkenyl substituted succinic anhydride derivative of polyethylene polyamine, e.g., where the alkenyl group is a polybutene of about a molecular weight of 1,200 and the amine is hexamethylene pentamine; and antioxidant such as phenyl naphthylamines, phenylenediamine, phenothiazine and diphenylamine; friction modifiers such as a modified carboxylic additive, e.g., N-acyl sarcosine compound represented by the formula:



where R is an aliphatic radical having from 12 to 70 carbons, antirust and anticorrosive agents such as a mixture of hydrolyzed C₆-C₁₈ alkenylsuccinic anhydride, phenol, mono and di-C₁₂ alkyl phosphoric acid esters, and friction modifier life extenders such as zinc di(alkylphenoxypolyalkoxylalkyl) dithiophosphate.

The finished lubricating oil compositions contemplated herein, particularly suitable for use as lubricants in internal combustion engines, will generally comprise between about 75 and 95 wt. % of the hydrocarbon lubricating base oil, preferably of an SUS viscosity between 95 and 150 at 100°F., and between about 1 and 5 wt. % of the interpolymer, the remainder of the engine oil compositions being composed of the standard lube oil additives for engines. These additional additives are found in the classes of supplementary detergent-dispersants, oxidation inhibitors, corrosion inhibitors, antifoamants, etc.

Examples of the supplementary detergent-dispersants contemplated herein for engine lubrication are the ethylene oxide derivatives of inorganic phosphorus acid free, steam hydrolyzed polyisobutene (700-500 m.w.)-P₂S₅ reaction product, overbased calcium alkyl aromatic sulfonate having a total base number of at least about 300 and sulfurized normal calcium alkylphenolate. These supplementary detergent-dispersants are disclosed in U.S. Pat. Nos. 3,087,956, 3,549,534 and 3,537,966.

Examples of suitable engine oil antioxidants contemplated herein are zinc and cadmium dialkyl dithiophosphates and diaryl dithiophosphates, the alkylated diphenylamines, sulfurized diphenyl amines, unsulfurized and sulfurized alkylphenols and phenolates and hindered phenols.

Examples of suitable engine oil corrosion inhibitors are zinc dialkyl dithiophosphates, zinc diaryl dithiophosphate, basic calcium and magnesium sulfonates; calcium, barium and magnesium phenolates.

The following examples further illustrate the compositions of the invention but are not to be construed as limitations thereof:

EXAMPLE I

This example illustrates the interpolymer component of the compositions contemplated herein. In the following procedure the quantities employed are on a weight basis unless otherwise stated.

To a 1-liter resin kettle there were added 40 parts vinyl toluene, 10 parts N,N-dimethylaminoethyl methacrylate, 100 parts technical lauryl methacrylate, 50 parts technical stearyl methacrylate and 100 parts of naphthenic lubricating oil of an SUS viscosity of about 145 at 100°F. The contents of the resin kettle were then purged with prepurified nitrogen for a 40 minute period at which point the mixture was then heated to 82° ± 1°C. and 0.4 parts of azobisisobutronitrile and 0.2 part lauryl mercaptan were added. The reaction was followed by monitoring the refractive index of the polymerizing solution every half hour. The reaction proceeded for a 3.5 hour period to completion at which point the temperature was raised to 100°C. and 186 parts of a mineral lubricating oil of an SUS viscosity of about 45 at 100°F. were added and an additional charge of 0.15 parts of azoisobutronitrile was added. This 100°C. temperature was maintained for an additional 2 hours as a finishing step and the resultant interpolymer concentrate was cooled to ambient temperature and analyzed. It was identified as a 41 wt. % interpolymer lubricating oil solution of an SUS viscosity of about 3,100, said interpolymer being the tetrapolymer of dimethylamino methacrylate, vinyl toluene, dodecyl methacrylate and octadecyl methacrylate in a respective monomer weight ratio of 1:4:10:5, said interpolymer per se having an intrinsic viscosity of about 0.85 at 77°F. in benzene.

Following Examples II, III and IV further illustrate the interpolymer component of the compositions contemplated herein. The quantities employed are on a weight basis unless otherwise stated.

EXAMPLE II

To a 1-liter resin kettle there was added 10 parts

N,N-dimethylaminoethyl methacrylate, 40 parts styrene, 100 parts technical lauryl methacrylate, 50 parts technical stearyl methacrylate and 100 parts of naphthenic lubricating oil of an SUS viscosity of about 145 at 100°F. The contents of the resin kettle were purged and polymerization was conducted as set forth in Example I. The product was identified as a 38 wt. % interpolymer lube oil solution, said interpolymer having an intrinsic viscosity in benzene at 77°F. of about 0.69 and having a monomer weight ratio that of the monomer weight ratio initially introduced.

EXAMPLE III monomer

A 1-liter resin kettle was charged with 8 parts N,N-dimethylaminoethyl methacrylate, 40 parts styrene, 100 parts technical lauryl methacrylate, 52 parts technical stearyl methacrylate and 100 parts of a naphthenic lubricating oil of an SUS viscosity of about 145 at 100°F. The contents of the resin kettle were purged and the polymerization was conducted as set forth in Example I. The product was identified as a 38 wt. % interpolymer lube oil solution, said interpolymer having an intrinsic viscosity in benzene at 77°F. of about 0.59 and having a monomer weight ratio that of the monomer weight ratio initially introduced.

EXAMPLE IV

A 1-liter resin flask was charged with 8 parts N,N-dimethylaminoethyl methacrylate, 44 parts styrene, 100 parts technical lauryl methacrylate, 48 parts technical stearyl methacrylate and 100 parts of a naphthenic lubricating oil of an SUS viscosity of about 145 at 100°F. The contents of the resin kettle were purged and polymerization was conducted as set forth in Example I. The product was identified as a 38 wt. % interpolymer lube oil solution, said interpolymer having an intrinsic viscosity in benzene at 77°F. of about 0.61 and having a monomer weight ratio that of the monomer weight ratio initially introduced.

EXAMPLE V

This example illustrates the lube oil compositions of the invention and the outstanding effect of the interpolymers contemplated herein in providing low temperature fluidity to said compositions.

Three automatic transmission fluid (ATF) base compositions were employed. They are as follows:

TABLE I

Composition	ATF-A	ATF-B	ATF-C
Ingredients, wt. %			
Mineral Lube Oil	86.5	100	88
Supplementary VI	8.5 ^a	—	4 ^b
Additive Package A-I ^c	5	—	—
Additive Package B-II ^d	—	—	8
Tests			
Brookfield Visc., cps.			
-10°F.	—	—	~2700
-20°F.	2270	—	—
-40°F.	59,200	300,000 ⁺	41,000
Visc. (SUS) cs. at 100°F.	30.9 (145)	(130)	43.4 (202)
210°F.	7.72 (51.5)	(42)	7.89 (52)
Viscosity Index	251	~95	167
Pour Point, °F.	-50	-0	-45

a). Terpolymer of methyl methacrylate, dodecyl methacrylate, octadecyl methacrylate in a weight ratio of 25/50/25 in a 43 wt. % concentration in hydrocarbon mineral lubricating oil of an SUS viscosity of about 100 at 100°F, said terpolymer having an intrinsic viscosity of about 0.68 in benzene at 77°F.

Table I—Continued

b). A tetrapolymer of butyl methacrylate, dodecyl methacrylate, octadecyl methacrylate, and dimethylaminoethyl methacrylate in a monomer weight ratio of 21:50:25:4 in a 41 wt. % concentration in hydrocarbon mineral lubricating oil of an SUS viscosity of about 120 at 100°F., said tetrapolymer having an intrinsic viscosity in benzene at 77°F. of about 0.86.

c). comprises about 18 wt. % overbased calcium petroleum sulfonates, 16 wt. % barium soaps of fatty acids, 12 wt. % overbased zinc salts of alkaryl phosphinodithioic acids and 54 wt. % mineral oil.

d). comprises about 2 wt. % alkylated polythiadiazoles, 5 wt. % diocryldiphenyl amine, 21 wt. % reaction product of a polyisobutenyl succinic anhydride-polyalkylene polyamine condensate with a phosphosulfurized hydrocarbon and neutralization thereof with a high molecular weight carboxylic acid and 72 wt. % mineral oil-aromatic stock.

Interpolymers contemplated herein and comparative 10
interpolymers were tested in the aforescribed three
ATF fluids. The test data and results are reported
below in Tables II and III. FISST in Tables II and III
stands for Fuel Injective Shear Stability Test and com-
prises injecting through a 0.006 inch nozzle at a pres-
sure of 3,000 psi the material to be tested. The viscosity 15
before and after injection is measured and the degree

of viscosity loss is proportional to the degree of shear.

In Table II, directly below, Runs 618, 621, 664, 665
and 680 are representative of the compositions of the
invention and comparison of their Brookfield Viscosity
at -40°F. with the base fluid demonstrates the low tem-
perature improving properties of the interpolymers
contemplated herein.

TABLE II

Run No.	Base Fluid	618	621	664	665	680
Interpolymer (I),						
wt. %						
N,N-Dimethylamino-ethyl methacrylate	—	4	5	5	4	4
Vinyl Toluene	—	21	20	—	—	—
Styrene	—	—	—	20	20	22
Lauryl Methacrylate	—	50	50	50	50	50
Octadecyl Methacrylate	—	25	25	25	26	24
Intrinsic Viscosity, 77°F. Benzene	—	0.71	0.80	0.69	0.59	0.61
Interpolymer Lube						
Conc. (ILC)						
Interpolymer, wt. %	—	41	41	38	38	38
Mineral Oil (145 SUS at 100°F.), wt. %	—	59	59	62	62	62
AFT-A + ILC						
Wt. % ILC	0	9	9	9	9	9
Visc. at 100°F., cs.	30.9	41.1	40.8	37.5	33.4	35.6
210°F., cs.	7.72	8.48	8.25	7.88	7.12	7.62
Pour Point, °F.	-50	-55	-50	-55	-55	-55
Brookfield Visc. (cps), at -20°F.	2270	3800	3510	2560	2360	2160
-40°F.	59,200	25,500	24,250	12,040	13,900	10,240
FISST (20 passes) 0.006						
Visc. at 100°F., cs.	—	30.3	30	27.2	26.1	26.7
210°F., cs.	—	6.11	6.17	5.68	5.54	5.72
ATF-B + ILC						
Wt. % ILC	0	3.5	3.5	3.5	3.5	3.5
Kin. Visc., 210°F., cs.	—	7.14	7.42	6.69	6.38	6.46
Brookfield Visc. -40°F., cps	—	46,000	42,500	35,500	38,750	32,900

TABLE III

Run No.	Base Fluid	7231	7232	7241	481	482
Interpolymer (I)						
wt. %						
N,N-Dimethylamino-ethyl methacrylate	—	10	20	25	4	6
Styrene	—	30	30	25	21	19
Lauryl Methacrylate	—	60	50	50	50	50
Octadecyl Methacrylate	—	—	—	—	25	25
Intrinsic Viscosity, 77°F. Benzene	—	0.55	0.49	0.55	0.73	0.92
Interpolymer Lube						
Conc. (ILC), wt. %						
Interpolymer	—	38	38	38	38	38
Mineral Oil (145 SUS at 100°F.)	—	62	62	62	62	62
ATF-C + ILC						
Wt. % ILC	0	4.3	4.3	4.3	4.3	4.3
Visc. at 210°F., cs	4.84	6.41	5.59	5.91	7.32	8.00
Pour Point, °F.	+5	0	-5	+10	-50	-55
Brookfield Viscosity -40°F., cps	300,000 ⁺	300,000 ⁺	300,00 ⁺	300,000 ⁺	32,624	34,500
FISST (20 passes) 0.006	5.86	5.90	5.73	5.42	5.88	6.02
Visc., 210°F., cs						

In Table III above by comparison of comparative Run Nos. 7231, 7232 and 7233 with representative Run Nos. 481 and 482 in respect to pour point and Brookfield Viscosity at -40°F . demonstrate the importance of the monomeric ratios of the interpolpolymer in respect of being in the ranges as defined.

EXAMPLE VI

The following illustrates the engine lubricating oil compositions contemplated herein and the effectiveness of the contemplated interpolpolymers in reducing pour point therein.

The base engine oil composition was as follows:

Base Oil	
Ingredients	Wt. %
Mineral Oil A, 130 SUS 100°F .	78
Mineral Oil B, 340 SUS 100°F .	8
Additive Package X*	14
Polysilicone Antifoamant	60 ppm

*Balance dispersant-inhibitor concentrate for heavy duty motor oils composed of ethoxylated, hydrolyzed polyisobutene- P_2S_5 reaction product, calcium carbonate overbased calcium petroleum sulfonate, zinc dialkyldithiophosphate, alkylated diarylamine antioxidant, polysilicone foam inhibitor (750 ppm) and mineral oil diluent.

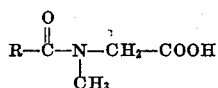
The pour points of the Base Oil and the Base Oil containing 9 wt. % of the interpolpolymer concentrate (ILC) of representative Run No. 482 in Example V, Table III were measured. The results are as follows:

Composition	Pour Point, $^{\circ}\text{F}$.
Base Oil	10
Base Oil + 9 wt. % ILC (Run No. 482)	-40

We claim:

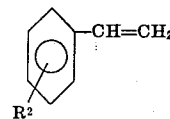
1. A mineral oil composition comprising a major amount of a hydrocarbon mineral lubricating oil and between about 0.1 and 10 wt. percent of an interpolpolymer consisting of a tetrapolymer composed of the following monomers:

a. dialkylaminoalkyl methacrylate characterized by the formula:



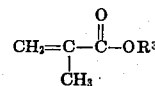
where R and R^1 are alkyl of from 1 to 2 carbons and A is alkanediyl of from 1 to 5 carbons;

b. styrene compound of the formula:



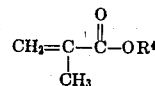
where R^2 is hydrogen or alkyl of from 1 to 10 carbons;

c. A first alkyl methacrylate of the formula:



where R^3 is alkyl of from 10 to 14 carbons;

d. A second alkyl methacrylate of the formula:



where R^4 is alkyl of from 16 to 20 carbons, said tetrapolymer having an intrinsic viscosity in benzene at 77°F . of between about 0.22 and 2.87 and consisting of a component content of between about 4 and 10 wt. percent of said dialkylaminoalkyl methacrylate, between about 15 and 25 wt. percent of said styrene compound, between about 40 and 60 wt. percent of said first alkyl methacrylate, and between about 20 and 30 wt. percent of said second alkyl methacrylate.

2. A composition in accordance with claim 1 wherein R^1 and R^3 are methyl, A is ethanediyl, R^2 is 4-methyl, R^3 is lauryl and R^4 is stearyl.

3. A composition in accordance with claim 1 wherein R^1 and R^3 are methyl, A is ethanediyl, R^2 is hydrogen, R^3 is lauryl and R^4 is stearyl.

4. A composition in accordance with claim 1 wherein said interpolpolymer content is between about 0.5-5 wt. percent and the viscosity of said lubricating oil is between about 40 and 150 SUS at 100°F .

5. A composition in accordance with claim 1 wherein said interpolpolymer content is between about 1 and 5 wt. percent and the viscosity of said lubricating oil is between about 95 and 150 SUS at 100°F .

* * * * *