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SYNTHETIC LUBRICANTS

John Arthur Wickings, Sutton, Surrey, Eric James Percy, Coulsdon, Surrey, and David John Rosevear, Epsom, Surrey, England, assignors to BP Chemicals (U.K.) Limited, London, England, a British company

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6 Claims

ABSTRACT OF THE DISCLOSURE

The invention is lubricant compositions which have as base compounds which may be made by esterifying with a mixture of acids the product of reaction of an alkylene oxide with a polyol. The compounds are found to have high viscosity indices and good resistance to shear stresses, and are therefore suitable for use in transmissions, gear boxes and the like.

The present invention relates to synthetic lubricant compositions.

Synthetic lubricant compositions based on adipic, sebacic or azelaic acid derivatives are known. These compositions are generally of low viscosity, i.e. of the order of 3 to 7 centistokes at 210° F. and find application as lubricants in jet engines where high temperatures and high journal speeds require the use of specialised lubricating compositions. The use more recently of synthetic lubricating compositions in internal combustion engines or associated equipment or stationary jet engines for specialised application e.g. the "filled for life" back axles of motor vehicles, is also known. These latter compositions for use in engines etc. having lower journal speeds than jet engines, require a higher viscosity than the 3 to 7 centistokes referred to previously and have been based on compounds such as pentaerythritol. Such lubricating compositions suffer from the disadvantage of high cost due to the basic cost of the raw materials.

It has now been found possible according to the present invention to cheapen the cost of the lubricant base without loss of any of the desirable properties of the lubricating composition.

In addition the lubricant bases of the present invention have high viscosity indices, of the order of 140. This compares very favourably with the viscosity index of mineral oils which are used as lubricant bases and have viscosity indices up to about 90. The viscosity index of the mineral oil can be improved by adding compounds such as polylauryl methacrylate. However, when the lubricant is subjected to shear stresses, for example when used in transmissions, gear boxes, back axles, the viscosity index improver breaks down, and the viscosity index of the lubricant tends to revert to the viscosity index of the base. Hence lubricants which rely on additives to improve the viscosity index must be replaced at fairly frequent intervals, whereas lubricant compositions comprising the lubricant base of our invention do not suffer from this drawback.

Accordingly the present invention is a synthetic lubricant composition wherein the lubricant base comprises a mixed ester of an alkoxyated polyol (as hereinafter defined) with at least two carboxylic acids, wherein the acids each contain from 3 to 15 carbon atoms.

Throughout the specification and claims by alkoxyated polyol is meant a compound which may be made by reaction of a polyol with an alkylene oxide having at least three carbon atoms. This is the preferred method for

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producing the alkoxyated polyol, but the invention is not confined to the use of compounds made in this particular manner.

Suitable polyols are trimethylol propane, glycerol, pentaerythritol, dipentaerythritol, sorbitol and neopentylglycol. A preferred polyglycol is trimethylolpropane.

The preferred alkylene oxide is propylene oxide, although it is possible to use mixtures of two or more different alkylene oxides for reaction with the polyol. The alkoxyated polyol may contain from 1 to 24, preferably 1 to 6 substituent alkoxy groups per hydroxyl group of the polyol.

The preferred carboxylic acids are aliphatic monobasic acids. Suitable acids may include high molecular weight acids such as heptonic, caprylic, pelargonic, capric and lauric acid and lower molecular weight acids such as propionic, butyric and valeric acid. It is preferred to use mixtures of lower and higher molecular weight acid for esterifying the alkoxyated polyol. By using mixtures of acids it is possible to exercise finer control over the viscosity of the final product.

The lubricant bases of the present invention may be prepared in any convenient manner. For example the polyol may be reacted with the desired alkylene oxide or mixtures of oxides, the reaction being carried out under pressure in an autoclave and preferably in the presence of a suitable alkaline catalyst e.g. caustic soda or sodium methoxide. On completion of the alkoxylation reaction indicated by the absorption of the desired number of moles of alkylene oxide by the polyol, the autoclave mixture is neutralised with acid, e.g. benzene sulphonic acid, an excess of acid added and the mixture esterified with the fatty acids. The esterification may be carried out in any convenient manner. The alkoxyated polyol may be esterified in a single step by reaction with a mixture of the higher and lower molecular weight fatty acids. Preferably the alkoxyated polyol is first partially esterified with a higher molecular weight acid or acids and the esterification completed by esterification with lower molecular weight acid or acids. It is desirable that the final product contains no hydroxyl groups, so the alkoxyated polyol is reacted with the higher acid and then subsequently with the more reactive lower acid to esterify all the remaining hydroxyl groups.

The lubricant base should be miscible with other base fluids for example the mineral oils commercially available as Pool 12 and Brightstock. As an approximate guide, it is found that the higher the ratio of carbon atoms to oxygen atoms in the molecule the more compatible the lubricant base of the present invention is with mineral oils. It is preferred to use compounds in which the ratio of carbon atoms to oxygen atoms is greater than 3.4:1. Compounds in which the ratio is greater than 3.9:1 are particularly preferred.

The synthetic lubricant compositions of the present invention show high performance and high temperature stability and have lubricating properties corresponding to those of a mineral lubricating oil. The compositions may comprise other conventional oil additives, e.g. antisludge agents, extreme pressure agents and antioxidants known in the art.

The compositions of the present invention are illustrated by the following examples:

EXAMPLES 1-3

Preparation of the polyol/alkylene oxide adduct

The polyol, dried to a water content of less than about 0.3% (solvent, where used) and flake sodium hydroxide catalyst were charged to the autoclave. The vessel was sealed, evacuated and the vacuum broken with nitrogen. This cycle was repeated once more. The vessel was then evacuated and the contents heated to 130-140° C. The

desired quantity of alkylene oxide was forced in, with stirring, the rate of addition being controlled so that the pressure in the vessel did not exceed ~40 p.s.i.g. Heating and stirring were continued until no further reduction in pressure took place. The reaction mixture was cooled to 50° C. and vacuum applied to the vessel to remove any unreacted alkylene oxide.

The details of the individual examples and the results are given in Table 1.

EXAMPLES 4-6

Esterification of the alkoxyated polyol

The alkoxyated polyol, higher fatty acid(s), catalyst and entrainer were charged to a suitable vessel. Refluxing was continued with the removal of water until the acid number of the reaction mixture was below about 5. The lower molecular weight acid was then added and reaction continued until the hydroxyl number of the product had fallen to a suitable level. Excess acid and entrainer were then removed under vacuum and the product purified by normal techniques used to purify esters. The details and results are given in Table 2.

comparison the test was run against an SAE 20, high V.I. aliphatic mineral oil.

The results are given in Table 3.

TABLE 3

Load (kg.):	Product of Example 6 Wear Scar diameter (mm.)	SAE 20 mineral oil Wear Scar diameter (mm.)
45	0.339	0.345
89	2.17	2.36
112	2.29	2.39
141	(1)	(1)
158	(2)	(2)

¹ Catastrophic damage.
² Weld point.

From these tests it is considered that the two fluids are practically identical in wear load characteristics.

Viscosity characteristics after shearing

Shear resistance was assessed by measuring the viscosity index before and after shearing. Shearing was carried out

TABLE 1

Example	Charge quantities			Reaction temp. (°C.)	Reaction time (hr.)	Product	Intermediate for preparation of example
	Polyol (g.)	Propylene oxide, (g.) (PrO)	NaOH (g.)				
1	Glycerol 920	~8,120	~20	140	22	Glycerol/14 propylene oxide	5
2	Trimethylol propane 1,206.	~7,350	~8.5	135-140	10	Trimethylolpropane/14 propylene oxide	6
3	Trimethylol propane/4 propylene oxide 358.	705	0.7	145	8	Trimethylolpropane/16 propylene oxide	7

TABLE 2

	Product			
	Example 4	Example 5	Example 6	Example 7
	Size of preparation (moles)			
	1.0	1.0	0.5	0.75
Polyol	Sorbitol/10 PrO	Glycerol/14 PrO adduct	Trimethylol propane/14 PrO adduct	Trimethylol propane/16 PrO adduct
Moles used	1.0	1.0	0.5	0.75
Grams used	680	871	465	772
Acid(s) in initial charge	(1)	(2)	(2)	(2)
Moles used	5.5	2.5	1.25	1.875
Grams used	787	500	250	375
Benzene sulphonic acid (g. of 33% solution)	11.1	31.8	12.0	28.5
Entrainer used	(3)	(3)	(3)	(3)
Grams used	220	137	230	230
Butyric acid (added later):				
Moles	1.0	0.5	1.25	0.375
Grams	88	2×44	110	2×33
Total reaction time (hrs.)	47	58	18	22
Reaction temp. range (° C.)	150-170	180-190	150-160	136-168
Additional entrainer added (ml.)	350	Nil	50	40
Analysis of product:				
Acid number	0.34	0.36	0.91	0.31
Ester number	262	184.8	136	119
Hydroxyl number	1.5	4.6	5.8	2.4
Water content (percent)	0.05	0.092	0.07	0.042
SG at 20° C.	0.987		0.969	0.970
RI at 25° C.			1.448	
Sodium content (p.p.m.)	20	8	2	Nil
Pour point (° F.)	-58	-27	-45	-51
Viscosity and miscibility characteristics:				
Viscosity at 210° F. (cs.)	6.85	7.95	11.40	12.01
Viscosity at 100° F. (cs.)	37.61	39.65	67.37	67.16
Viscosity index	142	153	142	146
Compatibility 1:1:				
With Pool 12	Yes	Yes	Yes	Yes
With Brightstock	Yes	Yes	Yes	Yes
Carbon to oxygen ratio	3.73	3.85	4.0	3.91

¹ Mixed (C⁸-C¹¹).
² Lauric.
³ Toluene.

Wear load characteristics

A sample of the product of Example 6 was tested using the Shell four ball tester and steel on steel specimens. For

75 by circulating the fluid through a diesel fuel injector for 45 minutes. It was calculated that the time of circulation was equivalent to 200 passes through the injector. The

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fluids tested were the product of Example 6 and a solvent neutral 150 oil containing 5% of a commercial V.I. improver, Hitec 960. The results are given in Table 4.

TABLE 4

	Product of Example 6		Solvent neutral 150 +50% Hitec 960	
	Before shearing	After shearing	Before shearing	After shearing
Viscosity at 210° F. (cs.)	11.40	10.78	10.74	7.12
Viscosity at 100° F. (cs.)	67.37	64.72	56.55	41.58
Viscosity index	142	141	150	134

It will be noted that the effect of shearing on the viscosity and viscosity index of the lubricants was very much greater with solvent neutral 150+Hitec 960. This clearly illustrates the superior stability to shear of the lubricant bases of our invention.

We claim:

1. A synthetic lubricant composition having a lubricant base which comprises a mixed ester of an oxypropylated polyol, which polyol contains only hydroxyl groups in addition to carbon and hydrogen, said oxypropylated polyol being obtained by reaction of propylene oxide with an aliphatic polyol and said ester being of liquid lubricating oil viscosity and having no free hydroxyl groups and the acids used to form the ester being a mixture of higher molecular weight acids selected from heptonic, caprylic, pelargonic, capric and lauric acids and lower molecular weight acids selected from propionic, butyric and valeric acids, the higher and lower molecular weight acids being present in amounts sufficient to adjust the viscosity of the lubricant composition.

2. A composition according to claim 1 wherein the ratio of carbon atoms to oxygen atoms in the base is greater than 3.4:1.

3. A composition according to claim 1 wherein the polyol is trimethylol propane, glycerol, pentaerythritol, dipentaerythritol, sorbitol or neopentylglycol.

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4. A composition according to claim 1 wherein the oxypropylated aliphatic polyol contains from 1 to 24 substituent oxypropyl groups per hydroxyl group of the polyol.

5. A composition according to claim 1 wherein the lubricant base has a high viscosity index of the order of 140 and has incorporated therein an additive selected from the group consisting of antisludge agents, antioxidants and extreme pressure agents.

6. A synthetic lubricant composition made up of a liquid lubricant base of lubricating oil viscosity and an additive wherein the lubricant base comprises a major proportion of an ester of an oxypropylated aliphatic polyol, obtained by reaction of propylene oxide with trimethylolpropane, with a mixture of carboxylic acids wherein one component of the mixture is selected from the group consisting of heptonic, caprylic, pelargonic, capric and lauric acids, and the other component is selected from the group consisting of propionic, butyric and valeric acids, each acid component being present in amounts sufficient to adjust the viscosity of the lubricant composition.

References Cited

UNITED STATES PATENTS

2,457,139	12/1948	Fife et al.	260—410.6
2,604,453	7/1952	Popkin	252—56
3,137,737	6/1964	Emrick et al.	
2,514,982	7/1950	Walters et al.	252—52
2,665,312	1/1954	Ohlmann et al.	260—410.6 X
3,062,671	4/1962	Kaupp et al.	260—410.6 X
3,337,595	8/1967	Lamont	260—410.6

DANIEL E. WYMAN, Primary Examiner

W. H. CANNON, Assistant Examiner

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260—410.6, 484