United States Patent Office 3,021,281 Patented Feb. 13, 1962

3,021,281

EXTREME PRESSURE LUBRICANT COMPOSITION

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No Drawing. Filed Aug. 4, 1958, Ser. No. 753,129
5 Claims. (Cl. 252—47.5)

This invention relates to additives for mineral oil compositions and more particularly to agents which impart extreme pressure, increased load carrying capacity and anti-wear properties to lubricants.

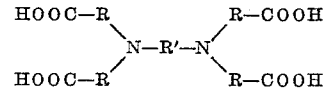
With the advent of higher compression internal combustion engines, much difficulty has been experienced in formulating a suitable lubricating oil composition capable of operating under severe conditions to protect effectively metal surfaces which come in contact with the oil. It is well known that the high pressure occurring in certain types of gears and bearings may cause a film of lubricant to rupture with subsequent damage to the machinery. It has been shown that base lubricants such as mineral oil and/or synthetic oil can be improved with regard to their protective effect on rubbing surfaces by the addition of certain substances so that excessive wear, scuffing and seizure which normally follow a break in the film of lubricant can be prevented even under the most unfavorable pressure and speed conditions. Lubricants possessing this highly desirable property are called extreme pressure lubricants.

Certain classes of compounds can be used to improve specific properties of base lubricants. Thus, compounds containing sulfur, chlorine, phosphorus or lead or combinations of these elements may improve the extreme pressure properties of a lubricant. However, the simple presence of these elements does not guarantee extreme pressure properties and various compounds that do have these properties can vary considerably in their effectiveness. It is generally believed that the effectiveness of an extreme pressure additive is related to its ability to react with and form a protective coating on a bearing surface to be lubricated. To be most effective, however, the additive must be present in sufficient concentration at the surfaces to form an adequate film. One method of realizing this surface concentration is the use of polar or other type molecules which have an affinity or attraction for surfaces. Thus, as opposed to the use of a non-polar molecule which will be present in more or less equal concentration throughout the bulk of the lubricant, a polar type molecule will tend to give a greater concentration of additive at the metal surface where it is needed.

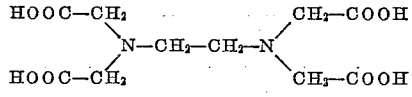
In a similar manner a strongly polar material which is not an extreme pressure additive can interfere with the function of a less polar extreme pressure additive. Thus, a strongly polar non-extreme pressure molecule may be preferentially deposited from its admixture with an extreme pressure additive and prevent the extreme pressure additive from forming its protective extreme pressure film.

My invention is directed to a lubricating oil composition containing essentially a major amount of a mineral lubricating base oil, an oil-soluble tetraester of a complex acid and a sulfurized fatty oil. The combined tetraester-sulfurized fatty oil additives have been found to improve load-carrying and anti-wear properties of the lubricating oil. The tetraester is formed from carboxylic acid groups preferably of lower aliphatic acids such as acetic, propionic, butyric, and the C_5 and C_6 aliphatic acids and an alcohol, saturated or unsaturated, preferably an aliphatic, saturated alcohol having about six carbon atoms

or more, preferably about 6 to 20 carbon atoms. In general the acid has the following molecular structure:



where R is a divalent hydrocarbon radical of say 1 to 5 carbon atoms and R′ is a divalent hydrocarbon radical preferably aliphatic, of 2 to 6 carbon atoms. The preferred acid for my invention is ethylene diamine tetraacetic acid and has the following molecular structure:



The ethylene diamine tetraacetic acid used in forming the reaction products is an efficient chelating agent; that is, a chemical which complexes polyvalent metallic ions with a multi-ring structure that inactivates the metal. This property has been used in a variety of applications.

The preferred alcohols of my invention are saturated alcohols having from about 6 to 20, advantageously 6 to 12, carbon atoms such as octyl alcohol, decyl alcohol, tetradecyl alcohol, hexadecyl alcohol, and the like. These aliphatic alcohols are preferred, but it will be understood that unsaturated, and aromatic alcohols may be substituted in whole or in part in formating the ester reaction product.

In making the ester the appropriate acid and methanol are mixed, and the solution is refluxed and heated for the necessary period of time to obtain esterification. The time of refluxing is related to the particular constituents employed and is not considered critical. The period of time will generally range from about 2 to 14 hours. After refluxing is completed excess alcohol is distilled off. Then a sufficient quantity of a low molecular weight alcohol such as isobutanol along with a catalyst such as octylene glycol titanate is added to facilitate transesterification. The constituents are heated for approximately 5 to 8 hours at a temperature of about 120 to 140° C. The product thus obtained is added to a higher molecular weight saturated alcohol and a solvent such as xylene and a catalyst such as octylene glycol titanate is added to facilitate ester interchange. The reaction is now maintained at a temperature from about 140 to 160° C. for a period of from about 5 to 10 hours. The solvent is then removed by distillation.

The mineral oil base stock used in the present invention is of lubricating viscosity and can be for instance a solvent extracted or solvent refined oil obtained in accordance with conventional methods of solvent refining lubricating oils. Generally, lubricating oils have viscosities from about 20 to 250 SUS at 210° F. The base oil may be derived from paraffinic, naphthenic, asphaltic or mixed base crudes, and if desired, a blend of solvent-treated Mid-Continent neutrals and Mid-Continent bright stocks may be employed. A particularly suitable base oil used in the preparation of the compositions described hereinafter is a solvent treated Mid-Continent neutral having a viscosity index of about 95.

The amount of tetraester added to the mineral oil will vary depending upon the particular characteristics of lubricating oil utilized although sufficient ester is present to give the desired effect. In general from about 0.1 percent of the ester by weight to about 10 percent of the final composition by weight is preferred in my lubricating oil composition. Although my additive has been de-

scribed as the tetraester it may contain portions of less esterified materials such as di- and tri-esters.

The sulfurized fatty oils preferred for my invention are for instance, sulfurized sperm oil, sulfurized rapeseed oil, sulfurized cotton seed oil and sulfurized palm oil. These oils in general are essentially esters, e.g. glycerides, of acids of 12 to 18 carbon atoms. A particularly effective material employed is sulfurized sperm oil which contains about 5 to 15 weight percent of combined sulfur. In general from about 1 percent of sulfurized fatty oil to about 15% of the final composition is employed with a preferred amount being from about 3 to 8 percent by weight of the final composition.

My composition can also contain other additives such as anti-oxidants, anti-foaming agents, etc.

The following examples, not to be construed as limiting are offered to further illustrate the novel lubricating oil composition of the present invention.

EXAMPLES

73 grams of ethylene diamine tetraacetic acid are added to 320 grams of methyl alcohol and refluxed with stirring for a period of about 3 hours. 10 grams of concentrated HCl is then added and the mixture is refluxed for about 8 hours and then the reaction product is recovered and the excess alcohol is distilled off. The light amber, viscous product is found to be insoluble in mineral oil, di-(2-ethylhexyl)-sebacate, toluene and 2-ethylhexanol, but soluble in isobutanol. 225 grams of isobutanol and 1 gram of octylene glycol titanate are added to the above product to facilitate ester interchange. The reaction is maintained at approximately 130° C. for a period of about 6 hours. 260 grams of ethylhexanol are added and the reaction is maintained at about 150° C. for five hours. This is accomplished by gradually recovering the reflux as distillate during the above period. The product is called product A and analyzed as containing 3.87% nitrogen.

The following tests were conducted on the indicated blended compositions:

Shell 4-ball wear test
Mean Hertz load
SAE load test
Timken L–20 test

These tests have been fully described in literature on the subject of anti-wear and extreme pressure testing. Thus, I will not attempt to go into any detail concerning the procedure of each. The possible exception to the above is the Timken L–20 test which is run using the Timken machine described in CRC L–18–545. The test block is continuously moved back and forth beneath the test cup in a direction paralleling the level arm for a distance for about 0.13 inch at an approximate rate of about 4 times per minute. Conditions for load, speed, time and temperature may be varied and are noted for a given test.

Table I

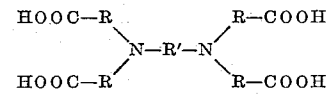
Composition	1	2	3	4
Base Oil,[1] Wt. Percent	100	98	95	93
Product A, Wt. Percent		2.0		2.0
Sulfurized Sperm Oil (12% S), Wt. Percent.			5.0	5.0
Tests:				
Shell 4-Ball Wear Test, Scar Diam., mm. (7 Kg., 2 hrs., 110° C., 640 r.p.m.).	0.687	0.500	0.445	0.400
Mean Hertz Load	13.7	20.0	33.7	37.6
SAE at 300 r.p.m., lbs.	"Fails at Start of Test."	84	136	333
Timken L–20 test, Total wear, mg.—				
(100 lbs., 200° F., 200 r.p.m., 16 hrs.).		89.5	32.2	15.3
(125 lbs., 200° F., 200 r.p.m., 16 hrs.).				

[1] Solvent refined Mid-Continent neutral oil, viscosity 33 SUS at 100° F.

The above data clearly demonstrate the improved anti-wear properties, increased load carrying capacity and extreme pressure properties of my compositions.

I claim:

1. A lubricating oil composition consisting essentially of a mineral lubricating base oil, and about 0.1 to 10% of an oil-soluble tetraester of an alcohol having about 6 to 20 carbon atoms and a carboxylic acid having the following molecular structure:



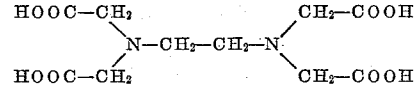
wherein R is a divalent hydrocarbon radical of 1 to 5 carbon atoms and R′ is a divalent hydrocarbon radical of 2 to 6 carbon atoms, and about 1 to 15 percent of a sulfurized fatty oil.

2. The lubricating oil composition of claim 1 wherein the sulfurized fatty oils are of acids of 12 to 18 carbon atoms and contain about 5 to 15% combined sulfur.

3. The lubricating oil composition of claim 1 in which the acid is ethylene diamine tetraacetic acid.

4. The lubricating oil composition of claim 3 in which the sulfurized fatty oil is sulfurized sperm oil containing about 5 to 15% combined sulfur.

5. A lubricating oil composition consisting essentially of a mineral lubricating base oil and about 0.1 to 10% of the final composition of an oil-soluble tetraester of an alcohol of 6 to 12 carbon atoms and a carboxylic acid having the following molecular structure:



and about 3 to 8% of sulfurized sperm oil containing about 5 to 15% combined sulfur.

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