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2,961,406

PENTAERYTHRITOL ESTER LUBRICANTS

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No Drawing. Filed Apr. 23, 1959, Ser. No. 808,318

2 Claims. (Cl. 252—56)

This invention relates to a lubricant composition. More particularly, it relates to a composition comprising an ester of pentaerythritol that is especially useful for high temperature lubrication.

With the development of gas turbine aircraft engines which must operate at peak efficiency at extremely high altitudes, there has developed a need for lubricants which are efficacious for long periods of time at the high temperature at which such engines operate. In the past, certain synthetic lubricants, particularly dibasic acid esters such as dioctyl sebacate, have been used successfully as lubricants for gas turbines but such lubricants are limited for practical purposes to a maximum bulk oil temperature of about 300° F. The newer gas turbines, however, operate at a bulk oil temperature considerably higher than 300° F. and the art is searching for a lubricant which operates satisfactorily at these higher temperatures.

As a result, many other materials of synthetic origin have been examined as possible lubricants in gas turbines. Although there are many materials which have looked promising from the standpoint of physical characteristics such as pour point, flash point, etc., actual performance of such materials in engine testing has been disappointing because of excessive sludge and coke deposits that form in the engine. For instance, esters of pentaerythritol and alkanolic acids have many properties that recommend them as lubricants for gas turbines. However, when this class of esters is employed in a gas turbine operating at a bulk oil temperature materially above 300° F., there occurs in time a build-up of coke and sludge on the bearings and in the bearing compartments of the engine.

The present invention is based upon the discovery that the tendency of the alkanolic acid esters of pentaerythritol to deposit coke and sludge in an engine in which they are employed as a lubricant can be reduced materially by adding thereto a relatively small amount of an alkanolic acid ester of pentaerythritol monoformal.

In accordance with this discovery the invention relates to a composition especially useful as a high temperature lubricant comprising (1) a pentaerythritol ester of an acid selected from the group consisting of alkanolic acids having from 5 to 10 carbon atoms or a mixture of alkanolic acids having from 2 to 12 carbon atoms in proportions to provide an average chain length of from 5 to 10 carbon atoms, and (2) a minor amount of an ester of pentaerythritol monoformal with at least one alkanolic acid having from 2 to 18 carbon atoms.

The preparation of compositions typical of those to which the invention relates is illustrated in the following examples. Parts and percentages are by weight unless otherwise specified.

Example 1

Into a reaction vessel equipped with a thermometer, an agitator, reflux condenser, and water trap, there was placed 17.1 parts of technical pentaerythritol (about 90

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parts monopentaerythritol and 10 parts dipentaerythritol), 1 part of pentaerythritol monoformal, and 74.3 parts of a mixture of acids having approximately the following composition averaging 6 carbon atoms in chain length:

	Parts
Butyric acid.....	45
Capric acid.....	18
Caprylic acid.....	37

There was next added one part of activated carbon and 1.7 parts of xylene. The resulting mixture was agitated and heated from 150° C. to 210° C. in 14 hours during which time 11 parts of water distilled as the result of esterification. During this 14-hour period the reflux condenser was maintained in place and water as it evolved from the reaction was collected in the water trap while xylene was refluxed back to the reaction. The reflux condenser was next substituted by a simple distillation head with condenser and the temperature was increased slowly to 240° C. over a period of 2 hours. This operation distilled xylene and part of the excess acids present. Next, the remainder of excess acids was stripped off using a nitrogen sparge at a minimum pressure of 20 millimeters of mercury; a maximum stripping temperature of 242° C. was reached in one hour and the mixture was held there for 30 minutes. The reaction product was next cooled to 170° C., slurried with 1.33 parts of lime for 30 minutes and then filtered with one to three parts of inert filter aid. The final product analyzed:

Acid number.....	0.05
Saponification number.....	414
Hydroxyl content.....percent...	0.18
Hazen color.....	425

Other properties of the product were as follows:

Specific gravity, 25/25° C.....	0.9974.
Refractive index, 20° C.....	1.4530.
Flash point.....	250° C.
Flame point.....	290° C.
Pour point.....	— 62° C.
Evaporation loss, 400° F.....	4.2% in 6.5 hours.

Viscosity (cs.):

At 400° F.....	1.3.
At 210° F.....	5.2.
At 100° F.....	26.8.
At 0° F.....	750.0.

Example 2

The procedure of Example 1 was followed with the exception that pentaerythritol monoformal was omitted from the initial charge and instead to the final reaction product there was added 2.25% of an ester of pentaerythritol monoformal which had been prepared by esterifying both of its hydroxyl groups with an equimolar mixture of caprylic and capric acid. After this addition the final composition had the following properties:

Acid number.....	0.18
Saponification number.....	408
Hydroxyl content.....percent...	0.30
Hazen color.....	400
Specific gravity, 25/25° C.....	0.9974

Viscosity, pour point, flash point, etc. were essentially the same as the product of Example 1.

The low tendency of the compositions of the invention to form coke and other solids at high temperatures is shown vividly by a test that has been devised to make possible a numerical comparison of the coking tendencies of a fluid at high temperatures. In this test, the test fluid to which has been added 0.5% phenothiazine as stabilizer

is splashed at a controlled rate on an aluminum panel maintained at 700° F. for a period of 8 hours, at the end of which time the amount of coke deposited on the panel is determined by weighing. When subjected to this test the compositions of Examples 1 and 2 deposited 83.9 and 51.3 milligrams of coke, respectively. This is to be compared with the behavior of pentaerythritol esters of the same alkanolic acids employed in the examples but without the incorporation of a pentaerythritol monoformal ester, in which case the amount of coke deposited in the above test averages 164 milligrams. The practical significance of this difference is observable in the relative cleanliness of a gas turbine operated with the composition of the invention as a lubricant at a bulk oil temperature of about 350° F.

The pentaerythritol esters employed in the invention can be prepared by the complete esterification of pentaerythritol with an alkanolic acid or mixture of alkanolic acids as hereinbefore described. Specific esters useful in the invention are, for example, pentaerythritol hexahydroheptanoate, pentaerythritol hexahydrovalerate and pentaerythritol hexahydroacaproate. Mixed esters useful in the invention can be prepared from a mixture of alkanolic acids such as acetic, propionic, butyric, isobutyric, caproic, caprylic, 2-ethyl hexanoic, heptanoic and pelargonic, provided the mixture of acids has an average chain length as prescribed.

The esters of pentaerythritol monoformal employed in the invention are complete esters with an alkanolic acid containing from 2 to 18 carbon atoms including, for example, acetic acid, propionic acid, butyric acid, isobutyric acid, caproic acid, heptanoic acid, α,α -dimethyl butyric acid, lauric acid, pelargonic acid, stearic acid, etc. Either a single acid or a mixture of such acids can be used.

As seen from the examples the compositions of the invention can be prepared either by esterifying pentaerythritol and pentaerythritol monoformal together or by esterifying them separately and then mixing the two esters.

The relative proportions of pentaerythritol ester and pentaerythritol monoformal ester are fairly widely vari-

able although for practical purposes the composition should comprise from about 85% to 99% pentaerythritol ester with the balance pentaerythritol monoformal ester.

In addition to the specified ingredients, the compositions of the invention can include relatively small amounts of other ingredients that are added for specific purposes. For instance, the compositions can contain stabilizers such as phenothiazine, diphenylamine, dipyrilamine; auxiliary lubricants such as dioctyl sebacate, silicones and phosphates; corrosion inhibitors; lubricating aids; and the like.

What I claim and desire to protect by Letters Patent is:

1. A lubricant composition consisting essentially of

(1) a pentaerythritol tetraester of an acid selected from the group consisting of alkanolic acids having from 5 to 10 carbon atoms and mixtures of alkanolic acids having from 2 to 12 carbon atoms in proportions to provide an average chain length of from 5 to 10 carbon atoms, and (2) a minor amount of a diester of 5,5-dimethylol dioxane-1,3 with at least one alkanolic acid having from 2 to 18 carbon atoms, the pentaerythritol tetraester comprising from 85% to 99% by weight of the composition.

2. A lubricant composition consisting essentially of a pentaerythritol tetraester of mixed alkanolic acids having from 2 to 12 carbon atoms in proportions to provide an average chain length of about 6 carbon atoms, and a minor amount of a diester of 5,5-dimethylol dioxane-1,3 with a mixture of alkanolic acids having from 2 to 18 carbon atoms, the pentaerythritol tetraester comprising from 85% to 99% by weight of the composition.

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