

1

2

3,510,426

LUBRICANTS COMPRISING ALKYL PHOSPHITES AND SULFURIZED OLEFINS

Andrew G. Papay, Woodbury, N.J., assignor to Mobil Oil Corporation, a corporation of New York
 No Drawing. Continuation-in-part of application Ser. No. 593,276, Nov. 10, 1966. This application Dec. 10, 1968, Ser. No. 782,732
 Int. Cl. C10m 1/48, 1/38, 5/24
 U.S. Cl. 252-46.6

3 Claims

ABSTRACT OF THE DISCLOSURE

Lubricant compositions are provided which contain a small amount, sufficient to impart extreme pressure properties thereto, of a mixture comprising, in synergistic proportions, an alkyl phosphite and at least one sulfurized olefin selected from the group consisting of sulfurized butylenes and sulfurized dipentene.

CROSS REFERENCE TO RELATED APPLICATIONS

Continuation-in-part of application Ser. No. 593,276 filed Nov. 10, 1966, now abandoned.

BACKGROUND OF THE INVENTION

(1) Field of the invention

This invention, which is a continuation-in-part of my application Ser. No. 593,276 filed Nov. 10, 1966, relates to improved lubricant compositions and, in one of its aspects, relates more particularly to improved lubricant compositions in the form of liquid and solid hydrocarbon lubricants which exhibit effective extreme pressure properties under conditions of use. Still more particularly, in this aspect, the invention relates to improved lubricant compositions in the form of lubricating oils and greases which exhibit this extreme pressure property under both high-torque, low speed and also at high speed environmental conditions.

(2) Description of the prior art

Among the desired characteristics of lubricating oils, and greases containing such oils as lubricating vehicles, is the ability of the lubricant compositions to exhibit the aforementioned effective extreme pressure properties under high-torque low speed and also at high speed operations. In this respect, it is also highly desirable that additives employed for imparting such characteristics to the lubricant be thermally stable under conditions of use without causing sludge formation, and that they can be used in relatively small amounts and yet be effective for their intended purpose. Prior to the present invention various additives have been available for the purpose of imparting extreme pressure properties at high-torque low speed operating conditions, while other additives have also been effective in imparting similar properties at high speed operating conditions to specified lubricants. No additive, however, has been provided which will successfully function in imparting effective extreme pressure properties to the lubricant at both high-torque low speed and also at high speed operating conditions. Furthermore, even when combinations of additives have been attempted to be used for such purpose, it has been found that relatively large amounts of such additives, individually, are required to achieve any significant degree of improvement.

SUMMARY OF THE INVENTION

It has now been found that the aforementioned desired effective extreme pressure properties of lubricant compositions, either in the form of liquid or solid lubricants, can be achieved by incorporating therein small amounts, suf-

ficient to impart extreme pressure properties thereto, at both high-torque low speed and high speed conditions, of a mixture comprising, in synergistic proportions, an alkyl phosphite and at least one sulfurized olefin selected from the group consisting of sulfurized butylenes and sulfurized dipentene. In general, as more fully hereinafter described, the present invention, in its preferred applications, contemplates the aforementioned lubricant compositions, having the above-described improved extreme pressure properties, which contain a small amount of the synergistic mixture, usually from about 0.02 percent to about 35 percent, by weight, and preferably from about 0.1 percent to about 15 percent, by weight, of the total weight of such composition. Insofar as the synergistic additive mixture itself is concerned, the alkyl phosphite is usually employed in an amount from about 5 percent to about 60 percent, by weight, and, correspondingly, the sulfurized olefin is usually employed in an amount from about 95 percent to about 40 percent, by weight, of the total weight of said synergistic additive mixture.

A field of specific applicability is the improvement of liquid hydrocarbons employed as lubricants, including any of the conventional hydrocarbon oils of lubricating viscosities. These may include mineral or synthetic lubricating oils, aliphatic phosphates, esters and diesters, silicates, siloxanes, oxyalkyl ethers, or esters. Mineral lubricating oils employed as the lubricating composition may be of any suitable lubricating viscosity and may range from about 40 SSU to about 6,000 SSU at 100° F., and preferably from about 50 SSU to about 250 SSU at 210° F. These may have viscosity indexes from below 0 to about 100 or higher. Viscosity indexes from about 70 to about 95 are preferred. The average molecular weight of these oils may be, for example, from about 250 to about 800. Mineral oils having an SAE grade from about 5 to about 140, and preferably from about 10 to about 120, have been found to possess particular commercial utility as lubricants, in which the aforementioned additive mixture may be incorporated.

As previously indicated, the aforementioned synergistic additive mixtures may also be incorporated as extreme pressure agents in grease compositions. Such greases may comprise a combination of a wide variety of lubricating vehicles and thickening or gelling agents. Thus, greases in which the aforementioned additive mixtures are particularly effective may comprise any of the aforementioned conventional hydrocarbon oils of lubricating viscosity as the oil vehicle, and may include any of the aforementioned mineral or synthetic lubricating oils of the type indicated.

With respect to the formation of improved grease compositions in which the aforementioned additive mixtures are to be incorporated, the choice of employing a mineral or a synthetic oil of lubricating viscosity can best be determined from the nature of the intended environmental use for the grease. Thus, when high temperature stability is not a requirement of the finished grease, mineral oils having a viscosity of at least 40 SSU at 100° F., and particularly those falling within the range from about 60 SSU to about 6,000 SSU at 100° F. may be effectively employed. In instances where synthetic vehicles are employed rather than mineral oils, or in combination therewith as the lubricating vehicle, various compounds of this type may be successfully utilized. Typical synthetic vehicles include: polypropylene, polypropylene glycol, trimethylol propane esters, neopentyl and pentaerythritol esters, di(-ethyl hexyl) sebacate, di(-ethyl hexyl) adipate, di-butyl phthalate, fluorocarbons, silicate esters, silanes, esters of phosphorus-containing acids, liquid ureas, ferrocene derivatives, hydrogenated mineral oils, chain-type polyphenyls, siloxanes and silicones (poly-siloxanes), alkyl-substituted diphenyl eth-

ers exemplified by a butyl-substituted bis(p-phenoxy phenyl) ether, phenoxy phenyl ethers, etc.

The lubricating vehicles of the aforementioned improved greases of the present invention containing the above-described synergistic additive mixtures are combined with a grease-forming quantity of a thickening agent. For this purpose, a wide variety of materials may be employed. These thickening or gelling agents may include any of the conventional metal salts or soaps which are dispersed in the lubricating vehicle in grease-forming quantities in such degree as to impart to the resulting grease composition the desired consistency. Other thickening agents that may be employed in the grease formulation may comprise non-soap thickeners, such as surface-modified clays and silicas, aryl ureas, calcium complexes and various other materials. In general, grease thickeners may be employed which do not tend to melt and dissolve when used at the required temperature within a particular environment; however, in all other respects, any material which is normally employed for thickening or gelling hydrocarbon fluids for forming greases can be used in preparing the aforementioned improved greases in accordance with the present invention.

The alkyl phosphite component of the novel additive mixtures of the present invention, employed as extreme pressure agents, has the function of imparting effective extreme pressure properties at high-torque low speed operating conditions to the lubricant composition. Any alkyl phosphite can be employed for this purpose, and particularly preferred are alkyl phosphites in which trivalent phosphorus is present. From a standpoint of practical commercial utility, alkyl phosphites which can supply the active phosphorus required to pass the standard CRC L-37 high-torque low speed gear test of standard specification MIL-L-2105B are of most importance for this purpose. Representative examples of these alkyl phosphites which can pass the aforementioned CRC L-37 gear test, even when employed in relatively small concentrations, include dibutyl phosphite, ethylhexyl phosphite, oleyl phosphites, tributyl trithiophosphites, tris 2-chloro ethyl phosphite, and dodecyl phosphite.

The sulfurized olefin component of the above-described synergistic additive mixture has the function of imparting effective extreme pressure properties at high speed operating conditions to the lubricating composition. The sulfurized olefins employed for this purpose are those which can pass the standard CRC L-42 high speed and shock test of the aforementioned standard specification MIL-L-2105B, even when employed in relatively small amounts. These sulfurized olefins include sulfurized butylenes, e.g., sulfurized triisobutylene, or sulfurized polyisobutylene, or sulfurized dipentene and mixtures thereof.

As previously indicated, the improved extreme pressure synergistic additives of the present invention represent a balanced mixture of the alkyl phosphite and the sulfurized olefin, within the aforementioned limits set forth, by maintaining these limits in such manner that the alkyl phosphite is present in the aforementioned amount from about 5 percent to about 60 percent, by weight, and, correspondingly, the sulfurized olefin is present in an amount from about 95 percent to about 40 percent, by weight, of the total weight of the additive mixture. By maintaining this balanced relationship, it is possible to realize effective extreme pressure properties of the lubricant composition when employed under varying operating conditions which include variations extending from high-torque low speed to high speed operating conditions without causing sludge formation. Thus, in this respect, the requirements of both the aforementioned CRC L-37 high-torque low speed and CRC L-42 high speed and shock tests of the MIL-L-2105B specification, can be effectively met.

DESCRIPTION OF SPECIFIC EMBODIMENTS

The following examples will serve to illustrate the effectiveness of the novel extreme pressure-imparting additive mixtures of the present invention, their utility when incorporated in various types of lubricant compositions and the synergistic effect obtained with respect to the combination of the above-described alkyl phosphite and sulfurized olefin components of the additive mixture.

In each of the following examples, a SAE 90 base lubricating oil, containing varying combinations of the above-described extreme pressure synergistic additive mixtures, comprising alkyl phosphites and sulfurized olefins, was subjected to the aforementioned standard CRC L-37 and CRC L-42 tests in order to establish their qualification for meeting the requirements of standard specification MIL-L-2105B gear oil lubricants. In each instance, the oil was also subjected to a thermal stability test for a determination of sludge content. In this latter test, 25 ml. of the respective oil samples were heated for 72 hours at 300° F. in the presence of a catalytic material comprised of elemental copper, iron and aluminum, under a blanket of nitrogen for the purpose of avoiding oxidation. At the end of this time, the oil was analyzed for sludge content.

EXAMPLE 1

A sample of the above-described SAE 90 base lubricating oil containing 0.7 percent dibutyl phosphite and 6.0 percent sulfurized triisobutylene, by weight, was subjected to the aforementioned respective CRC L-37 and CRC L-42 tests. In each instance the oil passed both tests and when subjected to the aforementioned thermal stability test revealed a sludge content of only .5 percent, by weight.

EXAMPLE 2

A sample of the above-described SAE 90 base lubricating oil containing 0.6 percent dibutyl phosphite and 4.4 percent sulfurized polyisobutylene, by weight, was subjected to the aforementioned respective CRC L-37 and CRC L-42 tests. In each instance the oil passed both tests and when subjected to the aforementioned thermal stability test revealed a sludge content of only 1.4 percent, by weight.

EXAMPLE 3

A sample of the above-described SAE 90 base lubricating oil containing 0.8 percent dibutyl phosphite and 4.4 percent sulfurized polyisobutylene, by weight, was subjected to the aforementioned respective CRC L-37 and CRC L-42 tests. In each instance the oil passed both tests and when subjected to the aforementioned thermal stability test revealed a sludge content of only 1.2 percent, by weight.

EXAMPLE 4

A sample of the above-described SAE 90 base lubricating oil containing 0.8 percent tris 2-chloroethylphosphite, 1.6 percent tributylphosphorotrithioate and 1.6 percent sulfurized dipentene together with 3.2 percent chlorinated wax (40% Cl), by weight, was subjected to the aforementioned respective CRC L-37 and CRC L-42 tests. In each instance the oil passed both tests and when subjected to the aforementioned thermal stability test, revealed a sludge content of only .3 percent, by weight.

In order to illustrate the aforementioned synergistic effect of the above-described novel additive mixtures, the following comparative experimental data were obtained in which a base lubricating oil (SAE 90), untreated; the same base oil containing sulfurized triisobutylene in an amount sufficient to impart a sulfur content to the base oil of 2 percent, by weight; the same base oil containing dibutyl phosphite in an amount sufficient to impart a phosphorus content of 0.1 percent, by weight; and the same base oil containing both the aforementioned amounts of the sulfurized triisobutylene and the dibutyl phosphite, were in-

dividually subjected to the standard CRC L-37 high-torque low speed gear test of standard specification MIL-L-2105B and the standard CRC L-42 high speed and shock test of the aforementioned standard specification MIL-L-2105B, for determination of extreme pressure properties. The data and the results obtained from conducting these tests are shown in the following table.

TABLE

	High-torque, low-speed E.P. prop- erties (CRC L-37)	High speed and shock E.P. prop- erties (CRC L-42)
Base oil (SAE 90).....	Fail	Fail
Base oil (SAE 90) plus sulfurized triisobutylene to impart a sulfur content of 2% by weight.....	Fail	Pass
Base oil (SAE 90) plus dibutyl phosphite to impart a phosphorus content of 0.1% by weight.....	Fail	Fail
Base oil (SAE 90) plus sulfurized triisobutylene to impart a sulfur content of 2% by weight and dibutyl phosphite to impart a phosphorus content of 0.1% by weight.....	Pass	Pass

The aforementioned CRC L-37 and CRC L-42 standard tests are characterized as "Pass-Fail" tests, and it will be apparent from the comparative data of the above table that the untreated base oil sample failed each of the high-torque low speed extreme pressure properties and the high speed and shock extreme pressure properties tests. The addition of the sulfurized triisobutylene only serves to improve the high speed and shock extreme pressure properties of the lubricant, with a rating of "Pass." The addition of the dibutyl phosphite to the base oil produced no improvement in extreme pressure properties with respect to either test. It will be noted, however, that the addition of the same aforementioned amount of the sulfurized triisobutylene and the addition of the same amount of the dibutyl phosphite to the base oil resulted in the lubricant passing each of the aforementioned tests. Thus, it will be noted that a clear synergistic effect is realized by the use of a mixture of the sulfurized triisobutylene and the dibutyl phosphite.

As will be seen from the data of the foregoing examples, the improved lubricant compositions of the present invention, containing the aforementioned extreme pressure synergistic additive mixture, are outstandingly effective

in obtaining satisfactory performance as lubricants under both high-torque low speed and high speed operating conditions in meeting the requirements of standard tests. The same advantages are also realized when lubricating oils containing the above-described improved extreme pressure synergistic additive mixtures are formulated with desired thickening agents to produce improved solid lubricants as in the form of grease compositions. It will be understood, moreover, that the improved lubricant compositions of the present invention may, if so desired, contain various other additives or mixtures of such additional additives in order to further enhance their properties. Thus, the lubricant compositions of the present invention may contain such additives as antioxidants, anti-foamants, detergents, anti-rusts, dispersants, and the like. It will also be understood that although the present invention has been described with preferred embodiments, various modifications and adaptations thereof may be resorted to without departing from the spirit and scope of the invention, as those skilled in the art will readily understand.

I claim:

1. A lubricant composition containing a small amount, sufficient to impart extreme pressure properties thereto, of a synergistic mixture of sulfurized triisobutylene, present in an amount sufficient to impart a sulfur content of at least about 2% by weight, and dibutyl phosphite, present in an amount sufficient to impart a phosphorus content of at least about 0.1% by weight.

2. A lubricant composition as defined in claim 1 wherein said lubricant comprises a grease.

3. A lubricant composition as defined in claim 1 wherein said lubricant comprises a lubricating oil.

References Cited

UNITED STATES PATENTS

2,441,496 5/1948 Lincoln 252-46.6
2,817,653 12/1957 Cole et al. 252-45

DANIEL E. WYMAN, Primary Examiner

I. VAUGHN, Assistant Examiner

U.S. Cl. X.R.

252-49.8