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NON-CORROSIVE LUBRICANT COMPOSITION

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18 Claims. (Cl. 252—32.7)

This invention relates to improved lubricant compositions and more particularly is directed to lubricant compositions having improved detergency and corrosion inhibiting properties.

Within recent years it has become common practice to impart improved properties to lubricants through the use of various types of additives or addition agents. Lubricating oils employed in internal combustion engines such as automotive and diesel engines require the use of one or more addition agents to improve their serviceability under certain adverse operating conditions. Among the more important additives employed are the type which function to prevent the formation and accumulation of sludge and varnish-like coatings on pistons and cylinder walls of the engine. Such additives which have the property of maintaining clean engines are referred to as "detergent-type" addition agents.

Corrosion inhibitors also play an important part in the formulation of efficient lubricants. Such corrosion inhibitors should effectively inhibit the corrosion of metal alloys of the type used in engine bearings and other engine parts. In recent years the increased use of silver and similar metals in internal combustion engines has created new problems in the use of sulfur-containing additives in lubricants because of the tendency of such sulfur compounds to corrode silver, silver alloys and similar metals.

It is an object of the present invention to provide a lubricant composition which possesses detergency and corrosion inhibiting characteristics. It is another object of the invention to provide a lubricant composition which is non-corrosive. A further object of the invention is to provide a composition which will inhibit the corrosion of silver and similar metals by sulfur and/or organic sulfur-containing compounds. Still another object of the invention is to provide a method of inhibiting the corrosion of engine parts in contact with lubricant compositions which contain sulfur and/or organic sulfur-containing compounds which are normally corrosive to metals. Other objects and advantages of the present invention will become apparent from the following description thereof.

In accordance with the present invention, the foregoing objects can be attained by incorporating in lubricant compositions from about 0.1% to about 10%, by weight, of a salt of the reaction product of a chlorinated sulfurized terpene and an alkyl phenol. In the preparation of such reaction products the chlorinated sulfurized terpene and the alkyl phenol are employed in the molar ratio of from about 1:1 to about 1:4 and preferably from about 1:1 to about 1:2, respectively. The reaction product is readily prepared by heating a mixture of the reactants at a temperature of from about 40° F. to about 100° F., and preferably from about 40° F. to about 60° F. for a period of about 2 hours. The temperature of the reaction mixture is then increased from about 60° F. to about 250° F. for a period of from about 1 hour to about 4 hours or until HCl is no longer detected with blue litmus. The reaction mass is then cooled to a temperature of about 150° F. and, if desired, diluted with a solvent, for example, benzene, dioxane, and the like, and the diluted product neutralized with stoichiometric amounts of the basic reagent. The neutralized product is then heated to a temperature sufficiently high to remove water from the product and then filtered. The diluent is then distilled from the filtrate.

The sulfurized terpene to be chlorinated can be prepared by any of the well known methods, such as by heating mixture of terpene and sulfur to reaction temperatures or by adding the terpene to molten sulfur in accordance with the method fully described in Watson U. S. 2,445,983 issued July 27, 1948. Sulfurized monocyclic, bicyclic, or acyclic terpenes as well as polyterpenes can be used. Examples of species falling within these classes of terpenes are pine oil, turpentine, cyamene, alphapinene, betapinene, allo-ocemene, tenchene, bornylenes, menthadienes such as limonene, dipentene, terpenine, terpinolenes, etc., sesquiterpenes, diterpene, and polyterpenes; mixtures of such terpenes can also be sulfurized. We prefer to use sulfurized dipentene. If the sulfurized terpene contains active sulfur (i. e., products which darken a copper strip submerged in a ½% solution of the sulfurized terpene in a hydrocarbon oil maintained at about 210° F.) it can be freed of such active sulfur or deactivated by treatment with an alkali metal sulfide, such as sodium sulfide, or by treatment with a mixture of an alkali metal sulfide and a small amount of sodium hydroxide in a manner described in U. S. 2,445,983. The term "sulfurized terpene" as used herein and in the claims refers to the deactivated product.

The chlorinated sulfurized terpene is readily prepared by diluting the sulfurized terpene with a suitable hydrocarbon solvent, for example hexane, and reacting the diluted sulfurized terpene with chlorine at a temperature of from about 32° F. to about 45° F. Preferably two moles of chlorine is reacted with each mole of the sulfurized terpene.

The alkyl phenol can be a monoalkyl phenol or a dialkyl phenol in which the alkyl radical contains from 2 to about 20 carbon atoms. In the case of dialkyl phenols the alkyl substituents may be the same or they may be different alkyl radicals. Examples of such alkyl phenols are diethyl phenol, dibutyl phenol, diamyl phenol, octyl phenol, dioctyl phenol, octyl amyl phenol, nonyl phenol, dinonyl phenol, lauryl phenol, lauryl octyl phenol, dilauryl phenol, hexadecyl phenol, octadecyl phenol, etc.

Salts of the reaction product above-described are prepared by neutralizing the reaction product with theoretical amounts of a basic reagent, such as for example, a hydroxide, carbonate or oxide of an alkali metal or a polyvalent metal, such as an alkaline earth metal or a heavy metal. Examples of suitable basic reagents are potassium hydroxide, sodium hydroxide, lithium hydroxide, calcium oxide, lime, barium hydroxide, barium oxide, strontium oxide, etc. Other basic reagents can be used, such as for example, ammonia or an alkyl or aryl-substituted ammonia such as an amine, a guanidine, etc. Heavy metal salts of such reaction products are prepared by employing hydroxide or oxides of the desired heavy metal, such as for example zinc oxide, lead oxide, chromium oxide, etc., although we prefer to form the heavy metal salt by double decomposition of the alkali metal or alkaline earth salt with a salt of the desired heavy metal. Examples of suitable heavy metal salts are the salts of tin, titanium, aluminum, chromium, zinc, iron, copper, etc. The preparation of the hereindescribed salts is illustrated by the following examples:

EXAMPLE I

Chlorinated sulfurized dipentene was prepared by treating one mole of sulfurized dipentene, diluted in hexane, with chlorine at 32° F.–45° F. until about 2 moles of chlorine were taken up. The chlorinated product was then blown with nitrogen for about 2 hours. The chlorinated sulfurized dipentene was reacted with one mole of 2,4-diamyl phenol 45–100° F. while blowing with nitrogen, until HCl was no longer detected with blue litmus. After stripping off the hexane, the product was diluted with benzene, neutralized with one mole of KOH, and then freed of the benzene.

EXAMPLE II

Chlorinated sulfurized dipentene was prepared as in Example I. The chlorinated product was then reacted with 2 moles of nonyl phenol at 45–100° F. while blowing with nitrogen until HCl no longer was detected with blue litmus. The reaction product was then neutralized with one mole of $Ba(OH)_2 \cdot 8H_2O$, diluted with about 33% of an SAE 5W oil and filtered.

The above-described reaction products can be used in amounts of from about 0.1% to about 10% and preferably from about 0.25% to about 5% in combination with lubricant base oils, such as hydrocarbon oils, synthetic hydrocarbon oils, such as those obtained by the polymerization of hydrocarbons, such as olefin polymers; synthetic lubricating oils of the alkylene-oxide type, for example, the "Ucon oils," marketed by Carbide and Carbon Corporation, as well as other synthetic oils, such as the polycarboxylic acid ester-type oils, such as the esters of adipic acid, sebacic acid, maleic acid, azelaic acid, etc.

While the above-described reaction products can be suitably employed alone in combination with a base oil, they are usually used in combination with other lubricant addition agents which impart various desired characteristics to the base oil. Usually, these reaction products are used in conjunction with detergent-type additives, particularly those which contain sulfur or phosphorus and sulfur addition agents. This type is usually used in amounts of from about 0.002% to about 10%, and preferably from about 0.01% to about 5%. Among the phosphorus- and sulfur-containing addition agents are the neutralized reaction products with a phosphorus sulfide and a hydrocarbon, an alcohol, a ketone, an amine or an ester. Of the phosphorus sulfide reaction product additives, we prefer to employ the neutralized reaction products of a phosphorus sulfide, such as a phosphorus pentasulfide, and a hydrocarbon of the type described in U. S. 2,316,082, issued to C. M. Loane et al. April 6, 1943. As taught in this patent, the preferred hydrocarbon constituent of the reaction is a mono-olefin hydrocarbon polymer resulting from the polymerization of low molecular weight mono-olefin hydrocarbons, such as propylene, butenes, amylenes or copolymers thereof. Such polymers may be obtained by the polymerization of mono-olefins of less than 6 carbon atoms in the presence of a catalyst, such as sulfuric acid, phosphoric acid, boron fluoride, aluminum chloride, or other similar halide catalysts of the Friedel-Crafts type.

The polymers employed are preferably mono-olefin polymers or mixtures of mono-olefin polymers and iso-mono-olefin polymers having molecular weights ranging from about 150 to 50,000 or more, and preferably from about 500 to about 10,000. Such polymers can be obtained, for example, by the polymerization in the liquid phase of a hydrocarbon mixture containing mono- and isomono-olefins, such as butylene and isobutylene at a temperature of from about −80° F. to about 100° F. in the presence of a metal halide catalyst of the Friedel-Crafts type, such as for example, boron fluoride, aluminum chloride, and the like. In the preparation of these polymers, a hydrocarbon mixture containing isobutylene, butylenes and butanes recovered from petroleum gases, especially those gases produced in the cracking of petroleum oils in the manufacture of gasoline can be used.

Another suitable polymer is that obtained by polymerizing in the liquid phase a hydrocarbon mixture comprising substantially C_3 hydrocarbons in the presence of an aluminum chloride-complex catalyst. The catalyst is preferably prepared by heating aluminum chloride with iso-octane. The hydrocarbon mixture is introduced into the bottom of the reactor and passed upward through the catalyst layer while a temperature of from about 50° F. to about 110° F. is maintained in the reactor. The propane and other saturated gases pass through the catalyst while the propylene is polymerized under these conditions. The propylene polymer can be fractionated to any desired molecular weight, preferably from about 500 to about 1000 or higher.

Other suitable polymers are those obtained by polymerizing a hydrocarbon mixture containing about 10% to about 25% isobutylene at a temperature of from about 0° F. to about 100° F., and preferably 0° F. to about 32° F., in the presence of boron fluoride. After the polymerization of the isobutylene together with a relatively minor amount of the normal olefins present, the reaction mass is neutralized, washed free of acidic substances, and the unreacted hydrocarbons subsequently separated from the polymers by distillation. The polymer mixture so-obtained, depending upon the temperature of reaction, varies in consistency from a light liquid to a viscous oily material and contains polymers having molecular weights ranging from about 100 to about 2,000 or higher. The polymers so-obtained may be used as such or the polymer may be fractionated under reduced pressure into fractions of increasing molecular weight and suitable fractions reacted with the phosphorus sulfide to obtain the desired reaction products. The bottoms resulting from the fractionation of the polymer which have Saybolt Universal viscosities at 210° F. ranging from about 50 seconds to about 10,000 seconds are well suited for this purpose.

Essentially paraffinic hydrocarbons, such as bright stock residuums, lubricating oil distillates, petrolatums, or paraffin waxes, may be used. There can also be employed the condensation products of any of the foregoing hydrocarbons, usually through first halogenating the hydrocarbons and reacting with aromatic hydrocarbons in the presence of anhydrous inorganic halides, such as aluminum chloride, zinc chloride, boron fluoride, and the like.

Examples of other high molecular weight olefinic hydrocarbons which can be employed are cetene (C_{16}), cerotene (C_{26}), melene (C_{30}) and mixed high molecular weight alkenes obtained by cracking petroleum oils.

Other preferred olefins suitable for the preparation of the phosphorus sulfide reaction products are olefins having at least 20 carbon atoms in the molecule of which from about 13 carbon atoms to about 18 carbon atoms, and preferably at least 15 carbon atoms, are in a long chain. Such olefins can be obtained by the dehydrogenation of alkyl halides, preferably long chain alkyl halides, particularly halogenated paraffin waxes.

As a starting material there can be used the polymer or synthetic lubricating oil obtained by polymerizing unsaturated hydrocarbons resulting from the vapor phase cracking of paraffin waxes in the presence of aluminum chloride which is fully described in U. S. Patents 1,955,260; 1,970,402 and 2,091,398. Still another type of olefin polymer which may be employed is the polymer resulting from the treatment of vapor phase cracked gasoline and/or gasoline fractions with sulfuric acid or solid adsorbents, such as Fuller's earth, whereby unsaturated polymerized hydrocarbons are removed. The reaction products of the phosphorus sulfide and the polymers resulting from the voltolization of hydrocarbons as described, for example, in U. S. Patents 2,197,768 and 2,191,787 are also suitable.

Other hydrocarbons that can be reacted with a phosphorus sulfide are aromatic hydrocarbons, such as for

example, benzene, naphthalene, toluene, xylene, diphenyl and the like, or an alkylated aromatic hydrocarbon, such as for example, benzene having an alkyl substituent having at least 4 carbon atoms and preferably at least 8 carbon atoms, such as a long chain paraffin wax.

The phosphorus sulfide-hydrocarbon reaction product can be readily obtained by reacting a phosphorus sulfide, for example, P_2S_5 with the hydrocarbon at a temperature of from about 200° F. to about 500° F., and preferably from about 200° F. to about 400° F., using from about 1% to about 50%, and preferably from about 5% to about 25% of the phosphorus sulfide in the reaction. It is advantageous to maintain a non-oxidizing atmosphere, such as for example, an atmosphere of nitrogen above the reaction mixture. Usually, it is preferable to use an amount of the phosphorus sulfide that will completely react with the hydrocarbon so that no further purification becomes necessary; however, an excess amount of phosphorus sulfide can be used and separated from the product by filtration or by dilution with a hydrocarbon solvent, such as hexane, filtering and subsequently removing the solvent by suitable means, such as by distillation. If desired, the reaction product can be further treated with steam at an elevated temperature of from about 100° F. to about 600° F.

The phosphorus sulfide-hydrocarbon reaction product normally shows a titratable acidity which is neutralized by treatment with a basic reagent. The phosphorus sulfide-hydrocarbon reaction product, when neutralized with a basic reagent containing a metal constituent, is characterized by the presence or retention of the metal constituent of the basic reagent.

The neutralized phosphorus sulfide-hydrocarbon reaction product can be obtained by treating the acidic reaction product with a suitable basic compound, such as hydroxide, carbonate, oxide or sulfide of an alkaline earth metal or an alkali metal, such as for example, potassium hydroxide, sodium hydroxide, sodium sulfide, calcium oxide, lime, barium hydroxide, barium oxide, etc. Other basic reagents can be used, such as for example, ammonia, or an alkyl or aryl-substituted ammonia, such as amines. The neutralization of the phosphorus sulfide-hydrocarbon reaction product is carried out preferably in a non-oxidizing atmosphere by contacting the acidic reaction product either as such or dissolved in a suitable solvent, such as naphtha with a solution of the basic reagent. As an alternative method, the reaction product can be treated with solid alkaline compounds, such as KOH, NaOH, Na_2CO_3, CaO, BaO, $Ba(OH)_2$, Na_2S, and the like, at an elevated temperature of from about 100° F. to about 600 °F. Neutralized reaction products containing a heavy metal constituent, such as for example, tin, titanium, aluminum, chromium, cobalt, zinc, iron and the like, can be obtained by reacting a salt of the desired heavy metal with the phosphorus sulfide-hydrocarbon reaction product which has been treated with the phosphorus sulfide-hydrocarbon reaction product, which has been treated with a basic reagent, such as above-described.

Other phosphorus sulfide reaction products which can be used are the reaction products of a phosphorus sulfide and a fatty acid ester of the type described in U. S. 2,399,243; the phosphorus sulfide-degras reaction products of U. S. 2,413,332; the reaction product of an alkylated phenol with the condensation product of P_2S_5 and turpentine of U. S. 2,409,877 and U. S. 2,409,878; the reaction product of a phosphorus sulfide and stearonitrile of U. S. 2,416,807, etc.

The silver corrosion inhibiting property of the above-described products is demonstrated by the data in Table I which were obtained by subjecting mixture of hydrocarbon oil, a neutralized reaction product of P_2S_5 and a polybutene, and various herein-described thiadiazole reaction products to the following test, hereinafter referred to as the modified EMD test:

A silver strip 2 cm. x 5.5 cm. with a small hole at one end for suspension is lightly abraded with No. 0 steel wool, wiped free of any adhering steel wool, washed with carbon tetrachloride, air-dried and then weighed to 0.1 milligrams. 300 cc. of the oil to be tested is placed in a 500 cc. lipless glass beaker and the oil is heated to a temperature of 300° F. (±2° F.) and the silver test strip suspended in the oil so that the strip is completely immersed therein. The oil in the beaker is stirred by means of a glass stirrer operating at 300 R. P. M. At the end of 24 hours, the silver strip is removed and while still hot rinsed thoroughly with carbon tetrachloride and air-dried. The silver strip is immersed in a 10% potassium cyanide solution at room temperature until the silver surface assumes its original bright or silver appearance. The silver strip is then washed successively with distilled water and acetone, air-dried, weighed and loss in weight noted.

The following samples were subjected to the above test and the results obtained given in Table I:

Sample A.—Solvent extracted SAE–30 base oil+3.3% barium-containing neutralized reaction product of P_2S_5 and a polybutene of about 1000 molecular weight

Sample B.—A+0.7% sulfurized dipentene

Sample C.—A+3% product of Example I

Sample D.—A+3% product of Example II

Table I

Sample No.	Silver Corrosion (Wt. Loss/Mg.)
A	1-3
B	100
C	5
D	10

Since a weight loss of 20 milligrams is allowable, the ability of the compounds of this invention to inhibit silver corrosion is demonstrated by the above data. Although it appears that sample A without the product of this invention is less corrosive toward silver than with the additive (samples D, etc.), the additive of this invention is necessary to inhibit corrosion toward other metals as demonstrated by the following test and the data in Table II.

A copper-lead test specimen is lightly abraded with steel wool, washed with naphtha, dried and weighed to the nearest milligram. The cleaned copper-lead test specimen is suspended in a steel beaker, cleaned with a hot trisodium phosphate solution, rinsed with water, acetone and dried, and 250 grams of the oil to be tested together with 0.625 gram lead oxide and 50 grams of a 30–35 mesh sand charged to the beaker. The beaker is then placed in a bath or heating block and heated to a temperature of 300° F. (±2° F.) while the contents are stirred by means of a stirrer rotating at 750 R. P. M. The contents of the beaker are maintained at this temperature for 24 hours, after which the copper-lead test specimen is removed, rinsed with naphtha, dried and weighed. The test specimen is then replaced in the beaker and an additional 0.375 gram of lead oxide added to the test oil. At the end of an additional 24 hours of test operation the test specimen is again removed, rinsed and dried as before and weighed. The test specimen is again placed in the beaker together with an additional 0.250 gram of lead oxide and the test continued for another 24 hours (72 hours total). At the conclusion of this time, the test specimen is removed from the beaker, rinsed in naphtha, dried and weighed.

The loss in weight of the test specimen is recorded after each weighing.

This test, known as the Stirring Sand Corrosion Test, is referred to hereinafter as "S. S. C. T."

The following oil samples were used in the above test:

Sample A'.—Solvent-extracted SAE–30 oil.
Sample B'.—A+3% of product of Example I.
Sample C'.—A+1.75% of product of Example II.
Sample D'.—A—3.3% barium-containing neutralized reaction product of P_2S_5 and a polybutene of about 1000 molecular weight.
Sample E'.—D+3% of product of Example I.
Sample F'.—D+2.25% of product of Example II.

Table II

Sample	S. S. O. T. (Mg. Weight Loss)	
	48 Hrs.	72 Hrs.
A'	734	1301
B'	65	91
C'	75	204
D'	698	1360
E'	150	160
F'	62	81

Since weight losses of 200 milligrams in 48 hours and 500 milligrams in 72 hours are allowable, the copper-lead corrosive inhibiting property of the herein-described compounds of this invention is clearly demonstrated by the above data.

The effectiveness of the herein-described salts of the present invention in inhibiting the oxidation of hydrocarbon oils is demonstrated by the data in Table III. These data were obtained by subjecting a hydrocarbon oil with and without the product of Example II to the oxidation test known as the Indiana Stirring Oxidation Test (I. S. O. T.). In this test 250 cc. of the oil to be tested are heated at 330–332° F. in a 500 cc. glass beaker in the presence of 5 square millimeters of copper and 10 square millimeters of iron. Four glass rods of 6 millimeters diameter are suspended in the oil which is stirred at about 1300 R. P. M. by means of a glass stirrer. At intervals of 24, 48 and 72 hours oil samples are withdrawn and sludge, acidity and varnish values determined. Varnish values or ratings are based upon a visual inspection of the glass rods, in which a rod free of any varnish deposit is given a rating of 10 while a badly coated rod is given a rating of 1. Rods having appearances between these extremes are given intermediate values. The following oil samples were subjected to this test:

Sample A''.—SAE–30 base oil.
Sample B''.—A''+2.25% reaction product of Example II.

Table III

Sample	Acidity (Mg. KOH/g. oil)			Naphtha Insoluble [1]			Varnish Rating		
	24 Hrs.	48 Hrs.	72 Hrs.	24 Hrs.	48 Hrs.	72 Hrs.	24 Hrs.	48 Hrs.	72 Hrs.
A''	11.2	14.0	14.0	2.2	7.1	7.6	5	4	4
B''	2.24	3.36	4.76	0.429	1.1	2.1	10	10	10

[1] Milligrams per 10 grams oil.

In addition to the aforementioned detergent-type additives and corrosion inhibitors, compositions containing the compounds of the present invention can contain other additives, such as anti-oxidants, pour point depressors, extreme pressure agents, anti-wear agents, V. I. improvers, etc.

While this invention has been described in connection with the use of the herein-described additives and lubricant compositions, their use is not limited thereto; but the same can be used in products other than lubricating oils, such as for example, fuel oils, insulating oils, greases, non-drying animal and vegetable oils, waxes, asphalts, and any fuels for internal combustion engines, particularly where sulfur corrosion must be combatted.

Concentrates of a suitable oil base containing more than 10%, for example up to 50% or more, of the products of this invention alone or in combination with more than 10% of the detergent-type additive and/or other additives, can be used for blending with hydrocarbon oils or other oils in the proportions desired for the particular conditions of use to give a finished product containing from 0.02% to about 10% of the reaction product of this invention.

Percentages given herein and in the appended claims are weight percentages unless otherwise stated.

Although the present invention has been described with reference to specific preferred embodiments thereof, the invention is not to be considered as limited thereto but includes within its scope such modifications and variations as come within the spirit of the appended claims.

We claim:

1. A lubricant composition comprising a major proportion of an oleaginous compound and from about 0.1% to about 10% of an oil-soluble salt of the reaction product of a chlorinated sulfurized terpene and an alkyl phenol selected from the group consisting of a monoalkyl phenol and a dialkyl phenol having 2 to about 20 carbon atoms in each alkyl radical, said reactants being reacted in the molar ratio of from 1:1 to about 1:4, respectively, at a temperature of from about 40° F. to about 100° F.

2. A composition as described in claim 1 in which the chlorinated sulfurized terpene is a chlorinated sulfurized monocyclic terpene.

3. A composition as described in claim 1 in which the chlorinated terpene is chlorinated sulfurized dipentene.

4. A composition as described in claim 1 in which the alkyl phenol is diamyl phenol.

5. A composition as described in claim 1 in which the alkyl phenol is dinonyl phenol.

6. A composition as described in claim 1 in which the alkyl phenol is dilauryl phenol.

7. A composition as described in claim 1 in which the salt is an alkali metal salt.

8. A composition as described in claim 1 in which the salt is an alkaline earth salt.

9. A composition as described in claim 7 in which the alkali metal is potassium.

10. A composition as described in claim 7 in which alkali metal is sodium.

11. A composition as described in claim 8 in which the alkaline earth is barium.

12. A composition as described in claim 8 in which the alkaline earth is calcium.

13. A lubricant composition comprising a major proportion of a hydrocarbon oil, from about 0.001% to about 10% of a phosphorus- and sulfur-containing detergent-type lubricant additive, and from about 0.1% to about 10% of an oil-soluble salt of the reaction product of a chlorinated sulfurized terpene and an alkyl phenol selected from the group consisting of a monoalkyl phenol and a dialkyl phenol having 2 to about 20 carbon atoms in each alkyl radical, said reactants being reacted in the molar ratio of from 1:1 to about 1:4, respectively, at a temperature of from about 40° F. to about 100° F.

14. A composition as described in claim 13 in which the chlorinated sulfurized terpene is chlorinated sulfurized dipentene.

15. A lubricant composition as described in claim 13 in which the phosphorus- and sulfur-containing detergent-type lubricant additive is an alkali metal-containing neutralized reaction product of a phosphorus sulfide and an olefin polymer.

16. A lubricant composition as described in claim 13 in which the phosphorus and sulfur-containing detergent-type lubricant additive is an alkaline-earth containing

neutralized reaction product of a phosphorus sulfide and an olefin polymer.

17. A lubricant composition described in claim 13 in which the phosphorus- and sulfur-containing detergent-type lubricant additive is a barium-containing neutralized reaction product of a phosphorus sulfide and an olefin polymer.

18. An addition agent concentrate for lubricating oils, comprising essentially a hydrocarbon oil containing more than 10% of an oil-soluble salt of the reaction product of a chlorinated sulfurized terpene and an alkyl phenol selected from the group consisting of a monoalkyl phenol and a dialkyl phenol having 2 to about 20 carbon atoms

in each alkyl radical, said reactants being employed in the molar ratio of from about 1:1 to about 1:4, respectively, at a temperature of from about 40° F. to about 100° F., said concentrate being capable of dilution with a lubricating oil to form a homogeneous mixture containing from about 0.1% to about 10% of said reaction product.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 2,799,655 July 16, 1957

Albert R. Sabol et al.

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

In the grant, lines 1 and 2, name of co-inventor, for "Robert E. Krall" read -- Robert E. Karll --; column 6, line 24, for "0.7%" read -- 0.75% --.

Signed and sealed this 24th day of September 1957.

(SEAL)

Attest:

KARL H. AXLINE
Attesting Officer

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Commissioner of Patents