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LUBRICATING OIL COMPOSITION

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7 Claims. (Cl. 252—48)

This invention relates to improved lubricating oils and to a composition of matter particularly useful in improving the characteristics of lubricating oils when dissolved therein. More particularly, the invention relates to the improvement of lubricating oils by the addition thereto of di-(acylphenyl)-dithiophosphates.

It has become generally recognized that conventionally refined lubricating oils of either paraffinic or naphthenic base stocks are unable to fully meet the drastic requirements of heavy duty service such as required in internal combustion engines for trucks, busses, aeroplanes, tanks, marine engines, etc., which operate for long periods of time at high temperatures.

It has been found that under such extreme service conditions most conventional oils tend to decompose with undesirable results. For example, certain acidic products are formed in the oil through oxidation or decomposition which tend to attack and corrode alloy bearings. Other decomposition products tend to polymerize in the oil and form sludge which may interfere with the circulation of the oil through the lubricating system and may accumulate on the heated metal surfaces forming lacquer-like deposits. These deposits mixed with carbon from the incomplete combustion of the oil tend to collect in the piston ring slots affecting their free movement and causing either excessive consumption of oil or scoring of the cylinder walls of the engine.

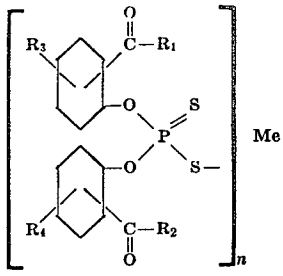
The addition of various chemical substances to lubricating oils to improve their resistance to oxidation and decomposition, to act as corrosion inhibitors, sludge dispersants, detergents, etc., has been proposed. While most of these compositions perform their intended functions very well, very few of them correct all of the difficulties encountered in heavy duty service. Some of the compounds which are added as anti-oxidants and corrosion inhibitors have very weak or no detergent properties and while they prevent corrosion they do not prevent sludge formation and varnish deposits on the engine parts. Certain other compounds which are added as detergents and dispersants disperse the sludge and prevent lacquer deposits and sticking of piston rings. Unfortunately, however, most of these latter substances increase the rate of oxidation of the oil and their presence therein results in an increase in the formation of acidic oxidation products and an increased rate of corrosion of alloy bearings and other metal parts. Adding both detergents and anti-corrosion agents as separate compounds does not usually give satisfactory results, since the two different types of compounds seem to offset the beneficial effects of each other.

The principal objects of the present invention are, therefore, to provide a composition of matter which can be added to lubricating oils whereby the oil is made more resistant to oxidation and sludge formation, non-corrosive to alloy bearings and other metal parts and which also possesses oiliness, detergent, dispersing, and sludge dissolving properties whereby the tendency of the oil to form varnish deposits and the tendency of the piston rings to stick under the conditions of heavy duty service is minimized. These objects, and others which will appear hereinafter, are attained by us by the addition to hydrocarbon lubricating oils of certain oil-soluble di-(acylphenyl)-dithiophosphates such as will be described hereinafter. Since these di-(acylphenyl)-dithiophosphates are new organic compounds their preparation will also be described in greater detail herein.

Although the addition of certain types of dithiophosphates to lubricating oils to improve their resistance to oxidation and to reduce their tendency to corrode alloy bearings and form sludge has been suggested most of the dithiophosphates heretofore employed have not had all of the oiliness, sludge dissolving, detergent, and oil solubility characteristics desired. Mixing 2,4-dialkylphenol sulfides, such as disclosed in our Patent No. 2,249,626, issued July 15, 1941, with the dithiophosphates has been resorted to to improve the detergency of the composition and the oil solubility of the dithiophosphates. While such mixtures are extremely effective as all-purpose lubricating oil additives, it is desirable that the composition be further improved and that the additive be a single chemical compound able to impart to the oil all of the desirable characteristics heretofore mentioned. The di-(acylphenyl)-dithiophosphates described herein possess all the desirable characteristics of an all-purpose additive and when dissolved in lubricating oils improve the oil in all important respects.

The di-(acylphenyl)-dithiophosphates which

may be employed by us to improve the operating characteristics of lubricating oils may be represented by the general formula



in which R_1 and R_2 are alkyl, cycloalkyl or aryl radicals, R_3 and R_4 are hydrogen or alkyl, cycloalkyl, or aryl radicals, Me is a metal, preferably an alkaline earth metal, and n is the valence of the metal.

The improved sludge dissolving, detergent, and oiliness characteristics imparted to the oil by the di-(acylphenyl)-dithiophosphates of the present invention are due in a large measure to the keto groups of the compounds. The

radical, particularly when in association with a long chain alkyl radical having 5 to 18 carbon atoms, improves considerably both the detergent powers of the resulting compound and its oiliness properties. Keto groups, particularly in association with phenyl groups and in mixed ketones such as aryl-alkyl ketones, are very effective in dissolving lubricating oil sludges. In our preferred alkaline earth metal salts as shown in the specific examples, it will be seen that there are present in the compounds four keto groups per molecule. The detergency and oiliness of the oil in which these compounds are dissolved is accordingly very greatly increased.

Other portions of the molecule of the di-(acylphenyl)-dithiophosphate have several important functions in the improvements of the lubricating oil. Alkyl groups of 5 or more carbon atoms and cycloalkyl groups increase considerably the oil solubility of the compounds and make it easy to incorporate them into lubricating oils. The anti-oxidation and anti-corrosion properties of the new compounds are due in large measure to the presence of sulfur and phosphorus atoms, particularly in the relationship in which they exist in our compounds. Also because of their sulfur and phosphorus content these compounds possess strong surface active properties making the oil adhere closely to metal surfaces and giving them properties useful in slushing oils and in extreme pressure lubricants, hypoid gear greases, and the like. Being of high molecular weight and complex structure the compounds are water-insoluble and have pour point depressant properties. They are also heat stable and stand up under the high temperatures encountered in crankcases of engines performing heavy duty service.

The di-(acylphenyl)-dithiophosphoric acids of the present invention are prepared by reacting approximately 4 mols of an acylated phenol with one mol of P_2S_5 as described hereinafter. The acylated phenols which may be employed in the reaction to prepare compounds useful in the improvement of lubricating oils are those obtained by acylating phenol or an alkyl, cycloalkyl or aryl substituted phenol with alkyl, cycloalky or aryl acyl halides. Suitable acylating agents for this latter reaction include acyl halides such as butyryl chloride, caproyl chloride, lauroyl chloride, myristoyl chloride, stearoyl chloride, naphthenoyl chloride, benzoyl chloride, amyl benzoyl chloride, octyl benzoyl chloride, phenylstearoyl chloride, and others. Particularly useful as acylating agents are those mixed acyl halides derived from mixed fatty acids obtained upon hydrolysis of animal, fish and vegetable oils. These latter products contain varying proportions of different long chain fatty acid halides in admixture. The naphthenoyl chloride mentioned above is composed of a mixture of various cycloaliphatic acyl halides such as are prepared from naphthenic acid, a product of the petroleum industry.

The alkyl, cycloalkyl and aryl substituted phenols which may be acylated and then reacted with P_2S_5 are easily obtained products of commerce such as cresol, isobutyl phenol, tertiary amyl phenol, ethylhexyl phenol, n-octyl phenol, dodecyl phenol, tetradecyl phenol, octadecyl phenol, cyclopentyl phenol, cyclohexyl phenol, p-hydroxy-biphenyl and the like. Although the preparation of acylated phenols has been previously described some of the acylated phenols employed by us are new compounds and accordingly, preparation of some of the typical ones is in the specific examples which follow. In general, however, the acyl phenols are prepared by treating phenol or an alkyl, cycloalkyl or aryl substituted phenol with an acyl halide of the type disclosed above in the presence of anhydrous aluminum chloride followed by decomposition of the resulting aluminum complex with dilute hydrochloric acid and washing.

As will be seen from the above, R_1 and R_2 in the general formula are aliphatic, aromatic or cycloaliphatic radicles such as methyl, ethyl, butyl, isobutyl, amyl, heptyl, nonyl, undecyl, tridecyl, pentadecyl, heptadecyl, cyclopentyl, cyclohexyl, methylcyclohexyl, dimethylcyclohexyl, phenyl, and the like. R_1, R_2, R_3 and R_4 may be the same or different radicals.

Since it is desirable that the di-(acylphenyl)-dithiophosphates be easily soluble in hydrocarbon oils it is preferred that R_3 and R_4 in the general formula above should be straight or branched chain, primary, secondary or tertiary alkyl radicals such as isobutyl, amyl, tertiary amyl, ethylhexyl, n-octyl, decyl, dodecyl, tetradecyl, octadecyl and the like, or cycloalkyl radicals such as cyclophentyl, cyclohexyl, methylcyclohexyl, and other alkyl substituted cyclohexyl radicals, such as the ethyl, propyl, butyl, and amyl mono-, di- and tri-substituted cyclohexyl radicals. Radicals of these types promote oil solubility. It should be understood, however, that our invention is not to be limited to the use of these particular compounds since compounds in which R_3 and R_4 are hydrogen or aryl radicals may be easily dissolved in lubricating oils particularly when R_1 and R_2 are alkyl or cycloalkyl radicals having 5 or more carbon atoms. In general, we may prepare an improved lubricating oil by dissolving therein any di-(acylphenyl)-dithiophosphate of the class described. Particularly advantageous because of their increased oil solubility are those having at least

one straight or branched chain alkyl or cycloalkyl radical having five or more carbon atoms.

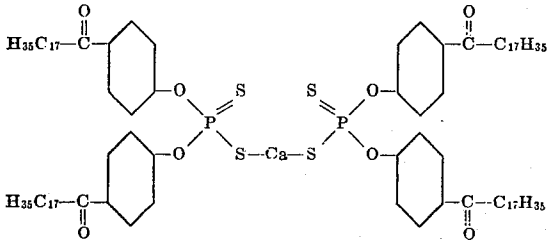
The compounds described may be prepared by mixing and heating approximately 4 mols of an acylated phenol or substituted acylated phenol as named above with one mol of P_2S_5 at temperatures between 80–140° C. until most of the P_2S_5 has dissolved and the evolution of H_2S has subsided. This usually requires 2 to 4 hours at these temperatures. If desired, an inert solvent such as toluene may be added to the reaction mixture, either before or after the reaction, to facilitate handling. The crude product may be decanted or filtered from unreacted P_2S_5.

Metal salts of the di-(acylphenyl)-dithiophosphoric acids may be prepared from the acids by simple neutralization of the above reaction product with a suitable salt-forming base or by double decomposition. A wide variety of salt-forming radicals including those of Ni, Al, Pb, Hg, Cd, Sn, Zn, Mg, Na, K, Ca, Sr, Ba and others may be introduced by neutralization of the acid with a corresponding oxide, hydroxide or carbonate or in some cases sulfide. Some of these salts may be more easily prepared by double decomposition of the sodium salt of the di-(acylphenyl)-dithiophosphoric acid with a desired metal salt, as for example, zinc chloride or the like.

Preparation of the di-(acylphenyl)-dithiophosphates will now be described in greater detail by means of the following examples in which the preparation and effectiveness of some of the representative ones are illustrated. All parts are by weight unless otherwise stated. It will be understood that our invention is not limited to the use of these particular compounds since the examples are given primarily for purposes of illustration and our invention is to be construed as broadly as the appended claims permit.

Example 1

Preparation of di-(stearoylphenyl)-dithiophosphoric acid and its calcium salts

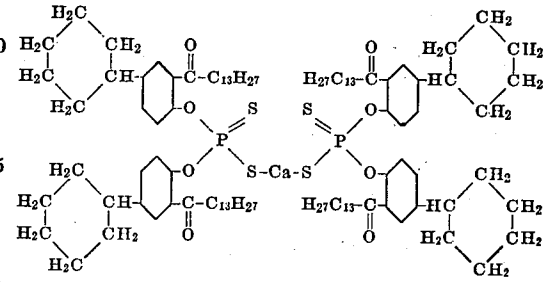


A mixture of 230 parts by weight of stearoyl chloride and 71 parts by weight of dry phenol was stirred at 95° C. for one-half hour, cooled and diluted with 100 parts of octane. When 33.6 parts by weight of anhydrous aluminum chloride had been added, the solution was heated with steam for two hours. After it had cooled, the product was stirred with warm dilute hydrochloric acid again, hot water three times, dilute sodium carbonate, and finally with sodium sulfate solution. Before it was washed with sodium carbonate, the solution was mixed with butanol to lessen emulsification. The stearoylphenol was isolated by distilling the solvents from it in vacuo. The product, a yellow colored liquid, solidified on standing and cooling.

A mixture of 248 parts by weight of stearoylphenol and 42 parts by weight of P_2S_5 was stirred for three hours at a temperature of 85–95° C. and the resulting di-(stearoylphenyl)-dithiophosphoric and decanted from the excess P_2S_5. The acid was diluted with 43 parts of 95% ethanol, 43 parts of absolute ethanol, and 172 parts of toluene and then 15 parts of calcium hydroxide was added slowly, followed by 3 parts of calcium hydroxide and 172 parts more of toluene. The solution of calcium di-(stearoylphenyl)-dithiophosphate was treated with activated carbon, filtered and the solvents removed by vacuum distillation. The product was a viscous reddish-brown liquid that became semi-solid on standing. It was readily soluble in lubricating oil.

Example 2

Preparation of di-(o-myristoyl-p-cyclohexylphenyl)-dithiophosphoric acid and its calcium salt



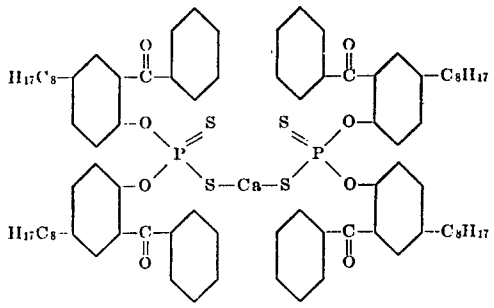
A mixture of 114 parts by weight of myristoyl chloride and 81 parts by weight of p-cyclohexylphenol was stirred on a steam bath for one-half hour, cooled and diluted with 50 parts of octane. When 21 parts of anhydrous aluminum chloride had been added, the mixture was stirred on a steam bath for two hours and after it had been cooled and diluted with 250 parts of toluene, the aluminum complexes were decomposed with warm dilute hydrochloric acid. The upper layer was washed successively with hot dilute hydrochloric acid, hot water twice, a warm solution of sodium carbonate with butanol to prevent emulsification, and finally with warm water.

The toluene, octane, butanol, and water were removed by vacuum distillation leaving 164 parts by weight of o-myristoyl-p-cyclohexylphenol, a yellowish-brown liquid.

A mixture of 35 parts by weight of o-myristoyl-p-cyclohexylphenol and 5.6 parts by weight of P_2S_5 was stirred at 95–110° C. for three hours and the resulting di-(o-myristoyl-p-cyclohexylphenyl)-dithiophosphoric acid was decanted from a slight residue of P_2S_5. The product was cooled, diluted with 26 parts of toluene, 6.5 parts of 95% ethanol, and 6.5 parts of absolute ethanol, and then reacted with 1.9 parts by weight of calcium hydroxide. The solution was treated with activated carbon, filtered, mixed with 40 parts of SAE 10 oil and vacuum distilled to remove the solvents from the calcium di-(o-myristoyl-p-cyclohexylphenyl)-dithiophosphate. The product, a brownish-yellow liquid was easily soluble in lubricating oil.

EXAMPLE 3

Preparation of di-(o-benzoyl-p-octylphenyl)-dithiophosphoric acid and its calcium salt

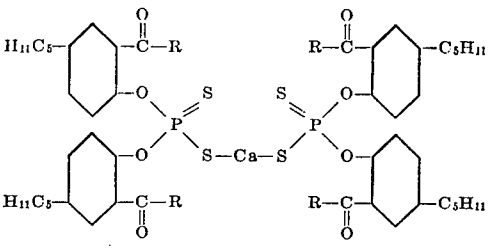


With 100 parts by weight of p-octylphenol was mixed 69 parts of benzoyl chloride and the solution was stirred at 95° C. for a half hour. When the liquid had cooled, 50 parts of octane and 21.7 parts of anhydrous aluminum chloride were slowly introduced. The mixture was stirred for two hours on a steam bath and then, after the products were at room temperature, 250 parts of toluene and an excess of warm dilute hydrochloric acid were added. The upper layer was washed again with warm dilute hydrochloric and then twice with hot water, once with warm aqueous sodium carbonate, and finally with warm water again. The solvents and water were removed by vacuum distillation, leaving 193 parts by weight of o-benzoyl-p-octylphenol, a viscous yellowish-brown liquid.

A mixture of 38 parts by weight of o-benzoyl-p-octylphenol and 7.4 parts of P_2S_5 was stirred at 95–110° C. for 2.5 hours, and then decanted from excess P_2S_5. The di-(o-benzoyl-p-octylphenyl)-dithiophosphoric acid was diluted with 28 parts of toluene, 7 parts of absolute ethanol, and 7 parts of 95% ethanol and then reacted slowly with 2.4 parts by weight of calcium hydroxide. Finally 28 parts of toluene and 0.5 part of calcium hydroxide more were added, the mixture treated with activated carbon, and filtered. After adding 40 parts of SAE #10 oil the solvents were removed by vacuum distillation leaving the calcium di-(o-benzoyl-p-octylphenyl)-dithiophosphate as a 50% oil solution.

EXAMPLE 4

Preparation of di-(o-naphthenoyl-p-amylphenyl)-dithiophosphoric acid and its calcium salt



Where R is a naphthenyl radical.

64 parts p-tertiary amyl phenol was dissolved in 40 parts warm octane and 100 parts naphthenoyl chloride added gradually with stirring. The solution was heated on the steam bath for 30 minutes, then cooled down to room temperature and 17 parts anhydrous aluminum chloride added with stirring. The mixture was gradually warmed up on the steam bath and heating continued for 2 hours. After cooling, the mixture was diluted with 150 cc. toluene and extracted with warm dilute hydrochloric acid three times, followed by warm dilute sodium carbonate solution and finally with water. The solvents were evaporated under reduced pressure leaving o-naphthenoyl-p-tertiaryamyl phenol as a fluorescent yellowish-green liquid.

69 parts by weight of o-naphthenoyl-p-amyl phenol was reacted with 13.2 parts by weight of P_2S_5 at 105–110° C. for three hours. The resulting di-(o-naphthenoyl-p-amylphenyl)-dithiophosphoric acid was decanted from the slight amount of P_2S_5 residue and, after dilution with 52 parts of toluene, 13 parts of 95% ethanol, and 13 parts absolute ethanol, it was reacted with 4.4 parts of calcium hydroxide. Finally, 0.8 part more of calcium hydroxide was added with 52 parts of Solvesso and the mixture was warmed to 60° C. for a half hour. Activated carbon was stirred in, the liquid filtered, and the solvents removed by vacuum distillation, leaving the calcium di-(o-naphthenoyl-p-amylphenyl)-dithiophosphate. The product was yellowish-brown and was easily soluble in oil.

The products prepared as thus described may be added directly to lubricating oils in which they act as detergents, sludge dispersants, corrosion inhibitors, etc. Ordinarily only from 0.1 to 3.0% of the di-(acylphenyl)-dithiophosphates is sufficient in a hydrocarbon oil to impart to the oil the improved characteristics desired. Although the compounds described are for the most part easily soluble in lubricating oils and may be easily blended therewith it is ordinarily more convenient to add a quantity of lubricating oil of suitable grade to the dithiophosphate reaction mixture as the solvent is being removed. This is shown in the specific examples. By following this procedure it is easily possible to obtain the di-(acylphenyl)-dithiophosphates dissolved in lubricating oil to the extent of about 50% or more. Solutions of the dithiophosphates of this strength in lubricating oils are particularly easy to blend with ordinary lubricating oil and are especially convenient for storing and shipping purposes.

The di-(acylphenyl)-dithiophosphate may constitute the sole additive in our lubricating oil or it may be used in conjunction with other materials added for special purposes such as alkylphenyl sulfides, particularly those of the type shown in our previously mentioned patent.

The effectiveness of some of the di-(acylphenyl)-dithiophosphates in the lubricating oils may be demonstrated by the results obtained upon testing a lubricating oil containing small amounts of the dithiophosphates by the standard Underwood oxidation test. In this test 1500 cc. of a hydrocarbon lubricating oil was heated for 5 hours at 325° F. while continuously spraying portions of the oil against the 2'' x 10'' freshly sanded copper strip and 2 freshly sanded alloy bearing while permitting free circulation of air during the operation. In these tests 0.5% by weight of the di-(acylphenyl)-dithiophosphate was added to the oil. As a corrosion catalyst iron naphthenate in amounts of 0.116% equivalent to 0.01% of Fe_2O_3 was also added. At the conclusion of the test the loss in weight of the bearings due to corrosion was determined. The neutralization number showing the formation of

acidic oxidation products during the test was also determined. For convenience the values obtained under the standard Conradson carbon test are also included in the table. Results of the test were as follows:

Table I—Standard Underwood oxidation test

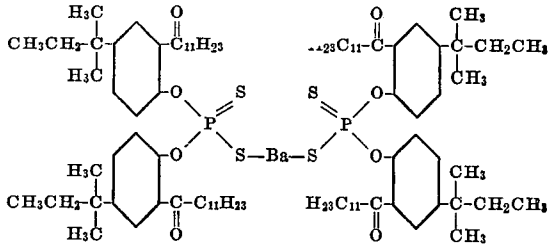
[Conventional refined, Midcontinent SAE 30 grade oil]

Composition	Bearing loss, mg.		Neutralization number	Conradson carbon
	Cu-Pb	Ag-Cd		
				Per cent
Oil, control	27	818	3.09	1.85
Oil+0.5% calcium di-(stearoyl-phenyl)-dithiophosphate	1	1	1.92	1.42
Oil+0.5% calcium di-(o-naphthenoyl-p-tertiaryamylphenyl)-dithiophosphate	0	0	0.88	1.13
Oil+0.5% calcium di-(o-myristoyl p-cyclohexylphenyl)-dithiophosphate	0	0	1.03	1.14

These results show the remarkable effectiveness of the di-(acylphenyl)-dithiophosphates of the present invention in reducing bearing corrosion and the formation of acidic oxidation products in the oil. The Conradson carbon values also show that the di-(acylphenyl)-dithiophosphates tend to reduce decomposition of the oil when heated to extremely high temperatures.

EXAMPLE 5

Preparation of di-(o-lauroyl-p-tertiaryamylphenyl)-dithiophosphoric acid and its barium salt



164 parts by weight of p-tertiaryamylphenol was warmed with 219 parts lauroyl chloride on the steam bath for 30 minutes. After cooling, 80 parts octane was added, followed by 45 parts anhydrous aluminum chloride. The mixture was stirred and slowly warmed up on the steam bath. Heating was continued for 2 hours. 200 parts of toluene was added and after cooling down was extracted and washed three times with dilute hydrochloric acid, followed with dilute sodium carbonate solution and finally with water. A quantity of butanol, about 50 parts by weight was added to prevent emulsification during the washing. After washing, the solvents were removed by evaporation under reduced pressure. o-Lauroyl-p-tertiaryamylphenol remained as a brownish-yellow liquid.

346 parts o-lauroyl-p-tertiaryamylphenol was stirred and warmed with 61 parts finely ground phosphorus pentasulfide at 125–135° C. for 2.5 hours. At the end of this time practically all of the P_2S_5 had disappeared and the evolution of H_2S had practically ceased. The crude di-(o-lauroyl-p-tertiaryamylphenyl)-dithiophosphoric acid was decanted from a small amount of unreacted P_2S_5. It was a brownish-yellow liquid.

78 parts di-(o-lauroyl-p-tertiaryamylphenyl)-dithiophosphoric acid was dissolved in a mixture of 40 parts toluene and 20 parts 97.5% alcohol. With stirring and cooling below 40° C. 10 parts finely ground barium oxide was added. As soon as neutral, which required about 10 minutes 40 parts toluene was added and the solution filtered from traces of inorganic barium salts. The solvent was removed by evaporation in vacuo. The barium di-(o-lauroyl-p-tertiaryamylphenyl)-dithiophosphate was a yellowish-brown, thick, viscous liquid.

This latter product was also evaluated by the Underwood Oxidation Test as previously described with the following results using a different type of oil.

Table II—Standard Underwood oxidation test

[Solvent refined, Midcontinent SAE 10 grade oil]

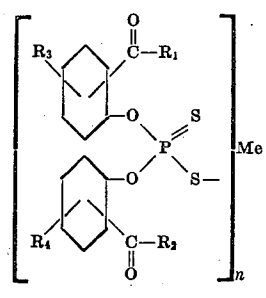
Composition	Bearing loss, mg.		Naphtha insol.	Neutralization number
	Cu-Pb	Ag-Cd		
			Per cent	
Oil, control	16	714	0.10	7.76
Oil+0.75% barium di-(o-lauroyl-p-amylphenyl)-dithiophosphate	11	1	0.01	0.42

Although this particular oil without a di-(acylphenyl)-dithiophosphate formed a high percentage of acidic decomposition products when heated as shown by the neutralization number and tended to form sludge as shown by the naphtha insolubles, the effectiveness of the barium di-(o-lauroyl-p-tertiaryamylphenyl)-dithiophosphate in reducing these tendencies is seen to be very pronounced.

We claim:

1. A hydrocarbon oil composition comprising a major proportion of a hydrocarbon oil and a minor proportion of a di-(acylphenyl)-dithiophosphate.

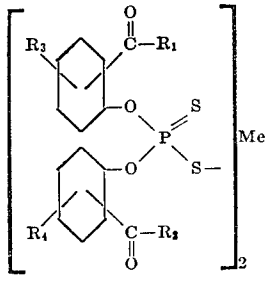
2. A hydrocarbon oil composition comprising a major proportion of a hydrocarbon oil and a minor proportion of a di-(acylphenyl)-dithiophosphate having the general formula



in which R_1 and R_2 are members of the group consisting of alkyl, cycloalkyl, and aryl radicals,

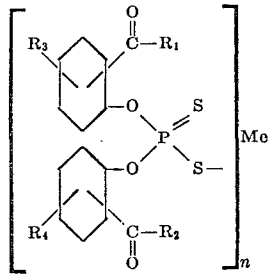
R_3 and R_4 are members of the group consisting of hydrogen, alkyl, cycloalkyl, and aryl radicals, Me is a metal and n is the valence of the metal.

3. A hydrocarbon oil composition comprising a major proportion of a hydrocarbon oil and a minor proportion of a di-(acylphenyl)-dithiophosphate having the general formula



in which R_1 and R_2 are members of the group consisting of alkyl, cycloalkyl, and aryl radicals, R_3 and R_4 are members of the group consisting of hydrogen, alkyl, cycloalkyl and aryl radicals and Me is an alkaline earth metal.

4. A hydrocarbon oil composition comprising a major proportion of a hydrocarbon oil and a minor proportion of a di-(acylphenyl)-dithiophosphate having the general formula



in which R_1, R_2, R_3 and R_4 are alkyl radicals, Me is a metal and n is the valence of the metal.

5. A hydrocarbon oil composition comprising a major proportion of a hydrocarbon oil and a minor proportion of di-(o-lauroyl-p-tertiary-amylphenyl)-dithiophosphate.

6. A hydrocarbon oil composition comprising a major proportion of a hydrocarbon oil and a minor proportion of di-(stearoylphenyl)-dithiophosphate.

7. A hydrocarbon oil composition comprising a major proportion of a hydrocarbon oil and a minor proportion of di-(o-naphthenoyl-p-amylphenyl)-dithiophosphate.

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