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MINERAL OIL LUBRICATING COMPOSITION AND AN IMPROVEMENT AGENT THEREFOR AND ITS METHOD OF PREPARATION

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This invention relates to improvement agents for lubricating oils and methods of making the same.

In the lubrication of internal combustion engines of all types, particularly when severe operating conditions are encountered, plain mineral oils often prove unsatisfactory due to the attendant deposition on the engine surfaces, such as cylinder walls, pistons and rings, of "varnish," gum, or "sludge." This effect is particularly serious in modern engines operating under severe conditions, such as Diesel engines, aviation, truck and tank engines. The problem has become increasingly serious due to the trend toward higher efficiency, or higher power output per unit weight per engine, reduction in amount of oil employed in the lubricating system of the engine, and other conditions which tend to accelerate the deteriorating influences on mineral lubricating oil.

Formation of so-called varnishes and sludges on engine surfaces is due to oxidation or polymerization effects (or both) on the lubricating oils, as well as to like effects on or from products of combustion of the fuels, which find their way by leakage into the engine crankcase and other parts of the engine.

The presence of these substances is disadvantageous for many reasons. In particular the oil and fuel oxidation products tend to increase ring sticking and production of deposits on piston surfaces and in fixed parts of the combustion chamber. Sludges are formed in the crankcase of the engine, and the rate of corrosion of bearing surfaces is increased, especially with bearing alloys of the types now in use.

Petroleum oils intended for use under the severe conditions described are almost always compounded with a mixture of substances each intended to counteract one or more of the undesirable effects noted. These substances, which fall into a wide variety of chemical classifications, are known by the general term "improvement agents" or addition agents. Preparation of satisfactory compounded lubricants is a matter of considerable difficulty. Some of the agents are of low potency in that a rather large amount has to be added to achieve the desired result. Often two or more agents are more or less incompatible with each other; and their rather low solubility in general further adds to the difficulties in preparing a useful lubricant.

One object of the present invention is the provision of an improvement agent for engine oils which can be incorporated in petroleum oils in effective proportions and which imparts to the oil a plurality of functions hitherto obtainable only by use of two or more agents. Another object is the provision of such an agent which, in the form of a solution in petroleum lubricating oils, serves the functions of (1) acting as a negative catalyst to suppress the formation of deterioration or oxidation products, such as varnish, sludge and gum, (2) acting as a loosening or suspending agent (detergent) to prevent ring sticking, "varnishing" or coating of metallic surfaces by such reduced amounts of deterioration products as may be formed and (3) acting as a suspending or dispersing agent for retaining very small particles of solid particles of deterioration or contaminating materials in the oil; for example, particles of carbonaceous matter that find their way into the oil by passing the rings, which cannot possibly be made absolutely tight. Another object is the provision of an improvement agent capable of increasing the solubility or dispersibility of other agents such as straight soaps or the like, when it is desired also to add such compounds to an oil.

According to this invention, new agents capable of achieving these and other objects are prepared by causing phosphorus pentasulfide to react with certain alkylated phenols (olefin-phenol reaction products) to form complex acid reaction products which in turn are treated with lime (or other polyvalent metal oxide or hydroxide) under conditions producing stable, complex, metallo compounds including polyvalent metal derivatives of acid thiophosphate esters of said alkylated phenols, of the character described in more detail below. That is, as shown post, the new agents are prepared from alkylated phenols containing at least one branched chain alkyl group. Likewise, as there shown, in preparing the new agents, such alkylated phenols are reacted with the phosphorus pentasulfide under controlled conditions to produce acid reaction products thereof and the acid reaction products so obtained are heated with polyvalent metal oxides and hydroxides until stable, substantially neutral, metal derivatives thereof are obtained; the stable, oil-soluble improvement agent so obtained being separated from the unreacted metal oxides and hydroxides and other insoluble compounds. The polyvalent metal derivatives so prepared, particularly di- and tri-valent metal compounds and mixtures thereof, are useful for the present purposes.

These compounds (so prepared) in solution in a lubricating oil provide four functions, so to speak, in a single molecular structure. (1) By

virtue of its combined metal (e. g. calcium) suspending and detergent properties are imparted as well as resistance to ring-sticking, (2) the combined phosphorus imparts anti-bearing-corrosion and anti-ring-sticking properties, (3) the combined sulfur serves to give pressure-carrying properties to the oil and to retard corrosion of alloy bearings, and (4) by virtue of the organic nucleus, the alkylated phenyl groups, the oil has good anti-oxidant and oiliness properties and suspending and solubilizing functions.

Thus according to the invention there is provided a compound which, dissolved in oil, imparts thereto all the many desirable features which we wish to obtain. It is in a form which is so soluble in motor oils of all types and degrees of refinement that we can incorporate any desired proportions to form a clear solution in lubricating oils; even in the most highly refined and most paraffinic of the heavy aviation oils, such as are specified for the most severe and exacting aviation service.

It is known that highly paraffinic oils, refined to the high degree desired to secure such properties as low carbon forming effects and the like as well as high viscosity index values (improved viscosity temperature relationships) that are preferred for such service, are relatively poor solvents for decomposition products formed in use. Also there is increased tendency to induce bearing corrosion. In the invention these undesirable properties of such oils are done away with by the use of moderate amounts of the compounding agent having cumulatively all the desired properties, in the way of ready solubility in the oil and the other effects mentioned above. We can employ very highly refined paraffinic type aviation oils that have the desired superior physical characteristics as regards the oil proper, and, at the same time, avoid any undesirable effects whatever from the use of such heavily refined paraffin type oils. In other words, the addition of our compound which is soluble in all proportions in the heavy highly refined paraffinic oils enables us to secure all of the properties desired in the way of the oils of superior performance characteristics, as well as the effects of detergency, avoiding of ring sticking, varnish formation, sludge formation, and corrosion of alloy bearings, that might otherwise be encountered, as well as to secure the desired effect of retarding oxidation due to the use of more highly refined oils of less tendency to oxidize combined with the use of a compound which actually retards oxidation.

Another important field of utility for the invention is in Diesel lubricating oils. Heretofore it has been considered best to use naphthene type oils, dosed heavily with less effective or less concentrated types of compounding agents which are not readily soluble even in the more naphthenic type of Coastal oils. It has often been necessary to make a compromise, even for the Coastal oils of greater solubility effect for compounding agents, between the degree of refining and the solubility effect for the addition agents. It is quite unsatisfactory for most of these agents to use even a moderately refined paraffin type oil, as all or part of the addition agents will often separate from the oil upon standing. For these reasons Diesel lubricating oils hitherto prepared have been rather unsatisfactory in that they employ lower grade oils of less degree of resistance to oxidation than would otherwise be desirable. With the present invention, we prepare an improved oil which performs satisfactorily in Diesel engines, by employing a highly refined paraffin type oil, with control of ring sticking effects and sludge forming and detergency effects by incorporation of the present compound in any desired proportion.

The agent has another important advantage in that in addition to being very oil-soluble itself it acts to disperse in mineral oils other improvement agents which are of themselves relatively insoluble. For example in accordance with the present invention one per cent or more of calcium oleate or aluminum stearate can be dissolved in oil to form a stable and permanent dispersion, with the aid of an equal quantity of the new agent. Thus the new agent is sometimes useful even where its own antioxidant and dispersive properties are not required, for its value in dissolving other agents.

The agent is conveniently prepared in the form of a concentrated oil solution, which can readily be stirred into an oil to prepare a compounded lubricant.

In preparing the new agent, the base material is advantageously a "heart cut" of phenol olefine reaction products, prepared from the 20 to 80 per cent cut separated by steam or vacuum distillation; vacuum distillation being preferred as it eliminates a step of drying the mixture before the next step-reaction thereof with phosphorus pentasulfide (P_2S_5). The heart cut described is a liquid, semi-liquid or crystalline material, soluble in all proportions in lubricating oils and gasoline, and soluble in most organic solvents, and insoluble in water and NaOH solutions. It has the following properties:

Gravity, °A. P. I.	18.0–24.0
Sp. gr., 60°/60° F.	0.9465–0.9100
Viscosity, S. U. V.:	
100° F.	60–100
Pour, °F.	−30–+15
Color, N. P. A.	Not darker than 8.0
Free phenol, per cent	Nil
Mineral acidity, per cent:	
SO_3 equivalent	Nil
Distillation, A. S. T. M. D 86–38:	
Over point, °F.	375–380
End point, °F.	525–540
10% at, °F.	415–450
50	470–495
90	495–525

This phenol-olefine reaction product contains varying amounts of 4-tertiary-butyl phenol, 2-tertiary-butyl-4-secondary butyl phenol, 2,4-di-tertiary-butyl phenol, 2,6-di-tertiary-butyl-4-secondary-butyl phenol, 2,4,6-tri-tertiary-butyl phenol, 2,6-di-tertiary-butyl-4-methyl phenol, 4,6-di-tertiary-butyl-2-methyl phenol and other alkylated phenols containing a branched chain alkyl group attached to the phenyl nucleus thereof.

We treat this reaction product with P_2S_5, in amount 8 to 40 parts per 100 parts of the reaction product, advantageously at a reaction temperature of about 250 to 450° F. This treatment so to speak puts the reaction product into a condition such that it can combine with the calcium or other metal in rather large proportion.

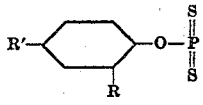
Phosphorus pentasulfide, P_2S_5, also known as phosphoric sulfide, thiophosphoric anhydride and phosphorus persulfide, is a light yellow crystalline mass, having a specific gravity of 2.03. It melts at 274° C. and boils at 518–520° C. It is

soluble in alkalies and carbon bisulfide and is decomposed by water.

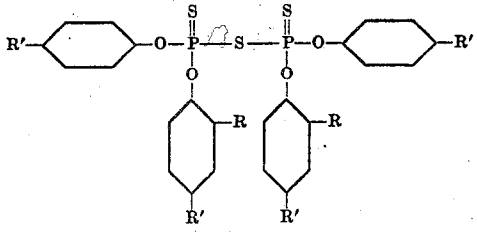
We prefer to incorporate a small quantity of phosphorus trisulfide to catalyze the reaction. The presence of this catalyst insures a product having a better color, etc. Phosphorus trisulfide, P_4S_3, sometimes known as phosphorus sesquisulfide or tetraphosphorus trisulfide, is a yellow crystalline, inflammable mass, having a specific gravity of 2.00, a melting point of 172° C. and a boiling point of 407.8° C. It is soluble in carbon bisulfide.

The reaction products so obtained are alkylated phenyl esters of thiophosphoric acids. That is, they are organic thiophosphates containing one or more alkylated phenyl groups; such groups having at least one branched chain alkyl substituent attached to the phenyl nucleus. The following formulae are representative of typical compounds of this class which are useful and advantageous in making our new improvement agents.

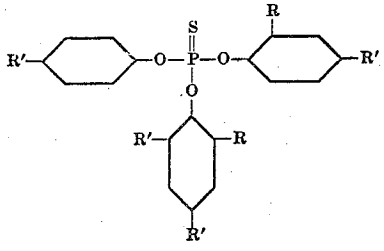
$RR'C_6H_3PS_2O$:



$(RR'C_6H_3)_4P_2S_3O_6$:



$(RR'C_6H_3)_3PSO_3$:



In the above formulae, R represents a secondary alkyl group (secondary butyl etc.) and R' represents a tertiary alkyl group (such as tertiary butyl etc.).

The esters represented by the above formulae are merely illustrative of the type of reaction products useful in practicing this invention. As a class, alkyl phenyl esters of thiophosphoric acids may be used. Such reaction products are viscous liquids having the following properties:

Average molecular weight	700
Gravity, ° A. P. I. (at 183° F.)	3.6
Viscosity, S. U. V.:	
210° F	90–110
Sulfur, B, per cent	7–9
Phosphorus, do	5–7

In order to facilitate ease in handling and in treating it is sometimes advisable to dilute one of the intermediate products or the final product with a mineral oil.

The P_2S_5-phenol-olefin reaction product is now neutralized with lime, quick or hydrated, added in proportion about 5 to 25 parts (figured as CaO) per 100 parts of the reaction product. In lieu of ordinary lime, dolomitic limes or magnesia or magnesium hydroxide can be employed; and other metallic oxides or hydroxides such as alumina or zinc oxide can be employed.

The reaction product is suitable for use directly in oils. The optimum amount to add to a given lubricating oil depends on the severity of the service conditions and on the degree to which the oil tends to form gum or varnish. Ordinarily from 0.25 to 5.0 per cent by weight of the agent suffices.

While the structural formulae outlined above are probably typical of the character of the compounds present in our preparation, they cannot be taken too literally. In practice we vary the proportions of the several reactants in accordance with the particular properties desired. For example if extreme pressure resisting qualities are desired we incorporate more combined sulfur. Thus the preparation exhibits relative proportions of combined Ca, P, S, etc. dependent on the particular effects desired.

Example I.—In a specific example illustrative of one good way of preparing an agent within the purview of the invention, 824 pounds of the above described phenol olefine reaction product were mixed with 222 pounds of P_2S_5, catalyzed with 10 pounds of P_4S_3. The mixture was agitated and heated to 450° F. for a period of four hours. The reaction product was diluted 1 part to 2 parts of mineral oil. The mixture had a neutralization number of 15.

The phospho-sulfo-derivative of phenol olefine reaction product was then treated with 28 pounds hydrated lime; an amount just sufficient to neutralize the acid derivative. After lime treatment, the material had an ash content (calculated as $CaSO_4$) of 7 per cent.

Example II.—A phenol-olefine reaction product as described above was treated with 50 per cent by weight of P_2S_5 at 450° F. The product had a neutralization number of 80. This reaction product was treated with 12 per cent hydrated lime at 300° F., and after treatment with this amount of lime had an ash of 16.0 per cent calculated as calcium sulfate.

Example III.—Thirty-eight parts by weight of phenol-olefine reaction product was treated with 9.8 parts by weight P_2S_5 and 0.5 part by weight P_4S_3 at 400° F. for 6 hours. The reaction product was treated with 3.4 parts by weight of hydrated lime at 450° F. for 2 hours. Then 52 parts of a 100 viscosity/100° F. naphthene type mineral oil were added at 350° F. and the mixture was filtered through filter aid. The product had the following properties:

Gravity, ° A. P. I.	11.8
Specific gravity, 60°/60° F	0.9874
Viscosity, S. U. V.:	
210° F	69
Sulfur, per cent	5.4
Phosphorus, do	3.7
Ash as sulfate, do	2.37

Example IV.—Forty-two parts by weight of a heart fraction from phenol-olefine reaction product, 0.5 part by weight P_4S_3, and 5.6 parts by weight P_2S_5, were heated at 450° F. for 4 hours. Then 3.8 parts of hydrated lime, $Ca(OH)_2$, were added and the mixture agitated at 450° F. for 2 hours. Then 52 parts by weight of a mixture of 50 parts sulfurized sperm oil and 50 parts mineral oil were added at 350° F. and the mixture was

filtered. This concentrate had the following properties:

Gravity, ° A. P. I.	16.5
Specific gravity, 60°/60° F.	0.9561
Viscosity, S. U. V.:	
100° F.	1701
Color, N. P. A.	7.5
Sulfur, per cent	5.10
Phosphorus, do	2.01
Ash as sulfate, do	2.11

The sulfurized sperm oil, mineral oil mixture used in preparation of this concentrate had the following properties:

Color, N. P. A.	5.5
Sulfur, per cent	5.5
Neutralization No.	5.0
Saponification No.	77.5

Example V.—One hundred parts by weight of phenol-olefine reaction product was treated with 50 parts by weight P_2S_5, and 1 per cent P_4S_3 at 400° F. for 4 hours. This acid phospho-sulfo-phenol-olefin reaction product, which had a neutralization number of 107, was treated with 60 parts by weight of aluminum hydroxide gel (Aluminum Ore Co.). The aluminum phospho-sulfo-phenol-olefin product had an ash content of 4.5 per cent. The product was mixed one part with two parts of 100 viscosity/100° F. naphthene type mineral oil.

Example VI.—In an iron distillation vessel equipped with a stirrer, means of heating and cooling and a reflux condenser were put 1760 pounds of heart fraction phenol-olefine product secured by fire distillation of the crude phenol-olefine 20–80 per cent cut. To this material was added 585 pounds of P_2S_5 and 4.4 pounds of P_4S_3, over a period of one hour. This mixture was then stirred and heated to a maximum temperature of 360° F., where it was maintained for 4 hours to digest the P_2S_5 and P_4S_3. At this point the intermediate product was cooled to 200° F. and further treated with 240 pounds of lime in a slurry (two parts of lime and 1 part water) which was run into the vessel over a period of one hour. During the lime treatment the temperature was maintained within the range of 200–230° F. for three hours to evaporate water from the lime as well as that derived from the reaction. Upon dehydration, the temperature was raised to 300° F., where it was maintained for two hours. At this stage, 2000 pounds of acid-treated Coastal distillate having a viscosity of 100 seconds S. U. V. at 100° F. were added to the vessel and blended with the reaction products. The oil mixture was then passed through a filter-aid filter to remove excess lime and other insoluble matter.

The finished product had the properties shown below:

Gravity, °A. P. I.	8.2
Viscosity, S. U. V.:	
100° F.	519
Sulfur, B, per cent	3.70
Phosphorus, do	3.90
Ash as sulfate, do	11.0

Example VII.—A batch of 1316 pounds of phenol-olefine reaction product was added to a vessel as in Example VI and 180 pounds of P_2S_5 and 4 pounds of P_4S_3 as catalyst and color stabilizer were added over a period of one hour. The mixture was stirred and heated to 360° F. for a period of 7 hours to digest the phosphorus compounds. At this stage the mixture was a clear amber viscous fluid. The reaction mixture was cooled at 200–220° F. and further treated with 75 pounds of zinc oxide in a slurry (2 parts ZnO and 1 part water). During the introduction of the slurry the temperature of the mixture was maintained at 230° F. to boil off the water. The temperature was raised to and maintained at 280° F. for eight hours to complete the digestion of ZnO. After the reaction was complete an equal weight of 100-second acid-treated Coastal oil was added to the product to facilitate filtering. The final product had the following properties:

Gravity, °A. P. I.	14.8
Viscosity, S. U. V.:	
100° F.	696
Sulfur, B, per cent	2.8
Phosphorus, do	2.3
Ash as sulfate, do	3.4

Example VIII.—A series of lubricating oil compositions was prepared by dissolving in a straight engine oil various quantities of the improvement agents of the present invention; in some cases in conjunction with certain old agents to show the marked solubilizing effect of the present agents. In the series of compositions,

A is the mineral oil base,

B is oil A containing 2 per cent of the reaction product of Example I,

C is another oil containing 2 per cent of the reaction product of Example I except that the metal is zinc instead of calcium,

D is oil A containing
- 1 per cent of the reaction product of Example I,
- 4 per cent of X—refined sulfurized liquid sperm oil derivatives of Ca-soap fluxed with an equal weight of petroleum oil (see below),
- 6 per cent of Y—a 50:50 mixture of mineral oil and sulfurized sperm oil (see below),

E is oil A containing 0.5 per cent of the reaction product of Example I and 6 per cent of Y,

F is oil A containing 2 per cent of a 50:50 mixture of the reaction product of Example I, and Y.

The viscosities and other conventional identifying characteristics of these compositions were as follows:

	A	B	C	D	E	F
Gravity, ° A. P. I.	26.5	26.5	28.5	25.7	25.8	26.4
Viscosity, S. U. V.:						
100° F.	1,819	1,761	1,453	1,581	1,517	1,724
210° F.	122.3	117.4	113.9	114.8	114.7	119.5
Viscosity index	93	91	102	96	99	95
Flash, OC, ° F.	525	510	535	435	455	485
Fire, OC, ° F.	595	585	600	545	545	595
Pour, ° F.	+5	0	−5	+5	+10	+5
Color, N. P. A.	5.5	5.5	3.25	5.5	5.5	5.5
Sulfur, per cent	-------	0.36	0.20	0.82	0.74	0.37
Carbon residue, do	0.46	0.43	0.31	0.70	0.68	0.45
Neutralization No.	0.03	0.18	0.36	0.06	0.32	0.08
Ash as sulfate per cent	-------	0.024	0.076	0.023	0.023	0.028

In order to evaluate their tendency to cause ring sticking in engines, all these compositions (except C) were subjected to a motor service test. For this test a standard Waukesha-CFR crankcase with special L head type cylinder block was employed, coupled to a cradle-type electric dynamometer. This engine has a bore of 3¼ inches, a stroke of 4½ inches, and a compression ratio of 4.7:1. The piston is of cast iron with four compression rings and one oil-control ring, all located above the wrist pin. Three liters of the oil to be tested are placed in the crankcase. The engine is then run at 900 R. P. M. with the spark and the air-fuel ratio adjusted to give

maximum power, and the coolant temperature is maintained at 350° F. At the end of 25 hours running, the engine is stopped and dismantled and the piston is examined. The performance of the oil is then rated according to the condition of the piston, in keeping with the following scale, in which the rating number increases as the piston condition becomes worse:

Ratings

No. 1—No ring stuck, and no heavy hard deposit.
2—Top or oil ring (or both) stuck on a small part of circumference.
3—Top or oil ring (or both) completely stuck.
4—Top or oil ring (or both) completely stuck, end ring partly stuck.
5—Top, 2nd and oil rings completely stuck.
6—Condition No. 5 with 3rd and 4th rings partly stuck.

In the test the ratings of the several compositions were as follows:

	Composition				
	A (plain oil)	B	D	E	F
Rating No	4	1	1	1.5	1

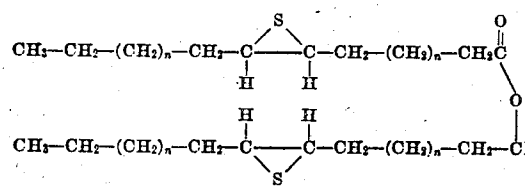
These compositions were also subjected to the well known Almen and Timken lubrication tests, with the following results:

	Composition				
	A (plain oil)	B	D	E	F
Almen test.....pounds..	6	16	18	16	14
Timken test.....do....	14	44	60+	60+	38

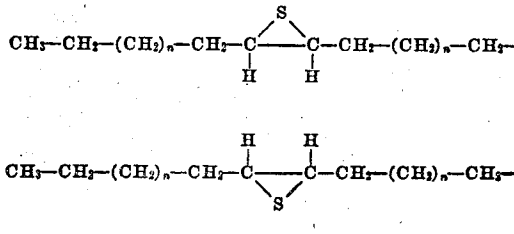
In further explanation of the improvement agents X and Y (which are not per se a part of the invention) employed in the above examples in conjunction with the agent of the present invention:

Agent X is a mixture of 45 per cent of a sulfurized sperm oil derivative, 2.5 to 3.0 per cent of a calcium soap of a sulfurized sperm oil derivative, 2.5 to 3.0 per cent of sulfurized sperm oil alcohols and 50.0 per cent of 100/2 Texas oil as a fluxing agent to facilitate handling. The formulas of the first three of these ingredients are given in typical or approximate form below (*n* being from 3 to 5).

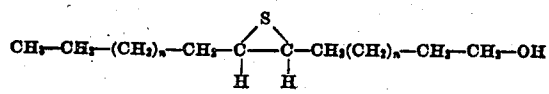
The sulfurized sperm oil derivative



The calcium soap of sulfurized sperm oil derivatives



The sulfurized sperm oil alcohols



Agent X had the following properties:

Gravity.....°A. P. I...	23.0
Viscosity, S. U. V.:	
100° F.....	255
210° F.....	60.4
Color N. P. A.....	8.0
Sulfur, B.....per cent..	3.84
Neutralization No.....	0.48
Ash (as sulfate).....per cent..	0.83

Agent Y is a 50:50 mixture by weight of mineral oil and sulfurized sperm oil, clay contacted with 2 pounds of filter clay per gallon of mixture at 260–275° F. This product normally tests 5.5 N. P. A. color, 5.5 per cent sulfur, 5.0 neutralization number and 77.5 saponification number.

While the agent of the present invention has been described primarily in reference to preventing ring sticking and other undesirable effects in engine oils, it is useful in other relations; for example, as described in aiding the dispersion of other improvement agents in oils.

What we claim is:

1. A process of preparing an improvement agent for lubricating oils, from alkylated phenols containing at least one branched chain alkyl group, which comprises heating 100 parts of said alkylated phenols with 8 to 40 parts of phosphorus pentasulfide at temperatures between 250° and 450° F. until substantially all of the phosphorus pentasulfide has reacted and acid reaction products thereof are obtained, and converting the acid reaction products so obtained into stable, substantially neutral metal derivatives thereof by further heating said acid reaction products with a reactive metal compound of the class consisting of the oxides and hydroxides of a polyvalent metal, the amount of said reactive polyvalent metal compound being sufficient to neutralize the acidity of said reaction products and the mixture being heated at temperatures between 230° and 450° F. until stable, substantially neutral, metal derivatives thereof are obtained, and separating the unreacted metal oxides and hydroxides and other insoluble compounds from the oil-soluble improvement agent so obtained.

2. A process for preparing an improvement agent for lubricating oils from alkylated phenols containing at least one branched chain alkyl group, which comprises heating 100 parts by weight of said alkylated phenols and 8 to 40 parts of phosphorus pentasulfide at temperatures between 250° and 450° F. until substantially all of the phosphorus pentasulfide has reacted and acid reaction products thereof are obtained, and then further heating the acid reaction product so obtained with a reactive metal compound of the class consisting of the oxides and hydroxides of a polyvalent metal, the proportion of said reactive polyvalent metal compound being stoichiometrically equivalent to 5 to 25 parts of CaO per 100 parts of said reaction products and the mixture being heated at temperatures between 230° and 450° F. until stable, substantially neutral, metal derivatives thereof are obtained, and filtering the oil-soluble reaction product so obtained to remove unreacted metal oxides and hydroxides and other insoluble compounds.

3. The process of claim 1 wherein the said reactive metal compound is lime.

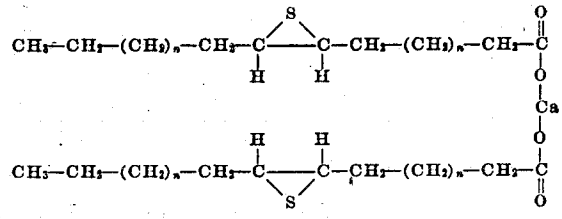
4. A process for preparing an improvement agent for lubricating oils from alkylated phenols containing at least one branched chain alkyl group, which comprises heating 100 parts by weight of said alkylated phenols and 8 to 40 parts of phosphorus pentasulfide at temperatures between 250° and 450° F. until substantially all of the phosphorus pentasulfide reacted and acid reaction products thereof are obtained, diluting the acid reaction products so obtained with mineral oil, adding to the so fluxed acid reaction products a reactive metal compound of the class consisting of polyvalent metal oxides and hydroxides in proportions stoichiometrically equivalent to 5 to 25 parts of CaO per 100 parts of said reaction products, maintaining this mixture at a temperature of 230° to 450° F. until stable, substantially neutral, derivatives thereof are obtained, and filtering the oil concentrate of the improvement agent so obtained to remove unreacted metal oxides and hydroxides and other compounds insoluble in the oil.

5. The process of claim 1 wherein the reaction between the phosphorus pentasulfide and said alkylated phenols are catalyzed by the presence of a small amount of phosphorus trisulfide.

6. An improvement agent soluble in petroleum oils and capable of inhibiting formation of deposits of varnish, gum and sludge in engines and of dispersing such deposits when present, comprising the stable, oil-soluble, substantially neutral metallo compounds obtained by the process of claim 1.

7. An improved lubricating composition comprising a major amount of a mineral lubricating oil and a small proportion of the stable, oil-soluble, substantially neutral metallo compounds obtained by the process of claim 1, the amount of said oil-soluble improvement agent being sufficient to inhibit the formation and deposition of gum, varnish and sludge in engines and to disperse such deposits when present.

8. An improved lubricating composition comprising a major amount of a mineral lubricating oil and a small proportion of the stable, oil-soluble, substantially neutral metallo compounds obtained by the process of claim 1 and of a calcium soap of sulfurized fatty acids having the formula



wherein n is from 3 to 5, the said mineral lubricating oil being a refined paraffinic oil, the said metal soap having a relatively low solubility in the paraffinic oil and being dispersed in the oil with the aid of said oil-soluble metallo compounds, and the amount of said oil-soluble metallo compounds being sufficient to inhibit the formation and deposition of gum, varnish and sludge in engines and to disperse such deposits when present.

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