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LUBRICANT

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10 Claims. (Cl. 252—46.7)

This invention relates to a lubricating oil composition having improved characteristics especially with respect to oxidation and corrosion.

In my co-pending application Serial No. 494,689, filed concurrently herewith, issued August 15, 1944, as Patent 2,356,073, I have described and claimed a novel class of materials resulting from the reaction of an aliphatic alcohol with the condensation product of a terpene, such as present in turpentine, and phosphorus pentasulfide, P_2S_5. The turpentine-P_2S_5 condensation product is the subject of my co-pending application Serial No. 494,688, also filed concurrently herewith.

I have now discovered that the materials to which my first said application is directed, particularly those prepared by reacting an aliphatic alcohol with a condensation product of turpentine and P_2S_5, are especially effective in repressing or inhibiting the deterioration of lubricating oil compositions and the corrosion of metal parts in contact therewith.

I am at present unable definitely to identify by chemical formula either the condensation products of turpentine and P_2S_5 or the products resulting from the reaction of said condensation products with the alcohol. For brevity, I shall herein refer to the former as the turpentine-P_2S_5 condensation product and to the products resulting from the reaction of said condensation products with the alcohol as my inhibitor.

The characteristics of the inhibitor used in the compounding of the lubricating oil composition of my present invention vary somewhat depending upon the characteristics of the turpentine-P_2S_5 condensation product and the nature and proportions of the alcohol used in its preparation. Generally, these compounds are relatively acidic and are highly soluble in mineral oils.

The lubricating oil composition of my present invention may consist solely of the lubricating oil constituent and my inhibitor. However, the inhibitors of my present invention have been found to be compatible with other desirable lubricating oil addends and the inclusion of such other addends, especially addends of the type known as detergents, is within the contemplation of my present invention and constitutes an important aspect thereof.

The inclusion of certain so-called detergents, for instance, in internal combustion engine lubricants, has been found highly advantageous. An especially effective lubricating oil composition for the lubrication of internal combustion engines and the like contemplated by the present invention is one comprising, in addition to the lubricating oil fraction and my inhibitor, a minor proportion of the calcium salt of iso-octyl salicylate, or the calcium salt of capryl salicylate. These calcium salts have been found particularly effective as detergents in lubricating oil compositions used in internal combustion engines, as more fully described in the patents, 2,347,547 and 2,339,692, issued April 25, 1944, and January 18, 1944, respectively, on applications of Willard L. Finley.

A further highly effective lubricating oil composition contemplated by my present invention is one comprising, in addition to the lubricating oil constituents and my adden i, a calcium petroleum sulfonate, as a detergen' Other detergents which may be used with advantage include the barium phenolate of sulfurized diamyl phenol, such as currently marketed under the trade name "Aerolube B," metallic phenolates of sulfurized tertiary amyl phenol, such as currently marketed under the trade names "Calcium Paranox" and "Barium Paranox," and various metallic soaps, either basic or neutral, metallic sulfonates, alcoholates and alkoxide and metallic derivatives of alkylated salicylic acid.

When used in conjunction with these detergents, particularly the calcium salts, including the calcium petroleum sulfonates previously mentioned, these detergents and my inhibitors have been found to complement each other so that the effectiveness of each is promoted. For example, the phosphorus acidity of the inhibitor appears to be neutralized by the calcium, thus minimizing any tendency of the former to promote sludge formation. Further, the tendency of the detergents to promote oxidation of the oil at the termination of its oxidation induction period is also minimized by the presence of my inhibitor. Each of these desirable ends is accomplished without destroying the effectiveness of either the detergent or the inhibitor.

The proportions of the inhibitor used in the compounding of my improved lubricating oil compositions may be varied somewhat but, in any case, only a minor proportion is used. In the absence of a detergent such as previously mentioned, generally satisfactory results have been obtained by using proportions within the range of about 0.05% to about 1% based on the weight of the lubricating oil constituent. In special cases, for example where such calcium salt detergents are present, somewhat higher proportions, say from about 0.25% to about 2%, may be used with advantage. However, these inhibitors are acidic

phosphorus derivatives and phosphorus acidity has been found to have a general tendency to promote polymerization and sludge formation in mineral lubricating oils. In internal combustion engine lubricants, where sludge formation must be minimized, the use of the inhibitor in proportions exceeding about 1% by weight, in the absence of detergents such as previously noted, is not generally advisable. However, proportions within the indicated range have not been found to cause noticeable or objectionable sludging under such conditions. In gear lubricants, for example, where polymerization and sludging is less critical, larger proportions of the inhibitor may be employed.

For optimum results, when used in conjunction with one of the previously-noted calcium detergents, the proportion of the inhibitor should not exceed that which will be neutralized by the calcium salt detergent, for, with an excess of the inhibitor, residual phosphorus acidity will remain with its characteristic tendency to promote sludge formation. The optimum ratio of the inhibitor to the detergent will depend upon the basicity of the detergent and upon the amount of P_2S_5 equivalent used in the preparation of the inhibitor and may be determined for any particular set of conditions by simple test.

Various petroleum lubricating oil fractions may be used, for instance solvent-treated Mid-Continent neutrals or a blend of such Mid-Continent neutrals with bright stock or a solvent-refined lubricating oil fraction from a Pennsylvania crude. Characteristics of two such oils which have been used with advantage, and which were used in the compounding of the lubricants hereinafter set forth as illustrative of my invention, appear in the following Table I, in which base oil A is a solvent-treated Mid-Continent 250 neutral oil and base oil B is a solvent-refined Mid-Continent S. A. E.-30 oil.

Table I

	Base oil	
	A	B
Gravity, °A. P. I.	29.5	27.1
Flash, °F.	405	465
Fire, °F.	480	535
Viscosity at 100° F. SUS	249.6	442.1
Viscosity at 210° F. SUS	49.0	57.5
Viscosity index	92.7	80.6
Pour, °F.	5	−10
Carbon residue	0.039	0.03

The invention will hereinafter be illustrated by specific examples of my improved lubricating oil composition. Since the characteristics of the inhibitor are to a substantial extent affected by the characteristics of the turpentine-P_2S_5 condensation product used in its preparation and the nature and proportions of the alcohol reacted therewith, the illustrations of my lubricating oil compositions will include a description of the preparation of the particular inhibitor used.

In the preparation of the intermediate turpentine-P_2S_5 condensation product to be used in preparing my inhibitor, the ratio of turpentine to P_2S_5 used is with advantage within the range from approximately 1:1 to about 3 parts of turpentine to 1 part P_2S_5. Where ratios higher than about 3:1 are used, the condensation product has been found to contain considrable unreacted turpentine. Ratios of about 3:2 have generally been found to be particularly satisfactory for this purpose.

The reaction of turpentine with P_2S_5 is highly exothermic and proceeds spontaneously after being initiated by slight heating. A desirable method of effecting the reaction is to heat the turpentine in a vessel to about 200° F. or slightly higher and then, without further heating, slowly stirring in the phosphorus penta-sulfide in the powdered form. The heat of the reaction is great and, consequently, the addition should be made slowly so as to avoid the possibility of the reaction's becoming uncontrollable. The characteristics of the inhibitor are favorably affected by the use in its preparation of the turpentine-P_2S_5 condensation product in the preparation of which the temperature during the mixing was not permitted to exceed about 250° F., although higher temperatures are permissible.

After the addition is completed, it is usually necessary to apply heat externally to complete the reaction. The temperature during this last stage is preferably maintained at about 300° F., though temperatures of about 200° F. to 400° F. may be employed. This second stage of the operation should be continued until all of the P_2S_5 is dissolved. The material thus prepared is a viscous liquid at elevated temperatures which solidifies on cooling to room temperature.

The turpentine-P_2S_5 condensation products, thus prepared, are, in the absence of excess turpentine, brittle, resinous solids which dissolve readily in lubricating oils or in excess turpentine to form liquids. Such solutions of high concentration are relatively viscous. However, the viscosity of the solution decreases rapidly as the proportion of the solvent is increased from 25% to 75%.

In general, my inhibitor may be prepared by adding the alcohol gradually to the turpentine-P_2S_5 condensation product prepared as previously described. Such addition is advantageously at a temperature of about 250° F. In reacting the alcohol with the turpentine-P_2S_5 condensation product, very little heat is evolved. After the alcohol has been added the mixture is maintained at an elevated temperature, advantageously about 200° F. to 220° F., for about 2 hours with stirring. The nature and proportions of the alcohol added may be varied over a considerable range without loss of the effectiveness of the resulting inhibitor. The optimum proportion of alcohol used is dependent upon the ratio of turpentine to P_2S_5 used in the preparation of the intermediate condensation product. Particularly desirable results have been obtained using about 2 moles of P_2S_5, 5 moles of turpentine and 3 moles of alcohol, assuming the molecular weight of the turpentine to be 136.

In the preparation of the turpentine-P_2S_5 condensation product used in preparing the inhibitor, I have found it desirable that no unreacted P_2S_5 remain in the product, as in the treatment of the condensation product with the alcohol, any unreacted P_2S_5 present has been found to react with the alcohol to form esters of thiophosphoric acid.

Aliphatic alcohols generally have been found suitable for use in the preparation of my improved inhibitor. For example, aliphatic alcohols ranging from methyl to octadecyl alcohol, i. e. containing from 1 to 18 carbon atoms per molecule, including secondary alcohols of 5 to 8 carbon atoms and branched chain primary alcohols containing 5 to 6 carbon atoms, have been

used with advantage. Lubricating oil compositions in which the inhibitor is prepared by reacting caprYl alcohol, i. e. octanol-2, or normal hexyl alcohol or lauryl alcohol with the turpentine-P_2S_5 condensation product, have been found to have particularly desirable properties. Generally, aliphatic alcohols having 5 or more carbon atoms per molecule are preferred.

In the preparation of the turpentine-P_2S_5 condensation product, either steam-distilled wood turpentine or gum spirits may be used. Such turpentine consists mainly of alpha pinene, a bi-cyclic terpene having the empirical formula $C_{10}H_{16}$. Pure alpha pinene and other more costly terpenes will react similarly with P_2S_5 but, for reasons including economic considerations, I prefer to use the more readily available turpentines. The turpentine used in the specific examples herein was a technical grade steam-distilled wood turpentine comprising about 90% alpha pinene.

The following specific examples of inhibitors used in the compounding of my improved lubricating oil compositions and the procedure by which such inhibitors may be prepared, are given as illustrative of my invention. It will be understood, however, that my invention is not limited to lubricating oil compositions containing the particular inhibitors illustrated.

Example I

In a 3-liter, 3-necked flask equipped with a stirrer, a thermometer and a funnel, there was placed 1360 grams (10 moles) of steam-distilled wood turpentine. The turpentine was heated to about 200° F., by means of an electric heater placed under the flask. The heat was then turned off, the stirrer started and 888 grams (4 moles) of powdered phosphorus pentasulfide was added portionwise. About 30 to 40 gram portions of the sulfide were added at first and, after about half the sulfide was added, the portions were increased to about 100 grams. The temperature was controlled by the rate of addition of the phosphorus pentasulfide so that it did not exceed 250° F. When all the phosphorus pentasulfide had been added and the exothermic reaction had stopped, as shown by a dropping of the temperature, the heater was again turned on and the mixture stirred and heated slowly to 300° F., and maintained at that temperature for about 4 hours. At the end of this period all of the phosphorus pentasulfide was dissolved and the product was a viscous amber-colored liquid. The heater was then turned off and the mixture allowed to cool to 250° F., and, while at this temperature, 780 grams (6 moles) of capryl alcohol (octanol-2) was added slowly over a period of 30 minutes. The heater was again turned on and the temperature held at 200–220° F. for 2 hours.

The resultant product was found by analysis to have an acid number of 71, a saponification number of 162.9 and to contain 8.07% phosphorus and 20.3% sulfur, each by weight.

Example II

In a 5-liter flask, similar to that used in Example I, there was placed 2040 grams (15 moles) of steam-distilled wood turpentine. The turpentine was then heated to about 200° F., by means of an electric heater placed under the flask. Thereafter, the heat was turned off and 1332 grams (6 moles) of powdered phosphorus pentasulfide was added portionwise and stirred into the turpentine. The first half of the phosphorus pentasulfide was added in about 50 gram portions and the second half in about 100–150 gram portions. The rate of addition was controlled so that the temperature of the reaction did not exceed 300° F. When the last of the sulfide was added and the heat of the reaction began to subside, the heater was again turned on and the reaction mixture stirred and heated to about 300° F., and maintained at this temperature for about 4 hours while stirring. At the end of this period, the phosphorus pentasulfide was dissolved. The heater was then turned off, the contents of the flask permitted to cool to 250° F., and, while at this temperature, 1170 grams (9 moles) of capryl alcohol was slowly added. After all the alcohol had been added, the temperature of the mixture was maintained at 200 to 220° F., for about 2 hours.

The product resulting from the above-described procedure was found by analysis to have an acid number of 64.9, a saponification number of 156.8 and to contain 8.2% phosphorus and 20.8% sulfur by weight.

Example III

680 grams (5 moles) of turpentine was placed in a 2-liter flask such as previously described and stirred while being heated to 225° F. 444 grams of powdered phosphorus pentasulfide was then added to the turpentine at such a rate that the temperature did not rise about 250° F. After all the sulfide had been added, the temperature of the mixture was raised to 300° F. and maintained at that temperature for about 3 hours, at the end of which period all of the phosphorus pentasulfide was dissolved. The mixture was then permitted to cool to 250° F. and 264 grams of "Pentasol," a commercial mixture of amyl alcohols, was added. The temperature was then maintained at 220° F. for an additional hour.

The product was found by analysis to have an acid number of 74.6 and a saponification number of 181.1 and to contain 9.23% phosphorus and 21.57% sulfur by weight.

Example IV

The process of Example III was repeated using 444 grams (2 moles) of P_2S_5, 666 grams (4.9 moles) of turpentine and 403 grams (3.1 moles) of capryl alcohol and the resultant product was found by analysis to have an acid number of 71.1 and a saponification number of 138.8 and to contain 8.04% phosphorus and 20.4% sulfur by weight.

Example V

A 3-liter, round-bottomed flask, fitted with a stirrer, was charged with 680 grams of turpentine. The turpentine was heated to 200° F., the heating discontinued and 444 grams of powdered phosphorus pentasulfide was added, with stirring, in portions of about 25 to 30 grams each, and at such a rate that the temperature did not rise above 250° F. After all of the phosphorus pentasulfide had been added, the temperature was raised to 300° F., and maintained at that temperature for 3 hours, at the end of which period all of the sulfide was dissolved. Thereafter, the mixture was cooled to 250° F. and 306 grams of normal hexyl alcohol was added and the temperature of the mixture held at about 200° F. for 1 hour.

The product was found by analysis to have an acid number of 74.0 and a saponification number of 172.1 and to contain 8.20% phosphorus and 21.57% sulfur by weight.

EXAMPLE VI

680 grams (5 moles) of steam-distilled wood turpentine was placed in a 3-liter flask, such as previously described, and heated to 225° F. There was then added 444 grams (2 moles) of powdered phosphorus pentasulfide, in 25 to 30 gram portions, at such a rate as to hold the temperature below 250° F. No external heat was applied during this addition. When all of the phosphorus pentasulfide was added, the mixture was heated to 300° F. and maintained at this temperature for 3 hours, at the end of which period the phosphorus pentasulfide had dissolved. The mixture was then cooled to 250° F., and 306 grams (3 moles) of 2-ethyl butanol-1 was added over a period of 30 minutes with continuous stirring. The temperature was held at about 200 to 220° F. for 1 hour after the addition of the alcohol.

The product was found by analysis to have an acid number of 74.9 and a saponification number of 176.2 and to contain 8.96% phosphorus and 22.1% sulfur by weight.

EXAMPLE VII

340 grams (2.5 moles) of turpentine was heated to 200° F. and 222 grams (1 mole) of powdered phosphorus pentasulfide was added slowly as previously described. After the phosphorus pentasulfide was all dissolved, 279 grams (1.5 moles) of "Lorol," a commercial mixture of alcohols made from hydrogenated fatty acid esters from palm oil and containing from 10 to 14 carbon atoms per molecule, was added and the mixture maintained for 1 hour at a temperature of 220° F.

The product was found by analysis to have an acid number of 67.2 and a saponification number of 147.3 and to contain 7.18% phosphorus and 19.80% sulfur by weight.

EXAMPLE VIII

To a turpentine-P_2S_5 condensation product prepared as in Example VII, there was added 405 grams (1.5 moles) of octadecyl alcohol and the mixture maintained at 220° F. for 1 hour. The product was found by analysis to have an acid number of 57.2 and a saponification number of 131.6 and to contain 6.52% phosphorus and 17.20% sulfur by weight.

EXAMPLE IX

In a 3-liter flask, such as used in Example I, 1360 grams (10 moles) of turpentine was placed and heated to 200° F. 888 grams (4 moles) of powdered phosphorus pentasulfide was then slowly added to the heated turpentine under conditions described in Example I. After the phosphorus pentasulfide was dissolved, 1300 grams (10 moles) of capryl alcohol (octanol-2) was added and the mixture stirred and heated as in Example I. The product was found by analysis to have an acid number of 114.2 and a saponification number of 146.9 and to contain 7.09% phosphorus and 17.4% sulfur, by weight.

EXAMPLE X

The procedure of Example IX was repeated, substituting methyl alcohol for the capryl alcohol and the product was found by analysis to have an acid number of 100.8 and a saponification number of 213.9 and to contain 10.9% phosphorus and 26.5% sulfur, by weight.

For the purpose of further illustrating my invention and the advantages derived therefrom, I have hereinafter set forth the results of conventional oxygen absorption tests and Chevrolet engine tests as applied to various of my improved lubricating oil compositions.

The advantages of my present invention with respect to oxidation and corrosion characteristics of my improved lubricating oil compositions are illustrated by their mean oxygen absorption rates as compared with the oxygen absorption rate of the base oil and the corrosion losses of bearing metal in contact with the respective lubricants.

For example, by the incorporation of 0.2% of the inhibitor of Example IV, the mean rate of oxygen absorption of the base oil A was reduced from 2.8 to 1.8 cc. per minute. By increasing the proportions of the inhibitor to 0.5%, the mean oxygen absorption rate was reduced from that of the base oil but to a less extent. The mean oxygen absorption rate of the base oil A in the presence of copper-lead bearing metal was 25.6 and, by the addition of 0.5% of the inhibitor of Example IV, the mean oxygen absorption rate was reduced to 2.7 cc. per minute. Further, the corrosion loss of the copper-lead bearing metal in the presence of the base oil was 5.8 milligrams while in the presence of the base oil compounded with 0.5% of the inhibitor of Example IV, no bearing corrosion loss was apparent.

The calcium iso-octyl salicylate and calcium capryl salicylate, previously noted as detergents, are themselves effective anti-oxidants over a considerable period. However, at the termination of their oxidation induction period, these calcium salts, in the presence of copper-lead bearing metal, have a tendency to act as pro-oxidants. This tendency to act as pro-oxidants upon the termination of a protective period is a characteristic of many of these so-called detergents. One aspect of my invention relates particularly to lubricating oil compositions including such detergents.

By compounding with such lubricating oil compositions a minor proportion of the inhibitor of my present invention, the pro-oxidant characteristics of these detergents, and especially the calcium salt detergents previously noted, following the induction period, is materially repressed and in effect the induction period is eliminated or substantially prolonged i. e. the oxygen absorption rate continues to rise gradually over a considerably prolonged period instead of a marked increase at the end of the induction period. The proportions of the inhibitor required to produce optimum results in this respect depend upon the characteristics of the particular detergent and of the inhibitor employed.

For example, the oxidation induction period of a 1% blend of the calcium salt of iso-octyl salicylate in base oil A was approximately 170 minutes. By the addition of 0.5% of the inhibitor of Example IV, the mean oxygen absorption rate, over a period of 518 minutes, was only 1.9 cc. per minute, the induction period being substantially eliminated. The oxidation induction period of the blend in the presence of copper-lead bearing metal was likewise substantially eliminated by the addition of 0.5% of the inhibitor of Example IV, and the mean rate of oxygen absorption was only 2.6 cc. per minute. The bearing corrosion loss of the blend in the absence of the inhibitor was 11 milligrams but, with 0.5% of the inhibitor included in the composition, no bearing corrosion loss was apparent. When the proportion of the inhibitor was reduced to about 0.3%, very little change in the induction period was observed. However, the bearing metal corrosion loss was

reduced from 11 milligrams to 6.8 milligrams by the inclusion of only 0.3% of the inhibitor.

Iron naphthenate is known to have strong pro-oxidant action for mineral oil. I have found that this pro-oxidant action of iron naphthenate is substantially repressed by the lubricating oil composition of my present invention. For example, the presence of iron naphthenate in an amount equivalent to 0.01% Fe_2O_3 was neutralized by the presence of 0.5% of this inhibitor, the mean rate of oxygen absorption being reduced from 45.6 to 3.5 cc. per minute.

The results of other oxidation and corrosion tests of my improved lubricating oil compositions compounded with various inhibitors described herein, as compared with the oxidation and corrosion characteristics of the base oil, are set forth in the following Table II. In these tests no detergent was present. These tests as well as those previously described were carried out at a temperature of 360° F. Copper-lead bearing metal was present in the oil during the respective tests. The identity of the particular inhibitor used has reference to the specific examples of my inhibitors previously set forth herein.

Table II

Test No.	Base oil	Inhibitor		Mean rate of O_2 absorption, cc./min./100 gms.	Bearing metal corrosion loss, mgs.
		Identity	Proportion, per cent		
1	A	None		25.6	5.8
2	A	Ex. IV	0.5	2.7	None
3	A	Ex. III	0.5	2.5	None
4	A	Ex. VI	0.5	2.8	None
5	A	Ex. V	0.5	2.6	None
6	A	Ex. VII	0.5	4.1	None
7	A	Ex. VIII	0.5	6.4	3.8
8	A	Ex. VIII	0.75	5.6	None
9	A	Ex. X	0.5	2.1	None

This base oil A compounded with 1% of the calcium salt of iso-octyl salicylate, previously referred to herein as a detergent, had an oxidation induction period of 107 minutes in the presence of copper-lead bearing metal and the corrosion loss of the bearing was 11 milligrams, as previously noted. By the addition of various inhibitors of my present invention in proportions as indicated in the following Table III, the oxidation induction periods and corrosion losses were as therein indicated. The identity of the particular inhibitor used has reference to the specific examples of my inhibitors previously set forth herein.

Table III

Test No.	Base oil	Inhibitor		Oxidation induction period, minutes	Bearing metal corrosion loss, mgs.
		Identity	Proportion, per cent		
1	A	Ex. IV	0.5		None
2	A	Ex. III	0.5		None
3	A	Ex. VI	0.5	163	None
4	A	Ex. V	0.5	183	1.6
5	A	Ex. VII	0.5	160	6.5
6	A	Ex. VIII	0.5	143	9.5
7	A	Ex. X	0.5	165	None

In tests #1 and #2 of Table III, the oxidation induction period was completely eliminated, i. e. the calcium of the detergent was completely deactivated by the inhibitor, and the mean oxygen absorption rates were, respectively, 2.3 cc. per minute and 2.9 cc. per minute. The results of tests #5 and #6 indicated that complete protection against corrosion was not obtained where only 0.5% of the inhibitor was used. However, these tests #5 and #6 were repeated using 0.75% of the respective inhibitors. In test #5, thus repeated, no corrosion loss was apparent while in test #6, thus repeated, the corrosion loss was only 0.1 milligram. In tests #3 to #7 inclusive, though the induction period was not completely eliminated, the rate of oxygen absorption progressively decreased toward the end of the tests indicating that calcium was being deactivated.

Tests of my improved lubricating oil compositions have consistently shown them effectively to inhibit the pro-oxidant characteristics of well-known oxidation catalysts such as iron naphthenate and copper-lead bearing metal as well as that of detergents such as previously noted following their respective induction periods.

The desirable characteristics of my improved lubricating oil compositions also appear from results of tests made in accordance with the procedure of the American Society of Testing Materials, published October 1942 and entitled "Proposed method of test for oxidation characteristics of heavy duty crankcase oils," conventionally known as the Chevrolet Engine Tests. These tests were run on the base oil alone and also on lubricating oil compositions of my present invention consisting of such base oil compounded with my inhibitors and various detergents.

Results, representative of such tests, are set forth in the following Table IV. In test #1, the base oil alone was used. The lubricating oil composition used in test #2 was prepared by compounding this same base oil with the indicated proportions of my inhibitor and of the detergent previously identified herein by its trade name "Aerolube B." In the compounding of the lubricating oil composition used in test #3, the same base oil was used together with the indicated proportions of my inhibitor and of the detergent previously identified herein as the calcium salt of capryl salicylate. The engine cleanliness rating, analysis of the lubricants at the end of the tests and the corrosion losses per half bearing of copper-lead bearing metal during the tests, were as indicated.

Table IV

	Test No.—		
	1	2	3
Base oil	B	B	B
Detergent, weight per cent	None	1.5	2.0
Inhibitor, do	None	0.4	0.9
Engine cleanliness rating	91	89	92
Used oil tests:			
Naphtha insol., per cent	1.31	0.93	0.29
$CHCl_3$ soluble, do	.44	.14	None
Neut. No.	1.7	0.50	0.28
Brg. metal corrosion loss, gms.	0.307	0.124	0.091

The acid number of the base oil was originally 0.05 and its sulfur content, 0.24%.

From the results of these tests, it appears that though the base oil had a high corrosion rate and a moderately high oil deterioration rate, the lubricating oil compositions prepared therefrom in accordance with my present invention show much lower corrosion losses and oil deterioration rates while the engine cleanliness ratings were not materially changed.

I claim:

1. A lubricating oil composition comprising a petroleum lubricating oil and a proportion effective to retard oxidation of the composition of an

inhibitor such as results from the reaction of a monohydroxy aliphatic alcohol with a condensation product of turpentine and phosphorus pentasulfide.

2. A lubricating oil composition comprising a petroleum lubricating oil and about 0.05% to about 2%, based on the weight of the oil constituent, of an inhibitor such as results from the reaction of a monohydroxy aliphatic alcohol with a condensation product of turpentine and phosphorus pentasulfide.

3. A lubricating oil composition comprising a petroleum lubricating oil and about 0.05 to about 1%, based on the weight of the oil constituent, of an inhibitor such as results from the reaction of a monohydroxy aliphatic alcohol with a condensation product of turpentine and phosphorus pentasulfide.

4. A lubricating oil composition comprising a petroleum lubricating oil, a calcium salt of an octyl salicylate as a detergent and from about 0.25 to about 2%, based on the weight of the oil constituent, of an inhibitor such as results from the reaction of a monohydroxy aliphatic alcohol with a condensation product of turpentine and phosphorus pentasulfide.

5. A lubricating oil composition comprising a petroleum lubricating oil and a proportion effective to retard oxidation of the composition of an inhibitor such as results from the reaction of a monohydroxy aliphatic alcohol containing not less than 5 carbon atoms per molecule with a condensation product of turpentine and phosphorus pentasulfide.

6. A lubrficating oil composition comprising a petroleum lubricating oil, a calcium petroleum sulphonate detergent and a proportion effective to retard oxidation of the composition of an inhibitor such as results from the reaction of a monohydroxy aliphatic alcohol of not less than 5 carbon atoms per molecule with a condensation product of turpentine and phosphorus pentasulfide.

7. A lubricating oil composition comprising a petroleum lubricating oil and a proportion effective to retard oxidation of the composition of an inhibitor such as results from the reaction of capryl alcohol with a condensation product of turpentine and phosphorus pentasulfide.

8. A lubricating oil composition comprising a petroleum lubricating oil and a proportion effective to retard oxidation of the composition of an inhibitor such as results from the reaction of normal hexyl alcohol with a condensation product of turpentine and phosphorus pentasulfide.

9. A lubricating oil composition comprising a petroleum lubricating oil and a proportion effective to retard oxidation of the composition of an inhibitor such as results from the reaction of lauryl alcohol with a condensation product of turpentine and phosphorus pentasulfide.

10. A lubricating oil omposition comprising a petroleum lubricating oil, a calcium derivative of alkylated salicylic acid as a detergent, and a proportion, effective to retard oxidation of the composition, of an inhibitor such as results from the reaction of a monohydroxy aliphatic alcohol of not less than 5 carbon atoms per molecule with a condensation product of turpentine and phosphorus pentasulfide.

ROBERT L. MAY.

CERTIFICATE OF CORRECTION.

Patent No. 2,392,252. January 1, 1946.

ROBERT L. MAY.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 1, second column, line 25, for "alkoxide" read --alkoxides--; page 2, first column, line 74, for "considrable" read --considerable--; page 3, second column, line 31, for "about" read --above--; page 6, first column, line 35, claim 6, for "lubrficating" read --lubricating--; and second column, line 26, claim 10, for "omposition" read --composition--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 9th day of April, A. D. 1946.

Leslie Frazer

(Seal) First Assistant Commissioner of Patents.

inhibitor such as results from the reaction of a monohydroxy aliphatic alcohol with a condensation product of turpentine and phosphorus pentasulfide.

2. A lubricating oil composition comprising a petroleum lubricating oil and about 0.05% to about 2%, based on the weight of the oil constituent, of an inhibitor such as results from the reaction of a monohydroxy aliphatic alcohol with a condensation product of turpentine and phosphorus pentasulfide.

3. A lubricating oil composition comprising a petroleum lubricating oil and about 0.05 to about 1%, based on the weight of the oil constituent, of an inhibitor such as results from the reaction of a monohydroxy aliphatic alcohol with a condensation product of turpentine and phosphorus pentasulfide.

4. A lubricating oil composition comprising a petroleum lubricating oil, a calcium salt of an octyl salicylate as a detergent and from about 0.25 to about 2%, based on the weight of the oil constituent, of an inhibitor such as results from the reaction of a monohydroxy aliphatic alcohol with a condensation product of turpentine and phosphorus pentasulfide.

5. A lubricating oil composition comprising a petroleum lubricating oil and a proportion effective to retard oxidation of the composition of an inhibitor such as results from the reaction of a monohydroxy aliphatic alcohol containing not less than 5 carbon atoms per molecule with a condensation product of turpentine and phosphorus pentasulfide.

6. A lubrficating oil composition comprising a petroleum lubricating oil, a calcium petroleum sulphonate detergent and a proportion effective to retard oxidation of the composition of an inhibitor such as results from the reaction of a monohydroxy aliphatic alcohol of not less than 5 carbon atoms per molecule with a condensation product of turpentine and phosphorus pentasulfide.

7. A lubricating oil composition comprising a petroleum lubricating oil and a proportion effective to retard oxidation of the composition of an inhibitor such as results from the reaction of capryl alcohol with a condensation product of turpentine and phosphorus pentasulfide.

8. A lubricating oil composition comprising a petroleum lubricating oil and a proportion effective to retard oxidation of the composition of an inhibitor such as results from the reaction of normal hexyl alcohol with a condensation product of turpentine and phosphorus pentasulfide.

9. A lubricating oil composition comprising a petroleum lubricating oil and a proportion effective to retard oxidation of the composition of an inhibitor such as results from the reaction of lauryl alcohol with a condensation product of turpentine and phosphorus pentasulfide.

10. A lubricating oil omposition comprising a petroleum lubricating oil, a calcium derivative of alkylated salicylic acid as a detergent, and a proportion, effective to retard oxidation of the composition, of an inhibitor such as results from the reaction of a monohydroxy aliphatic alcohol of not less than 5 carbon atoms per molecule with a condensation product of turpentine and phosphorus pentasulfide.

ROBERT L. MAY.

CERTIFICATE OF CORRECTION.

Patent No. 2,392,252. January 1, 1946.

ROBERT L. MAY.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 1, second column, line 25, for "alkoxide" read --alkoxides--; page 2, first column, line 74, for "considrable" read --considerable--; page 3, second column, line 31, for "about" read --above--; page 6, first column, line 35, claim 6, for "lubrficating" read --lubricating--; and second column, line 26, claim 10, for "omposition" read --composition--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 9th day of April, A. D. 1946.

Leslie Frazer

(Seal) First Assistant Commissioner of Patents.