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LUBRICANT

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1

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. 494,688, filed July 14, 1943, and since abandoned, I have described and claimed a novel class of materials resulting from the condensation of a terpene, such as present in turpentine, with a phosphorus sulfide such as P_2S_5 and P_2S_3 .

I have now discovered that certain of these materials, and especially the condensation products of turpentine and P_2S_5 , are particularly effective in repressing or inhibiting the deterioration of lubricating oil compositions and the corrosion of metal parts in contact therewith. The condensation products of turpentine and P_2S_3 have likewise been found effective in such lubricating oil compositions.

These condensation products are brittle, resinous solids which dissolve readily in mineral lubricating oils or in excess turpentine to form liquids. Such solutions are relatively viscous at high concentrations but the viscosity of the solution decreases rapidly as the proportion of the solvent is increased from 25% to 75%.

The condensation products of turpentine and P_2S_5 or P_2S_3 resulting from the use of about 30% or less of the sulfide in their production have been found to dissolve completely in lubricating oils. Those prepared using 33% to 40% or more of the sulfide have been found to contain matter not completely oil-soluble and, accordingly, are less desirable for use in lubricating oil compositions.

In accordance with my present invention, I have found the use of such turpentine- P_2S_5 condensation products as are prepared using 20% to 30% of P_2S_5 particularly advantageous. The combining equivalent of P_2S_5 with turpentine appears to be about 25% to 28% of the total weight of the reactants. When less P_2S_5 is used, some free turpentine remains in the product but may be distilled therefrom if desired. When more than about 28% of P_2S_5 is used, a part thereof remains as what appears to be an intermediate product, of high activity and relatively lower oil solubility, which would combine further with the turpentine if sufficient turpentine were present.

The lubricating oil composition of my present invention may consist solely of the lubricating oil constituent and the turpentine-phosphorus sulfide condensation product, the latter being hereinafter referred to as my inhibitor. However, my inhibitor has been found to be compatible with other desirable lubricating oil addends and the inclusion of such other addends is within the contemplation of my present invention and constitutes an important aspect thereof. The inclusion in internal combustion engine lubricants of addends of the type known as detergents has been found highly desirable. An especially effective lubricating oil composition, for the lubrication

2

tion 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 co-pending applications of Willard L. Finley, upon which United States Patents No. 2,347,547 and No. 2,339,692 were granted April 25, 1944, and January 18, 1944, respectively.

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

The proportion of the condensation product used in compounding my improved lubricating oil compositions may be varied somewhat but in any case only a minor proportion is used. In the absence of other addends, generally satisfactory results have been obtained by using proportions within the range of about 0.01% to about 0.5%, based on the weight of the lubricating oil constituent. In special cases, for example when calcium salt detergents are present, somewhat higher proportions, say up to about 1%, may be used with advantage. In gear lubricants, these turpentine- P_2S_5 condensation products have been found to increase the film strength of the lubricant and, for such purposes, proportions somewhat in excess of those previously noted may be used. However, these condensation products are acidic phosphorus derivatives and phosphorus acidity has been found to have a general tendency to cause polymerization and sludge formation in mineral lubricating oil. In internal combustion engine lubricants, where sludge formation must be minimized, the use of the condensation product in proportions exceeding 0.5% by weight, in the absence of detergents such as previously noted, is not generally advisable. However, proportions within the indicated range of about 0.01% to 0.5% have not been found to cause noticeable or objectionable sludging under such conditions. Proportions within the range of about 0.05% to 0.10% are particularly recommended in the preparation of my lubricating oil composition for use in internal combustion engines in the absence of a detergent.

3

For optimum results, when used in conjunction with one of the previously noted 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 may remain with its characteristic tendency toward 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 in the inhibitor, and may be determined for any particular set of conditions by simple tests.

The inhibitor used in the preparation of the lubricating oil composition of my present invention may be prepared as follows: The turpentine is heated to about 200° F. or slightly higher and then, without further heat, the phosphorus pentasulfide, preferably in powdered form, is slowly stirred into the turpentine. The reaction between turpentine and P_2S_5 is quite rapid and highly exothermic at temperatures above 200° F. If the reactants are admixed and heated to the reaction temperature, the resultant reaction is apt to become violent and uncontrollable. However, if the two reactants are slowly mixed together, the heat of the reaction may be dissipated and the temperature and rate of reaction controlled.

After all of the sulfide has been added, 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. The heating and stirring of the mixture should be continued until all of the P_2S_5 is dissolved which is usually accomplished in 2 to 4 hours. Though the inhibitor may be produced with reaction temperatures ranging from 200 to 400° F., there is a tendency toward the formation of insoluble by-products at temperatures as high as 400° F. Advantageously, the reaction temperature is maintained at from about 200° F. to about 300° F.

In the hereinafter related specific illustrations of my improved lubricating oil compositions, the particular inhibitors used were prepared in accordance with the previously described general procedure, the reaction conditions and the characteristics of the resulting inhibitor being as indicated in the following Table I:

Table I

Example.....	1	2	3	4
Reactants:				
Turpentine, weight per cent.....	90	80	75	75
P_2S_5 , weight per cent.....	10	20	25	25
Reaction Conditions:				
Temp. during mixing, ° F.....	200	200	250	250
Temp. during final stage, ° F.....	320	400	300	300
Duration of final stage, Hrs.....	2	3	3	3
Analysis of Product:				
Acid No.....	10.8	20	26.7	18.1
Saponification No.....	47.2	95.2	122.3	114.7
Weight Per Cent Phosphorus.....	2.81	5.7	6.94	6.95
Weight Per Cent Sulfur.....	6.82	14.1	18.3	18.4

The inhibitor of Examples 1 and 2 in which a substantial excess of turpentine was present were topped prior to use in my lubricating oil compositions by heating to a temperature of 250° F. under an absolute pressure of 2 millimeters of mercury. The final products of Examples 1 and 2 contain, respectively, 8.4% and 7.97% of phosphorus and 18.8% and 19% of sulfur, by weight. The acid number of Example 2 was 29.4 and its saponification number was 136.7.

To facilitate handling, the inhibitor may be obtained in solution by adding thereto, upon com-

4

pletion of the reaction, an equal weight of a light mineral oil, for instance a 100-pale oil. Characteristics of inhibitors used in the subsequent illustrations of my invention which were prepared in this manner together with the reaction conditions are set forth in the following Table II. In each instance the diluent used was an equal volume of 100-pale oil and the products were analyzed and used as 50% concentrates of the inhibitor in the oil.

Table II

Example.....	5	6	7	8
Reactants:				
Turpentine, Weight Per Cent.....	75	70	66.7	60
P_2S_5 , Weight Per Cent.....	25	30	33.3	40
Reaction Conditions:				
Temp. during mixing, ° F.....	250	250	250	250
Temp. during final stage, ° F.....	300	350	350	300
Duration of final stage, hrs.....	3	2	2	3
Analysis of Product:				
Acid No.....	13.4	20.7	29.6	67.5
Saponification No.....	57.6	83.4	95.5	139.2
Weight Per Cent Phosphorus.....	3.42	4.22	4.60	5.43
Weight Per Cent Sulfur.....	8.96	10.5	11.8	-----

The 100-pale oil used in the foregoing examples was a Gulf Coast neutral having the following characteristics:

Gravity, ° A. P. I.	22.1
Flash, ° F.	300
Fire, ° F.	350
Vis. at 100° F., S. U. S.	107.8
Vis. at 210° F., S. U. S.	38.4

The turpentine used in the preparation of the condensation products of the foregoing examples was a technical grade, steam-distilled wood turpentine comprising about 90% alpha pinene, a bi-cyclic terpene having the empirical formula $C_{10}H_{16}$. Gum spirits may likewise be used.

As the mineral oil constituent of my improved lubricating oil composition, various petroleum lubricating oil fractions may be used, for instance solvent-treated Mid-Continent neutral or a blend of such Mid-Continent neutral and bright stock or a solvent-refined lubricating oil fraction from a Pennsylvania crude. Characteristics of several 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 III in which oil A is a solvent-treated Mid-Continent neutral blended with 42% bright stock, oils B and E are unblended solvent-treated Mid-Continent neutrals, oil C is solvent-treated Pennsylvania S. A. E.-60 oil and oil D is a solvent-treated Mid-Continent S. A. E.-10 oil.

Table III

Base Oil.....	A	B	C	D	E
Gravity, ° A. P. I.....	26.5	27.1	28.4	29.5	26.9
Flash, ° F.....	425	470	540	405	490
Fire, ° F.....	485	525	610	480	545
Saybolt Viscosity at 100° F.....	628.1	448.6	1,526.7	249.6	556.7
Saybolt Viscosity at 210° F.....	67.5	57.7	121.7	49	62.6
Viscosity Index.....	86.7	80.0	106	92.7	79.2
Pour, ° F.....	10	5	10	5	0
Carbon Residue.....	.26	.04	.58	.039	.06
Ash.....	None	None	None	None	.004
Acid No.....	.05	-----	.025	.025	.075
Saponification No.....	-----	-----	.48	.16	-----
Color.....	6-	3½-	5-	2-	3½+
Sulfur, Percent.....	.26	.31	.11	.20	.28

As previously noted, the lubricating oil composition of my present invention may also contain addends in addition to my turpentine-phosphorous sulfide inhibitor, including various de-

5

tergents. Particularly desirable results are obtained by the inclusion, as detergents, of the previously-noted calcium salts of iso-octyl salicylate or of capryl salicylate. Other detergents which may be used with advantage include the barium phenolate of sulfurized diamyl phenol, 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"; a basic calcium detergent currently marketed under the trade name "C. M. 2A"; and various metallic soaps, metallic sulfonates, alcoholates or alkoxides and wax-alkylated salicylic acid salts.

For the purpose of further illustrating my present invention and the advantages derived therefrom, various samples have been prepared and subjected to tests including conventional Chevrolet engine tests and bus engine sludging tests.

In the Chevrolet engine tests, which were conducted in accordance with the procedure published by the American Society of Testing Materials in October 1942 entitled "Proposed Method of Test for Oxidation Characteristics of Heavy Duty Crankcase Oils," the lubricating oil composition being tested was placed in the crankcase of the test engine and the engine run for 36 hours at the specified temperature. At the end of the test period, the engine was examined for sludge and varnish deposits and the test rated for engine cleanliness, based on 100 for a clean engine. The lubricating oil, or lubricating oil composition, was tested after the run for naphtha insolubles, CHCl_3 solubility, neutralization number and viscosity rise, indicative of the extent of deterioration of the oil. Also, the loss in weight of the whole copper-lead bearing, due to corrosion during the test, was determined by carefully weighing the bearing before and after the test.

In the bus engine sludging test, the lubricating oil or lubricating oil composition was oxidized in the presence of bearing metal at 250° F., the oil being splashed against an iron cover maintained at 500° F., and inspections made at 45-hour intervals to observe oil deterioration, bearing corrosion and sludge and lacquer deposits in the test apparatus.

The results of the Chevrolet engine tests, using lubricating oil compositions of my present invention, of the indicated compositions and under the indicated test conditions, are set forth in the following Table IV, the identity of the base oil and of the inhibitor having reference to Tables I, II and III herein. For comparison, results of tests using the base oil alone are included.

Table IV

Test No.	1	2	3	4	5	6
Identity of Base Oil	A	A	B	B	B	B
Identity of Inhibitor	None	7	None	6	7	7
Percent of Inhibitor	None	0.1	None	.33	.50	.75
Detergent, Per Cent	None	None	None	.75	.75	.75
Oil Test Temp., °F.	265	265	280	280	280	280
Oil Charged Qts.	5	5	4	4	4	4
Engine Cleanliness Rating	85	84	86	91	91	88
Bearing/Corrosion Loss, gms./brg.	.253	.142	.809	1.006	.030	.138
Used Oil Tests:						
Naphtha Insoluble, mgs./10 gms.	80	109	152	154	44	68
CHCl_3 /Soluble, mgs./10 gms.	66	68	40	115	25	49
Neut. No. (Mgs. KOH)/gm. of oil	.65	.80	2.8	2.95	.35	.23
Vis. Rise (Sec. 100° F.)	234	364	195	282	4	-20

6

The detergent used in tests 4, 5 and 6 was the calcium capryl salicylate, previously noted.

From the foregoing tabulation, it appears in test 2 that, by the compounding of 0.1% of my inhibitor #7 with the base oil, the bearing corrosion loss was decreased from .253 gram to .142 gram. The advantages of the use of a detergent and the use of a sufficient proportion of my inhibitor therewith is clearly illustrated by tests 4, 5 and 6. In test 3, using the base oil alone, the oil rapidly deteriorated, as shown by an engine cleanliness rating of 86, a high bearing corrosion loss, high naphtha insoluble and high neutralization number. Test 4 indicates that, in the presence of 0.75% of the detergent, 0.33% of the inhibitor is insufficient to inhibit corrosion and oil deterioration. However, as shown by test 5, by increasing the proportion of inhibitor to 0.5%, the bearing corrosion loss and deterioration of the oil were materially diminished without affecting the sludge and varnish deposit within the engine.

The advantageous characteristics of my improved lubricating oil composition are further illustrated by the results of bus engine sludging tests of a single solvent-refined Pennsylvania S. A. E.-60 oil, previously identified herein as base oil C, and the same oil compounded with various proportions of my inhibitor, herein designated #7. The compositions and characteristics of these various lubricants, prior to the test and after a 180-hour test period together with bearing corrosion losses, are presented in the following Table V:

Table V

Test No.	1	2	3	4
Inhibitor, Per cent	None	0.05	0.075	0.1
Test on Fresh Lubricant:				
Vis. at 210° F., S. U. S.	121.7	121.0	121.0	121.9
Neutralization No., Mgs. KOH per gm. oil	0.025	0.025	0.05	0.05
After 180 Hr. Test:				
Vis. at 210° F., S. U. S.	177.7	147.5	142.9	130.3
Neutralization No.	5.0	1.0	0.3	0.3
Sludge Deposit on Roof, gms.	25.7	4.7	2.7	2.2
Cu-Pb Bearing Corrosion Loss, Mgs.	214	37	17	2

The total bearing area in these tests was 26.81 square centimeters.

The results of bus engine sludging tests of the Mid-Continent S. A. E.-10 oil, herein identified as base oil D, and of said oil compounded with a detergent and various proportions of the indicated inhibitor of my invention, are presented in the following Table VI together with the duration of the test in which the results were obtained.

Table VI

Test No.	1	2	3	4	5	6
Identity of Inhibitor	None	None	#3	#3	#4	#4
Percent Inhibitor	None	None	0.2	0.3	0.5	0.75
Detergent Percent	None	0.75	0.75	0.75	0.75	0.75
Test on Fresh Lubricant:						
Vis. at 210° F., S. U. S.	49.0	49.5	49.2	49.2	49.4	49.5
Neutralization No.	0.025	B 2.3	B 1.2	B 0.5	B 0.5	B 0.5
After Test Period, Hrs.	90	90	180	90	270	270
Vis. at 210° F., S. U. S.	66.6	61.8	62.4	51.6	53.1	51.7
Neutralization No.	6.7	5.4	2.7	0.5	1.2	0.8
Cu-Pb Bearing Corrosion Loss, Mgs.	197	575	55	1	0.5	None

The detergent used in the above lubricating oil compositions was the calcium salt of iso-octyl salicylate and each of the compositions containing the detergent showed basic reaction prior to use.

The results of tests recorded in Tables V and VI show that the increase in viscosity and in the neutralization number of the oil and the amount of sludge deposit and bearing corrosion loss occasioned thereby were materially decreased by the incorporation of the inhibitor of my present invention.

The advantages derived from my present invention with respect to the oxidation characteristics of my lubricating oil compositions are illustrated by their oxygen absorption rates. For example, by the incorporation of 0.15% of my inhibitor, previously identified as #1 in the D base oil having a mean oxidation rate of 2.8 c. c. per minute at 360° F., the mean oxidation rate was reduced to 1.4 c. c. per minute. The mean oxidation rate of this base oil alone, in the presence of Cu-Pb bearing metal catalyst at 360° F., was 25.6 c. c. per minute and, by the incorporation of 0.15% of this inhibitor, the mean oxidation rate was reduced to 10 c. c. per minute. The oxidation induction period of this same base oil, compounded with 1% of the calcium salt of iso-octyl salicylate, was increased from 167 minutes to 198 minutes by the inclusion of 0.15% of this inhibitor and was further increased to 268 minutes by the incorporation therein of 0.2% of this inhibitor. Further, the catalytic effect of iron naphthenate on the oxidation rate of this base oil was materially suppressed when compounded in accordance with my invention. For instance by the incorporation of 0.15% of this inhibitor, the mean oxidation rate in the presence of iron naphthenate was reduced from 45.6 to 28.5 c. c. per minute.

By the incorporation of 0.2% of my inhibitor, herein designated #4, in base oil D, the mean oxidation rate of the base oil at 360° F. was reduced from 2.8 to 2.0 c. c. per minute. The catalytic action of Cu-Pb bearing metal was completely neutralized, the mean oxidation rate being reduced from 25.6 to 1.9 c. c. per minute and, instead of a bearing corrosion loss of 5.8 mgs., no corrosion loss whatever was detected. Further, the mean oxidation rate in the presence of iron naphthenate, which normally acts as a pro-oxidant, was reduced from 45.6 to 4.8 c. c. per minute. Also, in a lubricating oil composition of this base oil compounded with 1% of calcium iso-octyl salicylate, the inclusion of 0.2% of this inhibitor resulted in an increase of the oxidation induction period from 167 minutes to 352 minutes and, even in the presence of Cu-Pb bearing metal, the induction period was increased from 107 minutes to 140 minutes.

The calcium iso-octyl salicylate and calcium capryl salicylate 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 Cu-Pb 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-oxidation 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, as shown by the re-

sults just described. 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, in a 1% blend of the calcium salt of iso-octyl salicylate in base oil D, tested at 360° F., I have found that between 0.6% and 1% of my inhibitor #5 was required for the elimination of the induction period. By the use of 1% of this inhibitor, the calcium salt was completely de-activated but, with 0.6% of the inhibitor, the induction period was still evident. Where my inhibitor #6 was used, slightly more than 0.6% was required for complete deactivation, while 0.4% or less of the inhibitor was relatively ineffective in deactivating the calcium. Using my inhibitor #8, the calcium of the iso-octyl salicylate was completely deactivated by 0.3% while 0.2% was found to be insufficient in this respect.

The presence of various metals, including iron and copper, has been found to tend to promote oxidation of the base oil. By the use of my improved lubricating oil compositions, such metals in contact with the lubricant may be substantially de-activated. This is illustrated by tests in which wire coils of the metal were immersed in the lubricant. Results of such tests, carried on at 360° F. on a lubricating oil composition comprising 0.15% of my inhibitor #3 in base oil D are presented in the following Table VII. Similar tests on the base oil without the inhibitor are included for comparison. The results are reported as mean oxygen absorption rate in c. c. per minute per 100 grams of oil.

Table VII

	Without Inhibitor	With Inhibitor
Base Oil.....	2.8	1.3
Base Oil + Iron Coil.....	10.5	2.0
Base Oil + Copper Coil.....	24.4	5.1
Base Oil + Iron-Copper Coil.....	15.8	

In the last of these tests coils of iron and copper were used welded together so as to make electric contact between them.

The effectiveness of the lubricating oil composition of my present invention in inhibiting the pro-oxidation action of detergents other than the calcium salts of iso-octyl salicylate and of capryl salicylate is illustrated by oxidation absorption tests of base oil E having compounded therewith 1.5% of the previously referred to "Aerolube B." The oxidation induction period of this compound was found to be 53 minutes after which the rate of oxygen absorption became even more rapid than that of the base oil alone in the presence of Cu-Pb bearing metal. However, by compounding therewith 0.5% of my inhibitor #1, the induction period was eliminated and the oxidation of the compound proceeded at a uniform rate much slower than the rate of oxidation of the base oil in the presence of Cu-Pb bearing metal, indicating that my inhibitor is effective not only against the pro-oxidant effect of the Cu-Pb bearing metal but also prevents the barium of the detergent from becoming pro-oxidant.

My invention has herein been illustrated specifically by lubricating oil compositions in which my inhibitor was prepared from turpentine and P₂S₅. It is to be understood that lubricating oil compositions comprising the condensation product of turpentine and P₂S₃ are also contemplated

by my invention. Such lubricating oil compositions have been found to have properties quite similar to those of the lubricating oil compositions previously described.

For instance, the condensation product of 70% turpentine and 30% P_2S_3 , prepared at a reaction temperature of 300° F. and having an acid number of 60.7, a saponification number of 191.6 and containing 8.79% phosphorus and 17.8% sulfur, has been effective blended with base oil D and 1% calcium iso-octyl salicylate and 0.6% of this inhibitor has been found completely to deactivate the calcium salt.

I claim:

1. A lubricating oil composition comprising a petroleum lubricating oil and a proportion, sufficient to repress oxidation of the oil composition, of an oil soluble phosphorus- and sulfur-containing reaction product obtained by reacting, at a temperature greater than about 200° F., turpentine and a phosphorus sulfide of the class consisting of P_2S_5 and P_2S_3 .

2. A lubricating oil composition comprising a petroleum lubricating oil and from about 0.01% to about 0.5%, based on the weight of the oil constituent, of an oil soluble phosphorus- and sulfur-containing reaction product obtained by reacting, at a temperature greater than about 200° F., turpentine and a phosphorus sulfide of the class consisting of P_2S_5 and P_2S_3 .

3. A lubricating oil composition comprising a petroleum lubricating oil and a proportion, sufficient to repress oxidation of the oil composition, of an oil soluble phosphorus- and sulfur-containing reaction product obtained by reacting, at a temperature greater than about 200° F., turpentine and phosphorus pentasulfide in the preparation of which reaction product the amount of the phosphorus pentasulfide present was equal to 20% to 30% of the total weight of the reactants.

4. A lubricating oil composition comprising a petroleum lubricating oil and from 0.05% to 0.1%, based on the weight of the oil constituent, of an oil soluble phosphorus- and sulfur-containing reaction product obtained by reacting, at a temper-

ature greater than about 200° F., turpentine and phosphorus pentasulfide.

5. A lubricating oil composition comprising a petroleum lubricating oil, a detergent of the class consisting of the calcium salt of iso-octyl salicylate and the calcium salt of capryl salicylate and a proportion, sufficient to repress oxidation of the oil composition, of an oil soluble phosphorus- and sulfur-containing reaction product obtained by reacting, at a temperature greater than about 200° F., turpentine with a phosphorus sulfide of the class consisting of P_2S_5 and P_2S_3 .

6. An improved mineral-oil composition comprising a viscous mineral oil and in admixture therewith a minor proportion, sufficient to stabilize said oil against oxidation, of a phosphorus- and sulfur-containing reaction product obtained by reaction of a phosphorus sulfide and a material selected from the group consisting of a dicyclic terpene and an essential oil predominantly comprised of a dicyclic terpene, at a temperature greater than about 100° C.

7. A lubricating oil composition as defined in claim 5 in which the phosphorus sulfide employed in the preparation of the phosphorus- and sulfur-containing reaction product is phosphorus pentasulfide.

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REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,844,400	Kobbe	Feb. 9, 1932
1,926,687	Palmer	Sept. 12, 1933
1,963,084	Gardner	June 19, 1934
2,043,961	Kaufman	June 9, 1936
2,111,882	Borglin	Mar. 22, 1938
2,125,218	Borglin	July 26, 1938
2,223,130	Prutton	Nov. 26, 1940
2,242,260	Prutton	May 20, 1941
2,262,813	Morway	Nov. 18, 1941
2,320,228	Frey	May 25, 1943