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LUBRICANT CONTAINING DI-ISO-BUTYL PHENOL

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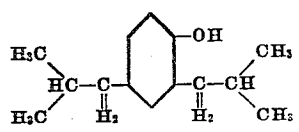
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4 Claims. (Cl. 87—9)

This invention relates to improved mineral oils and more particularly to mineral oils having an anti-sludging agent incorporated therein to obtain marked resistance of the oil to sludging and discoloration when the oil is subjected to oxidizing conditions, said improvement agent comprising di-iso-butyl phenol, and to methods of making such improved lubricants, all as more fully hereinafter set forth and as claimed.

It is a well established fact in the petroleum art that oils containing sulfur, nitrogen, and related components show a tendency to sludge and discolor when subjected to oxidizing conditions. This is true whether the said constituents occur naturally in the mineral lubricating oil, as is the case especially with some naphthenic base lubricants, or whether sulfur, nitrogen, chlorin, phosphorus and/or related compounds are artificially introduced for specific purposes as in the case of improved lubricants. It has been observed that improvement agents which are most effective for mineral lubricating oils and most economically employed to impart desired anti-corrosive and extreme pressure properties, while having no deleterious effect on the other original properties of the lubricating oil base, have been found to increase the tendency of the oil to sludge and discolor upon oxidation under extreme conditions of use. An object of my invention is to provide an improvement agent which will in no way alter the desirable properties of the original lubricant, but which, when incorporated with a mineral lubricating oil, will inhibit the tendencies of the improved oils to sludge and discolor when the lubricant is subjected to oxidizing conditions at high temperature and extreme pressure for a relatively long period of time. I have found, and herein disclose and claim, a highly effective improvement agent, which, when added to a mineral oil, as for example to improved mineral lubricating oil containing one of the aforesaid improvement agents, will overcome the aforementioned difficulties without adversely affecting the desirable properties of the oil.

The improvement agent of my invention is di-iso-butyl phenol. It is a chemical compound, having the following structural formula:



All of the various isomers appear to be effective and my invention comprehends the use of the various isomers either singly or in mixture with one another.

Di-iso-butyl phenol is a white crystalline solid, soluble in mineral oils to the extent of 0.50% by weight.

My invention is directed to the use of this compound as an anti-sludging agent for hydrocarbon oils, particularly for mineral lubricating oils. Mineral oils that develop a tendency to sludge and to discolor when used under oxidizing conditions such as occur at high temperature for long periods of time, when blended with a very small amount of di-iso-butyl phenol, show marked resistance to these undesired phenomena, with no deleterious effect upon the desirable properties of the oil. Resistance to color change is particularly marked with oils containing as little as 0.10 per cent by weight of di-iso-butyl phenol.

The improvement agency of my invention is readily incorporated in a mineral oil. The ordinary equipment and usual procedure employed in blending oils will suffice for incorporating this compound in any oil. Since heat lowers the viscosity of oils, solution of the compound in the selected oil is facilitated by heating the oil somewhat. I have found that heating the oil to 160° to 180° F., and adding thereto the required amount of di-iso-butyl phenol while blowing the mixture with a current of air for one hour is an advantageous procedure for blending the improvement agent with a selected oil. The improvement agent may be added all at one time and the mixture subsequently well agitated. The required proportion of di-iso-butyl phenol to lubricating oil is variable, being dependent upon the character of the oil to which it is added. The less stable mineral lubricating oils require higher percentages of di-iso-butyl phenol, the proportions ordinarily being from 0.02 per cent to 0.25 per cent by weight of the improvement agent to inhibit the ordinary tendencies to sludge and to discolor, although somewhat more is sometimes necessary.

The following examples are presented to illustrate the properties of improved lubricants prepared in accordance with my invention, comprising a major proportion of mineral lubricating oil and a minor proportion of di-iso-butyl phenol as the anti-sludging agent.

The data recorded below were obtained to determine the properties of improved lubricants comprising a major proportion of mineral lubricating oil, or a mineral lubricant base in which some anti-corrosive agent has been incorporated and a minor proportion of di-iso-butyl phenol to inhibit sludging and discoloration. Three samples of mineral lubricating oil were subjected to oxidation in the following corrosion test: A new cadmium-silver bearing shell was immersed in 300 cc. of mineral lubricating oil for 48 hours and throughout that period the oil was maintained at 347° F. (175° C.) and 2000 cc. of air per hour was bubbled through the heated oil and against the surface of the bearing. The first sample of oil, Number 1, was a highly refined paraffinic

base Pennsylvania motor oil. The second sample of oil, sample Number 2, was subjected to the same corrosion test. This sample Number 2 was an improved lubricating oil consisting of principally the same oil as sample Number 1, together with a small percentage of one improvement agent which has been found exceeding efficacious in inhibiting corrosion and in enhancing the lubricating value of the oil under extreme pressure conditions but which improvement agent has one deficiency in that when it is subjected to oxidizing conditions at high temperature and under extreme pressure for a long period of time it has the effect of increasing the rate of generation of sludge in the oil. Increased sludging with this particular anti-corrosion agent was also accompanied by discoloration of the oil, and these facts have been noted with respect to a number of other highly efficacious improvement agents for lubricating oils. A third sample, sample Number 3, consisting only of mineral lubricating oil and di-iso-butyl phenols was subjected to the same corrosion test to determine if di-iso-butyl phenol, when incorporated in a mineral oil, had any deleterious effects on the original oil. A fourth sample, sample Number 4, was an improved lubricant consisting of a portion of the same stock of oil as sample 1, 0.25 per cent of the anti-corrosion agent used in sample Number 2, and 0.10 per cent of di-iso-butyl phenol. The following results are tabulated for this study:

Composition	Sample 1	Sample 2	Sample 3	Sample 4
Pa. motor oil SAE 10 grade, percent weight	100	99.90	99.75	99.65
Anti-corrosion agent, percent weight	0	0.00	0.25	0.25
Di-iso-butyl phenol, percent weight	0	0.10	0.00	0.10
Cd-Ag bearing shell, weights in grams:				
Before test	27.7875	27.6667	27.4835	27.3975
At end of test	27.3670	27.5263	27.4830	27.3970
Total loss	−0.4205	0.1404	.0005	0.0005
Appearance after test	Severely corroded	Etched	Good	Good
Lubricant, after test: sludge, percent naphtha-insol. material	0.10	0.05	0.26	0.05

Samples 1, 2, 3, and 4 were all prepared with a highly refined Penna. SAE 10 grade mineral lubricating oil. Samples 5, 6, and 7 were prepared with a solvent refined Mid-Continent oil of SAE 20 grade and these samples compare, respectively, with samples 1, 3, and 4.

Composition	Sample 5	Sample 6	Sample 7
Mid-continent motor oil SAE 20 grade, percent weight	100	99.75	99.65
Anti-corrosion agent, percent weight	0	0.25	0.25
Di-iso-butyl phenol, percent weight	0	0	0.10
Co-Ag bearing shell weights in grams:			
Before test	27.5834	27.7876	27.8453
At end of test	27.3485	27.7821	27.8457
Total loss	.2749	0.0055	+0.0004
Appearance after test	Severe corrosion and pitting	Good	Good
Lubricant, after test: sludge, percent naphtha-insol. material at end of test	0.26	0.47	0.16

An inspection of physical properties was made before the oxidation test on each of the samples shown above to demonstrate that the addition of these improvement agents to a mineral lubricating oil does not adversely affect the physical properties of the oils to which they are added. The physical properties of the various samples were as follows:

Composition	Sample 1	Sample 2	Sample 3	Sample 4
Pa. motor oil SAE 10 grade; percent weight	100	99.75	99.90	99.65
Anti-corrosive agent, percent weight	0	0.25	0	0.25
Di-iso-butyl phenol, percent weight	0	0	0.10	0.10
Inspection				
Gravity, API	33.1	32.7	33.0	32.6
Viscosity, SUV:				
100° F	180	180	179	180
130° F	98	98	98	98
210° F	46.2	46.0	45.0	46.0
Flash, O. C.: °F	420	415	420	415
Fire, O. C.: °F	490	490	490	490
Pour, °F	0	0	0	0
Color, NPA	1.25	1.25	1.25	1.25
Carbon residue	.02	.02	.02	.02
Copper strip test	Satisfactory	Satisfactory	Satisfactory	Satisfactory

Composition	Sample 5	Sample 6	Sample 7
Mid-Continent motor oil SAE 20 grade; percent weight	100	99.75	99.65
Anti-corrosion agent, percent weight	0	0.25	0.25
Di-iso-butyl phenol, percent weight	0	0	0.10
Inspection			
Gravity, ° API	28.1	28.0	28.1
Viscosity, SUV:			
100° F	296	299	303
210° F	51	51.6	51
Flash, O. C.: °F	415	420	425
Fire, O. C.: °F	480	485	490
Pour, °F	0	+5	0
Color, NPA	3.50	3.75	3.75
Carbon residue	.12	.10	.07
Copper strip test	Satisfactory	Satisfactory	Satisfactory

Di-iso-butyl phenol is not highly effective as an anti-corrosion agent for preventing the destructive effect of lubricating oils on the newer bearing metals, but is highly effective as an anti-sludging agent for oils that have a tendency to sludge. Almost all corrosion inhibitors are compounds containing sulfur, nitrogen, phosphorus, or chlorin, some of which compounds frequently tend to produce sludge despite their marked and measurable influence upon the anti-corrosive and extreme pressure properties of mineral lubricating oils. Few organic compounds retain their original properties for any length of time when maintained under servere conditions of temperature and aeration and the newer laboratory tests advocated for evaluating the behavior of bearing alloys in aerated oils at elevated temperatures and under high pressure provide evidence of the fact that improved lubricants sometimes develop more sludge than is considered permissible. Development of excessive sludge occurs particularly when improved lubricants are subjected to some of the various test procedures currently proposed for evaluating corrosion inhibitors for extreme pressure lubricants. It is especially difficult to produce an improved lubricant which will satisfy all of the various corrosion tests in current use and still have the desired low sludging tendency. Di-iso-butyl phenol, as set forth herein, is peculiarly adapted for use as an auxiliary improvement agent with those improved lubricating oils which show special tendency to sludge and to discolor during use, and di-iso-butyl phenol is likewise highly efficacious in reducing the sludging tendency of those mineral lubricating oils which do not contain other improvement agents, as well

as other oils such as transformer oils, switch oils and the like.

Recently there have been developed improved lubricants containing certain improvement agents, which are particularly suitable for lubricating alloy bearings under relatively high temperatures, speeds and pressures without destructive action on the bearing surfaces. Due to the presence of the improvement agents employed, those new lubricants withstand drastic service conditions for relatively long periods, before they fail. I have found that when they fail, the primary cause is due to the formation of sludge. Thus with such improvement agents present, sludging is encountered with lubricating bases which without said improvement agents would fail from other causes before sludging occurs; this sludging being encountered because of the longer period of usefulness of such improved lubricants. By the present invention, those lubricants may be further improved and their utility extended for a much longer time. In applications Serial Nos. 98,826; 98,827; 99,156; 83,761; 83,760, now issued, respectively, as Patents Nos. 2,134,432, and 2,134,433 of October 25, 1938; 2,109,463 of March 1, 1938 and 2,145,971 and 2,145,970 of February 7, 1939, and 84,974 (filed by myself and one James O. Turner); and Serial No. 84,386 (filed by myself and one Herschel G. Smith) such improved lubricants are disclosed and claimed and I have found that the improved lubricants there described may be further improved by the addition of di-iso-butyl phenol. In those lubricants, various organic thio-ethers, organic poly-sulfides, alkoxy-hydroxy benzenes and sulfo-chlorinated monoesters of fatty acids are employed as the said improvement agents. Thus mineral lubricating oils containing any one of the following compounds as the improvement agent may be improved by adding the amounts of di-iso-butyl penhol given ante:

Alkyl thio-ethers of ortho-nitro benzene.
Alkyl thio-ethers of ortho-amino-benzene.
Alkyl thio-ethers of alpha-anthraquinone.
3-alkoxy-4 hydroxy benzaldehydes.
Iso-eugenol.
Diamyl poly-sulfides.
Sulfo-chlorinated sperm oil and like monoesters of fatty acids of the oleic series.

For instance, mineral lubricating oils containing small amounts, say a fraction of a per cent of alkyl thio-ethers, such as amyl thio-ethers of ortho-nitro-benzene, of ortho-amino-benzene or of the alpha-anthra-quinone, may be improved by the addition of from 0.02 to 0.25 per cent of di-iso-butyl phenol to obtain lubricating compositions improved as to sludging. Mineral compositions containing similar amounts of vanillin or iso-eugenol may be likewise improved as to their sludging characteristics. The addition of a similar amount of di-iso-butyl phenol, to improved lubricants containing diamyl-poly-sulfide, such as tri- and tetra-sulfides, or sulfo-chlorinated sperm oil, effects like improvements in their sludging characteristics.

What I claim is:

1. As an improved lubricant, the lubricating composition comprising a major amount of mineral lubricating oil, a fraction of a per cent of an alkyl thioether and from 0.02 to 0.25 per cent of a di-iso-butyl phenol, said composition having improved resistance to sludging.

2. A lubricating oil having improved extreme pressure and sludging characteristics comprising a mineral oil having added thereto a small amount of an alkyl thioether and 0.01 to 0.5 per cent of a di-iso-butyl phenol.

3. A lubricating oil having improved extreme pressure and sludging characteristics comprising a mineral oil having added thereto a small amount of an alkyl thioether of a nitrogen substituted benzene and 0.01 to 0.5 per cent of a di-iso-butyl phenol.

4. A lubricating oil having improved extreme pressure and sludging characteristics comprising a mineral oil having added thereto a small amount of an alkyl thioether of ortho-nitrobenzene and 0.01 and 0.5 per cent of a di-iso-butyl phenol.

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