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LUBRICANT

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This invention relates to an improved lubricant, more specifically to a lubricant of the type adapted for carrying extremely heavy loads such as are encountered in automotive transmission equipment and other similar heavy duty devices. The invention will be fully understood from the following description.

During the last few years there has been an increasing demand for transmission oils capable of withstanding heavy tooth and bearing pressures. In the automotive field the demand is becoming more insistent due to the greater use of worm and helical gear transmission. Leaded compounds have been used for this service, but there is great difficulty in obtaining a suitable compound capable of withstanding the heavy loads and which will not settle out or tend to harden and resinify. The tendency to drop out is especially to be noted where an oil base stock of high quality is used. For example, relatively stable oils can be produced in base stocks of low viscosity index, but if high viscosity index oils are used the difficulty of producing a suitable oil becomes much greater. Naturally occurring hydrocarbon lubricating oils have viscosity indices up to about 110 V. I. and the oils of this range cannot at present be used for making lead compounds. Almost all of the present leaded compounds on the contrary show viscosity indices of 50 or below, but it is the object of the present invention to produce oils of at least 80 V. I. and adapted to transmission service. Even oils with viscosity indices above 110 may be used for the present invention, such as are obtained in synthetic oils prepared by condensation of cracked waxes in presence or absence of aromatic hydrocarbons.

It has been found that active leaded compounds with viscosity indices equal to or better than those of Pennsylvania oils can be made using lead oleate, stearate or other fatty acids of which the oleate is at the present time the cheapest and most convenient of the lead compounds, if a sufficient amount of a second or a solubilizing compound is present. Among the solubilizing compounds are noted the naphthenic compounds, which are compounds of acids found naturally occurring in certain oils such as those obtained from Russia, Roumania, California and some of the Gulf Coast oils. Another class of solubilizing compound is the sulfonic type which is obtained by vigorous treatment of petroleum lubricating oil fractions with highly concentrated or fuming sulfuric acid, for example, as obtained in the preparation of highly refined "white oils" which are familiarly used for medicinal and technical purposes.

The lead compound and the solubilizing substances may be prepared in any preferred manner, for example, the acids, oleic and naphthenic as an illustration, may be added and the required quantity of lead oxide or litharge added to this mixture and heated to such temperature as to bring about the chemical combination. It is preferable to use an excess of the lead oxide and to separate that portion which does not react and which is therefore insoluble. Another method of producing the compound is to mix sodium or other salts of the oleate and sulfonate, for example, and to react with lead acetate or nitrate, at an elevated temperature so as to produce the corresponding lead compounds and to remove the sodium acetate or nitrate, as the case may be. It is also possible to separately produce the lead oleate and the naphthenic or sulfonic acids which include the acid, lead and other oil soluble salts of said acid, and to mix these materials with the oil base stock.

The exact nature of the solubilized ingredients, that is to say, the relation of the solubilizing materials to the normal oleates is not clearly understood, but it is believed that a double salt probably results especially in the case where the acids or their sodium salts are mixed and reaction with a lead compound is then conducted, but it should be understood that the present invention is not to be limited to that particular method of preparation. The relative proportions of the lead oleate or other insoluble compound and the solubilizing compound may vary within limits and the most favorable proportion depends on the particular base stock used. In general, if base stocks of higher viscosity index are employed, it is desirable to increase the proportion of the naphthenic or sulfonic solubilizing agent in proportion to the amount of the suitable lead salt. Ordinarily the two compounds may be used in proportion of one-third to one part of the solubilizing agent to one part of the lead oleate. If lead salts are prepared separately and then mixed, it is preferable to use more of the solubilizing agent than where the acids or their sodium salts are mixed and then cooked with litharge or some inorganic lead salt which presumably produces the double salt. The final composition should contain from 5 to 20% of the lead compound.

The base stock used is preferably a heavy residual, for example, a bright stock or a cylinder

oil. Pennsylvania or equivalent oils which have been raised to that grade by solvent extraction, hydrogenation, destructive hydrogenation or other processes of this type may also be used. In short, as a base stock it is preferred to use heavy residuals of a predominantly and strongly paraffinic structure as indicated by a viscosity index of 80 to 85, or even above 95, and with a viscosity upwards of 75 Saybolt viscosity at 210° F., as high as may be necessary for the particular service. The particular viscosity used depends naturally on the service to which the oil is adapted.

Another ingredient of the present composition is the sulfur, by which is meant free sulfur or organic sulfur, preferably in a "corrosive" form and by this latter term it is intended to include free sulfur and those oil soluble compounds of sulfur which will discolor a bright copper strip which is immersed therein. It is not necessary to use vigorously corrosive compounds such as will corrode metal gears or containers. If a base stock should contain an effective quantity of sulfur no more need be added, but insofar as applicants are aware, none of the predominantly paraffinic crudes contain a sufficient quantity of sulfur and furthermore such sulfur as is present is non-corrosive so that it is in all cases desirable to add "corrosive sulfur."

The "corrosive" compounds should be sufficiently high boiling so as not to be lost by evaporation, and some crude oils contain relatively large amounts of such compounds in natural form, particularly those oils obtained from the West Texas fields. Cuts derived from such crudes rich in sulfur may be added to the base stocks so as to obtain a proper amount of "corrosive sulfur". Pure compounds may also be used, for example, high molecular weight mercaptans, polysulfides—preferably the trisulfide or higher—and the like, as well as free sulfur itself which may be added in small amounts. Olefines, for example obtained from cracked wax, or vegetable, mineral and marine oils, may also be sulfurized by cooking with free sulfur or sulfur chloride and sulfurized terpenes are also employed. The necessary amount of "corrosive sulfur" is from .3 to 1.0% depending on the service and should be determined by the difference between total sulfur before and after heating with a finely divided metal such as copper which gives the amount of truly "corrosive sulfur". More corrosive sulfur may be added if desired say up to 3 or even 5% but this shows little advantage over 1 or 1.5%. Non-corrosive sulfur is not nearly so effective for the present purpose as the "corrosive" variety and in providing the proper amount of corrosive sulfur only that portion which is truly "corrosive" should be taken into account. It will be understood that corrosive selenium and tellurium compounds may be used along with or in place of the sulfur. While the leaded compounds above show some extreme pressure qualities themselves, this is feeble at best without the addition of sulfur. In addition to the lead compounds, stabilizers and sulfur compounds, there may also be added materials of the "oiliness" increasing class, by which is meant fatty oils, acids, or esters which increase the lubricating value of the compounds. These materials may be added in relatively small amounts, say 2 to 5%. The following examples illustrate the composition of the properties of our improved lubricants:

Example 1

To 100 parts by weight of a base stock of 110 sec. Saybolt viscosity at 210° F. and 100 V. I., is added 11 parts of a mixture of lead oleate and lead naphthenate in equal proportions. To this is added 6% of a sulfurized pine oil, 5% of "corrosive sulfur". The final mixture is the following composition:

Gravity	20.2
Viscosity at 100° F.—sec. Saybolt	1290
Viscosity at 210° F.—do	99.8
Viscosity index	93
Percent lead	3.1
Percent sulfur (corrosive)	.3

The above oil is found to have excellent extreme pressure characteristics when tested in the well known Almen machine constructed and operated according to the procedure described in National Petroleum News, November 16, 1933. The oil was found to carry the full 15 weights as described in the above. The friction curve was smooth throughout and the final friction value was 43. The oil temperature during the test did not rise above 150° F. and the condition of the test pin after the test was excellent. In comparison with the above, the base oil alone was run without the addition of either the lead compounds or the sulfur. In the Almen test the oil carried only 5 weights and the pin was sheared.

Example 2

To the base oil used in Example 1 is added 10 parts of a compounding agent made by adding sodium oleate and sodium sulfonate in weight proportion of 2 to 1 and precipitating the lead compound with an excess of lead acetate. About 30% of a sulfur compound made by heating 2 parts of chlorinated paraffin wax (25% Cl.) with 1 part of sodium polysulfide is then added. The sulfur compound should have an analysis of 2.4% total sulfur and .75% "corrosive" sulfur. The finished oil was stable and had a viscosity index of substantially 100.

The present application is a continuation in part of a prior application Serial No. 710,720 filed February 10, 1934.

The present invention is not to be limited to any theory of the extreme pressure characteristics nor to the causes or effect of the solubilizing agents, nor to any particular solubilizing agents, but only to the following claims in which it is desirable to claim all novelty inherent in the invention.

We claim:

1. An improved lubricating oil for extreme pressure service comprising a lubricating oil base characterized by a viscosity index of at least 80 and a viscosity above 75 sec. Saybolt at 210° F., a lead salt of a higher fatty acid normally insoluble in the oil and a solubilizing agent therefor in proportion of about ⅓ to 1 part per part of the lead salt, selected from the group consisting of sulfonic and naphthenic acids, and their heavy metal salts, the lead salt amounting to 5 to 20% of the final composition and a sulphur compound in quantity sufficient to furnish .3 to 3.0% of corrosive sulphur.

2. An improved lubricating oil for extreme pressure service comprising a heavy viscous hydrocarbon lubricating oil base characterized by a viscosity index of at least 80 and a viscosity of above 75 seconds Saybolt at 210° F., lead oleate and a solubilizing compound therefor in propor-

tion from ⅓ to 1 part per part of lead oleate selected from the group consisting of sulfonic and naphthenic acids and their heavy metal salts, the lead compound amounting to 5 to 20% of the final composition and a sulfur compound sufficient to furnish from 0.3 to 5% of "corrosive" sulfur.

3. An improved lubricating oil for extreme pressure service comprising a heavy viscous hydrocarbon lubricating oil base characterized by a viscosity index of at least 80 and a viscosity of above 75 seconds Saybolt at 210° F., lead oleate and a solubilizing compound therefor in proportion from ⅓ to 1 part per part of lead oleate selected from the group consisting of sulfonic and naphthenic acids and their heavy metal salts, the lead compound amounting to 5 to 20% of the final composition and a sulfur compound sufficient to furnish from 0.3 to 1.0% of "corrosive" sulfur.

4. An improved lubricating oil for extreme pressure service comprising a heavy viscous hydrocarbon lubricating oil base characterized by a viscosity index between about 80 and 100 and a viscosity of above 75 seconds Saybolt at 210° F., lead oleate and a solubilizing compound therefor in proportion from ⅓ to 1 part per part of lead oleate selected from the group consisting of sulfonic and naphthenic acids and their heavy metal salts, the lead compound amounting to 5 to 20% of the final composition and a sulfur compound sufficient to furnish from 0.3 to 5% of "corrosive" sulfur.

5. An improved lubricating oil for extreme pressure service comprising a heavy viscous hydrocarbon lubricating oil base characterized by a viscosity index between about 80 and 100 and a viscosity of above 75 seconds Saybolt at 210° F., lead oleate and a solubilizing compound therefor in proportion from ⅓ to 1 part per part of lead oleate selected from the group consisting of sulfonic and naphthenic acids and their heavy metal salts, the lead compound amounting to 5 to 20% of the final composition and a sulfur compound sufficient to furnish from 0.3 to 1.0% of "corrosive" sulfur.

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