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2,161,584

PETROLEUM LUBRICANT COMPOSITION

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3 Claims. (Cl. 87—9)

Advances in mechanical design of gears, adoption of new forms of gear teeth, higher speeds, and greater load demands in many mechanisms have recently resulted in the subjection of lubricated contact areas, as in gear teeth to loads of extremely high unit value. Petroleum fractions alone have been proven incompetent to carry these extremely high unit loads without serious rupture of the lubricating film between contacting areas, often resulting in seizure. To provide lubricants capable of maintaining lubricant films under such conditions, resort has been had to blends of major proportions of petroleum fractions with minor proportions of characterizing ingredients of various kinds. Such compounded lubricants for service under conditions of high unit loading are known as extreme pressure lubricants, and the characterizing ingredients are known as E. P. (extreme pressure) ingredients, or E. P. bases. This invention has to do with such lubricants and such bases.

This invention has for its object the provision of novel lubricants capable of maintaining a lubricant film under conditions of high unit pressure. A further object is the provision of novel ingredients or bases for use as characterizing ingredients, in admixture with petroleum fractions, to produce therefrom lubricants capable of maintaining a lubricant film under unit loadings much higher than those at which the petroleum fraction alone will fail. A most important object is the provision of a method of lubrication, making use of the novel extreme pressure lubricants and novel characterizing agents therefor which are herein disclosed.

This invention is based upon the discovery that a class of compounds comprising the dixanthyl ethyl ethers are novel and valuable extreme pressure characterizing agents, capable of conferring upon petroleum oil fractions, when in admixture therewith, the power to maintain a lubricating film under conditions of extreme pressure.

Dixanthyl ethyl ethers may be prepared by the reaction of alkali xanthates with beta, beta dichlor ethyl ether. Alkali xanthates or thiocarbonates are readily prepared by reacting an alcohol with carbon disulphide in the presence of an alkali such as potassium hydroxide. The reaction involved is of the type

$$ROH+CS_2+KOH \rightarrow ROSCSK+H_2O$$

where K may be potassium or sodium and where R is an alkyl, aralkyl, or other substituted alkyl radical. Many of these alkali xanthates are known and described in the literature. Examples of alkali xanthates useful for the purposes of this invention are the alkali salts of ethyl xanthate, isoamyl xanthate, isopropyl xanthate and benzyl xanthate. In general, the xanthic acid salts of any of the alkyl, aralkyl, or simply substituted alkyl radicals will be found useful, as for example, sodium n-butyl xanthate, potassium phenyl ethyl xanthate, potassium methyl xanthate, potassium oleyl xanthate, potassium n-amylxanthate, and sodium n-propyl xanthate.

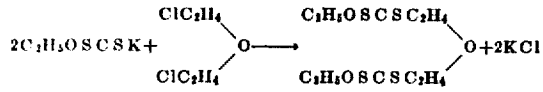
We have found that alkali xanthates of the above group may be reacted with beta, beta dichlor ethyl ether to form high boiling oils which are oil-miscible and substantially insoluble in water, and which possess the property of conferring upon petroleum fractions with which they are mixed an improved ability to maintain a lubricating film under conditions of extreme pressure loading.

As an example of the preparation of one of the novel E. P. ingredients of this invention, the preparation of diethyl xanthyl ethyl ether will be described. First, potassium ethyl xanthate was prepared in accordance with the reaction previously discussed. Then 32 parts of this potassium ethyl xanthate was dissolved in 320 parts (by weight) of ethyl alcohol. Then 14.3 parts of beta, beta dichlor ethyl was added at room temperature about 20°–25° C. and the mixture refluxed for 5½ hours, at approximately 78° C. The mixture was allowed to stand overnight to settle the precipitate of potassium chloride, which was then removed by filtration. The filtrate was distilled for the removal of alcohol, after which the distillation residue was taken up in benzol and washed with water to insure complete removal of any potassium chloride or of any remaining unreacted potassium ethyl xanthate. The benzol layer was then heated to 120° C. under 2.5 mm. pressure to remove benzol and unreacted beta, beta dichlor ethyl ether. The product so obtained was a yellow oil, miscible in petroleum fractions and substantially insoluble in water.

The proportions of ingredients used were in the ratio of 2 mols of the alkali xanthate for 1 mol of beta, beta dichlor ethyl ether. Sufficient ethyl alcohol was used to completely dissolve the alkali xanthate and to give a reaction mixture of suitable fluidity. Other solvents which might well be used are methyl alcohol, propyl alcohol, or any solvent which is a mutual solvent for the reacting compounds, and which does not itself react with the reagents.

It will be apparent to those skilled in the art

that the reaction product resulting from interaction of an alkali xanthate and beta, beta dichlor ethyl ether is probably a dialkyl xanthyl ethyl ether, formed according to the following reaction, in the case of an ethyl xanthate:



To illustrate the effectiveness of novel ingredients of the class herein disclosed as characterizing E. P. ingredients for the production of extreme pressure lubricants, the following tests are noted. These tests were made in the Almen Pin Test Machine, (Mougey & Almen, Proc. A. P. I. 1931, page 77), wherein a steel pin is rotated in a bearing so designed that increasing loads of known amount may be added, the assembly being lubricated by the oil undergoing test. This test is in wide use for the comparative evaluation of lubricants of this type. In the following tabulation of such tests, Table I, the first vertical column shows the loading in pounds per square inch of bearing area, and columns 2 and 3 show the torque in foot pounds applied to the Almen pin:

TABLE I

Almen pin test results

1	Torque, foot pounds	
Load, pounds per square inch	2 Straight mineral oil	3 Mineral oil + 2% compound
1,000	----------	----------
2,000	.7	.7
3,000	1.0	1.1
4,000	Seized	1.5
5,000	----------	1.8
23,000	----------	5.8
24,000	----------	6.1
25,000	----------	6.1 Released

In the above tests, column 2 shows the result of test on a mineral oil of 165'' Saybolt viscosity at 210° F., pour 20° F., used in the absence of any characterizing ingredient. Such oils are commonly used in the compounding of extreme pressure lubricants. The oil whose tests are reported in column 3 is the same oil as in column 2, except that there has been added to it 2% of diethyl xanthyl ethyl ether. It will be noted from these tests that the novel ingredients herein disclosed possess to a marked degree the ability to increase the power of a petroleum fraction to maintain a lubricating film under conditions of extreme pressure loading.

The novel E. P. characterizing compounds herein disclosed are described as oil-miscible, which term is used in the sense that they are sufficiently soluble in oil to accomplish the purposes of this invention. They are likewise described as substantially water-insoluble, which means that they are at least sufficiently water-insoluble to prevent their removal from admixture with oil by contact with water such as might occur during storage and/or handling and/or use.

The limits of concentration of the dixanthyl ethyl ether compounds in oil which will be found useful for the purposes of this invention will normally range from a minimum of about 0.5% by weight to a maximum of about 5.0% by weight in the oil.

It will be understood that the exemplary preparation of one of the novel E. P. characterizing compounds of this invention discussed herein is given only by way of example and is not in any way intended as a limitation. It is our intention to claim broadly all of the novelty inherent in this invention, subject only to those limitations expressed in the following claims.

We claim:

1. A lubricant oil composition capable of maintaining a lubricating film under conditions of high unit loading comprising a major portion of a mineral hydrocarbon lubricant not so capable, and in admixture therewith a small amount of an oil-miscible, substantially water-insoluble dialkyl xanthyl ethyl ether.

2. A lubricating composition comprising a major portion of a mineral hydrocarbon oil and in admixture therewith a small amount of diethyl xanthyl ethyl ether.

3. A lubricant oil composition capable of maintaining a lubricant film under conditions of high unit loading comprising a major portion of a mineral hydrocarbon lubricant not so capable, and in admixture therewith a small amount of an oil-miscible, substantially water-insoluble dialkyl xanthyl ethyl ether, wherein the alkyl xanthyl radicals are selected from the group consisting of ethyl xanthyl, iso-amyl xanthyl, iso-propyl xanthyl, and benzyl xanthyl.

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EVERETT W. FULLER.

CERTIFICATE OF CORRECTION.

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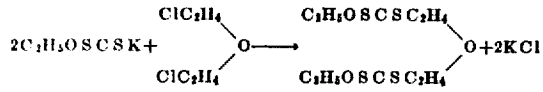
It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 1, first column, line 31, for the word "fall" read fail; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 8th day of August, A. D. 1939.

Leslie Frazer,
Acting Commissioner of Patents.

(Seal)

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