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SOAP THICKENED LUBRICATING GREASE CONTAINING FORMAMIDE

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This invention relates to improved lubricating greases. More particularly it relates to soap thickened lubricating greases containing minor amounts of formamide.

We have found that very superior rust preventive properties and other advantages are obtained by employing small amounts of formamide in soap thickened greases. The rust preventive action of formamide in soap thickened greases is a different result from that obtained with lubricating compositions of other types, such as, for example, water based cutting fluids, wherein formamide has little or no rust preventive action.

The lubricating compositions of this invention comprise essentially a lubricating oil as the chief component containing a fatty acid soap in sufficient amount to thicken the composition to a grease consistency and a minor amount of formamide. The composition may suitably contain about 0.1–10.0 percent by weight of formamide, and preferably from about 0.5 to about 5.0 percent by weight of formamide based on the weight of the composition. The fatty acid soap may be present in widely varying amounts, such as from about 5 to about 45 percent by weight, based on the weight of the composition, and ordinarily in amounts from about 7 to about 30 percent by weight of the grease composition. In addition to the formamide, other additives of various types commonly employed in lubricating greases, such as other rust inhibitors, oxidation inhibitors, extreme pressure agents, etc., may be present in these greases, insofar as no antagonistic effect with the formamide is produced. Suitable additives include particularly amines of various types, such as diphenylamine, aplha- and beta-naphthylamines, diphenyl para-phenylene-diamine and tetramethyl diamino diphenyl methane.

The fatty acid soaps employed in these greases may be any of those in general which have been employed as thickening agents in lubricating greases, wherein the metal component may be an alkali metal, an alkaline earth metal or other polyvalent metal such as aluminum, copper, cobalt, nickel, etc. The preferred soaps are those wherein the metal component is an alkali metal or an alkaline earth metal. It is not intended to imply that greases thickened with the different metal soaps are affected in exactly the same manner by the addition of formamide, since in addition to the rust preventive property which formamide imparts to fatty acid soaps generally, its effect otherwise is not necessarily the same, and special advantages are obtained in accordance with this invention with greases of certain types as will hereinafter appear.

The saponifiable materials employed in the preparation of the soaps may be any of those of the usual types, comprising higher fatty acids, containing at least 12 carbon atoms per molecule, which may be saturated, unsaturated or hydroxy substituted fatty acids, the glycerides or other esters of such acids or mixtures of the free fatty acids and their glycerides or other esters. The fatty acid materials may be naturally occurring or derived from naturally occurring materials, or they may be obtained synthetically, as for example by the oxidation of petroleum fractions. The preferred saponifiable materials are those which comprise at least a major portion of fatty acids containing from about 14 to about 22 carbon atoms per molecule, such as for example, myristic acid, palmitic acid, stearic acid, oleic acid, tallow fatty acids, 12-hydroxystearic acid, 9,10-dihydroxystearic acid, behenic acid, etc., or the glycerides or other esters of such acids.

The lubricating oil component of these greases may be any suitable oils of lubricating characteristics, which may be of mineral, vegetable, animal or synthetic origin, such as have been employed heretofore in lubricating greases. Suitable mineral oils include both distillate and residual fractions from paraffinic, naphthenic or mixed base crudes, ranging from fairly low to fairly high viscosities, such as from about 80 seconds Saybolt Universal at 100° F. to about 220 seconds Saybolt Universal at 210° F. These oils may be obtained by any of the usual refining methods such as acid treating, solvent extraction, and so forth. For certain purposes, such as, for example, the production of low temperature greases, various synthetic oils may be employed with advantage. As examples of suitable oils of this type may be mentioned high molecular weight aliphatic ethers, such as normal hexyl ether, the alkyl esters of benzoic and phthalic acids, and the aliphatic dicarboxylic acid di-esters, such as di-2-ethyl hexyl sebacate, di(secondary amyl) sebacate, di-2-ethyl hexyl azelate, di-isooctyl adipate, etc.

The grease preparation may be carried out in any suitable manner, either by merely mixing together the lubricating oil and preformed soap or by saponification in situ of the saponifiable material in a portion of the lubricating oil component of the grease. The formamide may be added during the grease preparation when the temperature of the mixture is below about 200° F., or it may be mixed into the finished grease.

A preferred embodiment of this invention is found in the manufacture of calcium base greases, wherein formamide is particularly effective as a rust inhibitor. As an example of this preferred embodiment of our invention, a grease was prepared comprising a mineral lubricating oil containing 14.5 percent by weight of calcium soap and 1.0 percent by weight of formamide. The mineral oil was a refined naphthene base distillate oil having a Saybolt Universal viscosity at 100° F. of about 310 seconds. The saponifiable material was Star Tallow, having a saponification number of 197, a neutralization number of 21.6, a titer, ° C., of 43.4 and an iodine number of 49. The preparation was carried out by the method comprising saponification in situ in the presence of about ⅓ of the mineral oil contained in the grease, at about 190° F., dehydration at 300° F., and addition of the remainder of the mineral lubricating oil during cooling. A small amount of water, equivalent to 1 percent by weight of the grease was also added when the grease was at a temperature below about 200° F. The formamide was added by mixing it into the finished grease at room temperature, then reheating the grease to 150° F. and maintaining it at that temperature for 30 minutes with stirring. The following table shows the rust inhibiting and other properties of this grease in comparison with those of the base grease containing no formamide.

Table I

Grease	Base Grease	Containing 1% Formamide
Water, Percent	1.0	0.59
Penetration at 77° F., ASTM: Worked (60 strokes)	314	370
Dropping Point, ° F	213	198
ASTM Bomb Oxidation Test (100 hrs.), lb. pressure drop	95	95
Water Washing and Rusting Test	Heavy Rust	No Rust
Dynamic Water Resistance Test, Percent loss	7.5	6.3

The water washing and rusting test comprises essentially rotating a standard X–469 Timken roller bearing packed with a sample of the test grease alternately in air and in water. The weighed bearing packed with 18 grams of the test grease is first rotated in air at room temperature for 30 minutes at 500 r.p.m. in order to uniformly coat the bearing surface with the grease. The bearing trough is then filled with water, which results in the bearing being about 33 percent immersed, and the bearing is rotated for 2 hours at 500 r.p.m. while the temperature is maintained at 60–70° F. The water is then drained from the trough and the bearing rotated in air for one hour, and again in water for two hours at 60–70° F. The bearing is then drained and stored in a humidity cabinet for 7 days, after which it is weighed and inspected to determine the grease loss and the amount of rusting. As shown by the above table, the calcium soap grease containing 1 percent of formamide prevented any rusting under the severe conditions of this test, whereas heavy rusting of the bearing occurred with the uninhibited grease.

Other greases wherein a special advantage is obtained by the addition of minor amounts of formamide are greases thickened with lithium soaps of hydroxy fatty acids. We have found that in these greases the formamide acts as both a rust preventive agent and an oxidation inhibitor, so that lithium hydroxy fatty acid soap greases having both excellent rust inhibiting and antioxidant properties are obtained with the use of a single inhibitor.

As an example of a grease of the above type, a grease was prepared comprising a mineral lubricating oil containing 7.6 percent by weight of lithium 12-hydroxy stearate and 5.0 percent by weight of formamide. The mineral lubricating oil was a blend in about a 6:1 ratio respectively of a refined paraffinic distillate oil having a Saybolt Universal viscosity at 100°.F. of about 182 seconds and a refined Manvel residuum having a Saybolt Universal viscosity at 210° F. of about 92 seconds. The saponifiable material was the methyl ester of 12-hydroxy stearic acid, sold under the trade name of Paricin 1. The preparation was carried out by saponification in situ, employing a small excess of lithium hydroxide, sufficient to give about 0.2 percent of free alkali to the finished grease. The saponification was carried out at about 200° F., and the dehydration at about 300° F. The formamide was added to the grease mixture at room temperature and the mixture then heated to 150° F. and stirred for 30 minutes at that temperature. The following table shows

the properties of this grease in comparison to those of the base grease without the inhibitor.

Table II

Grease	Base Grease	Containing 5.0% Formamide
Penetration at 77° F. ASTM:		
Unworked	267	339
Worked (60 strokes)	263	339
Dropping Point, ° F	383	337
ASTM Bomb Oxidation Test (100 hrs.), lb. pressure drop	88; 91	4; 5
Water Washing and Rusting Test:		
Condition of bearing	Heavy Rust	No Rust
Condition of grease	Poor	Good
Grease washed from bearing, percent	45	28

As shown by the above table, formamide imparted both a high degree of oxidation resistance and rust preventiveness to the lithium 12-hydroxystearate grease. Smaller amounts of formamide, such as about 1 percent by weight, imparted equivalent oxidation resistance to these greases but generally reduced rust preventiveness. Such smaller amounts are advantageously employed where the softening effect and reduction in dropping point resulting from the addition of formamide to these greases are objectionable, and a high degree of rust preventiveness is not required. However, the softening effect obtained in these greases on the basis of both unworked and worked penetration is desirable in some cases, where it is desired to obtain greases of a certain grade containing higher amounts of soap.

Obviously many modifications and variations of the invention, as hereinbefore set forth, may be made without departing from the spirit and scope thereof and only such limitations should be imposed as are indicated in the appended claims.

We claim:

1. A lubricating grease consisting essentially of a mineral lubricating oil thickened to a grease consistency with about 7–30 percent by weight of a lithium hydroxy fatty acid soap and containing formamide in an amount from above 1 percent to about 10 percent by weight, sufficient to impart rust preventiveness to the said grease in the water washing and rusting test.

2. A lubricating grease consisting essentially of a mineral lubricating oil thickened to a grease consistency with about 7–30 percent by weight of lithium 12-hydroxystearate and containing about 5 percent by weight of formamide, sufficient to impart rust preventiveness to the said grease in the water washing and rusting test.

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