Patented Feb. 19, 1952

2,586,693

UNITED STATES PATENT OFFICE

2,586,693

LUBRICATING GREASE

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No Drawing. Application November 1, 1949, Serial No. 124,941

7 Claims. (Cl. 252—41)

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This invention relates to lubricating greases. It relates particularly to lubricating greases having excellent structural stability to mechanical working over long periods of time when thickened with relatively small amounts of soap.

Prior art has shown greases thickened with the alkali metal soaps of combinations of high and low molecular weight fatty acids. The art has also shown that in order to obtain the desired structural stability and freedom from syneresis, or oil separation, these greases must have a soap content of at least 10%. Even in those cases in which this minimum amount of the mixed soap of low and high molecular weight fatty acids is present, structural stability to oil separation is generally poor.

It has now been found, and comprises the object of this invention, that an improved lubricating grease having improved structural stability and freedom from syneresis may be made by combining with the alkali metal soaps of high and low molecular weight fatty acids a soap of an aryl-aliphatic acid. In certain cases these improved greases are obtained when employing much lesser amounts of soap than are required in the greases of the prior art.

The improved lubricating grease of this invention consists essentially, in addition to the lubricating oil from which the grease is made, of three components. These components are:

(1) An alkali metal soap of a substantially saturated fatty acid of high molecular weight;

(2) An alkali metal salt of a fatty acid of low molecular weight; and

(3) An alkali metal soap of an aryl-aliphatic acid wherein the aliphatic portion has a carbon chain of from 8 to 20 carbon atoms.

The three soaps present in the grease as listed in the foregoing paragraph may be of the same alkali metal or they may be of different metals. Of the alkali metals, lithium and sodium are preferred. The preferred embodiment also contemplates that the three soap components be of the same alkali metal.

The high molecular weight fatty acid of component (1) may be any of the ordinary fatty acids used in grease making. Among a list of these will be found stearic acid, hydroxy-stearic acid, hydrogenated fish oil acids, beef fat acids, tallow acids, the saturated or unsaturated glycerides of the various fatty acids or mixtures of the above. Those acids having a carbon chain length of from 16 to 20 carbon atoms are preferred.

As the low molecular weight acid in component (2) saturated or unsaturated aliphatic acids may be employed, but it is preferred to use acetic acid. However, other of the low molecular weight fatty acids such as formic, propionic, butyric, acrylic, methacrylic, furoic, lactic, etc., or compounds which yield such acids, for example acrylonitrile, may be used.

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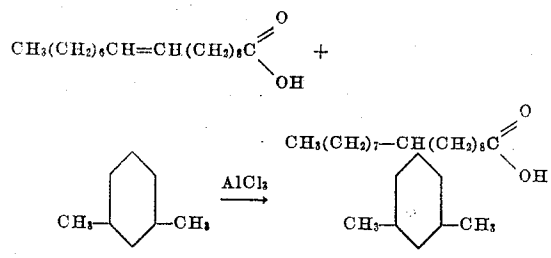
There are two requirements outstanding which must be met when choosing the acid for component (3). The acid chosen must have an aliphatic chain of at least 8 carbon atoms in length and there must be dependent from that chain an aryl group which may or may not contain substituent groups. These acids may be formed by the Friedel-Crafts condensation of an unsaturated acid and an aryl compound. Using as an example xylyl-stearic acid as a final product, their preparations may be illustrated by the following equation:



Operable aryl-aliphatic acids may be prepared by condensing the desired unsaturated acid with benzene, toluene, trimethyl benzene, tetramethyl benzene, cumene and the like. Among the unsaturated acids with which these aryl compounds may be condensed to give the desired aryl-aliphatic acids may be listed undecylenic acid, myristolenic acid, palmitolenic acid, oleic acid, petroselic acid, erucic acid, and the like. The preferred embodiment contemplates oleic acid, however.

The proportions in which these three components are mixed will depend upon the characteristics of the final grease composition desired. For the most part it will be desirable to utilize only minor proportions of the aliphatic-aryl soap component, it being the most expensive of the three soaps. It has been found that with increasing amounts of the aryl-aliphatic acid soap, the final grease product increases in elasticity and cohesiveness. For optimum results, especially in the case of lithium soap greases, about 0.25% to 2.0% of the aryl-aliphatic acid soap should be used with from 2.25% to 17.0% by weight of the high molecular weight fatty acid soap and from 0.5% to 4.0% of the low molecular weight acid soap. In the case of sodium soap greases, greater amounts of aryl-aliphatic acid may be employed, for example equal to but preferably not greater than the amount of fatty acid employed. The mol ratio of low molecular weight acid to the combined aryl-aliphatic and fatty acids is preferably 1/1 or greater, for example, 2/1 or 3/1.

To more clearly explain the concept of this invention, the following examples are given.

EXAMPLE I

A grease composition having the following formulation was prepared:

	Percent by weight
Hydrogenated fish oil acids	4.5
Acetic acid	1.0
Xylyl-stearic acid	0.5
Lithium hydroxide monohydrate	1.3
Conventionally refined Coastal distillate having a viscosity at 210° F. of 55 S. U. S.	92.7

One-third of the oil, all of the hydrogenated fish oil acids, and the xylyl-stearic acid were charged to a fire-heated grease kettle and heated to 150° F. The acetic acid was then added and the acids co-neutralized with the lithium hydroxide dissolved in boiling water. The temperature was then raised to 225°–250° F. to dehydrate the mixture. After dehydration the balance of the oil was added and the temperature raised to 450°–500° F. The fluid molten grease was then rapidly chilled in thin layers. The cold grease was then returned to the kettle for homogenization.

This grease was tested in accordance with the ASTM procedure for testing penetration and gave a test result of 250 mm./10 at 77° F. After working for 100,000 strokes in an ASTM worker using the fine hole plate (having 325 1/6'' holes) the penetration at 77° F. increased only 5 mm./10 (255 mm./10). This indicates an outstanding structure stability. The dropping point of this grease was 370° F. Employing the same formulation as Example I excluding the xylyl stearate, the product is soft and structurally unstable both to mechanical working and oil separation.

EXAMPLE II

A similar grease was prepared employing a mixture of acrylic acid and acetic acid as the low molecular weight constituent:

	Percent by weight
Acrylonitrile	0.65
Acetic acid	0.50
Xylyl-stearic acid	0.50
Hydrogenated fish oil acid	4.50
Lithium hydroxide monohydrate	1.40
Conventionally refined Coastal distillate having a viscosity at 210° F. of 55 S. U. S.	91.95
Phenyl alpha naphthylamine (oxidation inhibitor)	0.50

Inspection tests on this grease composition were as follows:

Dropping point, °F., 375
Unworked penetration, 260 mm./10
Worked penetration (100,000 strokes 325 1/6'' hole worker plate), 347 mm./10
Percent free alkalinity, 0.08 as NaOH

EXAMPLE III

The effect of the presence of the aryl-aliphatic acid soap upon a grease structure is clearly shown by the following:

According to the method outlined in Example I above a grease with the following formulation was prepared.

	Percent by weight
Hydrogenated fish oil acids	10.0
Xylyl-stearic acid	10.0
Acetic acid	3.5
Lithium hydroxide monohydrate	5.1
Phenyl alpha naphthylamine	1.0
Conventionally refined Coastal distillate having a viscosity at 210° F. of 55 S. U. S.	70.4

The grease formed was very hard and of an elastic-cohesive nature. Using this grease material as a base, successive additions of the same oil used in the manufacture of the base were made (heating to 500° F. after each addition and cooling to room temperature), thus, in effect, forming a series of greases with decreasing amounts of the xylyl-stearic acid soap contained therein. For convenience in examination the following table gives a chart of these greases and their components along with the properties of the greases so formed:

Table I

PROPERTIES OF GREASE STRUCTURES CONTAINING ARYL-ALIPHATIC ACID

	Parts by Weight of Acids			Properties	Penetration	
	Fish Oil Acid	Acetic Acid	Xylyl-Stearic Acid		Unworked	mm./10 Worked
a	10	3.5	10.0	Heavy, elastic consistency.	105	Too hard to work.
b	5	1.75	5.0	do	198	210 (aerated).
c	2.5	0.875	2.5	do	245	255 (aerated).
d	0.5	0.175	0.5	Good stable grease structure. Soft consistency, only slightly cohesive.	285	368.

The data reported in Table I above shows that reduction of the xylyl-stearic acid content reduced the elastic-cohesive nature of the grease structure. It also shows that excessive amounts of the xylyl lithium stearate in combination with lithium aliphatic acid soaps will give products generally unsuitable due to excessive elasticity and cohesiveness. It also shows that greases having stable structures may be obtained with extremely small amounts of soap thickener present, with the elastic cohesive nature practically eliminated in the lower soap content products.

EXAMPLE IV

Using sodium hydroxide to form the acid soaps, and following the procedure outlined in

Example I above, greases having the following formulation were made:

	Per Cent by Weight	
	(a)	(b)
Xylyl-stearic acid	20.0	10.0
Hydrogenated fish oil acid		10.0
Acetic acid	3.0	3.5
Sodium hydroxide	4.8	5.0
Phenyl alpha naphthylamine	1.0	1.0
Conventionally refined Ccastal distillate having a viscosity at 210° F. of 55 S. U. S	71.2	70.5

These sodium soap grease formulations gave the following inspections:

Properties	(a)	(b)
Per Cent Free Alkalinity (as NaOH)	0.66	0.34
Unworked Penetration at 77° F. (mm./10)	179	153
Worked Penetration at 77° F. 63,000 strokes fine hole worker (mm./10)	195	202
Dropping Point, °F	480	496
Physical Appearance	(1)	(2)

[1] Very elastic and cohesive.
[2] Smooth, short fiber, good adhesiveness to metal, very slightly cohesive, no tendency to separate oil.

It will be noted that the sodium soap greases containing the xylyl-stearic acid soap shows a slightly higher unworked penetration than does the lithium soap greases (153 mm./10 for grease (b) of Example IV compared to 105 mm./10 for grease (a) of Example III). This indicates some slightly lower thickening effect of the sodium soap of the xylyl-stearic acid. However, the sodium based greases are very satisfactory from the point of view of both structural and thermal stability.

To summarize briefly, this invention comprises an improved lubricating grease which is made by combining with a lubricating oil a mixture of the alkali metal soaps of a high molecular weight substantially saturated fatty acid, a low molecular weight fatty acid and an aryl-aliphatic acid. The use of the aryl-aliphatic acid soap permits better soap dispersion in the oil and is obviously an economic advantage since it permits the manufacture of a mixed acid base soap grease with excellent structural stability with a smaller proportion of soap than was heretofore believed pos-

What is claimed is:

1. An improved lubricating composition which comprises a major proportion of a lubricating oil thickened to a grease consistency with the alkali metal soaps of a mixture of about 2.25% to 17% by weight of a high molecular weight substantially saturated fatty acid having from 16 to 20 carbon atoms, from about 0.5% to 4.0% by weight of a low molecular weight fatty acid having from 1 to 4 carbon atoms, and about 0.25% to 2% by weight of an aryl-aliphatic acid containing in the aliphatic portion a hydrocarbon chain of from 8 to 20 carbon atoms.

2. An improved lubricating composition according to claim 1 wherein the alkali metal is lithium.

3. An improved lubricating composition according to claim 1 wherein the alkali metal is sodium.

4. An improved lubricating composition which comprises a major proportion of a lubricating oil thickened to a grease consistency with the alkali metal soaps of a mixture of about 2.25% to 17% by weight of a high molecular weight substantially saturated fatty acid having from 16 to 20 carbon atoms per molecule, from about 0.5% to 4.0% by weight of a low molecular weight fatty acid having from 1 to 4 carbon atoms per molecule, and about 0.25% to 2% by weight of an aryl-aliphatic acid wherein the aliphatic portion contains 17 carbon atoms and the aryl portion is selected from the class consisting of substituted and unsubstituted benzenes.

5. An improved lubricating composition which comprises a major proportion of a lubricating oil thickened to a grease consistency with about 4.5% by weight of the lithium soap of hydrogenated fish oil acid, about 1.3% by weight of the lithium soap of a low molecular weight fatty acid having from 1 to 4 carbon atoms per molecule and about 0.5% by weight of the lithium soap of an aryl-aliphatic acid wherein the aliphatic portion contains from 8 to 20 carbon atoms, said weight percents being based on the weight of the total grease composition.

6. An improved lubricating composition which comprises a major proportion of a lubricating oil thickened to a grease consistency with the sodium soaps of a mixture of acids, said mixture essentially of equal proportions of an aryl-aliphatic acid wherein the aliphatic portion contains from 8 to 20 carbon atoms and a high molecular weight substantially saturated fatty acid having from 16 to 20 carbon atoms per molecule combined with a low molecular weight fatty acid having from 1 to 4 carbon atoms per molecule, the mol ratio of the low molecular weight fatty acid to the combined aryl-aliphatic acid and high molecular weight acid being within a range of from 1/1 to 3/1.

7. An improved lubricating composition which comprises a major proportion of a lubricating oil thickened to a grease consistency with the sodium soap of a mixture of acids, said mixture consisting essentially of about 10.0% hydrogenated fish oil acids, about 3.5% acetic acid, and about 10.0% xylyl-stearic acid, said percentages being by weight based on the weight of the total composition.

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