

UNITED STATES PATENT OFFICE

2,450,254

LITHIUM BASE GREASE AND METHOD OF PREPARING THE SAME

Oney P. Puryear, Fishkill, and Harry V. Ashburn, Glenham, N. Y., assignors to The Texas Company, New York, N. Y., a corporation of Delaware

No Drawing. Application August 10, 1944, Serial No. 548,940

2 Claims. (Cl. 252-41)

1

This invention relates to an improved lubricating grease composition and particularly to a texture-stable lithium base grease and methods of preparing the same.

In general, lithium base greases, prepared from saturated fats or fatty acids, possess a high melting point and are water-resistant. These properties are particularly desirable in that they permit lubrication over a wide temperature range and minimize the solubilizing or leaching effect of water and moisture condensation on the grease. However, there are certain difficulties associated with the production of these greases, together with certain undesirable performance characteristics, which prevent any large scale acceptance thereof as a commercial lubricating grease.

In the preparation of the conventional lithium base grease, a slurry of a lithium soap, such as the stearate, which is either preformed or prepared in situ, and a mineral oil, are heated to temperatures of around 360-500° F. until a homogeneous solution is obtained. This solution of lithium soap in oil is then drawn from the kettle and allowed to cool in open pans until a gel structure is set up. The cooled gel is then subjected to a milling operation where it is homogenized to a homogeneous grease composition. It becomes quite apparent that the efficiency and economies of such a method of manufacture are quite poor, due to the high temperatures of operation, special equipment required, and the lack of control over the consistency and yield of the final product.

One of the most important drawbacks of the conventional lithium base greases is, however, the inability of these greases to maintain their structure and lubricating qualities under conditions of operation where shearing stresses are involved and particularly at temperatures above atmospheric. In such installations the greases gradually lose their consistency until they become liquid and cause bearing failures. Attempts have been made to correct this deficiency by the incorporation of various stabilizing and thickening agents, such as other metallic soaps and high molecular weight esters.

In accordance with the discovery of the present invention, it has been found possible to pro-

2

vide a lithium base grease composition of exceptional texture and oxidation stability which is capable of continued operation under conditions of high shearing stresses with negligible change in structure and very little change in consistency, together with an efficient and economical method of preparation which requires a minimum of equipment, provides an accurate control of consistency and yield of product, and is operated at temperatures attainable in a steam-jacketed kettle.

In its broadest aspect, the invention contemplates the use of a soap-forming hydroxy fatty acid glyceride as the acidic component in the preparation of a lithium base grease composition. It has been found that a lithium soap grease prepared from a hydroxy fatty acid glyceride possesses exceptional texture stability and maintains its structure with little change in penetration even after extensive working at either high or low temperatures. Furthermore, the hydroxy fatty acid glyceride permits the use of a simple low temperature method of preparation as distinguished from the high temperature complex methods required by the use of other well-known fats and fatty acids. The preparation of the grease is carried out by saponifying a soap-forming hydroxy fatty acid glyceride with a basic lithium compound, dehydrating the reaction product, and adding mineral oil until the desired consistency is obtained. The total operation is easily conducted in a steam-jacketed open kettle and the resulting finished lithium base grease requires no homogenization or further treatment prior to actual use.

The hydroxy fatty acid glycerides used in accordance with the invention are the glycerides of hydroxy fatty acids containing 12 or more carbon atoms and one or more hydroxyl radicals which are separated from the carboxyl group by at least one carbon atom. These glycerides may either be used as the sole acidic component of the lithium soap or in combination with other fats or fatty acids, in which case the hydroxy fatty acid glycerides are preferably present in a predominant amount. In the latter case, the glycerides of the invention may either be blended with other fats or fatty acids prior to saponifica-

tion or the preformed lithium soaps thereof may be blended in the desired proportions.

In the following description of the preferred form of the invention hydrogenated castor oil is used as the representative soap-forming hydroxy fatty acid glyceride. For practical application at the present time, hydrogenated castor oil possesses the advantage of availability and cost which renders it competitive to the more common fats and fatty acids. However, it is to be understood that the invention is not limited to hydrogenated castor oil and that other soap-forming hydroxy fatty acid glycerides may be used in accordance with the invention. Examples of such glycerides are the glycerides of the hydroxy fatty acids produced by catalytic oxidation of hydrocarbon oils and waxes which have been extracted and fractionated to the desired molecular range.

The oil component of the subject grease compositions may be either a mineral oil, a synthetic oil, or an organic oily medium, such as the benzoate or phthalate esters, with a wide range of viscosities, depending upon the type of lubrication required of the finished grease. For example, in the preparation of a low temperature lithium base grease employed in the lubrication of bearings and the like at temperatures below -60°F. , an oil component possessing a viscosity in the range of 40–70 S. U. S. at 100°F. , a low pour point, and a viscosity index of 30 or more is preferred. The soap content may also be varied in accordance with the grade or consistency desired. Normally, the soap content of the lithium base grease of the invention possessing an ASTM penetration in the range of 300–350 varies between 10–20% soap.

Where a high degree of oxidation stability is required an oxidation inhibitor must be used. For these lithium base greases the aromatic amine type of inhibitor has been found particularly effective. Of this class of inhibitors the polynuclear aromatic amines, such as tetramethyl diamino diphenyl methane, diphenyl amine, and phenyl alpha naphthylamine are preferred.

The procedure involved in the preparation of these lithium base greases may be outlined as follows:

A steam-jacketed open kettle is charged with a mixture of lithium hydroxide and water and heated with stirring to $160\text{--}180^{\circ}\text{F.}$ until all the lithium hydroxide is dissolved. The required amount of hydrogenated castor oil in equal weight of oil is then added and stirring is continued for three to five hours at $170\text{--}200^{\circ}\text{F.}$, or until the hydrogenated castor oil is completely saponified. The kettle contents are then heated within the range of $270\text{--}330^{\circ}\text{F.}$, preferably $270\text{--}300^{\circ}\text{F.}$, and held at such temperatures until the soap mass becomes translucent, which usually requires from two to five hours. The heat is then reduced and oil addition is begun. When the temperature reaches approximately $180\text{--}200^{\circ}\text{F.}$ the oxidation inhibitor, if desired, is then added and thereafter oil addition is continued until the product possesses the desired consistency.

It has been found particularly desirable to effect the dehydration of the saponified product at temperatures above 270°F. At temperatures of around 270°F. it is necessary to heat for several hours in order to cause the soap mass to become translucent and lose its cloudy-white appearance, whereas at temperatures of around 300°F. a shorter dehydration time may be used. At lower

temperatures the yields of the product fall off materially.

Example

15 pounds of water and 2.18 pounds of lithium hydroxide were charged to a steam-jacketed open kettle, which was heated to 190°F. , with stirring at 35 R. P. M. After the lithium hydroxide was dissolved, a mixture of 14 pounds of hydrogenated castor oil (saponification No.—182, iodine No.—5, OH No.—158, titer— 75.7°C.), and 14 pounds of a mildly-refined mineral oil of the following characteristics were added:

Gravity, $^{\circ}\text{API}$	26.9
Flash, $^{\circ}\text{F.}$	310
Fire, $^{\circ}\text{F.}$	340
Vic. at 100°F. S. U.	60.2
Pour $^{\circ}\text{F.}$	—55
Color ASTM	3½

This mixture was then heated with stirring, for six hours, at $182\text{--}192^{\circ}\text{F.}$ and shut down over night. The temperature was then brought up to dehydration temperature and the contents of the kettle heated with stirring for five hours at $277\text{--}285^{\circ}\text{F.}$ The steam heat was then cut down to 75 pounds' pressure and the temperature gradually reduced, with simultaneous addition of the mineral oil. When the temperature of the kettle reached approximately 200°F. , 189.5 grams of phenyl alpha naphthylamine, dissolved in oil, were added. Oil addition was then continued until 67 pounds of oil had been added and the worked penetration at 78°F. was 325. The grease thus obtained was a smooth, buttery, semi-transparent, very light brown grease. The tests obtained thereon are as follows:

Lithium soap	per cent.	16.1
Free alkali (LiOH)	¹ do.	0.09
Free fatty acid (as oleic)	¹ do.	0.03
Free neutral fat	do.	0.67
Phenyl alpha naphthylamine (calc.)	do.	0.5
Penetration at 77°F. worked		336
Dropping point $^{\circ}\text{F.}$ (ASTM)		356
Navy water absorption	per cent.	10
Norma-Hoffman oxidation (100 hrs. at 210°F.), pounds pressure drop	max.	5

¹"The Institute Spokesman," National Lubricating Grease Institute, January, 1944. "Test methods for determining free acid and free alkali in greases."

In order to determine the texture-stabilizing properties and the resistance to shear at high temperatures, the grease prepared in the foregoing example was subjected to a so-called dynamic shear test. This test was conducted in an apparatus consisting of a perforated piston reciprocating within a closed cylinder maintained at a constant temperature. In the test the grease was charged to the cylinder maintained at a temperature of 225°F. , and the piston then reciprocated at 49 strokes per minute for a period of 8 hours. At the conclusion of the test the grease was removed and miniature penetrations taken. This miniature penetration was then compared with the miniature penetration of the grease taken before the test to determine the effect of the shearing action upon the grease structure. The results obtained on the subject grease, designated as "Grease No. I," as compared to the lithium stearate grease prepared by the conventional high temperature manufacture, designated as "Grease No. II," are as follows:

5

Shear test

	Miniature Penetration ¹	
	Before	After
Grease No. I.....	133	107
Grease No. II.....	114	Liquid

¹ "Miniature Penetrometer for Determining the Consistency of Lubricating Greases." Ind. Eng. Chem., analytical edition, vol. II, page 108, February 15, 1939.

As may be seen from the comparative results obtained, the lithium soap grease prepared from hydrogenated castor oil possesses a structure which does not break down under the severe conditions of the test and, if anything, formed a tighter grease structure which accounts for the harder consistency. This phenomenon is characteristic of the lithium soap greases prepared from hydroxy fatty acid glycerides. On the other hand, the lithium stearate grease which is representative of the conventional fats and fatty acids, breaks down to a liquid or semi-liquid product.

The low temperature method of manufacturing a lithium base grease from a hydroxy fatty acid, such as 12-hydroxystearic acid, is disclosed and claimed in our co-pending application, Serial No. 588,890, filed April 17, 1945.

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

We claim:

1. A method of preparing a low temperature gel-type lithium base grease containing a lithium

6

soap of a soap-forming hydroxy fatty acid glyceride as the essential soap constituent thereof, said method enabling the use of temperatures attainable in a steam-jacketed kettle without fire hazard, which comprises saponifying with a basic lithium compound at a temperature within the range of about 170-200° F. of a soap-forming hydroxy fatty acid glyceride in the presence of a small proportion of the oleaginous vehicle included in the grease, then raising the temperature of the saponified product to about 270-330° F. to dehydrate the product and render the same translucent, thereafter reducing the temperature and adding the balance and large proportion of the oleaginous vehicle as the product cools and the desired consistency is attained, and finally drawing the resultant gel-type grease as a final product.

2. The method according to claim 1, wherein the hydroxy fatty acid glyceride is hydrogenated castor oil, and the oleaginous vehicle comprises a mineral lubricating oil having a viscosity within the range of 40-70 S. U. S. at 100° F.

ONEY P. PURYEAR.
HARRY V. ASHBURN.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
2,283,602	Fiero	May 19, 1942
2,308,599	Fraser	Jan. 19, 1943
2,351,384	Woods et al.	June 13, 1944
2,397,956	Fraser	Apr. 9, 1946