

UNITED STATES PATENT OFFICE

2,245,702

GREASE COMPOSITION

Arnold J. Morway, Roselle, N. J., assignor to
Standard Oil Development Company, a corpo-
ration of Delaware

No Drawing. Application November 24, 1937,
Serial No. 176,222

3 Claims. (Cl. 252—40)

The present invention relates to an improved grease composition which has a smooth structure and which is stable both in respect to its structure and to oxidation characteristics. The method of preparing the grease and its composition will be fully understood from the following description.

Industrial greases are prepared by dissolving certain metallic soaps in lubricating oils, and many different soaps have been used. In addition, certain mixed base greases which contain mixtures of different soaps have been known in the art and by suitable mixtures it is possible to obtain properties which are not obtained by the use of any single soap. Soda soaps are used to make hard greases which while desirable for many purposes are not suitable for others. Such greases often contain glycerine and are characterized by a markedly fibrous structure. Barium soap greases, on the other hand, are soft, buttery and without fibre, but such greases must contain water and in consequence are non-reversible; that is to say they lose their structure on heating and do not regain it on subsequent cooling because of the loss of water.

It is desirable to make a grease for use in the lubrication of small gears; for example, in the use of household appliances such as mixers, kneaders, and for timing gears and the like. Such a grease should be buttery, soft, without fibre, reversible and capable of maintaining its structure up to temperatures of about 200° F. or somewhat higher. Such a grease is not limited to the particular uses mentioned above, but may be used also in anti-friction bearings and the like.

It has been found that an excellent grease of the type desired can be prepared from the mixed soaps of soda and baryta, which are free from the difficulties found in greases containing either the one or the other of such soaps alone. The present grease is made from a fatty acid such as stearic acid and the hydroxide of the two metals barium and sodium. It is preferred to use the acids rather than fats. It will be understood that other acids may be employed, for example oleic acid, but a saturated acid is preferred; also mixed acids such as are obtained from commercial fats, lard, tallow and the like can be used as well as acids prepared by oxidation of hydrocarbon oils and waxes.

In the manufacture of the grease, the acid is admixed with hydrocarbon lubricating oil so as to obtain a thorough solution and it is usually necessary to raise the temperature to about 150°

F. in order to accomplish this. The hydroxides of the metals are then added, preferably in aqueous solution. It has been found best to add barium hydroxide first while maintaining a temperature of from 130° to 180° F. The mixing is continued for about an hour at this temperature and thereafter caustic soda is added preferably in an aqueous solution. The mixture is then thoroughly stirred and heated up to about 350° F. or higher, at which temperature the grease becomes smooth but ropery, and steam is allowed to escape so that the final product will be substantially anhydrous. The mixture is then cooled to about 180–200° F. and is worked at this temperature for about one hour. Under such conditions, very little, if any basic barium soap is produced.

The amount of soap in the present composition may vary, say from 10% to 40%, depending on the particular type of grease desired, its penetration and other qualities, and the balance of the composition is a mineral lubricating oil. Any particular type of lubricating oil may be used, but those having viscosities from say 90 to 350 seconds Saybolt at 100° F. are preferred. The ratio in which the two soaps are used may also vary considerably, but the amount of soda soap should not be more than about ten times the amount of the barium soap, so as to prevent the product from becoming too hard and grainy. Similarly, it is preferred that the amount of soda soap should not be less than the amount of barium soap, as such materials are too soft and become too fluid at elevated temperatures. The preferred ratio falls within the range between 2 to 1 and 8 to 1 parts of the sodium soap per part by weight of the barium soap, and by varying this ratio slightly between the limits given, the properties may be gradually changed as desired for specific uses.

The finished grease preferably contains no glycerine or water and has a preferred free alkali content of between 0.05 and 0.3% but not over 0.5% and is for this reason of a non-fibrous, smooth, buttery structure. It is reversible and has an A. S. T. M. penetration between about 200 and 300 at 77° F., and a melting point in the neighborhood of 360° F.; it keeps its body up to temperatures close to the melting point tending to become semi-fibrous around 230–240° F.

As an example of the manufacture of the grease, the following may be considered:

425.0 parts by weight of stearic acid and 500 parts by weight of a mineral lubricating oil having a viscosity of 200 seconds Saybolt at 100° F.

are thoroughly mixed in a grease kettle at a temperature of 140-150° F., until the acid is completely melted and dissolved. Stirring is then begun and 25 parts by weight barium hydroxide (dry basis) are added in approximately saturated aqueous solution. Stirring is continued for about an hour and temperature held as indicated above. 62.0 parts by weight caustic soda are then added (30% aqueous solution) and while stirring the temperature is raised to about 360° F., the balance of the oil being added in small dosages and thoroughly incorporated before further additions. When saponification is completed and water has been substantially, completely eliminated, the product is cooled to about 200° F. while stirring and is worked at that temperature for two to three hours. The grease has the following composition:

	Percent
Barium stearate-----	2.75
Sodium stearate-----	21.25
Mineral oil-----	76.00

The product made according to the above procedure and having the above mentioned composition was found to have an A. S. T. M. penetration test of 250-270 at 77° F.

The material was then tested in the Mechanical stability testing machine of the Bearing Engineers Committee. The machine and the test method was described by C. R. Gillette, at a meeting of the National Association of Grease Manufacturers, Inc., at Chicago, Ill., October 12-13, 1936, the following is quoted from his paper:

"The machine consists of a base, a vertical column, a constant speed 3450 R. P. M. motor and an extension to the motor spindle, upon which is mounted a plate shielded, Conrad type, size 204 S. A. E. bearing. This bearing is mounted in a grease cup which is free to turn with the bearing. A flexible cord connecting this cup with the torque scale spring, provides a means for determining the torque. The grease cup is surrounded by an oil bath which is a counterweighted, adjustable container in which the oil is maintained at the various test temperatures. The oil temperature is recorded by the dial type thermometer.

"The best procedure consists of mounting the bearing upon the shaft with the shield side down and packing the test quantity of grease into the bearing and around the shaft. The grease cup is then assembled on the bearing and the grease in the cup leveled off.

"The oil bath is raised to the stop on the column, with the damping vane on the grease cup in front.

"The flexible cord from the torque spring is attached to the cup by means of the slot provided.

"For the start of the initial test, the oil temperature is adjusted to 80°-85° F. The motor is started and the starting torque is observed. The running torque is recorded at intervals throughout the test. The test is continued for twenty minutes, at which time the torque approaches a constant.

"With the bearing still running, the temperature of the oil bath is raised to 150° F. at the

rate of 10°-12° F. per minute and another 20 minute test is conducted.

"With soda soap and mixed base greases the temperature of the oil bath is again raised, this time to 220° F., where another 20 minute test is made.

"During all of these tests, separation of oil and soap, change in structure, change in consistency and aeration are noted, in addition to the torque readings."

The results of the tests and the temperatures are given in the following tabulation. Torque values are in grams centimeters.

Temperature of test---	80-85° F.	150° F.	220° F.
Starting torque-----	25	-	-
Avg. running torque---	8	4	2½
Rating-----	Good	Good	Good

The grease did not become fibrous, aerate, or materially change its consistency. There was no tendency to leak through the bearing.

For comparison a similar series of tests were made on a typical soda lime grease made from animal fat such as is now used commercially. The ratio of soda to lime was about 5 to 1.

Temperature of test---	80-85° F.	150° F.	220° F.
Starting torque-----	25	-	-
Avg. running torque---	10	6	3
Rating-----	Poor	Poor	Poor

This grease became quite fibrous during the 150° and 220° F. tests, began to climb the shaft and was partly thrown out of the bearing. It had also become much harder after the tests.

In preparing these greases it has been found that excellent products may be prepared when the ratio of sodium to barium soap is in the range of from 2:1 to 3½:1 or from 5:1 to 10:1 and although suitable products have likewise been obtained with intermediate ratios, it is felt that the best products have ratios within one or the other of these ratios.

It will be understood that while the grease is preferably made up as indicated above, the soaps may be made in the absence of the oil and may then be admixed in the desired proportions, or each soap may be made up in a separate portion of the oil and the solutions mixed as desired. In every case, however, it is necessary to thoroughly mix the product, preferably at an elevated temperature.

The present invention is not to be limited to any theory of the effect of either of the ingredients, nor to any particular proportions thereof.

I claim:

1. An improved substantially anhydrous lubricating grease consisting of mineral oil, from 10 to 40% of a soda soap and barium soap mixture, the said soaps being in a proportion of from about 10 to 1, to about 1 to 1.

2. An improved substantially anhydrous grease composition comprising mineral oil and from 10 to 40% of sodium and neutral barium soaps, the latter ingredients being in relative proportions from about 2:1 to 3½:1.

3. An improved substantially anhydrous grease composition comprising mineral oil and from 10 to 40% of sodium and neutral barium soaps, the latter ingredients being in relative proportion from 5:1 to 10:1.

ARNOLD J. MORWAY.