This invention relates to improved calcium base greases. More particularly it relates to anhydrous calcium hydroxy fatty acid soap greases and to a method of preparing them.

This invention is a continuation-in-part of our copending application, Serial No. 388,426, filed October 26, 1953, now abandoned, wherein it is disclosed that stable anhydrous calcium base greases may be successfully prepared by employing a hydroxy fatty acid material as the saponifiable material. By carrying out the preparation by a method which involves saponification in the presence of a small amount of a naphthenic lubricating oil and dehydration at an elevated temperature above the melting point of the soap. This continuation-in-part application involves the additional discovery that in order to obtain a grain-free product of good marketable appearance it is necessary to employ in the saponification a hydroxy fatty acid material which contains a minimum amount of estolide polyesters of the hydroxy fatty acid.

Anhydrous calcium hydroxy fatty acid soap greases have been successfully prepared, as disclosed in the copending application of O. P. Puryear, H. V. Ashburn and J. P. Dilworth, Serial No. 247,558, filed September 20, 1951, now abandoned, by a low temperature process wherein the saponification and subsequent dehydration steps are carried out in a minor portion of the lubricating oil included in the soap at a temperature below the melting point of the soap, and the remainder of the lubricating oil added gradually with continuous stirring while the mass is allowed to cool. Earlier attempts which were made by Fraser, U. S. 2,380,960, and others to prepare calcium hydroxy fatty acid soap greases without the use of water or other stabilizing agent, employing the high temperature method wherein the soap and the lubricating oil are heated together at a temperature above the melting point of the soap until the soap is completely melted, were unsuccessful, separation of the oil and soap taking place during the cooling down process.

Anhydrous calcium base greases of Puryear et al. represent a considerable advance in the art, since they are free from the serious objection of thermal instability due to the loss of water from water-stabilized greases at temperatures above about 212° F., which has placed a severe limitation upon the usefulness of calcium base greases in the past.

However, the method of preparation employed by Puryear et al. has the disadvantage that very careful temperature control is necessary during the dehydration step in order to avoid local overheating and partial melting of the grease mixture, which results in a lumpy and unsatisfactory product. Also, it is difficult to obtain these greases free from graininess; and a special technique, involving cold premixing of the reactants in the saponification mass, in addition to close control of the other operating conditions, is used to obtain greases of marketable appearance.

We have now found that the above difficulties are avoided and anhydrous calcium base greases of excellent smoothness and texture are consistently produced by a method which involves saponifying a hydroxy fatty acid material, containing a small proportion of hydroxy fatty acid estolides, in the presence of a small amount, equivalent to only a minor proportion of the oleaginous liquid lubricating base employed in the grease, of a naphthenic lubricating oil, and then dehydrating the mass at a temperature above the melting point of the soap. The temperature at which the dehydration is carried out will depend somewhat upon the particular calcium hydroxy fatty acid soap in the saponification mass, but in general it is preferable to carry out this process at a temperature of at least about 280° to 300° F. Following the dehydration, additional lubricating oil, sufficient to give a grease of the desired consistency, is added slowly with continuous stirring while the mass is allowed to cool. Additives of the usual types may be added during the cooling down process, ordinarily when the temperature of the mass is below about 200° F.

The hydroxy fatty acid material employed in the saponification is a soap forming hydroxy fatty acid material selected from the group consisting of hydroxy fatty acids containing at least 12 carbon atoms and one or more hydroxy groups, and the glycerides of such acids. The acids are preferably substantially saturated acids containing from 12 to 24 carbon atoms and one or two hydroxy groups, such as mono- and di-hydroxy stearic, oleic, myristic and palmitic acids. A particularly suitable material of this character is 12-hydroxy stearic acid. Other acids of this type which may be mentioned include 9- and 10-hydroxy stearic acids, 9,10-dihydroxy stearic acid and 8-hydroxy palmitic acid.

The estolides which must be present in the hydroxy fatty acid material in order to obtain grain-free greases are intermolecular esters and polyesters formed by the reaction of the hydroxy group of one molecule of the hydroxy fatty acid with the carboxy group of another. The estolides of monohydroxy fatty acids containing 12 to 24 carbon atoms are represented by the formula:

$$\text{R} - \text{O} - (\text{CH}_2\text{OH})_n - \text{R}$$

wherein R is hydrogen or an aliphatic hydrocarbon radical containing from 1 to 21 carbon atoms, and n is an integer having a value of 1 to 22 and n is an integer having a value of 2 to about 10. The estolides of the stearic acid are represented by the above formula wherein R is an alkyl radical containing 6 carbon atoms and x is equal to 10. The 12-hydroxy stearic acid employed in the grease making suitably contains such estolides having an average molecular weight in the range from about 800 to about 1,200, represented by the above formula wherein n has a value of 2 to about 5.

The minimum percentage of estolide which must be present in the hydroxy fatty acid material in order to obtain grain-free greases is about 3 percent by weight, and is preferably in the range from about 4 to about 10 percent by weight, based on the total weight of the hydroxy fatty acid material. Larger amounts of estolides may be present in the saponifiable material without impairing the properties of the grease, but the presence of such large amounts of estolides has the disadvantage of materially decreasing the yield, and amounts in excess of about 15 percent by weight of the fatty acid material are regarded as objectionable for this reason.

The naphthenic oil employed in the saponification mixture is suitably a distillate fraction in the low or medium viscosity lubricating oil range, such as an oil having a Saybolt Universal viscosity below about 600 seconds at 100° F., and preferably below about 350 seconds at 100° F. With particular advantage, it may be very light.
naphthenic lubricating oil having a Saybold Universal viscosity in the range from about 40 to about 200 seconds at 100° F. Suitable proportions of the oil and saponifiable material may be from about 0.5:1 up to about 5:1 by weight, and are preferably from about 1:1 to about 3:1 by weight.

The lubricating oil which is employed for finishing off the grease, and which comprises the chief oil component of the grease, may be of the same type as that employed in the saponification mixture, or a different oil may be employed, such as a paraffinic oil or an oil of higher or lower viscosity. Suitable oils for this purpose include both distillate and residual oils of naphthenic or paraffinic character, having viscosities in the range from about 50 to about 1,200 seconds Saybold Universal at 100° F., as well as blends of such oils. Also, other types of liquid oleaginous lubricating bases can be employed for this purpose either in whole or in admixture with mineral lubricating oils. For example, any of the so-called synthetic lubricants, such as aliphatic dicarboxylic acid esters of the character of 2-ethyl hexyl sebacate, can be used.

In accordance with our preferred procedure, a saponifiable hydroxy fatty acid material containing 3–15 percent by weight of estolides and a naphthenic oil of the character described above and in the disclosed proportions are charged to a grease kettle together with a small amount of water and lime in the calculated amount for saponifying the fatty acid material, together with a slight excess if desired. The grease kettle may suitably be an open type grease kettle equipped with stirrers and a steam heating jacket, or a pressure kettle may very advantageously be employed. The charge is heated with stirring to about 150–210° F., and preferably to about 180–210° F., under either atmospheric or increased pressure, and maintained at this temperature until the saponification is substantially complete, which usually requires from about 2 to about 4 hours. The temperature is then increased up to above the melting point of the soap in the open kettle with continued stirring and held at this temperature for a period sufficient to substantially dehyrate the mass, which usually requires from about one half to about 2 hours or more. The temperature at which the dehydration is carried out will depend upon the melting point of the soap. For example, when 12-hydroxy fatty acid or a glyceride thereof is employed in the saponification, the dehydration is suitably carried out at at least about 280° F., and preferably at a temperature between about 300° F. and about 330° F. The dehydrated mass, with water content below about 0.2 percent and generally not more than about 0.1 percent by weight on the basis of the final grease, is then brought to the desired consistency by the addition of the major quantity of the liquid oleaginous lubricating base with continuous stirring as the mass slowly cools. The grease is preferably stirred down to about 200° F. or lower, and discharged from the kettle at about 160–180° F. by pumping it through straining screens in the conventional manner.

In carrying out the oil addition during the cooling down process, at least a major portion of the oil is preferably added while the saponified mass is at a temperature below the melting point of the soap. When a hydroxy fatty acid is employed as the saponifiable material, the oil addition may be started at a temperature above the melting point of the soap if desired and continued during the entire cooling down process. However, when a hydroxy fatty acid glyceride is employed as the saponifiable material a difference has been noted in this respect, it being necessary for best results to cool the saponified mass down to a temperature below the melting point of the soap and to stir until a grease consistency is obtained before starting the oil addition. For example, in preparations made using castor oil as the saponifiable material, it was necessary to cool to about 250° F. and to stir for at least about an hour at that temperature before adding the additional oil. By employing this precaution, very satisfactory greases have been prepared according to the method of our invention from hydroxy fatty acid glycerides as the saponifiable material.

According to a modification of the above procedure which may be employed very advantageously as a means of avoiding difficulties in stirring the stiff soap base at a temperature below the melting point of the soap, all or a portion of the lime required for the saponification may be added to the mixture of saponifiable hydroxy fatty acid material and oil after the mixture has been heated to a temperature above the melting point of the soap. Where all or a major portion of the lime is added at such higher temperature, the saponification is preferably carried out in a closed kettle under at least moderately elevated pressure. The dehydration and finishing steps may then be carried out in the open kettle in the manner described above. Preferably, a major portion of the lime required for the saponification is added to the original charge and the remainder of the lime is added after at least a partial melting of the soap has taken place. For example, grease preparations have been carried out very successfully employing 12-hydroxy stearic acid as the saponifiable material wherein three-fourths of the amount of lime required for complete saponification was added to the original charge, saponification carried out at 150–210° F., and the remainder of the lime required for completion of the saponification added after the mass was heated to about 270° F.

A particularly valuable application of this invention is found in the preparation of anhydrous calcium hydroxy fatty acid soap greases from very low viscosity oils, such as lubricating oils having viscosities below about 100 seconds Saybold Universal at 100° F. Conventionally, in the preparation of calcium base greases, the soap is prepared in the presence of an oil of about 300 seconds Saybold Universal viscosity at 100° F., and oils substantially below about 100 seconds Saybold Universal viscosity at 100° F. have not been employed for this purpose because of the poor solubility of the calcium hydroxy acid greases in the very low viscosity oils and the very poor yields obtained. In order to obtain greases of satisfactory stability, the use of these light oils in calcium base greases has been limited to only minor amounts which have been employed in blends with heavier oils. We have found that, due to the superior solubility of the calcium hydroxy fatty acid soaps in such oils, these very light oils can also be employed in the soap preparation according to the method of our invention, and very stable anhydrous calcium base greases can be prepared in excellent yield employing these light oils as the chief oil component. A combination of very valuable lubricating properties is thereby obtainable which is not obtainable when either higher viscosity oils or the conventional calcium fatty acid soaps are employed, including outstanding low temperature properties combined with suitability for use at temperatures above 212° F. Also, these greases are highly shear stable and resistant to softening or other deterioration under conditions of high shearing stress, and greases of excellent shear stability, suitable for use as wheel bearing lubricants, are obtained with only relatively low amounts of the calcium hydroxy fatty acid soap in these light lubricating oils, such as about 10 percent or less of the soap for a NLGI No. 2 grade grease. We have found that greases having the difficulty obtainable combination of lubricating properties necessary to meet the requirements of Government Specification MIL–G–10924 for a water resistant, wide temperature range of use grease can be produced by employing a combination of a calcium hydroxy fatty acid soap and a very low viscosity lubricating oil, having a Saybold Universal viscosity at 100° F. below 100 seconds.

Greases of the above type may suitably comprise about 5 to 20 percent by weight of a calcium hydroxy fatty...
acid soap and a mineral lubricating oil having a viscosity in the range from fatty acid to about 100 seconds Saybolt Universal at 100° F. as the oil component. The oil may comprise a blend of lighter and heavier oils, but preferably it contains no more than a minor proportion, such as less than about 25 percent by weight, of an oil having a viscosity outside of the viscosity range discussed above.

A preferred grease composition of this type comprises about 8–15 percent by weight of calcium hydroxy fatty acid soap, including calcium soap of estolides, and a distillate mineral lubricating oil having a Saybolt Universal viscosity at 100° F. of about 50 to about 75 seconds, which may be a blend of lighter and heavier oils in the viscosity range from about 40 to about 100 seconds Saybolt Universal at 100° F., as the oil component. The grease preferably contains a small amount of free alkalinity, suitably about 0.05 to about 0.5 percent by weight, calculated as Ca(OH)₂. A small amount of free fatty acid, such as up to about 0.5 percent by weight may also be present, although this is not necessary for imparting stability or other desired lubricating properties to the grease.

Additives of conventional type may be employed, such as extreme pressure agents, pour depressants, oxidation inhibitors, corrosion inhibitors, dyes and the like. Preferably, the grease contains both a corrosion inhibitor and an oxidation inhibitor, oxidation inhibitors of the amine type, such as diphenylamine, alpha- and beta-naphthylamines and N,N'-diphenyl para phenylenediamine being especially suitable. A particularly effective additive combination of this type has been found to comprise sorbitan monooleate, which may be suitably employed in amounts of about 1–5 percent by weight of the grease, and a commercial oxidation inhibitor (Ortholeum 300) consisting of 95 percent diphenylamine and 5 percent salicylaminoquinuclaine oleate, which may suitably be employed in amounts of about 0.1–1.0 percent by weight of the grease. In addition, a small amount, such as about 0.2–2 percent by weight, of a Manvel residuum (residual oil from Manvel crude) having a Saybolt Universal viscosity of about 600–800 seconds at 210° F. may very advantageously be employed in order to impart a desirable green color to the grease.

A number of anhydrous greases of the above type were prepared very successfully in both laboratory and plant size batches according to the method of this invention from hydroxy fatty acid materials, employing light naphthenic distillate oils having viscosities from about 40 to about 100 seconds Saybolt Universal at 100° F. in the kettle charge and finishing by blending in oils of the same or different types, such as paraffinic oils and oils of different viscosities within the above range. In every case the preparation proceeded without separation of the soap and oil during the finishing process or other difficulties and greases of good stability and other lubricating properties were obtained. In addition, greases which were entirely free from graining were obtained when hydroxy fatty acid materials containing at least 3 percent by weight of estolides were employed in the saponification.

The preparation of a grease of a preferred type in accordance with this invention is described in detail in Example 3 below. Example 1 is representative of unsuccessful attempts which were made previously to prepare anhydrous calcium hydroxy acid greases, and shows the criticality of employing a naphthenic type oil in the saponification and dehydration steps. Example 2 is representative of greases prepared in accordance with the method of this invention but employing estolide-free hydroxy fatty acid materials.

EXAMPLE 1
An open type laboratory grease kettle equipped with stirrers and a steam heating jacket was charged with 2,892,884 pounds of distillate paraffin base mineral lubricating oil of SAE 20 grade having a Saybolt Universal viscosity at 100° F. of 325 seconds, 9 pounds of Hydrofol acids 200, 6.5 pounds of water and 605 grams of dry powdered lime. Hydrofol acids 200 is a product of the Archer-Daniels Midland Co., comprising in major proportion 12-hydroxy stearic acid and containing approximately 5 percent of 12-hydroxy stearic acid estolides having an average molecular weight of about 2,000. The material employed in this preparation had a neutralization number of 174, a saponification number of 187, and iodine number of 3, a titer, ° C. of 74.7 and a hydroxyl number of 148. The kettle contents were heated with stirring to about 185–197° F. and maintained at that temperature for 1 hour to obtain complete saponification. The heat was then increased and the temperature of the saponified mass brought up to about 300° F. in about 1.3 hours, and maintained at 308–320° F. for about 1.1 hours longer to dehydrate. The mass was then allowed to cool gradually while it was attempted to stir in additional mineral oil of the same type as that employed in the original charge. However, the mass rejected oil starting at about 275° F. and broke completely into soap lumps and free oil at about 250° F. The grease was finally stirred down to 200° F. before the oil addition was carried out, in an attempt to smooth out the soap base. An extremely lumpy and grainy product was obtained in a very poor yield, 19.9 percent of the calcium 12-hydroxy stearate being required to give a number 2 grade grease, having an ASTM worked penetration at 77° F. of 298.

EXAMPLE 2
The grease preparation was carried out in substantially the same manner as described in Example 1, except that a naphthenic distillate oil was employed, having a Saybolt Universal viscosity at 100° F. of 59.4, a flash point, COC, of 305° F. and a pour point of -58° F. Also, a 12-hydroxy stearic acid was employed which was free from estolides, as indicated by its hexane insolubility at 32° F. This material had a neutralization number of 180, a saponification number of 186 and a hydroxyl number of 161. The kettle was charged with 14.0 pounds of the lubricating oil, 14.1 pounds of the 12-hydroxy stearic acid, 10.0 pounds of water and 964 grams of dry powdered lime. The charge was heated with stirring to 182–194° F., and maintained at this temperature for 4 hours to complete the saponification. The temperature of the saponified mass was then brought up to about 308° F. in about 2.5 hours and maintained at 308–322° F. for an additional 2.2 hours to dehydrate. The heat was then cut off and additional lubricating oil of the same type as that employed in the original charge was mixed in with continuous stirring while the mass cooled from about 322° F. to about 120° F. during about 20 hours. When the grease was at about 200° F. 4.1 pounds of sorbitan monooleate (Span 80) and 0.69 pound of Ortholeum 300, dissolved in a part of the lubricating oil, were added. About 1.4 pounds of Manvel residuum having a Saybolt Universal viscosity of 731 seconds at 210° F. were also mixed in at this temperature. The grease was finally drawn from the kettle at about 100° F. and pumped through three 60-mesh screens.

No separation of the oil and soap or lump formation occurred during the above process, and a grease was obtained having a dropping point of 286 and a worked penetration at 77° F. of 283 for a soap content of 10.8 percent. However, the grease was very grainy and clogged the screens during the screening process.

EXAMPLE 3
A grease preparation was carried out in substantially the same manner as described in Examples 1 and 2, employing a naphthenic distillate oil of the same character.
as described in Example 2, and an estolide-containing 12-hydroxy stearic acid of the same character as described in Example 1 (Hydrolf acids 200). The kettle was charged with 9.43 pounds of the lubricating oil, 9.43 pounds of the estolide-containing 12-hydroxy stearic acid, 6.5 pounds of water and 629 grams of dry powdered lime. The charge was heated with stirring to 182–197° F., and maintained at this temperature for 4 hours to complete the saponification. The temperature of the saponified mass was then brought up to about 300° F. in about 1.3 hours and maintained at 303–309° F. for an additional 0.5 hour to dehydrate. The heat was then cut off and additional lubricating oil of the same type as that employed in the original charge was mixed in with continuous stirring while the mass cooled from about 309° F. to about 176° F. during about 12 hours. When the grease was at about 190–200° F., 1352.5 grams of sorbitan monooleate (Span 80) and 227.3 grams of Ortholeum 300, dissolved in a part of the lubricating oil, were added. About 450.8 grams of Marveln resin having a Saybolt Universal viscosity of 731 seconds at 210° F. were also mixed in at this temperature. The grease was finally drawn from the kettle at about 170° F. and pumped through three 60-mesh screens. No difficulties of any kind were encountered in this preparation and a smooth glossy butter product was obtained in excellent yield. The following Table 1 shows the composition and tests on this grease.

Table 1

<table>
<thead>
<tr>
<th>Specification Limits</th>
<th>MIL-G-10924</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ca Glaxo stearate (including Ca soaps of estolide)</td>
<td>10.1%</td>
</tr>
<tr>
<td>Extra Ole (OH)</td>
<td>0.1%</td>
</tr>
<tr>
<td>Naphthenic distillate lubricating oil having an SUS viscosity at 100°F of about 60</td>
<td>85.8%</td>
</tr>
<tr>
<td>Sorbitan monolaurate</td>
<td>0.5%</td>
</tr>
<tr>
<td>&quot;Ortholeum 300&quot;</td>
<td>3.0%</td>
</tr>
<tr>
<td>Marveln resin</td>
<td>3.0%</td>
</tr>
<tr>
<td>Test</td>
<td></td>
</tr>
<tr>
<td>Water</td>
<td>0.1%</td>
</tr>
<tr>
<td>Free alkali, as Ca(OH)₂</td>
<td>0.2%</td>
</tr>
<tr>
<td>Free fatty acid, as oleic</td>
<td>0.16%</td>
</tr>
<tr>
<td>ASTM penetrant at 77°F</td>
<td>29°F ± 2°F</td>
</tr>
<tr>
<td>ASTM penetrant at 77°F</td>
<td>25°F ± 2°F</td>
</tr>
<tr>
<td>ASTM penetrant at 100°F</td>
<td>20°F ± 2°F</td>
</tr>
<tr>
<td>ASTM penetrant at 130°F</td>
<td>17°F ± 2°F</td>
</tr>
<tr>
<td>ASTM penetrant at 210°F</td>
<td>10°F ± 2°F</td>
</tr>
</tbody>
</table>

All of the tests listed in the above table were carried out in accordance with the methods prescribed by specification MIL-G-10924.

The apparent viscosity test is a test indicating the suitability of a grease for use at very low temperatures, and is carried out by forcing the grease, at a temperature of 45°F, through a series of capillaries of different sizes by means of a floating piston actuated by a hydraulic system, at shear rates of 25, 100 and 300 reciprocal seconds. The apparent viscosity is calculated from the predetermined flow rate and the force developed in the system. As shown by the table, the grease of Example 3 very satisfactorily met these low temperature requirements of the specification.

The bomb copper corrosion test is carried out by placing a cleaned and polished strip of electrolytic copper (1.75 x 0.25 x 0.02 inch in size and bent to a V-shape) on edge in a ⅛ inch layer of the test grease in a glass dish and placing the dish in a Norma-Hoffmann oxidation bomb for 20 hours at 210°F and 110 p. s. i. After the test the copper strip and the grease are examined for discoloration. As shown by the table, the grease of Example 3 met the oxidation and corrosion resistance requirements of this rigorous test.

The salt spray test is a test commonly employed to show the rust resisting properties of greases under conditions of high humidity and in the presence of rust accelerating salts such as are present in sea water. The test is carried out by thinly coating steel panels with the test grease and maintaining them in contact with a spray of synthetic sea water, containing sodium, calcium and magnesium chlorides and sodium sulfate, at a temperature of 95°F and atmospheric pressure for 100 hours. As required by the specification, the grease of Example 3 permitted no rusting of the panels during 100 hours exposure to the severe rusting conditions of this test.

The wheel bearing test is a test indicating the ability of a grease to maintain satisfactory lubrication without undue softening or other deterioration at elevated temperatures and high shearing rates under service conditions. The test is carried out as described in Ordnance Department Specification AXS-1574 by packing a front wheel hub and spindle assembly with 90 grams of the test grease and rotating the wheel for 6 hours at a speed of 430–450 r. p. m. and at a spindle temperature of 220°F. At the end of the run, the condition of the bearing parts and the appearance of the grease are noted and the amount of leakage is determined by weighing the grease or oil collected in a retainer at the back of the hub. The grease of Example 3 passed this test very satisfactorily, excellent lubrication being maintained with no more than slight deterioration of the grease and with only a small amount of leakage as shown by the table.

The Norma-Hoffmann oxidation test is a well-known test for measuring the resistance to oxidation of a lubricating grease when stored under static conditions for long periods of time. Samples of the grease are tested in a stainless steel bomb sealed in an atmosphere of oxygen under initial pressure of 110 p. s. i. at 210°F, with the pressure drop at the end of 100 hours being recorded. A grease giving a pressure drop of less than 5 p. s. i. per hundred hours up to 400 hours in this test is generally considered to have excellent oxidation resistance properties.

As shown by the data given in the table, the grease of Example 3, representative of the preferred greases of our invention, had the combination of exceptional low temperature properties and good lubricating properties at elevated temperatures, including satisfactorily low evaporation loss, stability, and excellent oxidation and corrosion resistance, necessary to meet the difficult requirements of specification MIL-G-10924. Other tests not listed have also shown this grease to have excellent resistance to softening and oil separation upon severe working and resistance to change upon working in the presence of water.

In addition to the above tests, greases of substantially the same composition as the grease of Example 3 have been tested very successfully as wheel bearing lubricants in actual service tests carried out under severe conditions, including extreme atmospheric temperatures and the presence of water. For example, a grease of this composition gave very outstanding performance as a wheel bearing grease in a 10,000-mile test in Army trucks carried out in the California desert, at an ambient tempera-
ture ranging from 85° F. to 120° F. and hub temperatures as high as 233° F. developed under the severe braking employed. At the end of this test the grease appeared to be unchanged in consistency, the bearings being well lubricated and in good condition and no leakage having occurred from the hubs.

While the method of this invention is of particular utility in the production of stable anhydrous calcium base greases, as discussed above, it is understood that other alkaline earth metal soap greases also may advantageously be prepared by this method, such as, for example, barium 12-hydroxy stearate grease, strontium 12-hydroxy stearate grease, barium 10-hydroxy myristate grease, strontium 12-hydroxy oleate grease, barium 9-hydroxy stearate grease and strontium 10-hydroxy stearate grease. The presence of the prescribed amount of estolide in the hydroxy fatty acid material employed in the saponification is also critical in the production of such other alkaline earth hydroxy fatty acid greases to obtain greases of superior smoothness.

Mixed base greases containing hydroxy fatty acid soaps of two or more different alkaline earth metals or a major proportion of an alkaline earth metal hydroxy fatty acid soap with a minor proportion of an alkali metal hydroxy fatty acid soap are included in the present invention where the type and proportion of the other soap mixed with the major proportion of the alkaline earth metal hydroxy fatty acid soap does not seriously impair the advantageous properties obtained with the straight alkaline earth metal hydroxy fatty acid soap grease. For example, greases of higher dropping points have been obtained without substantial sacrifice of water resistance or other desirable properties of these greases by employing a soap containing about 10 percent by weight of sodium hydroxy fatty acid soap based on the total soap in addition to calcium hydroxy fatty acid 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 therefore only such limitations should be imposed as are indicated in the appended claims.

We claim:

1. The process of preparing a stable substantially anhydrous calcium base lubricating grease which comprises saponifying a hydroxy fatty acid material selected from the group consisting of substantially saturated mono- and dihydroxy fatty acids containing from about 12 to about 24 carbon atoms, and the glycerides thereof, with calcium hydroxide in the presence of a small amount equivalent to only a minor proportion of the liquid oleaginous lubricating base employed in the grease of a naphthenic distillate lubricating oil having a viscosity in the range from about 40 to about 600 seconds Saybolt Universal at 100° F., heating the saponified mass at a temperature above the melting point of the soap for a time sufficient to substantially dehydrate the said mass, and thereafter gradually adding additional lubricating oil with continuous stirring as the said mass is cooled at a temperature below the melting point of the said soap, said hydroxy fatty acid material containing 3–15 percent by weight of estolides having an average molecular weight in about the range 800–1200.

2. The process of claim 1 wherein the said naphthenic distillate lubricating oil and the said hydroxy fatty acid are employed in the saponification in a proportion between about 0.5:1 and about 5:1 by weight.

3. The process of claim 1 wherein the said naphthenic distillate lubricating oil is an oil having a viscosity in the range from about 40 to about 200 seconds Saybolt Universal at 100° F.

4. The process of claim 1 wherein the said hydroxy fatty acid material is 12-hydroxy stearic acid.

5. The process of preparing a stable substantially anhydrous calcium base lubricating grease which comprises saponifying 12-hydroxy stearic acid with lime at 150–210° F. in the presence of about 0.5 to about 5 times the quantity of a naphthenic distillate lubricating oil having a viscosity in the range from about 40 to about 100 seconds Saybolt Universal at 100° F., heating the saponified mass at a temperature from about 280° F. to about 350° F. for a time sufficient to substantially dehydrate the said mass, and thereafter gradually adding additional lubricating oil with continuous stirring as the said mass is cooled down to about 200° F., said 12-hydroxy stearic acid containing 4–10 percent by weight of estolides having an average molecular weight in about the range 800–1200.

6. The process of claim 5 wherein the 12-hydroxy stearic acid is reacted at 150–210° F. with a major portion of the lime required for the saponification and the remainder of the lime required is added to the saponification mass at a temperature about 270° F.

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