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PRODUCTION OF LUBRICATING GREASES FROM MONOHYDRIC ALCOHOL ESTERS OF HYDROXY FATTY ACIDS

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This invention relates to an improved process for manufacturing lubricating greases with the gelling agent being derived from monohydric alcohol esters of hydroxy fatty acids, and to the improved products formed thereby. This invention applies particularly to the case where the soap component is manufactured in the presence of the lubricant base material, this process being referred to herein as in situ lubricating grease formation. This application is a continuation-in-part of Serial No. 211,581, which was filed on February 17, 1951, now abandoned.

It has been known in the prior art to manufacture lubricating greases by processes in which the saponifiable material was a glyceride oil or a monocarboxylic acid. When a glyceride oil serves as the saponifiable material, glycerine is formed and is removed only with difficulty from the reaction mixture. Greases containing glycerine readily pick up moisture, and the presence of such moisture results in corrosion of the metal surfaces with which the greases are in contact. The presence of glycerine also softens the grease undesirably, this being evidenced by an appreciable lowering of the dropping point. This means that a larger amount of the soap component is required to achieve a given hardness in the case of greases containing free glycerine. Another adverse effect of the presence of glycerine is that a number of antioxidants, including phenylene diamine, are thereby rendered less effective in protecting the lubricating grease compositions.

When free monocarboxylic acids serve as the saponifiable material for this type of reaction, water is formed in the saponification reaction, and it is necessary to heat the reaction mixture to temperatures higher than those required in the process of this invention in order to effect the dehydration of such a prior art mixture. The presence of water in the reaction mixture, even for short periods, frequently leads to non-homogeneity of the mixture, and to formation of finished products which are not uniform in quality.

Another difficulty with the use of free monocarboxylic acids in situ formation of lubricating greases is that the elevated temperatures required for this reaction cause some decarboxylation of the acid, and this results in an undesirable lowering of the yield of the soap component. A further difficulty is encountered when free hydroxy fatty acids are used; namely, it is not possible to obtain commercially hydroxy fatty acids which have hydroxyl values approximating the theoretical values. Thus, when hydroxystearic acid is prepared by present commercial methods from hydrogenated castor oil, the acid has a hydroxyl value as low as 152, whereas the theoretical value is 170. This loss in hydroxyl value occurs because of the elevated reaction temperatures required in the preparation of the acid; the processing steps include the boiling of the glyceride with caustic, and boiling of the resulting soap with dilute mineral acid. Reactions such as dehydration and interesterification occur during this processing. However, no lowering of the hydroxyl values is

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encountered in the preparation of the starting esters of this invention. Since the outstanding performance of hydroxy fatty acid soaps in greases is directly dependent on the hydroxyl content, the superior process for grease manufacture is the one that can utilize starting materials with the highest hydroxyl values. That is to say, the gelling efficiency of the soap component in greases is directly related to the hydroxyl content of said component.

It is an object of this invention to overcome the prior art difficulties cited above by the use of a monohydric alcohol ester of a hydroxy fatty acid as the saponifiable material in the in situ formation of lubricating greases. A further object is to provide an in situ process for lubricating grease manufacture in which a monohydric alcohol is formed during the formation of the soap component, the presence of said alcohol aiding in the dispersion of said component so as to obtain a smooth, homogeneous grease. Another object of this invention is to provide a process in which the in situ formed monohydric alcohol is removed from the grease composition after its function therein has been performed. A still further object is the production of markedly improved and novel lubricating greases according to the process of this invention. Additional objects will be apparent from the following description of the invention.

According to the present invention, lubricating greases are produced by a fusion (as opposed to a precipitation) process in which metal compounds are caused to react with monohydric alcohol esters of hydroxy fatty acids in the presence of the unsaponifiable material which is to serve as the base for the final grease composition. Esters of this type have not hitherto been used as the saponifiable material in the in situ production of lubricating greases. They are readily available by low temperature, catalytic alcoholysis of glyceride oils, a suitable process of this type being that shown in my U. S. Patent No. 2,486,444.

The acid portion of the esters utilized in the process of this invention is suitably derived from hydroxy fatty acids having from 9 to 24 carbon atoms per molecule. Examples of suitable acids include the following: 12-hydroxystearic acid, polyhydroxystearic acids, ricinoleic acid, hydroxypelargonic acid, dimethyl hydroxycaprylic acid, dimethyl hydroxycapric acid, 11-hydroxy-9-undecenoic acid, hydroxypalmitoleic acid, ricinelaidic acid, linolic acid, sativic acid, dihydroxygadoleic acid, hydroxybehenic acid, and hydroxylignoceric acid. Suitable hydroxy fatty acids also include those formed by the hydroxylation of unsaturated fatty acids in the indicated range; for example, hydroxylation may be effected by such oxidizing agents as peracetic acid, potassium permanganate, and the like.

The monohydric alcohol portion of the esters used in the process of this invention is required to be low-boiling, as in the in situ formation of the soap component the alcohol is liberated, and is thereafter separated from the reaction mixture by vaporization. These alcohols can be removed from the reaction mixture at lower temperatures than those required for the dehydration of lubricating grease compositions when soaps are formed by the saponification of free monocarboxylic acids. The preferred monohydric alcohol for the present purpose is methyl alcohol, which boils at about 149° F. If higher temperatures are not objectionable in the formation of specific lubricating grease compositions, then it is possible to use higher alcohols up to and including the amyl alcohols as the alcoholic constituent of the esters used in this invention. It is, of course, possible to lower the effective boiling point of these higher alcohols by resort to azeotropic distillation, or by other means. For example, isopropyl alcohol boils at about 180° F., while its azeotrope with n-hexane boils at 142° F. There is, in most cases,

no advantage in using alcohols other than methyl alcohol for forming the esters used in the process of this invention.

The metal compounds used herein for the saponification reaction are selected from the group consisting of hydroxides, oxides, and carbonates of alkali metals, alkaline earth metals, and aluminum. An exemplary list of metals included in the foregoing category includes: lithium, sodium, potassium, rubidium, caesium, beryllium, magnesium, calcium, strontium, barium, and aluminum. The choice of the particular metallic constituent to be used will depend on the exact use to which the final lubricating grease composition is to be put. Thus, for example, greases containing a lithium soap of a saturated hydroxy fatty acid, together with small proportions of a zinc, tin, barium, or aluminum soap, are able to withstand wide variations in temperature and humidity. Greases containing lithium hydroxystearate are more uniform in their characteristics than greases produced with non-hydroxy fatty acids; such greases containing lithium hydroxystearate are water-resistant, have high dropping points, and are further characterized by retention of their consistency over long periods of mechanical working, whereas greases containing the lithium soap of a non-hydroxy fatty acid exhibit much lower mechanical stability under similar conditions. On the other hand, sodium soap greases are quite effective at high temperatures, but are water-soluble and tend to disintegrate under humid conditions. The addition of magnesium or aluminum soaps of hydroxystearic acid to sodium soap greases is capable of rendering such greases water-repellant. Calcium soap greases are characterized by being water-insoluble, but are not normally capable of withstanding high temperatures. However, the melting point and useful lubricating range of calcium soap greases is raised by 100° F. or more by the use of a hydroxy fatty acid, instead of a non-hydroxy fatty acid, in forming the calcium soap component. Barium soap greases are water-resistant, and have comparatively high dropping points. Aluminum soap greases are water-insoluble, but have relatively low transient points. The addition of a lithium soap of a hydroxy fatty acid to a grease containing an aluminum soap of a saturated fatty acid stabilizes the colloidal structure of the aluminum soap grease, thereby preventing syneresis during the storage and use of the grease, restrains the tendency of the aluminum soap grease to assume a rubbery texture at high temperatures, and produces a smoother and more unctuous texture. The use of the aluminum soap of hydroxystearic acid in amounts of about 0.25% to about 10% is effective in preventing syneresis in greases containing same.

With relation to the process of this invention, a desirable preliminary procedure to be followed in the case of metallic compounds which are hydrates or otherwise contain water in that covered in Beerbower et al. Patent No. 2,434,539. This procedure provides for the elimination of water from such compounds by the heating thereof in the presence of a liquid hydrocarbon until the water is driven off.

The in situ lubricating grease formation is preferably effected in the presence of a base material, which should be unsaponifiable. Normally, in the production of lubricating greases, this base material is a mineral oil. Mineral oils of a naphthenic or paraffinic nature are particularly suitable for use in lubricating grease manufacture, the former type of oil being especially useful in the production of low temperature greases. Examples of other suitable base materials include paraffin wax, chlorinated aromatic hydrocarbons, e. g. chlorinated diphenyl, compounds of the type of chlorinated diphenyl oxide, high boiling, liquid polyorganosiloxanes, and the like. The indicated polyorganosiloxanes are especially useful in the formation of texture-stable grease compositions. Typical examples of such compounds are the polymers of dimethyl

silicone, diethyl silicone, ethyl phenyl silicone, and methyl phenyl silicone.

The temperatures used for effecting the in situ lubricating grease formation of this invention are preferably lower than the temperatures shown in the prior art for this general type of reaction. A suitable temperature range for effecting the process of this invention is from 100° F. to 400° F. In one exemplification of the invention, the saponification step is conducted at from about 135° F. to about 190° F. at atmospheric pressure; this temperature range can be suitably modified when operating at subatmospheric or superatmospheric pressures. Thus, the reaction can be carried out at temperatures substantially lower than those required for the reaction when free monocarboxylic acids are used as the saponifiable material, since, in the latter case, water is formed and must ordinarily be removed from the reaction mixture.

The exact temperature to be used in a particular case will be determined by the soap being formed, the base material used, and the use to which the product is to be put. The saponification reaction can be effected at a temperature lower than that required for the evaporation of the liberated monohydric alcohol, but the reaction will go more rapidly to completion if the alcohol is removed as the reaction proceeds. It should be noted that, in order to obtain the full effect of the liberated monohydric alcohol in producing a well dispersed, smooth grease composition, it is preferable, in some instances, to arrange the conditions of temperature and pressure so that the liberated alcohol is not immediately flashed off from the reaction mixture. Also, for the production of a homogeneous composition, it is desirable that the temperature of the reaction mixture, during the reaction or subsequently, approach, but not necessarily be as high as, the melting point of the lubricant composition. Following the completion of the saponification step and of any subsequent heating step, the lubricant composition is cooled, frequently with stirring until the temperature of the mixture is below the critical transition temperature of the grease. Alternatively, the cooling of the lubricating grease composition may be effected by the method of the Woods et al. Patent No. 2,470,965.

In general, substantially stoichiometric amounts of the monohydric alcohol ester and of the metal compound are charged to the reaction vessel. In the case of polyvalent metal compounds, the amounts of reactants will depend on the particular type of metallic soap, or mixture of soaps, desired. The in situ lubricating grease formation can be conducted in amounts of the unsaponifiable base material such as will result in the production of a concentrate containing 50% by weight (a practical upper limit) or more of the soap. In some cases, it will be desired that the final lubricating grease contain a concentration of soap as high as 50% by weight. In most instances, however, it is desired to dilute the concentrate with additional quantities of the base material during the cooling step or while homogenizing the cooled grease. Alternatively, the in situ lubricating grease formation can be conducted in sufficient quantities of the unsaponifiable base material so as to yield a finished lubricating grease with the desired soap content. With reference to lithium hydroxystearate greases, from about 6% to 40% or more by weight of this soap can suitably be incorporated in mineral oil for the production of greases. As regards greases prepared from other soaps, the acceptable range of soap content will depend on the particular soap and particular lubricant base used, and on the processing conditions to which the grease ingredients are subjected during the manufacture of the grease as well as on the conditions to which the finished grease is to be subjected during storage or actual use. said acceptable range for many of such other soaps used in the process of this invention extends from 3% to 50% by weight of the ultimate grease, consistent with the ingredients and resultant requirements stated above.

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Generally, it is desired to maintain the water concentration in the final lubricating grease composition at a minimum. This is quite feasible with the process of the present invention, since it is preferred to use substantially anhydrous reagents in the production of the finished compositions, and since no water is formed in the reaction mixture during the in situ lubricant formation of this invention.

Other ingredients which may be added to the compositions formed by the process of this invention include various corrosion inhibitors, extreme pressure additives, anti-wear agents, stabilizers, V. I. improvers, and the like.

The following examples illustrate various aspects of the process and products of this invention:

Example 1.—A mixture of 50 pounds of lithium hydrate ($\text{LiOH} \cdot \text{H}_2\text{O}$), 49 pounds of mineral oil, and one pound of ricinoleic acid was placed in a steam-jacketed kettle equipped with a stirrer. This mixture was heated to 300° F. with stirring until all of the water was completely driven out. Dehydration of the mixture was complete at the end of about 1.5 hours.

A separate mixture of 372 pounds of the methyl ester of hydrogenated castor oil fatty acids and 375 pounds of paraffin oil was prepared, and heated to approximately 175° F. in a small kettle. Thereafter, the hydroxide slurry as prepared above was added slowly, over about 5 minutes, to the mixture in the kettle. After the reaction was complete, some 1550 pounds of 100 Coastal oil were added over a period of 1 hour, the temperature of the reaction mass being raised during this time to about 325° F. Subsequently, the temperature of the reaction mass was raised to about 415° F., the mass then being allowed to cool, preferably with agitation. The cooling, as well as the other steps of this process, can be carried out by batch or continuous methods.

The process illustrated by this example is distinguished from prior art processes in that it proceeds smoothly to completion. The product of this example is more uniform than is the case when the saponifiable material is hydrogenated castor oil fatty acids, and has a firmer consistency than is the case when the saponifiable material is hydrogenated castor oil.

Example 2.—In this example, comparative data were obtained on the effect of the liberation of a lower monohydric alcohol during the in situ production of lubricating greases. The following table gives the ingredients used in making up the lubricating greases:

Table I

Ingredients	Weight Percent
Saponifiable Material.....	12.00
Lithium Hydroxide (Monohydrate).....	1.68
Phenyl alpha-naphthylamine.....	0.50
Mineral Oil.....	85.82

The saponifiable materials used were methyl hydroxystearate, hydrogenated castor oil, and 12-hydroxystearic acid. The mineral oil had a S. U. S. viscosity of 508 at 100° F. The mineral oil and the saponifiable material were charged to a fire-heated kettle equipped with means for efficient mixing. The temperature of the ingredients was raised to 135-140° F. and the lithium hydroxide was charged. Heating was continued, and the temperature was raised to 400° F. At this temperature, the greases were molten with all of the soap dissolved or dispersed in the mineral oil. The grease was then cooled while mixing to 350° F. The phenyl alpha-naphthylamine was then added with mixing, and cooling was continued without further agitation. The unworked cold grease was homogenized by passage through a Gaulin homogenizer at 3000 p. s. i.

During the manufacture of these greases, it was noted

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that the soaps formed by saponification of methyl hydroxystearate and of hydrogenated castor oil were more easily dispersed in the mineral oil at somewhat lower temperatures than that required for dispersion of the soap formed by saponification of 12-hydroxystearic acid. Also, the finished greases prepared from the former saponifiable materials were smoother and more pleasing in appearance than the grease prepared from the latter material, even after homogenization at high rates of shear.

The data obtained on the finished greases are as follows, the data for the grease from methyl hydroxystearate being in column A; from hydrogenated castor oil, in column B; from 12-hydroxystearic acid, in column C:

Table II

Properties	A	B	C
Appearance.....	Excellent..	Excellent..	Slightly Grainy.
Penetrations, 77° F., mm./10:			
Unworked.....	215.....	310.....	215.
Worked 60 strokes.....	217.....	310.....	256.
Dropping Point, °F.....	342.....	330.....	346.

It should be noted that the finished grease prepared from methyl hydroxystearate had a harder worked consistency than did the other two greases. This advantageous property is due to the greater ease of dispersion of the soap formed from the methyl ester during manufacture.

The ease of preparation and the better appearance of the grease prepared from methyl hydroxystearate appear to be due to the modifying effect of the methyl alcohol, released in the saponification of the methyl ester, on the soap. In the case of the hydrogenated castor oil grease, the glycerine released during saponification is retained in the grease and acts as a softening agent, giving the finished grease an undesirably lower dropping point and requiring a larger amount of expensive thickener (soap) for a given hardness. With methyl hydroxystearate, the methyl alcohol released during saponification imparts beneficial qualities to the grease during manufacture; and these qualities are retained by the grease after manufacture and in the absence of the vaporized methyl alcohol.

Example 3.—Another series of greases was prepared from the same saponifiable materials used in Example 2. A paraffin base mineral oil having an S. U. S. viscosity of about 345 at 100° F. was used, and the three greases were made up to an 8% lithium hydroxystearate content by the same in situ procedure. After the greases had been worked to constant consistency, their penetrations were determined by the A. S. T. M. method with the following results:

Grease from—	A. S. T. M. Worked Penetration at at 77° F.
Methyl Hydroxystearate.....	278
12-Hydroxystearic Acid.....	329
Hydrogenated Castor Oil.....	351

The great improvement obtained when methyl hydroxystearate was the saponifiable material was unexpected, and is the more striking when it is considered that the only variable in these preparations was the saponifiable material and that the content of lithium hydroxystearate in each grease was identical.

Example 4.—Greases were prepared, in the same manner as in Example 3, from methyl hydroxystearate and from hydrogenated castor oil in the presence of a paraffin base mineral oil having a S. U. S. viscosity of about 1025 at 100° F. The content of lithium hydroxystearate

in these greases was 6%, and the dropping points of these greases were as follows:

Grease from—	Dropping Point, ° F.
Methyl Hydroxystearate.....	387
Hydrogenated Castor Oil.....	368

Again, the superiority of the grease prepared from methyl hydroxystearate is evident.

Example 5.—Another set of greases was prepared by the same in situ procedure, the greases having a 6.0% content of lithium hydroxystearate, and the mineral oil utilized having a S. U. S. viscosity of about 470 at 100° F. Data obtained on these greases were as follows:

Grease from—	ASTM Bomb Oxidation Test	Penetration at 0° F.
Methyl Hydroxystearate.....	9, 9 p. s. i. drop in 100 hrs.	201
Hydrogenated Castor Oil.....	13, 14 p. s. i. drop in 100 hrs.	184

It is noteworthy that the grease prepared from methyl hydroxystearate shows greater resistance to oxidation than does the grease prepared from hydrogenated castor oil, and also displays less tendency to harden at low temperatures. This latter result is especially striking when it is considered that the penetration data at 77° F. (supra) show that the consistency of the grease prepared from methyl hydroxystearate is greater than those for the other two greases prepared for comparative purposes.

Example 6.—A mixture of 197 pounds of barium carbonate and 375 pounds of naphthenic mineral oil was prepared, and heated to about 230° F. A separate mixture of 620 pounds of methyl ricinoleate and 375 pounds of naphthenic mineral oil was made up, and heated in a similar manner. The carbonate composition was then added gradually, with agitation, to the ester-oil blend. When the saponification was complete, the reaction mixture was heated to about 300° F. Subsequent cooling of the grease was accompanied by stirring until the grease temperature was less than about 200° F. Additional oil could then be added to bring the soap content of the grease to the desired figure.

As in the preceding examples, the process and product of this example are superior to what can be achieved by prior art processes in which monohydric alcohol esters are not used as the saponifiable material.

Example 7.—157 pounds of methyl hydroxystearate were charged to a steam-jacketed kettle and 570 pounds of mineral oil, having a S. U. S. viscosity of 300 at 100° F., were added. The mixture was stirred and heated to 190° F. 36 pounds of strontium hydroxide (non-hydrated) and 0.67 pound of caustic soda were then added while stirring, and the stirring was continued at this temperature for an hour or longer to complete the reaction. Further mineral oil was then worked into the soap stock to give a grease containing about 7% of strontium 12-hydroxystearate, and this grease was allowed to cool. This process proceeded smoothly to yield a grease having optimum worked penetration properties and dropping point.

Example 8.—2480 gms. of butyl ricinoleate were mixed at about 120° F. with 1750 gms. of a mineral oil having a S. U. S. viscosity of 498 at 100° F., with 750 gms. of n-hexane. 280 gms. of anhydrous flake caustic were dispersed in 550 gms. of the same mineral oil at about 70° F. by ball-milling for 18 hrs. in a 1 gallon capacity porcelain ball-mill having flint pebbles about 1.5 inches in diameter, the mill being about 1/3 filled with the balls. The caustic-oil dispersion was added to the ester-oil mixture, and the resulting mixture was heated to about 190° F. to effect saponification.

750 gms. of the resulting grease base were charged

to a kettle and heated with stirring at 235° F. The temperature was then maintained in the range from 150° to 175° F. and 2000 gms. of a dimethyl silicone polymer, having a flash point above 600° F. and a kinematic viscosity of 82 cs. at 100° F., were slowly added over a period of about 10 hrs. The resulting grease was smooth, showed excellent mechanical stability, had a fairly high dropping point, and possessed good low temperature characteristics.

Example 9.—430 gms. of methyl 11-hydroxy-9-undecenoate, 1000 gms. of paraffinic mineral oil having a S. U. S. viscosity of 300 at 100° F., and 12.5 gms. of zinc naphthenate were blended together at 180° F. 80 gms. of aluminum hydroxide were blended into 750 gms. of the same mineral oil, in which 12.5 gms. of phenyl alpha-naphthylamine had been previously dissolved. The separate blends were metered into a high speed homogenizer. The effluent was then heated to 425° F., and allowed to cool slowly to room temperature without agitation. The resulting grease is smooth, water-proof, and heat stable. In general, aluminum soap greases made from hydroxy fatty acids are superior to aluminum soap greases made from non-hydroxy fatty acids, in that the former are exceedingly stable relative to oil separation, retain their consistency better when worked, and cause no granulation difficulties.

As has been indicated, the greases of this invention are superior to corresponding greases prepared from glycerides or free fatty acids. One factor in this result is the fiber length found in greases prepared from monohydric alcohol esters of hydroxy fatty acids. An industry rule of thumb would predict that greases prepared from such esters would correspond to greases prepared from free fatty acids as regards fiber length. However, electron microscope data show that, in the case of lithium greases prepared from methyl hydroxystearate, 12-hydroxystearic acid, or hydrogenated castor oil, the grease from the methyl ester contained small loosely twisted fibers which were similar to the type found in the glyceride grease, and differed markedly in its superior structural characteristics from the grease prepared from the acid, which existed in granular form with a very few primitive fibers. Thus, the prediction based on the industry rule of thumb proved to be incorrect, and, instead, the greases of this invention are eminently satisfactory as regards fiber length and structure.

The lower processing temperatures which can be achieved by the use of the process of this invention give highly desirable results. The use of higher processing temperatures results in break-down in the fiber structure of the greases, and also has a deleterious effect on the working characteristics thereof. Also, some grease manufacturers do not have the high temperature equipment which is required for the manufacture of, e. g., lithium greases by prior processes; but they can produce lithium greases with their existing equipment at the lower processing temperatures made possible by this invention.

The mechanical stability of greases prepared from monohydric alcohol esters by the process of this invention is superior to that for greases prepared from the corresponding glycerides. Also, as has been indicated, the dispersion of the soap component in the grease is effected more smoothly when operating per the process of this invention than when operating according to prior art processes.

In general, greases prepared from hydroxy fatty acid esters are superior to greases prepared from non-hydroxy fatty acid esters in that, for example, the former can maintain their structure with little change in penetration after extensive working at high or low temperatures, have improved stability under conditions of static heating and under storage, and have improved water-proofing characteristics, as is evidenced especially when water-soluble rust-proofing agents, e. g., sulfonates, are present in the grease.

Under some circumstances, it may be desired to add water to the lubricating greases prepared by the process of this invention. For example, depending on the method of preparation, it may be desirable to stabilize calcium soap greases by the incorporation therein of up to about 2.5% of water. Where, in special cases, it is desired to carry out the in situ lubricating grease formation in the presence of water, results which are superior to those previously obtainable may be achieved by the process of this invention. However, this latter procedure involving the use of monohydric alcohol esters in the presence of water is not preferred.

The process of this invention comprises the formation of soaps from metallic soap-forming materials and hydroxy fatty acid esters of low-boiling monohydric alcohols in the presence of unsaponifiable base materials. This process goes smoothly to completion, can be readily controlled, and yields products which are uniform in quality. The products are not undesirably water-sensitive, as is the case with compositions prepared from glyceride oils and containing glycerine; the glycerine present in such prior art compositions is difficult to remove. Also, it is known that a number of anti-oxidants are more effective in protecting lubricating grease compositions in the absence of glycerine. The process of this invention can proceed at lower temperatures than those required when free monocarboxylic acids are used as the saponifiable constituent of the reaction mixture, since the low-boiling alcohols, such as methanol, formed in the process of the present invention boil at much lower temperatures than does the water formed in the saponification reaction involving free monocarboxylic acids. The desirable absence of water from the reaction mixtures of this invention results in more uniform and more homogeneous finished compositions. Further, the use of the monohydric alcohol esters as the saponifiable constituent avoids the partial decarboxylation and consequent decrease in the soap yield which result when free monocarboxylic acids are used, and enables the production of soap components having maximum hydroxyl values.

The reactions described herein may be carried out either in a batch or continuously. Also, these reactions may be carried out under reduced or increased pressures, the reaction temperatures being adjusted for the particular pressure used.

The superiority of the products of this invention over prior art products is clearly demonstrated by the data in the foregoing examples. This superiority appears to be a result of the process of this invention which involves the liberation of a lower monohydric alcohol during the in situ production of lubricating greases.

Obviously many modifications and variations of the invention, as hereinbefore set forth, will be apparent to those skilled in the art and are within the spirit of the appended claims.

What is claimed is:

1. In the manufacture of lubricating greases, the method which comprises heating about stoichiometric proportions of a metal compound selected from the group consisting of hydroxides, oxides, and carbonates of alkali metals, alkaline earth metals, and aluminum together with an ester of an aliphatic monohydric alcohol having from 1 to 5 carbon atoms and a hydroxy fatty acid having from 9 to 24 carbon atoms in an unsaponifiable lubricant base to a soap-forming temperature, said metal compound and said ester being in sufficient amount so that the resulting soap imparts a grease consistency to the lubricant base, and then heating the mixture to a higher temperature sufficient to form a grease upon subsequent cooling.

2. The process of claim 1, in which the metal component of said metal compound is an alkali metal.

3. The process of claim 1, in which the metal component of said metal compound is lithium.

4. The process of claim 1, in which the metal component of said metal compound is an alkaline earth metal.

5. The process of claim 1, in which said monohydric alcohol is methyl alcohol.

6. The process of claim 1, in which said fatty acid is 12-hydroxystearic acid.

7. The process of claim 1, in which said fatty acid is ricinoleic acid.

8. The process of claim 1, in which said fatty acid is 11-hydroxy-9-undecenoic acid.

9. The process of claim 1, in which said ester is methyl hydroxystearate.

10. The process of claim 1, in which said unsaponifiable lubricant base is mineral oil.

11. In the manufacture of lubricating greases, the method which comprises heating, in an unsaponifiable lubricant base, about stoichiometric proportions of a metal compound selected from the group consisting of hydroxides, oxides, and carbonates of alkali metals, alkaline earth metals, and aluminum together with the ester of an aliphatic monohydric alcohol having from 1 to 5 carbon atoms and a hydroxy fatty acid having from 9 to 24 carbon atoms in sufficient amount so that the resulting soap imparts a grease consistency to the mixture, and then heating the mixture to a temperature sufficient to effect substantial homogeneity thereof.

12. In a process of preparing a lubricating grease composition, said composition comprising an unsaponifiable lubricant base and a metal soap component, the improvement in the step of forming said metal soap component in situ which comprises reacting, in said unsaponifiable lubricant base, substantially stoichiometric amounts of (a) a metal compound selected from the group consisting of hydroxides, oxides, and carbonates of alkali metals, alkaline earth metals, and aluminum and (b) an ester of a monohydric alcohol having from 1 to 5 carbon atoms and a hydroxy fatty acid having from 9 to 24 carbon atoms in sufficient amount so that the resulting soap imparts a grease consistency to the mixture and at a temperature in the range from about 100° to about 400° F.

13. A process of producing a lubricating grease composition which comprises: (a) mixing a metal compound selected from the group consisting of hydroxides, oxides, and carbonates of alkali metals, alkaline earth metals, and aluminum with a methyl ester of a hydroxy fatty acid having from 9 to 24 carbon atoms in about stoichiometric proportions, said mixture being formed in an unsaponifiable lubricant base, the amounts of soap-forming ingredients being such that the final composition contains up to about 50% of the metal soap component and at least a sufficient amount thereof to impart a grease consistency to said composition; (b) heating the resulting mixture under substantially anhydrous conditions to a soap-forming temperature in the range from about 135° F. to about 190° F. at atmospheric pressure, and removing the methyl alcohol from the reaction zone substantially as formed; and (c) completing the heat treatment of the reaction mixture at a higher temperature sufficient to form a grease upon subsequent cooling.

14. In the manufacture of lithium greases, the method which comprises heating, in mineral oil, about stoichiometric proportions of a lithium compound selected from the group consisting of the hydroxide, oxide, and carbonate and methyl hydroxystearate in sufficient amount so that the resulting soap imparts a grease consistency to the mixture, and then heating the mixture to a temperature sufficient to effect substantial homogeneity thereof.

15. In the manufacture of lithium greases, the method which comprises heating about stoichiometric proportions of a lithium base and methyl hydroxystearate in mineral oil to a soap-forming temperature, said base and said stearate being in sufficient amount so that the resulting soap imparts a grease consistency to the oil, and then heating the mixture to a higher temperature sufficient to form a grease upon subsequent cooling.

16. In a process of preparing a lithium grease com-

position, said composition comprising a mineral oil and lithium hydroxystearate, the improvement in the step of forming said lithium soap in situ which comprises reacting, in mineral oil, substantially stoichiometric amounts of (a) a lithium compound selected from the group consisting of the hydroxide, oxide, and carbonate and (b) methyl hydroxystearate in sufficient amount so that the resulting soap imparts a grease consistency to the mixture and at a temperature in the range from about 100° to about 400° F.

17. A process of producing a lithium grease composition which comprises: (a) mixing a lithium compound selected from the group consisting of the hydroxide, oxide, and carbonate with methyl hydroxystearate in about stoichiometric proportions, said mixture being formed in a mineral oil, the amounts of soap-forming ingredients being such that the final composition contains up to about 50% of lithium hydroxystearate and at least a sufficient amount thereof to impart a grease consistency to said composition; (b) heating the resulting mixture under substantially anhydrous conditions to a soap-forming temperature in the range from about

135° F. to about 190° F. at atmospheric pressure, and removing the methyl alcohol from the reaction zone substantially as formed; and (c) completing the heat treatment of the reaction mixture at a higher temperature sufficient to form a grease upon subsequent cooling.

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