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## LITHIUM HYDROXY STEARATE GREASE COMPOSITIONS

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This invention relates to the art of manufacturing lubricating greases comprising mineral oil or other suitable oleaginous vehicles and a metal salt of a hydroxy fatty acid. More particularly, it is concerned with the production of lithium hydroxy fatty acid soap greases of improved yield having minimum bleeding characteristics. The term "yield" as used herein is intended to denote the relation between ASTM penetration and the soap content of the grease. In the manufacture of lithium greases using lithium hydroxy stearate, for example, it has been generally proposed to proceed in three stages. First, the lubricating oil and soap are heated at an elevated temperature to form a homogeneous solution of the ingredients. The resulting composition is cooled to cause gelation. Finally, the gel product is homogenized to impart suitable grease texture. In many instances, the second step, namely that of cooling has been conducted in the past by shock chilling from the homogenizing temperature to the packaging temperature.

Greases prepared by the steps defined above showed evidence of bleeding or poor yield. More recently the need for closer control of operating conditions has received some recognition. Greater advances have been made, however, in connection with sodium, potassium, aluminum and calcium soaps rather than with lithium hydroxy fatty acid soap.

The properties of a grease are dependent not only on the method of its manufacture, but also upon the identity of the components and their ratio to each other. It is evident from these considerations that any description of a set of operating conditions must necessarily be in terms of a general operating range, and that optimum operating conditions exist within said range for a given set of grease forming components.

It is an object of the present invention to improve greases which employ lithium hydroxy fatty acid soaps as the sole or material gelling agent. It is a specific object of this invention to improve the yield of said greases. A further object of this invention comprises the improvement in bleeding characteristics thereof. Other objects will become apparent during the following discussion.

Now, in accordance with this invention a lubricating grease comprising an oleaginous vehicle and a soap consisting of or containing at least 35% of a lithium soap of a higher hydroxy fatty acid is made by heating the ingredients until a homogeneous composition is formed; cooling this

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composition to a temperature between about 110° C. and about 170° C., isothermally gelling the composition between said temperatures, and subsequently cooling and homogenizing the grease thus produced.

The temperature range within which the composition is isothermally gelled is an important criterion determining the eventual properties of the grease. Within the limits of a given composition, the isothermal gelling may take place over a relatively wide range provided that the whole of the composition is in the particular condition which is stable at said temperature. To ensure that the whole of the composition is in its desired condition when isothermally gelled, it is highly desirable that the rate of cooling to the equilibration temperature (hereinafter referred to as the isothermal gelling temperature) be sufficiently slow to enable the grease to complete any changes occurring therein at higher temperatures. Since, however, the compositions are liable to suffer from oxidation if held at high temperatures for long periods and since the oxidation products have a deleterious effect on the yield and on the bleed values, it is preferable to adopt a rate of cooling which will ensure a stable condition but minimize oxidation. The optimum rate of cooling to the equilibration temperature will thus vary with the particular composition employed and with the tendency of the composition to oxidize. In actual practice, under usual conditions of operation, it has been found that the optimum rates of cooling to the equilibration temperature lie between 1 and 3° C. per minute, and preferably between about 1½ and 2½° C. per minute.

During the period of cooling to the equilibration temperature, the composition may be left static or it may be stirred either continuously or intermittently. If left static, gel formation takes place before the equilibration temperature is reached and it may become necessary to break the gel by agitation of the composition. However, if the grease is cooled in thin layers, for example, on a belt, such agitation may not be necessary. It is preferred practice to permit the grease to remain in a static condition both during this cooling period and the equilibration period which follows rather than employing stirring or other means of agitation.

The isothermal gelling of the composition is accomplished by maintaining it statically or with stirring at an equilibration temperature which, as stated above, lies between about 110° C. and about 170° C. Although equilibration periods of

one hour or more are generally sufficient to obtain the results contemplated by this invention, the practical limits on the time of equilibration are from one to six hours and usually from one to four hours. Equilibration periods longer than about two hours usually show little if any advantage over those obtained by the use of the latter period. If the grease is cooled below the equilibration temperature before gelling is complete, the resulting product has a poor yield and a poor bleeding value. As noted hereinbefore, greases gelled by means of a lithium hydroxy fatty acid soap (or of a soap mixture containing at least 35% of said hydroxy fatty acid soap) must be equilibrated within the recited temperature range. The exact optimum temperatures, as mentioned above, will vary somewhat depending on the individual components employed, but preferably equilibration occurs between about 120° C. and about 160° C., and optimum isothermal gelling conditions are found for most systems to be between 125° C. and 150° C.

Prolonging the isothermal gelling period after gelation is complete appears to have no harmful effects, although it is to be avoided (especially if the grease is to be stirred during the isothermal gelling) in order to reduce oxidation to a minimum.

After gelling, the rate of cooling is preferably rapid in order to fix the particular soap structure created at equilibration temperature. This is not, however, essential, since simple air cooling may be employed after gelation is complete. Following the isothermal gelling period, the greases are preferably cooled at a relatively rapid rate to a temperature at least below 60° C. By the term "rapid rate" is meant a cooling rate of from about 5° C. to about 15° C. per minute. This cooling may be conducted by stirring or forcing the grease through apparatus having a high heat transfer capacity, such as through tubular assemblies permitting the rapid dispersal of heat. A second method comprises the use of an agitated film cooler. In such an apparatus the grease is introduced into the interior of a drum whose interior walls are continuously scraped by a screw conveyor or paddle arrangement whereby the grease is forced against the walls of the drum and continuously moved from the injection point to the outlet of the apparatus. By means of the use of such an apparatus the film of grease deposited on the interior surface of the drum is continuously sheared by the action of the screw conveyor or paddles and is thus continuously altered so as to present an ever-changing surface to the heat exchanger for the maximum rate of heat dispersion. It will be evident that such an apparatus permits simultaneous shearing action upon the grease composition which enables the formation of a suitable grease structure.

This same apparatus may, in fact, be used not only as a cooling apparatus but also may be used during the initial steps of raising the mixture of soap and oil to the homogenizing temperature. In the latter instance, the exterior of the drum is heated in order to drive off any water which may be added or formed during soap formation and which must be removed. Due to the constant agitation of the film of the mixture, the maximum rate of water removal occurs and subsequently the maximum rate of heat to the homogenizing temperature takes place. The homogenizing temperature is normally above about 200° C. (preferably above about 210° C.) but is chosen to permit a minimum of thermal de-

composition during the period of heating and cooling to or from the homogenizing temperature.

The process of this invention is applicable to greases wherein the thickening agent is one or more lithium soaps of hydroxy fatty acids having grease forming properties. While unsaturated hydroxy fatty acids may be employed as the sole, principal or minor acid used in forming the soap, saturated higher hydroxy fatty acids are preferred since they appear to have better gelling values than their unsaturated homologs. By the expression "saturated higher hydroxy fatty acid," as used in this specification and in the claims appearing hereinafter, is meant an acid wherein the hydroxy alkyl radical contains more than 10 carbon atoms, and preferably more than 12 carbon atoms in the chain. The invention is thus particularly applicable to greases based on the lithium soaps of hydroxy stearic acids, such as 9-, 10-, 11-, 12- and 13-hydroxy stearic acids or their homologs and analogs, such as hydroxy palmitic acids, including 10- and 15-hydroxy palmitic acid. Other suitable acids for use in the formation of grease forming lithium soaps include the following:

Hydroxy myristic acids e. g., 1-hydroxy myristic acid  
 Hydroxy behenic acid  
 5,6-dihydroxy stearic acid  
 2,11-dihydroxy palmitic acid

Other di- or polyhydroxy fatty acids may be employed such as 6,7-, 7,8-, 8,11-, 9,10-, and 10,11-dihydroxy stearic acids. The most common variety (due to its natural source) is 12-hydroxy stearic acid, which is derived by the saponification of hydrogenated castor oil or hydrogenated castor oil fatty acids.

It has been determined that the process of the present invention is applicable to soap mixtures containing a minimum of 35% lithium hydroxy fatty acid soaps. Preferably, however, the amount of soaps other than the lithium hydroxy fatty acid soaps is less than about 50% of the total soap content. Mixtures of lithium hydroxy fatty acid soaps and lithium fatty acid soaps exhibit maximum response to the process of this invention, as will be shown by the examples appearing hereinafter.

Other soaps which may be mixed with the lithium hydroxy fatty acid soaps include lithium soaps of stearic, myristic, palmitic, oleic and behenic acids; aluminum naphthenate; and sodium, aluminum or calcium stearate or palmitate may be present. Some sodium soaps of hydroxy fatty acids are normally present in the commercial mixtures of soaps formed from commercial lithium hydroxide and hydrogenated castor oil acids. Saponification of the natural glycerides (such as hydrogenated castor oil) increases the gelling properties of the soaps. The grease should usually contain between about 5% and about 25% by weight of soap, and preferably between 6.5% and 15% soap.

The base lubricant for forming lithium soap greases by the process of this invention may be selected from a wide variety of natural and synthetic oils. Mineral oils of wide viscosity range varying from about 50 to 2000 SUS at 100° F. and having a viscosity index of from below zero to about 80 can be used as well as their mixtures. A preferred mineral oil base may be prepared by redistilling a gas oil over caustic soda, extract-

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ing the distillate with sulfur dioxide and refining the raffinate with a suitable adsorbent material to give an oil having the following properties:

Specific gravity at 60° F.-----	0.861
Flash point, ° F.-----	275
Pour point, ° F.-----	-70
Final B. P., ° C. (ASTM)-----	370

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phosphonates, phosphinates, as well as the corresponding oxides. Typical species include:

Tricresyl phosphate  
 Trioctyl phosphate  
 Tributyl phosphate  
 Bis(3,5,5-trimethylhexyl) 2,4,4-trimethylpentene phosphonate

	S. G. Oil (60° F.)	Flash Point Closed (°F.)	Viscosity Red. I (secs.)		K. V. I.	Pour Point, °F.	Leendertse Type Analysis		
			140° F.	70° F.			Aro- matics	Paraf- fins	Naph- thenes
A.-----	0.861	275	-----	61	64	-70	2	50.5	47.5
B.-----	0.828	275	-----	44	-----	25	6	67	27
C.-----	0.867	300	45	-----	55	-40	1	50	49
D.-----	0.934	370	95	-----	10	-20	18.6	50.5	30.9
E.-----	0.904	410	168	-----	46	-15	10	53	37

Mixtures of mineral oil and fixed oils such as castor oil, lard oil and the like can be used as well as organic synthetic lubricants and mixtures thereof such as:

I. Synthetic lubricants produced by the Fischer-Tropsch, Synthol, Synthine and related processes, e. g.:

A. Polymerization of olefins such as ethylene, butylene, and the like, and their mixtures in presence of a Friedel-Crafts or other type condensation catalyst under elevated temperatures and pressures.

B. Polymerization of unsaturated hydrocarbons in presence of a catalyst and then condensing said polymerized product with an aromatic hydrocarbon such as xylol, benzol, naphthalene, etc.

C. Oxidation of polymerized olefins of lubricating range as noted under A and B.

D. Process of converting natural gas to carbon monoxide and hydrogen, followed by catalytic reaction under elevated temperature and pressure to produce hydrocarbons of lubricating range (Synthol process).

II. Synthetic lubricating products produced by the Bergius process, e. g., by:

A. Hydrogenation of coal, peat, and related carbonaceous materials under pressure and elevated temperature in presence of a catalyst.

B. Hydrogenation of asphalts, petroleum residues and the like.

III. Synthetic lubricants produced by the Voltolization process, e. g., by:

A. Voltolization of fatty materials such as fatty oils.

B. Voltolization of mixtures of fatty oils and petroleum hydrocarbons.

C. Voltolization of unsaturated hydrocarbons, their mixtures, and the like.

IV. Organic synthetic lubricants:

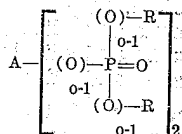
A. Alkyl esters of organic acids, e. g.—  
 Alkyl lactates  
 Alkyl oxalates  
 Alkyl sebacates (2-ethylhexyl sebacate)  
 Alkyl adipates  
 Alkyl phthalates (dioctyl phthalates)  
 Alkyl ricinoleates (ethyl ricinoleate)  
 Alkyl benzoates

B. Alkyl, alkylaryl esters of inorganic acids, e. g., such as the phosphorus esters.

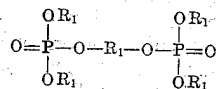
This particularly desirable class of oleaginous bases for the present compositions comprises organic phosphorus esters including phosphates, 75

Tris(3,5,5-trimethylhexyl) phosphate  
 N-heptenyl bis(3-butylpentane) phosphinate  
 Bis(3,5,5-trimethylhexane) octane phosphine oxide

Another highly desirable type of phosphorus lubricants includes the diphosphorus compounds including the four classes referred to above. Preferably, the diphosphorus compounds have a configuration as follows:



wherein A is an organic radical preferably aliphatic or aromatic hydrocarbon radical or alternatively an oxahydrocarbon radical or the corresponding sulfur, selenium or tellurium containing hydrocarbon radicals preferably saturated aliphatic hydrocarbon radicals having from 4 to 12 carbon atoms. The above configuration contemplates diphosphates, diphosphonates, diphosphinates and diphosphine oxides. A particularly desirable configuration comprises those diphosphates having the following configuration:



wherein each R<sub>1</sub> is an aliphatic hydrocarbon radical having from 2 to 6 carbon atoms. It has been found that lubricants of this particular configuration possess unexpectedly extreme low temperature operating characteristics. Species of such lubricants include:

1,4-butanediol bis(dibutyl phosphate)  
 1,3-propanediol bis(diamyl phosphate)

V. Synthetic lubricants made by polymerization of alkylene oxides and glycols at elevated temperatures in the presence of catalysts such as iodine, hydriodic acid, etc.

A. Polymers of alkylene glycol:

Trimethylene glycol  
 Propylene glycol  
 Tetramethylene glycol  
 Hexamethylene glycol  
 Pentamethylene glycol

- B. Copolymers of:
- Trimethylene glycol and triethylene glycol
  - Trimethylene glycol and hexamethylene glycol
  - Trimethylene glycol and B-methyltrimethylene glycol
  - Trimethylene glycol and diethylene glycol

- C. Copolymers prepared from certain epoxides at elevated temperatures and in presence of KOH or BF<sub>3</sub>-ether catalyst, e. g.:

- Ethylene oxide and propylene oxide
- Isobutylene oxide and propylene oxide
- Epichlorohydrin and propylene oxide

- D. Sulfur containing polymers obtained by treating allyl alcohol, divinyl ether, diallyl ether, diallyl sulfide, dimethylallyl ether, glycols, with H<sub>2</sub>S in presence of a catalyst such as toluene sulfonic acid, peroxides, ultraviolet light, e. g.:

- Dihydroxy diethyl sulfide
- Dihydroxy dipropyl sulfide
- Trimethylene glycol and dihydroxy dipropyl sulfide
- Trimethylene glycol and dihydroxy diethyl sulfide

- VI. Polymers obtained from oxygen-containing heterocyclic compounds, e. g., polymerization of tetrahydrofuran in the presence of a catalyst.

- VII. Silicon polymers, e. g.:

- Polyalkyl siloxane and silicate polymers
- Alkylaryl siloxane and silicate polymers
- Dimethyl siloxane and silicate polymers, etc.

Summarizing the steps of the present process, lithium hydroxy soap greases may be prepared by the process of this invention by dispersing in a suitable kettle or vessel a calculated amount of lithium hydroxy fatty acid soap or the materials used for forming the soap in a base oil to form a cold slurry and heating the mixture with agitation to a temperature of about 210-250° C. until a homogeneous mass is obtained. Additional oil may be added to the mixture as required together with additives such as anti-corrosion, antioxidant, anti-bleeding agents, thickeners, etc. The liquid homogeneous mass is cooled from its homogenizing temperature to the equilibrating temperature range and held there for the time as discussed hereinbefore. Following the equilibration, the grease is then cooled preferably at a rapid rate and homogenized prior to packaging. The examples which follow illustrate the process of this invention.

#### EXAMPLE I

93.5 parts of mineral lubricating oil and 6.5 parts of a soap mixture consisting of 20% lithium stearate and 80% lithium 12-hydroxy stearate are mixed together at 210° C. until a homogeneous solution is obtained. The lubricating oil had the following characteristics:

Specific gravity 0.906 at 60° F.  
Flash point 420° F.  
Viscosity 170 seconds  
Redwood I at 140° F.  
Pour point -5° F.

The equilibration temperature was chosen between about 110 and about 150° C., the rate of cooling from the homogenizing temperature being about 2° C. per minute. The greases were equilibrated for two hours at the chosen equilibration temperature, chilled at a rapid rate sub-

sequently to room temperature and milled to obtain a satisfactory grease structure. The process was repeated with the exception that the grease was rapidly cooled from the homogenizing temperature (210° C.) to room temperature without any equilibration period. The table which follows illustrates the effect obtained by following the process of the present invention as compared with the effect of the latter process.

Table I

Equilibration Temperature, °C.	ASTM Penetration, Worked, 60 Strokes	Percent Bleeding
130.....	225	2.9
140.....	221	2.7
150.....	230	3.7
None.....	283	7.9

It will be apparent from the above comparative data that equilibration between 130° C. and 150° C. caused the production of a grease exhibiting a substantially higher yield and lower bleeding than occurred in a grease prepared by rapid chilling from the homogenizing temperature to room temperature.

#### EXAMPLE II

Greases were prepared according to the procedure of Example I with the following modifications: The soap mixture consisted of 35% lithium stearate and 65% lithium 12-hydroxy stearate. These greases were cooled at equilibration temperatures given in Table II below and were compared with a grease prepared by rapid chilling from the homogenizing temperature to room temperature.

Table II

Equilibration Temperature, °C.	ASTM Penetration, Worked, 60 Strokes	Percent Bleeding
120.....	230	3.7
130.....	225	3.1
138.....	221	2.7
None.....	286	8.0

Examination of the above data showed the same relationship exhibited in Table I and indicated the superiority of the process of the present invention.

#### EXAMPLE III

Greases were prepared according to the process described in Example I from mixtures of lithium hydroxy stearate and lithium stearate soaps. Table III which follows presents the data obtained from greases isothermally gelled at the indicated optimum equilibration temperatures:

Table III

Ratio of Lithium Stearate to Lithium 12-hydroxy stearate	Optimum Equilibration Temperature	ASTM Penetration at 25° C.	Percent Bleeding
50-50.....	130	214	1.9
35-65.....	140	221	2.7
20-80.....	140	221	2.7
15-85.....	150	225	4.1

#### EXAMPLE IV

Greases were prepared according to the process described in Example I using 8.5% lithium 12-hydroxy stearate in an oil mixture comprising

three parts by weight of bis(2-ethylhexyl)sebacate and one part by weight of a low viscosity mineral oil. Table IV which follows illustrates the effect of equilibrating the grease within the temperature range of the present process as compared with equilibration at 180° C. which is outside the limits of the present invention:

Table IV

Equilibration Temperature, °C.	ASTM Penetration at 25° C.	Percent Bleeding
140	263	2.3
160	272	2.9
180	338	11.2

## EXAMPLE V

High temperature greases were prepared by following the process of Example I using 15% soap in a Duosol raffinate oil having a Redwood I viscosity at 140° F. of 522 seconds. One grease prepared by mixing 92 parts of lithium 12-hydroxy stearate and 8 parts sodium 12-hydroxy stearate was compared with a second grease containing only the former soap. Table V which follows presents the pertinent physical characteristics of the resulting greases:

Table V

Soap, Li/Na Ratio	ASTM Penetration	Percent Bleeding
92/8	179	Nil
100/0	151	Nil

When similar greases were prepared by the use of a chilling process omitting the equilibration step used in the preparation of the greases illustrated in Table V the resulting products were soft and exhibited bleeding. A cooling rate from the homogenizing temperature (220° C.) at 2° C. per minute was used for all samples and the equilibration temperature of the two greases in Table V was 160° C. whereat the greases were isothermally gelled for about 2 hours.

## EXAMPLE VI

A grease was prepared containing 10% lithium 12-hydroxy stearate and 0.25% 2,4-dimethyl-6-tertiary butyl phenol in a medium viscosity Duosol raffinate. The process for the preparation of these greases was essentially that described in Example I. For the purpose of comparison a grease was prepared employing an equilibration temperature of 210° C. Table VI which follows presents the physical characteristics of these greases and proves the unsuitability of equilibrating such a grease at a temperature outside the range specified by the process of the present invention.

Table VI

Equilibration Temperature, °C.	ASTM Penetration	Percent Bleeding
100	317	9.2
110	291	5.5
120	285	6.6
160	282	5.6
210	410	21.9

## EXAMPLE VII

If greases are prepared containing from 6.5 to 14% lithium hydroxy palmitate in a mineral

lubricating oil, it will be found that maximum yields are obtained by equilibrating said greases at a temperature between 120° C. and 160° C. for a period from about 1 to about 4 hours. If equilibration temperatures are in the order of 90° C. the resulting greases are soft and exhibit bleeding tendencies.

## EXAMPLE VIII

When a grease is prepared containing 8.5% of 9,10-dihydroxy stearic acid lithium soap, maximum yields are obtained when said grease is equilibrated at a temperature of about 145° C. A similar grease chilled from the homogenizing temperature of about 215° C. to room temperature will be found to be unduly soft.

## EXAMPLE IX

One preferred type of apparatus for accomplishing cooling comprises tubes (preferably of glass) having an inside diameter less than one inch and preferably  $\frac{1}{2}$ - $\frac{3}{4}$  inch. In order to demonstrate the use of such equipment, a grease was prepared by homogenizing 5% lithium 12-hydroxy stearate in an Edeleanu raffinate oil at about 210° C., filling the tubes with the homogeneous composition, immersing the tubes in an oil bath maintained at 155-160°, and allowing the grease to assume this temperature for about two hours. After this equilibration period the tubes of grease were transferred to a cold water bath in order to cool the grease. The product was forced from the tubes through a 120 mesh screen to form a grease having an ASTM worked penetration of 253.

A similar grease prepared by chilling from 210° to room temperature without disturbance and without the equilibration period had an ASTM worked penetration of 288.

To stabilize grease of the type described against oxidation it is advisable to add minor amounts of oxidation inhibitors to the grease. Among the anti-oxidants which are effective with grease compositions of the type disclosed are: N-alkyl para-phenylene diamine, condensed polynuclear aromatic mono-amines and alkylolamines. Such inhibitors are N-butyl para-phenylene diamine, N,N'-dibutyl para-phenylene diamine, triethanol amine, etc. Also effective as oxidation inhibitors are alpha or beta-naphthylamine, phenyl-alpha or beta-naphthylamine, alpha-alpha, beta-beta, or alpha-beta-dinaphthylamine, diphenylamine, tetramethyl diamino diphenyl methane, petroleum alkyl phenols, 2,4-ditertiary butyl 6-methyl phenol, 2,4-dimethyl 6-tertiary butyl phenol, polyalkylene glycols, methyl cellulose, carboxymethyl cellulose, glycols and water, etc.

Corrosion inhibitors which are particularly applicable with compositions of this invention are N-primary amines containing at least 6 and not more than 18 carbon atoms in the molecule such as hexylamine, octylamine, decylamine, dodecylamine, octadecylamine, heterocyclic nitrogen containing organic compounds such as alkyl substituted oxazoline salts of fatty acids.

Extreme pressure agents can be added to such grease and the preferred comprise esters of phosphorus acids such as triaryl, alkyl-hydroxy aryl, or aralkyl phosphates, thiophosphates or phosphites, etc., neutral aromatic sulfur compounds such as diaryl sulfides and polysulfides, e. g., diphenyl sulfide, dicresyl sulfide, dibenzyl sulfide, methyl butyl diphenol sulfide, etc., diphenyl selenide and diselenide, dicresyl selenide and polyselenide, etc., sulfurized fatty oils or

esters of fatty acids and monohydric alcohols, e. g., sperm oil, jojoba oil, etc., in which the sulfur is tightly bound, sulfurized long-chain olefins obtained by dehydrogenation or cracking of wax, sulfurized-phosphorized fatty oils, acids, esters and ketones, phosphorus acid esters having sulfurized organic radicals, such as esters of phosphoric or phosphorus acids with hydroxy fatty acids, chlorinated hydrocarbons such as chlorinated paraffins, aromatic hydrocarbons, terpenes, mineral lubricating oil, etc., or chlorinated ester of fatty acids containing the chlorine in position other than alpha position.

Additional ingredients which can be added are anti-wear agents such as oil-soluble urea or thio-urea derivatives, e. g., urethanes, allophanates, carbazides, carbazones, etc., or rubber, polyisobutylene, polyvinyl esters, etc.; viscosity index (V. I.) improvers such as polyisobutylenes having a molecular weight above about 800, voltolized paraffin wax, unsaturated polymerized esters of fatty acids and monohydric alcohols, etc.; oiliness agents such as stearic and oleic acids and pour point depressors such as chlorinated naphthalene to further lower the pour point of the lubricant.

The above additives can be added to grease composition of this invention in amounts of from about 0.01% to less than 10% by weight, and preferably 0.1 to 5.0% by weight.

Lithium soap greases made by the process of this invention are stable, non-bleeding products applicable for numerous purposes such as aircraft greases, automotive greases, and wherever a wide temperature, water-resistant grease is required.

We claim as our invention:

1. A method of manufacturing a lubricating grease which comprises heating an oleaginous vehicle mixed with a grease forming amount of alkali metal aliphatic monocarboxylic acid soaps containing at least 35% lithium hydroxy fatty acid soaps until a homogeneous composition is formed, cooling said composition to a temperature between about 110° C. and about 170° C. at a rate between 1° C. and 3° C. per minute, isothermally gelling the composition between said temperatures and subsequently cooling the grease.

2. In a process of preparing a stable grease comprising an oleaginous vehicle and alkali metal aliphatic monocarboxylic acid soaps containing at least 35% by weight of lithium hydroxy fatty acid soaps having at least 10 carbon atoms in the hydroxy aliphatic hydrocarbon radical thereof, the steps comprising heating said mixture to a temperature above about 210° C. until a homogeneous composition is formed, cooling said composition to a temperature between about 110° C. and about 170° C. at a rate between 1° C. and 3° C. per minute, isothermally gelling the composition between said temperatures for a period between about 1 and about 6 hours and cooling and homogenizing the grease.

3. In a process of preparing a stable grease comprising an oleaginous vehicle and lithium soaps, said soaps containing at least 35% by weight of lithium hydroxy fatty acid soaps, the steps comprising heating said mixture to a temperature above about 210° C. until a homogeneous composition is formed, slowly cooling the homogenized composition to a temperature between about 110° C. and about 170° C. at a rate between 1° C. and 3° C. per minute, isothermally gelling the composition between said temperatures and subsequently rapidly chilling the grease.

4. A method of manufacturing a lubricating

grease which comprises heating a mineral oil and lithium higher fatty acid soaps, said soaps comprising at least 35% of lithium hydroxy higher fatty acid soaps at a temperature above about 210° C. until a homogeneous composition is formed, agitating and cooling said composition to a temperature between about 120 and 160° C. at a rate between 1° C. and 3° C. per minute, isothermally gelling the composition between said temperatures for a period between about 1 and about 6 hours and subsequently cooling and agitating the grease.

5. A method of manufacturing a lubricating grease which comprises heating a mineral lubricating oil and grease forming amount of alkali metal aliphatic monocarboxylic acid soaps containing at least 35% by weight of lithium 12-hydroxy stearate to a temperature above about 210° C. until a homogeneous composition is formed, cooling the composition at a rate of from about 1 to about 3° C. per minute to a temperature between about 110 and about 170° C., isothermally gelling the composition between said temperatures and subsequently cooling the grease so formed.

6. In a process of preparing a stable grease composition comprising a mineral lubricating oil and a grease forming amount of alkali metal aliphatic monocarboxylic acid soaps containing at least 35% by weight of lithium 12-hydroxy stearate, the steps comprising heating said composition to a temperature above about 210° C. until a homogeneous composition is formed, slowly cooling this composition to a temperature between about 110 and 170° C. at a rate between 1° C. and 3° C. per minute, isothermally gelling the composition between said temperatures for a period from about 1 to about 6 hours, and subsequently cooling and homogenizing the grease.

7. A method of manufacturing a lubricating grease which comprises heating an oleaginous aliphatic diester and a grease forming amount of alkali metal aliphatic monocarboxylic acid soaps containing at least 35% by weight of lithium 12-hydroxy stearate to a temperature above about 210° C. until a homogeneous composition is formed, slowly cooling the composition to a temperature between about 110 and about 170° C. at a rate between 1° C. and 3° C. per minute, isothermally gelling the composition between said temperatures for a period between about 1 and about 4 hours and subsequently cooling and homogenizing the grease so formed.

8. A method of manufacturing a lubricating grease which comprises heating a mineral lubricating oil and a grease forming amount of soaps comprising lithium stearate and at least 35% by weight of said soaps of lithium hydroxy stearate to a temperature above 210° C. until a homogeneous composition is formed, cooling the composition at a rate between about 1 and about 3° C. per minute to a temperature between about 110 and about 170° C., isothermally gelling the composition between said temperatures and cooling and homogenizing the grease.

9. In a process of preparing a stable grease composition comprising a mineral lubricating oil and a mixture of a major amount of alkali metal aliphatic monocarboxylic acid soaps containing at least 35% by weight of lithium 12-hydroxy stearate and a minor amount of sodium 12-hydroxy stearate, the steps comprising heating said composition to a temperature above about 210° C. until a homogeneous composition is formed, slowly cooling the composition to a temperature

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between about 120 and about 160° C. at a rate between 1° C. and 3° C. per minute, isothermally gelling the composition between said temperatures for a period between about 1 and about 6 hours and subsequently cooling the grease.

10. A method of manufacturing a lubricating grease which comprises heating a mineral lubricating oil and a grease forming amount of alkali metal aliphatic monocarboxylic acid soaps containing at least 35% by weight of lithium hydroxy stearate to a temperature above about 210° C. until a homogeneous composition is formed, agitating and cooling the composition to a temperature between about 125 and 150° C. at a rate between 1° C. and 3° C. per minute, isothermally gelling the composition between said temperatures for a period from about 1 to about 4 hours and subsequently cooling and homogenizing the grease.

11. In a process of preparing a stable grease comprising a mineral oil and a grease forming amount of alkali metal aliphatic monocarboxylic acid soaps containing at least 35% by weight of lithium 12-hydroxy stearate, the steps comprising heating said components to a temperature above about 210° C. until a homogeneous composition is formed, cooling the composition at a rate between about 1 and about 3° C. per minute to a temperature between about 120 and 160° C., isothermally gelling the composition between said temperatures for a period between about 1 and about 6

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hours and cooling the grease at a rate from about 1 to about 15° C. per minute.

12. In a process for preparing a grease comprising mineral lubricating oil and 6.5-15% by weight of alkali metal aliphatic monocarboxylic acid soaps containing at least 35% by weight of lithium 12-hydroxy stearate, the steps comprising heating the components to about 210° C. until a homogeneous composition is formed, cooling the composition at a rate between about 1½ and 2½ degrees centigrade per minute to a temperature between 125 and 150° C., isothermally gelling the composition between said temperatures for about 2 hours, and cooling and homogenizing the grease.

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