

PATENT SPECIFICATION

(11) 009
1364 648

1364 648

- (21) Application No. 52658/71 (22) Filed 12 Nov. 1971
(31) Convention Application No. 90877 (32) Filed 18 Nov. 1970 in
(33) United States of America (US)
(44) Complete Specification published 29 Aug. 1974
(51) International Classification C10M 5/14, 7/20
(52) Index at acceptance
C5F 321 326 343 362 370 452 50X 522 523 532 537 53X
544 545 55X 595 632 807 80X C D
(72) Inventors DONALD WILLIAM MURRAY and WARREN
CHARLES PATTENDEN



(54) LITHIUM SOAP GREASE

(71) We, ESSO RESEARCH AND ENGINEERING COMPANY, a Corporation duly organised and existing under the laws of the State of Delaware, United States of America, of Linden, New Jersey, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention concerns a modified lithium soap grease of premium quality with outstanding properties which include a high dropping point, excellent oxidation stability, long lubrication life and water resistance.

Lithium greases have been known and widely used for many years. The lithium soaps that are used as the thickening agents for these greases are ordinarily prepared by reaction of lithium hydroxide or other suitable lithium base with a conventional high molecular weight acid or acids. The principal advantages of lithium greases have been high water resistance and ease of dispersion of the soaps in all types of lubricating oil base stocks. Particularly useful have been greases prepared from lithium hydroxystearate, since the soaps of the hydroxystearic acids and related hydroxy fatty acids have been found to be more mechanically stable than the corresponding soaps of the conventional fatty acids.

There are many fields of application of grease compositions where a high dropping point is required, as for example in the lubrication of traction motor bearings. Such traction motors are used to propel modern diesel locomotives. The bearings of these locomotives may be required to operate for periods of as much as three years without any maintenance, and temperatures as high as 250°F. can be reached in such bearings.

In accordance with the present invention, a lithium base grease composition having excellent oxidation and mechanical stability and a dropping

point of 500°F. or higher, comprises a major proportion of a lubricating oil and from 2 to 30 wt. %, based on the total composition, of (a) a lithium soap of a C₁₂ to C₂₄, preferably C₁₆ to C₂₀, hydroxy fatty acid, (b) a second hydroxy carboxylic acid of from 3 to 14 carbon atoms, wherein the hydroxy group is attached to a carbon atoms not more than 6 carbon atoms removed from the carboxyl group, and (c) a dilithium salt of a C₄ to C₁₂ dicarboxylic acid; the weight ratio of each hydroxy acid to the dicarboxylic acid being as hereinafter specified.

It has already been taught in U.S. Patent 2,940,930 that high dropping point greases (500°F. or greater) can be prepared from mixtures of monocarboxylic and dicarboxylic acids. However, in preparing the greases described in that patent, it was necessary to also include a glycol. The presence of a glycol is undesirable because it renders the grease prone to oxidation and makes the water resistance of the grease undesirably low in some applications. The present invention makes possible the preparation of high dropping point greases from a combination of hydroxy fatty acid and dicarboxylic aliphatic acid without the necessity of incorporating a glycol.

While U.S. Patents 3,223,633 and 3,223,624 teach the preparation of high dropping point greases from a 3-component mixture of acids, the presence of a C₂ to C₄ fatty acid salt, which is an essential component of those greases, is frequently undesirable because of the tendency of such greases to undergo surface hardening on standing.

The hydroxy fatty acid employed in preparing the greases of this invention will preferably have from 16 to 20 carbon atoms, and will most preferably be a hydroxystearic acid, e.g. 9-hydroxy, 10-hydroxy, or 12-hydroxystearic acid. Ricinoleic acid, which is an unsaturated form of 12-hydroxystearic acid, having a double bond in the 9—10 position, can also be used.

2
The hydroxy carboxylic acid forming the second acid component of the grease of this invention is one having an OH group attached to a carbon atom that is not more than six carbon atoms removed from the carboxyl group. This acid has from 3 to 14 carbon atoms and can be either an aliphatic acid, such as lactic acid, 6-hydroxydecanoic acid, 3-hydroxybutanoic acid, or 4-hydroxybutanoic acid, or an aromatic acid such as parahydroxybenzoic acid, salicylic acid, 2-hydroxy-4-hexylbenzoic acid, meta-hydroxybenzoic acid, 2,5-dihydroxybenzoic acid (gentisic acid); 2,6-dihydroxybenzoic acid (gamma-resorcylic acid); 4-hydroxy-3-methoxybenzoic acid, or a hydroxy-aromatic aliphatic acid such as ortho-hydroxyphenyl-, meta-hydroxyphenyl-, or parahydroxyphenyl-acetic acid. A cycloaliphatic hydroxy acid, such as hydroxycyclopentyl carboxylic acid or hydroxynaphthenic acid, can also be used.

5
10
15
20
In place of the free hydroxy acid of the latter type when preparing the grease, one can use a lower alcohol ester, e.g. the methyl, ethyl, or propyl, isopropyl or sec-butyl ester of the acid, e.g. methyl salicylate, to give a better dispersion when the salt is insoluble. The amount of lithium salt of the second hydroxy acid will range from 0.1 to 10 wt. % of the finished grease, or preferably from 0.5 to 5 wt. %.

25
30
The dicarboxylic acid will have from 4 to 12 carbon atoms, preferably 6 to 10 carbon atoms. Such acids include succinic, glutaric, adipic, suberic, pimelic, azelaic, dodecanedioic, and sebacic acids. Sebacic acid and azelaic acid are preferred.

35
40
The total soap and salt content of the grease will be in the range of from 2 to 30 weight percent and preferably 5 to 20 weight %.

45
50
55
The weight ratio (calculated as free acid) of the C_{12} to C_{24} hydroxy fatty acid to the dicarboxylic acid will be in the range of 0.5 to 15 parts by weight of the former to one part by weight of the latter, and preferably in the range of 1.5 to 5 parts by weight of the hydroxy fatty acid to one part by weight of the dicarboxylic acid. The weight ratio (calculated as free acid) of the second hydroxycarboxylic acid to the dicarboxylic acid will be from 0.025 to 2.5 parts by weight of the former to one part by weight of the latter, preferably 0.125 to 1.25 parts by weight of the former per part by weight of the latter.

60
The lubricating oil base used in preparing the grease compositions of this invention can be any of the conventionally used mineral oils, synthetic hydrocarbon oils, or synthetic ester oils, and will generally have a viscosity within the range of 35 to 200 SUS at 210°F. Synthetic lubricating oils that can be used include esters of dibasic acids such as di-2-ethylhexyl sebacate, esters of glycols such as the C_{12} Oxo

acid diester of tetraethylene glycol, or complex esters such as a complex ester formed by reacting one mole of sebacic acid with two moles of tetraethylene glycol and two moles of 2-ethylhexanoic acid. Other synthetic oils that can be used include synthetic hydrocarbons, such as alkyl benzenes, e.g. alkylate bottoms from the alkylation of benzene with tetrapropylene, or the copolymers of ethylene and propylene; silicone oils, e.g. ethyl phenyl polysiloxanes, methyl polysiloxanes, etc.; polyglycol oils, e.g. those obtained by condensing butyl alcohol with propylene oxide; carbonate esters, e.g. the product of reacting C_8 Oxo alcohol with ethyl carbonate to form a half ester followed by reaction of the latter with tetraethylene glycol, etc. Other suitable synthetic oils include the polyphenyl ethers, e.g. those having from about 3 to 7 ether linkages and about 4 to 8 phenyl groups.

65
70
75
80
85
90
95
100
105
110
The greases of this invention can be formed in a number of different ways. One convenient way when the second acid is salicylic acid is to co-neutralize the hydroxy fatty acid and the dicarboxylic acid in at least a portion of the oil with lithium hydroxide. This neutralization will take place at a temperature in the range 180 to 220°F. When the soap stock has thickened to a heavy consistency, the temperature is raised to 260 to 300°F. to bring about dehydration. The soap stock is then cooled to 190 to 210°F. and the ester of salicylic acid is added; then, additional lithium hydroxide is added gradually to convert the salicylate ester to the dilithium salicylate salt. Reaction is conducted at 220 to 240°F., preferably with agitation so as to facilitate the reaction. In this reaction, the alcohol is evolved, and dilithium salicylate forms. Dehydration is then completed at 300 to 320°F. after which the grease is heated at 380—390°F. for 15 minutes to improve its yield and is then cooled while additional oil is added to obtain the desired consistency. Alternatively, the additional oil can be added to the soap concentrate prior to the in situ formation of the dilithium salicylate.

115
120
An alternative method is to co-neutralize all three types of acid used in making the grease, or to saponify a lower ester of the second type of hydroxy acid, e.g. methyl salicylate, simultaneously with the neutralization of the hydroxy acid of the first type, e.g. hydroxystearic acid and the dicarboxylic acid. Still another alternative is to co-neutralize the hydroxy fatty acid and the ester of the second hydroxy acid followed by neutralization of the dicarboxylic acid.

EXAMPLE 1.

This example illustrates the preparation of a grease of this invention on a plant scale in a 1000 lbs. kettle. The ingredients used and their proportions are given in the following

TABLE I

Ingredient	Weight (lb) per 100 lb. Finished Base Grease
12-Hydroxystearic Acid	11.50
Azelaic Acid	3.97
Lithium Hydroxide Monohydrate(c)	3.34
Methyl Salicylate	2.86
Lithium Hydroxide Monohydrate(d)	1.55
Lubricating Oil(a)	81.29
	104.50(b)

- (a) Mixture of solvent refined and dewaxed paraffinic distillates, hydrofinished, having a viscosity of 520 SUS at 100°F. and a V.I. of 93.
- (b) 4.50 lb. of water and methanol are formed through reaction of the base with the fatty acids and methyl salicylate. These are lost during the dehydration cycle, leaving a net yield of 100 lb. of finished base grease.
- (c) This portion of the $\text{LiOH} \cdot \text{H}_2\text{O}$ required to neutralize the 12-hydroxystearic and azelaic acids only.
- (d) This portion of the $\text{LiOH} \cdot \text{H}_2\text{O}$ required to react with the methyl salicylate to produce dilithium salicylate.

The 12-hydroxystearic acid and the azelaic acid were charged to the kettle with about 55 weight % of the base oil, and the mixture was heated at 195–200°F. until the carboxylic acids had dissolved. Then while the kettle temperature was maintained at 200 to 205°F., a hot saturated aqueous solution containing sufficient lithium hydroxide to react with the hydroxystearic acid and the azelaic acid was added gradually over a period of one hour. When the soap stock had thickened to a heavy consistency, the temperature was raised to about 270°F. as quickly as the dehydration process would permit. When dehydration was essentially complete, as indicated by a rapid increase in temperature to 300°F., the soap stock was cooled to 200°F. by circulating cold water through the jacket of the kettle; then the methyl salicylate was added and mixed with the soap stock at 200°F. Following this, the remainder of the lithium hydroxide monohydrate (1.55 lbs. per 100 lbs. of the finished grease) was added very gradually as a hot saturated solution. At this stage the heat input and mixing rate were increased to speed up the dehydration process (conducted at 212–275°F.) and to avoid accumulation of free water in the kettle. When all of the lithium hydroxide solution had been added and dehydration was complete as noted by rapid increase in temperature, the entire mass of grease was heated to a temperature of 385°F.

and maintained there for 15 minutes to improve the grease structure. The grease mixture was then rapidly cooled to about 300°F. by circulating cold water through the jacket of the kettle and while adding a small amount of oil. After the temperature had been reduced to less than 300°F., the remaining oil was blended into the grease while it cooled gradually. After the base grease had been cooled to a temperature of between 150° and 200°F. it was passed through a 100 mesh screen to complete its preparation. The product was a smooth uniform grease, beige in color, with an unworked penetration of 230 at 77°F. The worked penetration of this base grease at 77°F. was 230 after 60 strokes, 240 after 10,000 strokes and 251 after 100,000 strokes.

EXAMPLE 2.

To prepare a grease suitable for lubrication of locomotive traction motor bearing, the base grease of Example 1 was modified by adding thereto by simple mixing 0.5 wt. % of phenyl alpha-naphthylamine to serve as an oxidation inhibitor, 3.5 wt. % of the base oil described in Table I, and 1.75% of a rust inhibitor concentrate identified as Na Sul BSN, which consisted of 50 wt. % of a light mineral lubricating oil and 50 wt. % of a neutral barium dinonyl naphthalene sulfonate.

The properties of this grease are com-

pared in Table II with a conventional lithium hydroxystearate grease and with a lithium hydroxystearate grease, containing dilithium salicylate, identified as DLS grease. The conventional lithium hydroxystearate grease was a commercial product containing lithium 12-hydroxystearate as the only thickener. The grease DLS also had lithium 12-hydroxystearate as the only thickener but it additionally contained 3 weight percent of finely ground preformed dilithium salicylate powder as an antioxidant.

It is to be noted that the grease of Example 2 had a dropping point of over 500°F., as compared with 394°F. for the conventional lithium hydroxystearate grease and 370°F. for the grease into which powdered dilithium salicylate had been incorporated.

TABLE II
COMPARISON OF PROPERTIES OF GREASES

	Example 2 Product	Conventional Lithium Hydroxy- stearate Grease	DLS Grease
Oil Content, Wt. %	80.1	86	82.1
Oil Viscosity, SUS at 100°F.	520	485	520
ASTM Worked Penetration, 77°F., 60 strokes			
5,000	248	253	234
10,000	265	—	250
100,000	265	—	250
	277	264	304
ASTM Roller, 100 Hr. at 160 rpm			
Δ ASTM Pen. (77°F.)/Adhesion:	+28/Pass	+38/Pass	+120/Pass
	+58/Pass	—	—
Water Absorption in ASTM Roller			
% H ₂ O Absorbed/Δ ASTM Pen. (77°F.)/Adhesion:	54/+12/Pass	—	66/+108/Pass
	58/+44/Pass	67/+76/Pass	36/+92/Pass
ASTM Dropping Point, °F.	500+	394	370
*ASTM Wheel Bearing, 6 Hr. at 200°F.			
Leakage, g/Slump	1.1/Nil	0.7/Nil	0.9/Nil
Oil Separation, 30 Hr. at 212°F., Wt. %	1.9	1.1	3.9
NLGI Spindle Life, 10,000 rpm, 300°F., Hr.	2,388	288	1,566
ASTM Bomb Oxidation, 210°F.			
Pressure Loss, psig, after:			
100 Hr.	1.5	5.0	2.5
500 Hr.	5.0	10.0	11.5
ASTM D 1743 Rust Test	Pass	Fail	—
Copper Corrosion Test, 24 Hr. at 212°F.	Pass	Fail	Pass

*ASTM D—1263—53T.

EXAMPLE 3.

Using the procedure of Examples 1 and 2, a grease similar to that of Example 2, but using a lower viscosity oil (250 SUS at

100°F. and 90 V.I.) was prepared. The composition of this grease, on the basis of weight percent of reactants, is given in Table III.

TABLE III

	Wt. %
Mineral Oil, 90 V.I., 250 SUS at 100°F.	81.01
12-Hydroxystearic acid	8.77
Azelaic acid	2.67
Methyl salicylate	1.09
Lithium hydroxide monohydrate	3.21
NaSul BSN (rust inhibitor)	1.75
Phenyl α -naphthylamine (oxidation inhibitor)	0.50
Pour depressant(*)	1.00

*Oil concentrate, 20% active ingredient, of 2 parts wax-alkylated naphthalene and 1 part C_8 — C_{12} alkyl methacrylate polymer.

10 This grease had a dropping point of more than 600°F. and a worked penetration of 283 at 77°F. after 60 strokes. It had an operating range of -40°F. to +400°F. and successfully passed the copper corrosion test (24 hours at 212°F.) and the ASTM D-1743
15 rust test. The starting and running torque values of this grease at -40°F. compared

very favorably with those of a commercial grease which had been prepared from a lower viscosity oil (90 V.I., 200 SUS at 100°F.) with a calcium-lithium 12-hydroxy stearate soap mixture. The comparison is shown in Table IV. Taking into account the viscosity of the oil used, the grease of Example 3 is considered to have excellent torque properties.

TABLE IV

ASTM D-1478 TORQUE
(g. cm. at -40°F.)

	Starting	Running
Example 3 Grease	8300	2360
Commercial Ca-Li Grease	10,400	3000

EXAMPLE 4.

(Base Grease Preparation).

30 This example describes the preparation of a base grease using a mole ratio of 1 mole of hydroxystearic acid, and 0.75 mole of azelaic acid, first forming a soap concentrate and then a grease. Into 300 grams of 90 V.I. mineral lubricating oil having a viscosity at 100°F. of 550 SUS there were dispersed 92 grams of
35 12-hydroxystearic acid and 43.2 grams of azelaic acid at a temperature of 200°F. Then 31.7 grams of lithium hydroxide hydrate

(LiOH.H₂O) was added and the reaction was continued at 200—240°F., after which the reaction product was dehydrated by heating it to 300°F. The free alkali content of the dehydrated product was 0.18%, which indicated that neutralization of all of the carboxyl groups was essentially complete. The temperature of the dehydrated product was raised to 390—400°F. for 30 minutes in order to improve the dispersion. The soap concentrate product was then cooled while additional oil was added to give the desired consistency.

This product is identified as a base grease in Table V.

product that was obtained was identified as Grease D.

EXAMPLE 5.

To make a grease of the present invention the same procedure was followed as for the soap concentrate of Example 4. After the soap concentrate had been heat treated at 390—400°F., 30.4 grams of methyl salicylate and 17 grams of lithium hydroxide monohydrate were added at 220°F. and the reaction was continued for one hour at this temperature. In this reaction methyl alcohol was evolved and dilithium salicylate was formed in the concentrate. After dehydration had been conducted at 300°F. the grease was cooled and oil was added to obtain the desired consistency. The grease is identified in Table V as Grease A.

EXAMPLE 6.

A second grease was prepared by starting with the base grease of Example 4 rather than with the soap concentrate. Methyl salicylate and lithium hydroxide monohydrate were reacted in the base grease at 220°F. in the same manner as described for the preparation of grease A, Example 5. The difference was that the methyl salicylate and lithium hydroxide were reacted in the finished base grease rather than in the soap concentrate. The

COMPARATIVE EXAMPLE — I.

The effect of omitting the dicarboxylic acid soaps from the grease was determined by preparing a grease similar to greases A and D omitting the dilithium azelate. The grease is identified as Grease E.

The properties of the base grease and of greases A, D and E are given in Table V. Greases A and D both have a mole ratio of 1 mole of hydroxystearic acid, 0.75 mole of azelaic acid, and about 0.7 mole of salicylic acid. The data obtained with grease E show the presence of the dicarboxylic acid soap is necessary in order to obtain a satisfactorily fine dilithium salicylate in the grease as well as to insure that the grease will have a dropping point of at least 500°F.

The static oxidation test for which data are given in Table V was conducted in the following manner. Five grams of the grease being tested was packed into an open ball bearing of known weight which was then hung in an oven maintained at 350°F. At periodic intervals the bearing was removed from the oven, and weighed again to determine weight loss of the grease. The results were reported in terms of the number of hours for a 35% weight loss.

TABLE V
PROPERTIES OF LITHIUM HYDROXYSTEARATE-DILITHIUM AZELATE GREASES

GREASE	Base Grease	A	D	E
Composition, Parts by Weight				
Lithium 12-hydroxystearate	93.6	93.6	93.6	102.0
Dilithium Azelate	45.9	45.9	45.9	—
Mineral Oil, 90 V.L., 550 SUS at 100°F.	917.0	830.5	917.0	885.0
Dilithium Salicylate (3%), made in situ via methyl salicylate	—	30.0	32.7	31.0
	1056.5	1000.0	1089.2	1018.0
DLS Particle Size	None	Fine	Fine	Coarse
ASTM Penetration, 77°F., mm/10: 60 strokes 100,000 strokes	291	305 335	304	248
	—	—	—	—
ASTM Dropping Point, °F.	390	525	500+	385
Static Oxidation Test, 350°F., hours to 35% weight loss	80	280	295	—
Water Resistance in ASTM Roller, 150°F.				
% H ₂ O Absorbed/ASTM Penetration Change, 77°F.	—	78/+32	—	—
Oil Separation, Federal Test Method, 30 hr.				
212°F., Wt. %	—	2.7	—	—
ASTM Wheel Bearing, 6 hr. at 220°F., Leakage in grams/slump	0.6/Nil	1.6/Nil	—	—

COMPARATIVE EXAMPLE — IA.

5 The formulation of Grease D was repeated, using the same amounts of each component as for that grease, except that the dilithium salicylate was added to the base grease as a fine performed powder (all particles less than 150 microns in size) rather than being formed in situ in the grease. The resulting comparative grease was of substantially the same consistency as Grease D (ASTM penetration 300) but it had a dropping point of only 374°F. and had a life of only 175 hours in the Static Oxidation Test. These data show that the second acid component must be a component of the thickener system. It is desirable but not necessary that it be formed in situ but, in any case, the second acid cannot be added as preformed salt after the thickener system has been formed.

20 EXAMPLE 7.

Using the same general procedure as

described in Example 1, several grease formulations were made in which, in one instance, lower mole ratios of dibasic acid and methyl salicylate were used (Grease F), and in other instances a C_{10} or a C_{18} dibasic acid was used in place of azelaic acid (Greases G and H). The formulations of these greases and their properties are given in Table VI. An additional grease formulation (Grease I) wherein stearic acid was employed instead of hydroxystearic acid resulted in a fluid product containing undispersed soap rather than a grease. Formulation J shows that a high dropping point grease can also be obtained by forming dilithium salicylate in situ from salicylic acid instead of methyl salicylate.

The data for greases G and H show that sebacic acid can be satisfactorily used in place of azelaic acid but that a C_{18} acid (brassylic) did not produce a high dropping point grease.

TABLE VI
PREPARATION OF LITHIUM HYDROXYSTEARATE — DILITHIUM DICARBOXYLATE GREASES

Formula	A	F	G	H	I	J
Composition, Mole Ratio						
LiOH, H ₂ O	3.83	3.10	3.94	3.83	3.35	3.0
Stearic Acid	—	—	—	—	1.0	—
12-Hydroxystearic Acid	1.0	1.0	1.0	1.0	—	1.0
Azelaic Acid (C ₉)	0.75	0.55	—	—	0.75	0.55
Sebacic Acid (C ₁₀)	—	—	0.75	—	—	—
Dimethyl Brassylate (C ₁₃)	—	—	—	0.75	—	—
Methyl Salicylate	0.67	0.50	0.72	0.67	0.43	—
Salicylic Acid	—	—	—	—	—	0.50
Mineral Oil, 90 V.I., 550 SUS at 100°F., Wt. %	83.1	84.9	84.0	81.0	77.4	84.5
ASTM Penetration, 77°F. 60 Strokes, mm/10	305	317	300	317	Fluid	360
ASTM Dropping Point, °F.	525	500+	500+	396	—	520+

COMPARATIVE EXAMPLE — II.

Substituting 0.5 mol of lithium acetate for 0.5 mole of dilithium salicylate in Grease F gave a grease having a dropping point of only 420°F.

COMPARATIVE EXAMPLE — III.

A grease similar to grease G was prepared from one mole of 12-hydroxy stearic acid, 0.75 mole of sebacic acid, and 0.80 of benzoic acid. The total amount of oil in the grease was 83.5%. The grease had an ASTM penetration of 336 at 77°F. and a dropping point of 450°F. This compares with a dropping point of more than 500°F. for grease G showing that the substitution of a non-hydroxylated acid such as benzoic acid in place of a hydroxylated acid such as salicylic acid reduces the dropping point of the grease.

EXAMPLE 8.

Using the procedure of Example 6 a grease was prepared in which the acids used had a mole ratio of 1 mole of 12-hydroxystearic acid, 0.75 mole of azelaic acid and 0.75 mole of parahydroxy benzoic acid. A portion of the base grease of Example 4 was heated to 180°F. and then the benzoic acid was added along with sufficient lithium hydroxide to convert the acid to a dilithium salt. The mixture was then heated for 1/2 hour at 230°F. and then dehydrated for one hour at 300°F. The grease had a dropping point in excess of 500°F. and had an ASTM penetration of 325 at 76°F. (330 after 60 strokes). The total thickener content of this grease was 18.1 wt. %.

EXAMPLE 9.

Following the procedure of Example 8, a grease was made using 0.75 mole of lactic acid instead of the parahydroxy benzoic acid. In this case, the base grease was made from an 87 V.I. oil having a viscosity of 600 SUS at 100°F. rather than 550 SUS at 100°F. and 90 V.I. The total thickener content of the grease as lithium salts was 15.3 wt. %. The dropping point of the grease was 515°F and the grease had a penetration at 82°F of 328 (332 after 60 strokes).

WHAT WE CLAIM IS:—

1. A lubricating grease composition which comprises a major proportion of a lubricating oil and from 2 to 30 weight %, based on the

total compositions, of a thickener system having three essential components.

(a) a lithium soap of a C_{12} to C_{24} hydroxy fatty acid;

(b) a lithium salt of a second hydroxy carboxylic acid of from 3 to 14 carbon atoms, wherein the hydroxy group is attached to a carbon atom not more than 6 carbon atoms removed from the carboxyl group;

and

(c) a dilithium salt of a C_4 — C_{12} dicarboxylic acid, wherein there exists a weight ratio, calculated as free acids, of 0.5 to 15 parts of the C_{12} to C_{24} hydroxy fatty acid per part of said dicarboxylic acid, and a weight ratio, calculated as free acids, of 0.025 to 2.5 parts of said second hydroxy carboxylic acid per part of dicarboxylic acid.

2. A grease composition as claimed in claim 1, wherein the proportion of said C_{12} to C_{24} hydroxy fatty acid is from 1.5 to 5 parts by weight per part by weight of said dicarboxylic acid.

3. A grease composition as claimed in claims 1 or 2, wherein the proportion of said C_3 to C_{14} hydroxy carboxylic acid is from 0.125 to 1.25 parts by weight per part by weight of said dicarboxylic acid.

4. A grease composition as claimed in any of claims 1 to 3, wherein the lithium salt of said second hydroxy carboxylic acid has been formed in situ in the grease.

5. A grease composition as claimed in any preceding claim, wherein said second hydroxy carboxylic acid is salicylic acid.

6. A grease composition as claimed in any one of claims 1—4, wherein said second hydroxy carboxylic acid is lactic acid or parahydroxy benzoic acid.

7. A grease composition as claimed in any preceding claim, wherein said hydroxy fatty acid is 12-hydroxystearic acid.

8. A grease composition as claimed in any preceding claim, wherein said dicarboxylic acid is azelaic acid or sebacic acid.

9. A grease composition as claimed in any preceding claim and substantially as herein described.

10. A grease composition as claimed in claim 1, and substantially as herein described in any of Examples 1 to 3 or 5 to 9.

K. J. VERYARD,
15, Suffolk Street, S.W.1.
Agents for the Applicants.

COMPARATIVE EXAMPLE — II.

Substituting 0.5 mol of lithium acetate for 0.5 mole of dilithium salicylate in Grease F gave a grease having a dropping point of only 420°F.

COMPARATIVE EXAMPLE — III.

A grease similar to grease G was prepared from one mole of 12-hydroxy stearic acid, 0.75 mole of sebacic acid, and 0.80 of benzoic acid. The total amount of oil in the grease was 83.5%. The grease had an ASTM penetration of 336 at 77°F. and a dropping point of 450°F. This compares with a dropping point of more than 500°F. for grease G showing that the substitution of a non-hydroxylated acid such as benzoic acid in place of a hydroxylated acid such as salicylic acid reduces the dropping point of the grease.

EXAMPLE 8.

Using the procedure of Example 6 a grease was prepared in which the acids used had a mole ratio of 1 mole of 12-hydroxystearic acid, 0.75 mole of azelaic acid and 0.75 mole of parahydroxy benzoic acid. A portion of the base grease of Example 4 was heated to 180°F. and then the benzoic acid was added along with sufficient lithium hydroxide to convert the acid to a dilithium salt. The mixture was then heated for 1/2 hour at 230°F. and then dehydrated for one hour at 300°F. The grease had a dropping point in excess of 500°F. and had an ASTM penetration of 325 at 76°F. (330 after 60 strokes). The total thickener content of this grease was 18.1 wt. %.

EXAMPLE 9.

Following the procedure of Example 8, a grease was made using 0.75 mole of lactic acid instead of the parahydroxy benzoic acid. In this case, the base grease was made from an 87 V.I. oil having a viscosity of 600 SUS at 100°F. rather than 550 SUS at 100°F. and 90 V.I. The total thickener content of the grease as lithium salts was 15.3 wt. %. The dropping point of the grease was 515°F and the grease had a penetration at 82°F of 328 (332 after 60 strokes).

WHAT WE CLAIM IS:—

1. A lubricating grease composition which comprises a major proportion of a lubricating oil and from 2 to 30 weight %, based on the

total compositions, of a thickener system having three essential components.

(a) a lithium soap of a C_{12} to C_{24} hydroxy fatty acid;

(b) a lithium salt of a second hydroxy carboxylic acid of from 3 to 14 carbon atoms, wherein the hydroxy group is attached to a carbon atom not more than 6 carbon atoms removed from the carboxyl group;

and

(c) a dilithium salt of a C_4 — C_{12} dicarboxylic acid, wherein there exists a weight ratio, calculated as free acids, of 0.5 to 15 parts of the C_{12} to C_{24} hydroxy fatty acid per part of said dicarboxylic acid, and a weight ratio, calculated as free acids, of 0.025 to 2.5 parts of said second hydroxy carboxylic acid per part of dicarboxylic acid.

2. A grease composition as claimed in claim 1, wherein the proportion of said C_{12} to C_{24} hydroxy fatty acid is from 1.5 to 5 parts by weight per part by weight of said dicarboxylic acid.

3. A grease composition as claimed in claims 1 or 2, wherein the proportion of said C_3 to C_{14} hydroxy carboxylic acid is from 0.125 to 1.25 parts by weight per part by weight of said dicarboxylic acid.

4. A grease composition as claimed in any of claims 1 to 3, wherein the lithium salt of said second hydroxy carboxylic acid has been formed in situ in the grease.

5. A grease composition as claimed in any preceding claim, wherein said second hydroxy carboxylic acid is salicylic acid.

6. A grease composition as claimed in any one of claims 1—4, wherein said second hydroxy carboxylic acid is lactic acid or parahydroxy benzoic acid.

7. A grease composition as claimed in any preceding claim, wherein said hydroxy fatty acid is 12-hydroxystearic acid.

8. A grease composition as claimed in any preceding claim, wherein said dicarboxylic acid is azelaic acid or sebacic acid.

9. A grease composition as claimed in any preceding claim and substantially as herein described.

10. A grease composition as claimed in claim 1, and substantially as herein described in any of Examples 1 to 3 or 5 to 9.

K. J. VERYARD,
15, Suffolk Street, S.W.1.
Agents for the Applicants.

