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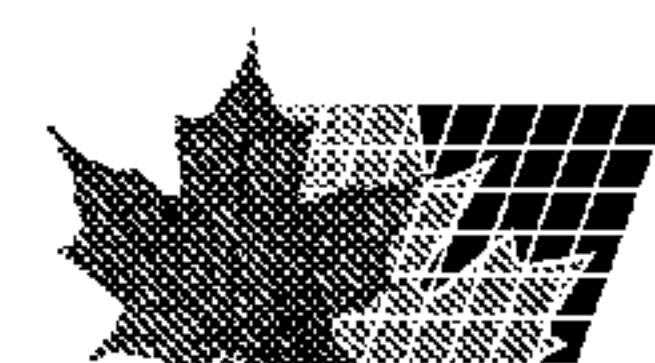
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(54) Titre : COMPOSITION DE GRAISSE
(54) Title: GREASE COMPOSITION

(57) **Abrégé/Abstract:**

A lubricating grease having improved noise properties and high dropping point. The grease comprises a base oil, from 2 to 30 wt.% of a lithium soap complex thickener and from 0.1 to 10 wt.% of a polyalkenyl succinic anhydride, polyisobutenyl succinic acid or mixture thereof.



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(21) International Application Number: PCT/US95/07471 (22) International Filing Date: 12 June 1995 (12.06.95) (30) Priority Data: 08/261,491 17 June 1994 (17.06.94) US (71) Applicant: EXXON RESEARCH AND ENGINEERING COMPANY [US/US]; 180 Park Avenue, P.O. Box 390, Florham Park, NJ 07932-0390 (US). (72) Inventors: IKEUCHI, Hiromitsu; 3-8-2, Aizawa, Seya, Yokohama 246 (US). KOBAYASHI, Takeo; 2364-2, Kamit-suruma, Sagamihara 228 (JP). SHIGANO, Shin; 343-1, Kamikodanaka, Nakahara, Kawasaki 211 (JP). (74) Agents: ALLOCCA, Joseph, J. et al.; Exxon Research and Engineering Company, P.O. Box 390, Florham Park, NJ 07932-0390 (US).		(81) Designated States: CA, JP, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
(54) Title: GREASE COMPOSITION (57) Abstract A lubricating grease having improved noise properties and high dropping point. The grease comprises a base oil, from 2 to 30 wt.% of a lithium soap complex thickener and from 0.1 to 10 wt.% of a polyalkenyl succinic anhydride, polyisobutenyl succinic acid or mixture thereof.		

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GREASE COMPOSITIONBACKGROUND OF THE INVENTION1. Field of the Invention

This invention relates to grease compositions having low noise properties and high dropping points.

2. Description of the Related Art

The bearings in modern automobiles and electric appliances are typically sealed bearings. Greases for sealed bearings must provide lubrication for the life of the bearing which places high demands on greases used in such service. Sealed bearing greases must meet numerous performance requirements including extending bearing life, high temperature performance, high dropping point, low oil separation, oxidation stability, fretting wear protection and low noise.

Recently, lithium complex soap based greases have been used in sealed bearing applications. Such greases offer long service at high temperatures but suffer from generally higher noise than simple lithium soap based greases.

It would be desirable to have a sealed bearing grease containing a lithium complex soap which does not cause high noise in sealed bearings. For high temperature service improvement in grease dropping point is also desirable.

SUMMARY OF THE INVENTION

This invention relates to a grease composition having improved noise properties and high dropping point which comprises:

- a) a major amount of a lubricating base oil,

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b) from 2 to 30 wt%, based on grease, of a lithium soap complex thickener, and

c) from 0.1 to 10 wt%, based on grease, of a polyalkenyl succinic anhydride, a polyalkenyl succinic acid or a mixture thereof.

The greases according to the invention provide long bearing life and high dropping point while providing low bearing noise.

DETAILED DESCRIPTION OF THE INVENTION

A wide variety of lubrication base oils can be employed in preparing the grease compositions of the present invention. Accordingly, the lubricating oil base can be any of the conventionally used mineral oils, synthetic hydrocarbon oils or synthetic ester oils, or mixtures thereof depending upon the particular grease being prepared. In general these lubricating oils will have a viscosity in the range of about 5 to about 400 cSt at 40°C, although typical applications will require an oil having a viscosity ranging from about 10 to about 200 cSt at 40°C. Mineral lubricating oil base stocks used in preparing the greases can be any conventionally refined base stocks derived from paraffinic, naphthenic and mixed base crudes. Synthetic lubricating oils that can be used include esters of glycols such as a C₁₃ oxo acid diester of tetraethylene glycol, or complex esters such as one formed from 1 mole of sebacic acid and 2 moles of tetraethylene glycol and 2 moles of 2-ethylhexanoic acid. Other synthetic oils that can be used include synthetic hydrocarbons such as polyalphaolefins; 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 C₈ 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. The amount of lubricating oil in the grease can also vary

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broadly, but, typically, will range from about 50 wt% to about 98 wt%, preferably from about 75 wt% to about 95 wt%, of the grease.

The grease composition contains a thickener dispersed in the lubricating base oil to form a grease. Preferred thickeners are lithium soap complex thickeners containing a lithium soap of a C₁₂ to C₂₄ hydroxy fatty acid and a second lithium salt compound which is a lithium salt of a C₂ to C₁₂ aliphatic or cycloaliphatic dicarboxylic acid, a lithium salt of a C₃ to C₁₄ hydroxy carboxylic acid which has the hydroxy group separated from the carboxyl group by six or less carbon atoms or a mixture thereof. The lithium salt of boric acid may be added as a third thickener component. Preferred hydroxy fatty acids include hydroxystearic, hydroxyricinoleic, hydroxybehenic and hydroxypalmitic. Especially preferred is 12-hydroxystearic acid. The second lithium salt compound is preferably a C₆ to C₁₀ aliphatic dicarboxylic acid, more preferably azelaic or sebacic acids, especially azelaic acid. The hydroxy-carboxylic acid is preferably lactic acid, salicylic acid or other hydroxy-benzoic acids, more preferably salicylic acid. The amount of lithium soap complex thickeners is preferably from 5 to 20 wt%, based on grease. The ratio of hydroxy fatty acid to aliphatic dicarboxylic acid and/or hydroxy-carboxylic acid is from 10:0.5 to 10:15, preferably 10:1.5 to 10:6.

The grease composition will also contain a polyalkenyl succinic anhydride, a polyalkenyl succinic acid or mixture thereof as dispersant. The polyalkenyl group will have a number average molecular weight of from 300 to 4,000, preferably 400 to 2,500. Preferred polyalkenyl groups are poly-C₃ and poly-C₄ olefins, especially polyisobutylene. Polyisobutylene succinic anhydride and/or acid will hereinafter be collectively referred to as PIBSA. The amount of dispersant is preferably from 0.1 wt% to 5 wt%, based on grease.

Lithium complex soap greases are prepared by heating hydroxy fatty acid and base oil, adding a concentrated aqueous solution of lithium hydroxide. Heating is continued at elevated temperature to remove most of the water. The reaction mixture is then cooled to about 100°C and dicarboxylic acid is added followed by a second

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portion of concentrated lithium hydroxide. This mixture is again heated to elevated temperature to again remove water. After heating is completed, the grease mixture is cooled, remaining oil and any other additives added and the grease is worked up using conventional techniques. The preparation may also be accomplished in a single step process by heating, base oil, fatty acid and dicarboxylic acid, then adding lithium hydroxide solution followed by dehydration and adding remaining oil.

The grease composition may contain small amounts of supplemental additives which include, but are not limited to, anti-corrosive agents, extreme pressure antiwear agents, pour point depressants, tackiness agents, oxidation inhibitors, dyes and the like, which are incorporated for specific purposes. The total amount of these additives will typically range from about 2 wt% to about 10 wt% based on total weight of the grease composition.

This invention will be further understood by reference to the following examples which include a preferred embodiment of the invention.

Example 1

This example illustrates the preparation of a low noise grease. A laboratory kettle was charged with 700 g of refined paraffinic base oil having a viscosity of 90 cSt at 40°C, 139.2 g of 12-hydroxystearic acid, 69.5 g of azelaic and 12.0 g of PIBSA having a polyisobutenyl radical of number average molecular weight of 950. The resulting mixture was heated with stirring until the temperature reached 90°C. At this point, heating was stopped and 345 g of a 15% solution of lithium hydroxide (heated to 90°C) was added with stirring. When addition of the lithium hydroxide solution was complete, heating was resumed to remove water. An additional 100 g of oil was added during the dehydration process to control grease hardening. Heating was continued to a temperature of 210°C at which time heating was discontinued and an additional 128 g of oil was added. The heated mixture was allowed to cool slowly to a temperature of 70°C or less.

The cooled greases was milled at 0.005 inch clearance and deaerated. The grease samples described in Table 1 of Example 3 were manufactured using this procedure. Component amounts were varied as described in Samples 1-3 and Comparative Samples 4-14.

Example 2

Grease noise was measured according to the following procedure which is more fully described in Japanese Patent 53-2357. Solid particles in grease result in vibrations which can be converted into an electrical signal. After correcting for race noise of bearings under standard conditions, the electrical signal due to solid particles including soap particles in grease can be measured according to the following procedure. A vibration detection device is attached to the outer ring of the bearings. The detection device converts the vibrations into an electrical signal which is amplified and sent to a zone wave filter. The zone wave filter compares the amplified signal received from the bearings with the known vibration frequency from the bearings alone, filters out the vibration frequencies from the bearings and sends the filtered electronic signal through an amplifier to a clipper circuit. The clipper circuit "clips" the electrical signal from the amplifier at a given level. This level is set so as to measure only peak voltages which correspond to solid particle noise from particles in the grease. The clipping level is set at a value greater than the peak value corresponding to the race noise from the bearings.

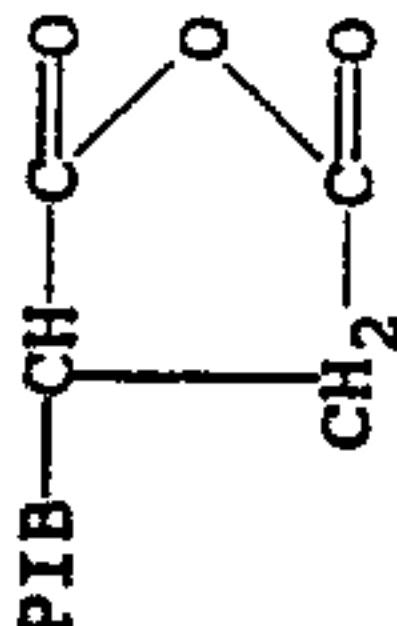
Example 3

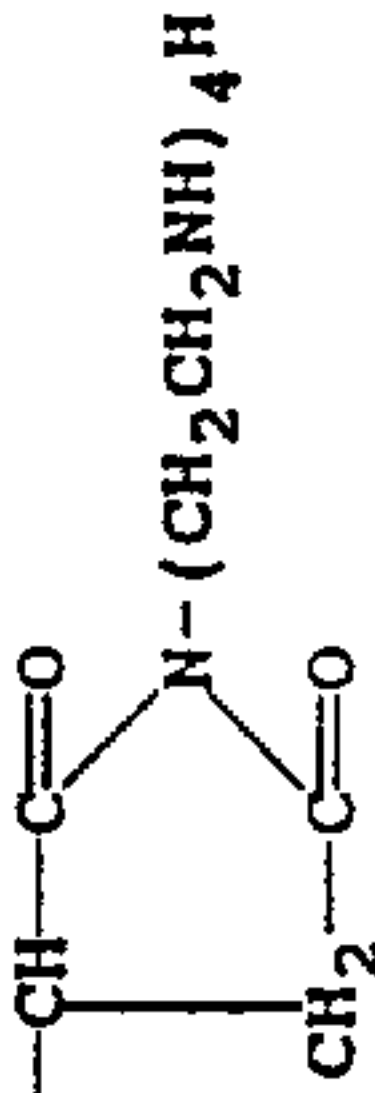
This example compares the noise properties and dropping point of a grease formulated with a polyisobutenyl succinic anhydride/acid dispersant according to the invention with polyisobutenyl succinic imide type dispersants described in Japanese Patent A2 58125794. Greases were formulated using the procedures of Example 1. Noise was tested using the procedure of Example 2 and dropping point was measured according to test method ASTM D566. The results are set forth in Table 1.

TABLE 1

Composition (Wt% Based On Grease)	Samples			Comparative Samples										
	1	2	3	4	5	6	7	8	9	10	11	12	13	14(6)
12-hydroxystearic acid	11.6							same						-
Azelaic acid	5.8							same						-
Lithium hydroxide	4.3	4.3	4.4	4.2				same					4.3	-
Dispersant A (1)	1.0	1.5	2.0											-
Dispersant B (2)					1.5	2.0								-
Dispersant C (3)							1.0	1.5	2.0					-
Dispersant D (4)										1.5	2.0	2.5	3.0	-
Property														
Worked consistency (ASTM D217)	242	229	263	260	249	239	234	239	252	255	252	224	221	235
Dropping point, °C (ASTM D566)	>270	>270	>270	234	264	247	204	241	208	239	257	270	255	>270
Noise (5)	452	128	379	7,808	480	372	3,255	326	562	599	517	83	100	2,725

Notes:

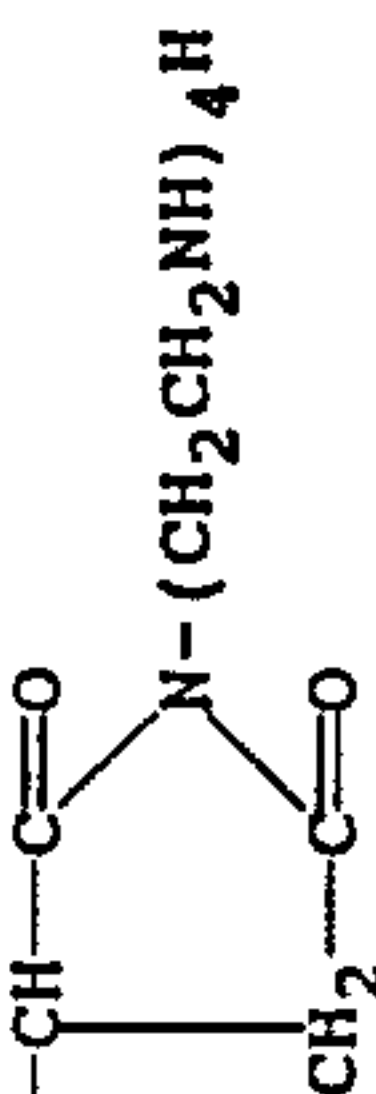
- (1) 80 wt% solution of  in refined mineral oil; average molecular weight of PIB is 950

- (2) 50 wt% solution of  in refined mineral oil, average molecular weight of PIB is 950

in refined mineral oil, average molecular weight of PIB is 950

- (3) 52 wt% solution of  in refined mineral oil, average molecular weight of PIB is 950

in refined mineral oil, average molecular weight of PIB is 950

- (4) 60 wt% solution of borated  in refined mineral oil, average molecular weight of PIB is 950

in refined mineral oil, average molecular weight of PIB is 950

- (5) Counts/120 second as determined by method of Example 2

- (6) Commercial sample of lithium complex grease

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Greases formulated with a polyisobutenyl succinic anhydride/acid type dispersant (Samples 1-3) showed significantly better noise property and higher dropping point compared to a lithium complex grease formulated without dispersant (Sample 4). Samples 1-3 also demonstrated consistently higher dropping points compared to greases formulated with polyisobutenyl succinic imide type dispersants (Comparative Samples 5-13). A commercial lithium complex grease (Sample 14) demonstrated a much inferior noise property compared to Samples 1-3 according to this invention.

CLAIMS:

1. A method for improving noise reduction and providing high dropping point in a grease which comprises incorporating into a base oil from 2 to 30wt%, based on grease, of a lithium soap complex thickener and from 0.1 to 10wt%, based on grease, of a polyalkylene succinic anhydride, a polyalkylene succinic acid or mixture thereof.
2. The method of claim 1 wherein the lithium soap complex thickener contains a lithium soap of a C₁₂ to C₂₄ hydroxy fatty acid and a lithium salt of a C₂ to C₁₂ aliphatic or cycloaliphatic dicarboxylic, a lithium salt of a C₃-C₁₄ hydroxy carboxylic acid or a mixture thereof.
3. The method of claim 2 wherein the lithium soap complex thickener contains a lithium soap of 12-hydroxystearic acid and a lithium salt of azelaic acid or lithium salt of salicylic acid.
4. The method of claim 2 wherein the ratio of hydroxy fatty acid to aliphatic or cycloaliphatic dicarboxylic acid or hydroxy carboxylic acid is from 10:0.5 to 10:15.
5. The method of claim 1 wherein the polyalkenyl moiety is polyisobutenyl having a number average molecular weight of from 300 to 4,000.