

UNITED STATES PATENT OFFICE

2,684,944

LITHIUM POLYORGANO SILOXANE POLYMER GREASE COMPOSITIONS

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No Drawing. Application June 2, 1952,
Serial No. 291,323

18 Claims. (Cl. 252—42.1)

1

This invention relates to high temperature greases and more particularly pertains to lithium soap silicone polymer greases having high temperature stability characteristics and to the method of preparing such greases.

The preparation of stable lithium soap silicone greases by conventional methods is very difficult requiring the use of milling operations and/or the use of solvents which must be removed. In addition, such greases require a relatively high soap content.

It is an object of the present invention to provide a lithium soap silicone polymer grease which is stable at high temperatures with respect to consistency and which is resistant to leakage at high temperatures. Another object of the invention is to provide a lithium soap silicone polymer grease having a high dropping point. Still another object of the invention is to provide a lithium soap silicone polymer grease which will effectively lubricate bearings operating at high temperatures and which will not melt and/or lose consistency at such operating temperatures. A further object of the invention is to provide a method of preparing lithium soap silicone polymer greases without the use of volatile solvents. Other objects and advantages of the invention will become apparent from the following description thereof.

I have discovered that the foregoing objects, as well as others, can be attained with a lubrication grease comprising a major proportion of a silicone polymer oil, 1% to about 20%, and preferably from about 2% to about 10% of a lithium soap of a high molecular weight fatty acid, and a free basic compound in amounts of from about 10% to about 500%, and preferably from about 50% to about 300% based on the lithium soap content of the grease. The lithium soap employed in the grease composition of the present invention is a soap of an unsaturated, partially unsaturated or saturated fatty acid or a hydroxy fatty acid having at least 12 carbon atoms, and preferably from about 12 to 22 or more carbon atoms. Examples of suitable fatty acids are lauric acid, myristic acid, palmitic acid, stearic acid, behenic acid, oleic acid, Standard Fatty Acids, hydrogenated fish oil fatty acids, hydrogenated castor oil, hydroxy stearic acid, e. g., 12-hydroxy stearic acid, and the like. The term "high molecular weight fatty acid," as used herein and in the appended claims, means a fatty acid having at least 12 carbon atoms. Standard Fatty Acids is a product well known in the grease making trade, and comprises about 40% animal fatty acids and about 60% cottonseed fatty acids.

The specific effectiveness of the various fatty acids varies to some extent, the lithium soaps of

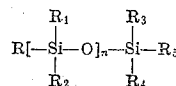
2

all of them, however, being effective to various degrees. It is, therefore, not to be implied that the lithium soaps of all of the herein-described fatty acids are equivalents.

5 The required excess alkalinity of the grease can be supplied by incorporating in the grease composition from about 10% to about 500%, or more, and preferably from about 50% to about 300% based on the soap content of the grease, of a basic compound such as basic alkali metal compounds, basic alkaline earth compounds, and high boiling basic organic nitrogen compounds, such as aliphatic or aromatic amines boiling above about 250° F.

15 Examples of suitable basic compounds are sodium hydroxide, potassium hydroxide, lithium hydroxide, lithium oxide, barium hydroxide, barium oxide, calcium hydroxide, calcium oxide, triphenyl amine, octylamine, etc.

20 The silicone oil polymers employed as the grease vehicle is suitably a polymeric liquid compound having the general formula



25 in which R, R₁, R₂, R₃, R₄, and R₅ are the same or different hydrocarbon radicals, such as alkyl, aryl, alkaryl or aralkyl radicals and n is an integer greater than 1. Depending upon the extent of condensation or polymerization, the polymer products vary from relatively light liquids to solid resins. However, for the purpose of the present invention, polymers which are high boiling liquids within the lubricating oil viscosity range are preferred. Commercially available compounds of this type are polymers of dimethyl silicone, diethyl silicone, ethylmethyl silicone, diphenyl silicone, ditolyl silicone, methyl phenyl silicone, ethyl phenyl silicone, and the like. Other silicone polymers produced from high molecular weight hydrocarbon groups, such as butyl, amyl, hexyl and above, when economically available can also be used. Polymeric organic silicon compounds of this type are also referred to as polymeric dihydrocarbon siloxanes or polyorgano siloxanes.

30 Greases of the present invention are easily prepared and do not require the use of solvents which must be subsequently removed as is made necessary by prior art methods of preparing such greases. In preparing greases of the present invention, all of the lithium soap, a portion of the polyorgano siloxane and all of the basic compound, preferably in the form of an aqueous solution or suspension, of about 5% to about 35%, are mixed together, slowly heated to a temperature of about 212° F., and maintained at this temperature until the mixture is substantially free of

3

water. When the mixture is substantially anhydrous, and foaming, if any, subsides, the remaining portion of the polyorgano siloxane is added, the batch heated to a temperature of from about 450° F. to about 550° F., and preferably to a temperature of about 500° F. to 510° F., and maintained at said temperature until the reaction is completed. The grease is then filled and cooled. The cooled product, if desired, can be homogenized by well known means.

A preferred grease stable at temperatures of at least about 450° F. is illustrated by the following composition:

	Per cent
Lithium hydroxy stearate.....	1-10
Alkali metal or alkaline earth hydroxide..	2-10
Polyorgano silicone.....	80-97

Grease compositions made in accordance with the hereindescribed invention are illustrated by the following examples in Table I:

Table I

Example No.	I	II	III	IV	V	VI
N. L. G. I. Grade ¹	00	2	3	5	-----	-----
Lithium 12-hydroxy stearate, percent.....	1	2	3	5	7.5	10
Lithium hydroxy monohydrate, percent.....	2	2	2	3	7.5	10
Silicone polymer oil ² , percent.....	97	96	95	92	85	80
Micro-penetration, unworked.....	258	68	43	35	30	7
Micro-penetration, worked.....	257	69	44	34	-----	-----
Dropping Point, °F.....	+550	+554	+552	+557	+553	+555

¹ National Lubricating Grease Institute Grades.

² Dow Corning Silicone Oil No. 550—SSU—300-400 at 100° F.

In each of the above examples, there was no change in the appearance or structure of the grease during the dropping point test made in accordance with the A. S. T. M. method D-566-42. Examples I to IV are transparent, smooth and buttery and light-colored greases. The greases of Examples V and VI are opaque, light-colored, and while mealy in structure, can be molded for high temperature plug valve lubricants and for brick greases.

The present invention is not only applicable to greases containing lithium hydroxy stearate but is also applicable to greases compounded with lithium soaps of other fatty materials. The following examples in Table II illustrate the application of the present invention to silicone polymer greases containing lithium soaps of various fatty materials.

Table II

Example No.	I-A	II-A	III-A	IV-A
N. L. G. I. Grade.....	5	00	1	3
Lithium soap of Hydrogenated castor oil.....	4.0%	4.0%	-----	-----
Oleic acid.....	-----	-----	5.0%	-----
Standard Fatty Acid ¹	-----	-----	-----	3.0%
12-hydroxy stearic acid.....	4.3%	4.3%	5.0%	3.4%
Lithium monohydrate.....	91.7%	91.7%	90.0%	93.6%
Silicone Polymer oil ²	33	340	119	50
Micro-penetration, unworked.....	34	344	118	52
Micro-penetration, worked.....	+547	+551	+552	+550
Dropping Point, °F.....	White.	Amber.	Mahogany	White.
Color.....	-----	-----	-----	-----

¹ Approximately 40% animal fatty acid; 60% cottonseed fatty acid.

² Dow Corning Silicone Oil No. 550.

Although in the foregoing examples excess alkalinity of the grease was imparted by the use

4

of lithium hydroxide monohydrate, other alkaline materials can be effectively employed for this purpose. Greases prepared with alkaline agents other than lithium hydroxide, are illustrated by the examples of Table III:

Table III

Example No.	I-B	II-B	III-B	IV-B	V-B
10 Lithium 12-hydroxy stearate, percent.....	5.0	2.0	2.0	4.0	2.0
Alkaline agent:					
Sodium hydroxide.....do.....	5.0	-----	-----	-----	-----
Potassium hydroxide.....do.....	-----	2.0	-----	-----	-----
Barium hydroxide.....do.....	-----	-----	2.0	-----	-----
Hydrated lime.....do.....	-----	-----	-----	4.0	-----
Triphenyl amine.....do.....	-----	-----	-----	-----	6.0
15 Silicone polymer oil ¹do.....	90.0	96.0	96.0	92.0	92.0
Micro-penetration, unworked.....	60	41	289	170	451
Micro-penetration, worked.....	56	-----	307	231	463
Dropping Point, °F.....	+420	+429	293	282	-----

¹ Dow Corning Silicone Oil No. 550.

In the foregoing examples the silicone polymer oil used was a particular grade silicone oil marketed as Dow Corning Silicone Oil No. 550. However, other grades of silicone polymer oils can be suitably used, as demonstrated by the examples in Table IV:

Table IV

Example No.	I-C	II-C
30 N. L. G. I. Grade.....	2	0.
Lithium 12-hydroxy stearate.....	3.0%	2.0%
Lithium hydroxide monohydrate.....	3.0%	2.0%
Silicone Polymer Oil ¹	94.0%	-----
Silicone Polymer Oil ²	-----	96.0%
Micro-penetration, unworked.....	80	170
Micro-penetration, worked.....	83	181
Dropping Point, °F.....	+554	+550
Color.....	Light Brown	White.

¹ Dow Corning Silidone Oil No. 200 SSU at 100° F.—355.

² Dow Corning Silicone Oil No. 710 SSU at 100° F.—1060.

The micro-penetration data given in the foregoing tables were determined by the proposed method described on page 339 of the November 1949 issue of "A. S. T. M. Standard on Petroleum Products and Lubricants."

Grease compositions prepared by the method described herein exhibit essentially the same consistency at room temperatures as at temperatures as high as 500° F. This property makes such greases especially suitable for use in the lubrication of moving parts operating at temperatures ranging from room temperatures to temperatures in excess of about 300° F.

A high temperature bearing test, conducted at 10,000 R. P. M. and at 450° F., was made on a 3 gram sample of a grease comprising 1% lithium 12-hydroxy stearate, 2% lithium hydroxide monohydrate and 97% Dow Corning Silicone Oil No. 550. At the end of 135½ hours the bearing was still in good condition.

In the foregoing examples the silicone polymers designated "Dow Corning Silicone Oil No. 550 and No. 710" were methylphenylpolysiloxanes, the latter having a higher phenyl/methyl ratio. The silicone polymer designated "Dow Corning Silicone Oil No. 200" was a methylpolysiloxane.

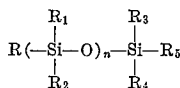
Percentages given herein and in the appended claims are weight percentages unless otherwise specified.

While I have described my invention by reference to specific preferred embodiments thereof, it is not intended that the invention be limited thereby but includes within its scope such modifications and variations as come within the spirit of the appended claims.

5

I claim:

1. A lubricant grease composition comprising a major proportion of a liquid polyorgano siloxane, from about 1% to about 20% of a lithium soap of a fatty acid, and a free basic compound lithium soap content of said composition, said polyorgano siloxane having the general formula



in which R, R₁, R₂, R₃, R₄ and R₅ are hydrocarbon radicals.

2. A lubricant grease composition described in claim 1, in which the lithium soap is lithium stearate.

3. A lubricant grease composition as described in claim 1, in which the lithium soap is lithium 12-hydroxy stearate.

4. A lubricant composition described in claim 1, in which the lithium soap is a soap of hydrogenated castor oil.

5. A lubricant grease composition described in claim 1, in which the lithium soap is lithium oleate.

6. A lubricant composition described in claim 1, in which the basic compound is a basic alkali metal compound.

7. A lubricant composition described in claim 1, in which the basic compound is lithium hydroxide monohydrate.

8. A lubricant composition described in claim 1, in which the basic compound is sodium hydroxide.

9. A lubricant composition as described in claim 1, in which the basic compound is a basic alkaline earth compound.

10. A lubricant grease composition described in claim 1, in which the basic compound is barium hydroxide.

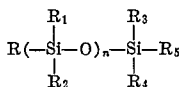
11. A lubricant grease composition described in claim 1, in which the basic compound is hydrated lime.

12. A lubricant grease composition described in claim 1, in which the basic compound is a basic organic amine.

13. A lubricant grease composition described in claim 1, in which the polyorgano siloxane is a methylpolysiloxane.

14. A lubricant grease composition described in claim 1, in which the polyorgano siloxane is a methylphenyl polysiloxane.

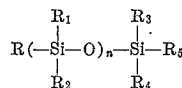
15. A lubricant grease composition comprising a major proportion of a liquid polyorgano siloxane, from about 1% to about 20% of a lithium soap of a high molecular weight hydroxy fatty acid, and lithium hydroxide in an amount equivalent to at least 10% of the lithium soap content of said grease composition, said polyorgano siloxane having the general formula



in which R, R₁, R₂, R₃, R₄ and R₅ are hydrocarbon radicals selected from the class consisting of methyl radicals, phenyl radicals and mixtures thereof.

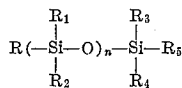
6

16. A lubricant grease composition comprising a major proportion of a liquid polyorgano siloxane polymer, from about 1% to about 10% of a lithium 12-hydroxystearate and from about 2% to about 10% of an alkali metal hydroxide, said polyorgano siloxane having the general formula



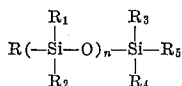
in which R, R₁, R₂, R₃, R₄ and R₅ are hydrocarbon radicals selected from the class consisting of methyl radicals, phenyl radicals and mixtures thereof.

17. A lubricant grease composition comprising from about 80% to about 97% of a liquid polyorgano siloxane polymer, from about 1% to about 10% lithium hydroxystearate and from about 2% to about 10% of an alkaline earth hydroxide, said polyorgano siloxane having the general formula



in which R, R₁, R₂, R₃, R₄ and R₅ are hydrocarbon radicals selected from the class consisting of methyl radicals, phenyl radicals and mixtures thereof.

18. A method of preparing a lubricant grease composition comprising a major proportion of a liquid polyorgano siloxane having the general formula



in which R, R₁, R₂, R₃, R₄ and R₅ are hydrocarbon radicals, from about 1% to about 20% of a lithium soap of a fatty acid, and a free basic compound in an amount equivalent to about 10% of the lithium soap, which method comprises mixing together all of the lithium soap, a portion of said polyorgano siloxane, and all of the basic compound in aqueous solution, heating said mixture to a temperature of about 212° F., maintaining the mixture at said temperature until the mixture is substantially anhydrous, adding the remaining portion of said polyorgano siloxane to said mixture, heating the mixture to a temperature of from about 450° F. to about 550° F., maintaining said mixture at said temperature until the reaction is complete, and filling and cooling said finished grease product.

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