United States Patent Office 2,946,749 Patented July 26, 1960

2,946,749

OPEN GEAR LUBRICANTS CONTAINING SILICONES

Lorne W. Sproule and Thomas M. Rogers, Sarnia, Ontario, Canada, assignors to Esso Research and Engineering Company, a corporation of Delaware

No Drawing. Filed Mar. 28, 1955, Ser. No. 497,436

9 Claims. (Cl. 252—28)

This invention relates to lubricating compositions and more particularly relates to open gear lubricants of a grease consistency containing silicone compositions, which lubricants have long lubrication lives.

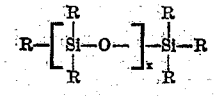
In the prior art, lubricants designed for heavy-duty service, for example for heavy and relatively large and rough open-type gears, have commonly consisted of viscous mineral lubricating bases of a grease consistency either alone or compounded with additive materials, such as fatty acid soaps, sulfonates and the like. Such lubricants when employed in open-type gears are exposed to the atmosphere, and because of this exposure the lubrication life of such gear lubricants has been relatively limited.

It has now been found that the addition of relatively high concentrations, at least about 0.05% by weight, of certain silicone compositions to these types of lubricants substantially increases their lubrication life. By incorporating these silicone compositions at relatively high concentrations in such lubricants in accordance with this invention, it has been found that the lubrication life of the lubricants, both in the presence of and the absence of water, is greatly improved. Although silicones have been added to normally liquid lubricating oil compositions heretofore, they have been added in substantially lower concentrations, generally less than about 0.01%, and for a different purpose, namely, to reduce foaming of the liquid oil compositions. This foaming problem does not exist with the viscous mineral lubricating bases used in the present compositions as the compositions of necessity are non-fluid at gear operating temperatures to thereby prevent loss of the lubricant from the open gears as well as to provide proper lubrication.

The gear lubricant compositions of this invention are lubricants having a grease consistency which comprise a major proportion of a viscous mineral lubricating base and a minor, but lubricating life extending proportion, of a non-fluid silicone composition consisting essentially of a fluid dimethyl silicone polymer thickened with a small amount of finely divided silica. In addition to the viscous mineral lubricating base and the silicone composition, the gear lubricant compositions of this invention may contain conventional gear lubricant additives such as fatty acid soaps, sulfonates, and the like. The gear lubricants may be blended with a volatile solvent to reduce the viscosity of the lubricant compositions so that the lubricant compositions may be more easily applied to open gears.

The silicone compositions useful in this invention consist essentially of a mixture of a dimethyl silicone polymer having an S.U.S. viscosity at 210° F. in the range of about 300 to 3,000 thickened with about 4.0 to 20.0 wt. percent (based on the silicone composition) of finely

divided silica. The dimethyl silicone polymers have the following general formula:



where R represents a methyl (CH_3—) group and x is an integer sufficient to provide a polymer having the aforementioned viscosity. Preferred silicone compositions consist essentially of a dimethyl silicone polymer having an S.U.S. viscosity at 210° F. in the range of about 500 to 2,000 thickened with about 8 to 12 wt. percent of finely divided silica. It will be understood of course that mixtures of dimethyl silicone polymers of different molecular weights may be employed in preparing the aforedescribed silicone compositions. The finely divided silica used as the thickener is preferably an amorphous substantially anhydrous SiO_2 having an ultimate particle size of about 5 to 100 millimicrons, for example 10 to 50 millimicrons. The preparation of these silicone polymers is well known to the art and is described for example in detail in the book "Chemistry of Silicones" by Eugene G. Rochow.

The viscous mineral lubricating bases useful in this invention are those having a grease consistency which are preferably derived from petroleum crude oils having a high asphalt and/or resin content. The viscosity of the viscous mineral lubricating base should be about 1,000 to 6,000 and preferably about 1,500 to 5,000 S.S.U. at 210° F.

The preferred viscous mineral lubricating base is one which contains a high asphalt content. This particular type of lubricant base is well known in the art and may be obtained from natural asphalts such as natural Trinidad, Bermudez, gilsonite, grahamite and Cuban or may be obtained from an asphaltic petroleum crude oil, such as those of South America, California or Mid-Continent crudes, by removing the lighter fractions from the crude oil. The removal of these lighter fractions from a petroleum crude oil may be accomplished by well-known methods, for example, by steam distillation, by vaciuum flashing in pipe stills, or by solvent separation. Steam distillation is carried out by treating a crude oil residuum, remaining after atmospheric distillation of light stocks, with steam sprays to remove additional light stocks, thereby producing a concentrated asphalt residuum. Vacuum flashing in pipe stills is a more efficient method for removing the light stocks from crude oil residua than is steam distillation as it may be carried out under vacuum at a lower temperature and thus prevents degradation of the products of this operation. Suitable viscous mineral lubricating bases may be also obtained by solvent separation of the asphalt content of crude residua from the light stock content. This method, which is also well known in the art, is accomplished by employing a light hydrocarbon solvent such as butane and propane. Also, suitable viscous mineral lubricating bases may be prepared by blending products of different viscosities, obtained by one or more of the above three mentioned methods, to thereby prepare a viscous mineral lubricating base of a desired viscosity. The asphaltic residua obtained by any of the above methods may be oxidized with air to produce lubricating bases having improved properties. Also, if desired, a heavy asphalt residua or natural asphalt may be "cut back" or blended with a heavy mineral oil to reduce the viscosity of the asphalt residua in order to prepare a mineral lubricating

base having a suitable viscosity for the purposes of this invention.

Another type of viscous mineral lubricating base which may be utilized in this invention is a petroleum residuum having a high resin content, or a solvent precipitated resinous fraction derived therefrom. These residua or resinous precipitates may be blended with a heavy distillate if desired to prepare a mineral lubricating base of this invention. Such resin-containing residua may be obtained for example by solvent refining (e.g. propane precipitation of the resin) a Pennsylvania crude oil, which contains a substantial proportion of the resinous constituent. Certain crude oils may contain a substantial proportion of both asphalt and resin constituents and they may likewise be used in this invention.

Generally, therefore, the viscous mineral lubricating bases of this invention which have a grease consistency are preferably petroleum residua of high asphalt and/or resin content obtained by the removal of the light stocks from a crude oil by such means as distillation, solvent separation and the like. In certain applications, the viscous mineral lubricating bases may be combined with tars or pitches such as wood tar, coal tar, vegetable pitch, and the like, if desired.

The viscous mineral lubricating base represents a major proportion of the lubricant composition of this invention. Preferably, it will represent about 80.00 to 99.95% by weight of the total lubricant composition. The silicone compositions of this invention are utilized in proportions sufficient to extend the lubrication life of the gear lubricant. More specifically, they should be employed at a concentration in the range of about 0.05 to 3.0% by weight, and preferably at a concentration in the range of about 0.08 to 1.5% by weight, based on the total lubricant composition. In addition to the viscous mineral lubricating bases and the silicone compositions described above, it will be understood that various conventional additive materials may be also incorporated into the lubricants of this invention as will be apparent to those skilled in the art. Thus, anti-oxidants such as phenyl alpha naphthylamine, rust inhibitors such as oil-soluble sodium petroleum sulfonates or other similar alkali or alkaline earth sulfonates, tackiness agents, extreme pressure agents such as lead oleate, sulfurized fatty oils, etc., film-forming agents such as colloidal graphite and the like, may be added for particular purposes without departing from the spirit of the present invention. The silicone compositions and other additives, if any, may be simply incorporated into the viscous mineral lubricating base by mixing. The total lubricant composition should be of a grease consistency and should have an S.S.U. viscosity at 210° F. in the range of about 1,000 to 6,000 and the ingredients should be selected to formulate such a composition.

Since the lubricants of this invention are non-fluid and relatively viscous, it is preferred to blend them, especially the higher viscosity lubricants, with a volatile solvent such as trichlorethylene, carbon tetrachloride, naphtha, etc. so that they may be readily applied to open gears. A non-flammable volatile solvent is preferred. After a relatively short period of time (e.g. 4–5 hours at 100° F.), the volatile solvent evaporates, leaving the lubricant compositions of this invention adhering as a film to the open gears. By employing such volatile solvents, the necessity of heating such compositions prior to their application to open-gears is eliminated. The amount added to the lubricant compositions of this invention should be sufficient to produce a semi-fluid composition, generally about 5 to 30% by weight based on the total solvent-free composition. Particularly preferred compositions containing a volatile solvent include a viscous mineral lubricating base having a viscosity in the range of about 1500 to 5000 S.S.U. at 210° F., trichlorethylene, colloidal graphite and a silicone composition of this invention. These preferred compositions have the following formulation:

LUBRICANT COMPOSITION [1]

Ingredient	Weight percent (based on the total solvent-free composition)
Viscous mineral lubricating base (1,500–5,000 SSU at 210° F.).	About 80 to 95.
Trichlorethylene (solvent)	About 5 to 30.
Colloidal graphite	About 5 to 15.
Silicone composition	About 0.08 to 1.5.

[1] Composition may also include minor amounts of conventional gear lubricant additives.

The following examples of this invention are presented to set forth the invention in greater detail, but it will be understood that it is not intended that the invention be limited solely thereto.

EXAMPLE 1

The lubrication life of a viscous mineral lubricating base was determined both in the presence and absence of water. The mineral lubricating base, which will hereinafter be termed Lubricating Base I, consisted essentially of South American (Tia Juana) reduced asphaltic crude oil having an S.S.U. viscosity of about 2,000 at 210° F. and a softening point of about 100° F. Lubricating Base I was evaluated in the following lubricant life test:

The lubricant was applied to open gears operating at a temperature of 100° F., carrying a load of 260 lbs. per tooth, and operating at a peripheral speed of 134 ft. per minute. A zenith pump driven through a variable speed transmission was used to drip water on the gear at a given rate. The lubricant life was arbitrarily chosen as the point when a ½" bare area on the bearing surface was visible when viewed under ultra-violet light.

The following results were obtained when evaluating the lubricant life of Lubricating Base I in the above-described lubricant life test:

Table I

LUBRICANT LIFE OF LUBRICATING BASE I

Water Contact Rate (cc./min.)	Lubricant Life (hrs.)
0	72
5	48
15	23
30	15

It will be noted that the lubricant life of Lubricating Base I was substantially reduced in the presence of water.

A number of conventional surface-active agents or water displacing agents, including oleic acid, castor oil, pine tar, barium sulfonate, and calcium acetate, were added to Lubricating Base I and the resultant compositions were evaluated in the aforedescribed lubricant life test. The following results were obtained with these conventional additives:

Table II

LUBRICANT LIFE OF LUBRICATING BASE I PLUS CONVENTIONAL ADDITIVES

Additive	Additive Concentration [1]	Water Contact Rate (cc./min.)	Lubricant Life (hrs.)
Oleic acid	5.0	30	15
Castor oil	5.0	30	<15
Pine tar	15.0	30	15
Barium sulfonate	10.0	30	12
Calcium acetate	20.0	30	4

[1] Weight percent, based on total composition.

It will be noted that none of the above conventional agents or additives were effective in increasing the lubricant life of Lubricating Base I in the presence of water. In fact several of the conventional additives actually reduced the lubricant life.

A non-fluid silicone composition of the present invention was then evaluated as a lubricating life extending agent in Lubricating Base I. This silicone composition, which will hereinafter be referred to as Additive A, consisted essentially of dimethyl silicone polymer having an S.U.S. viscosity at 210° F. of about 1234 seconds (S.U.S. viscosity at 100° F. was about 3100 seconds) thickened with about 10 wt. percent (based on total silicone composition) of finely divided silica (having a particle size in the range of about 10 to 50 millimicrons). For comparison purposes, a fluid dimethyl silicone polymer (not containing any finely divided silica) having an S.U.S. viscosity at 100° F. of about 350 seconds, hereinafter referred to as Additive B, was also evaluated. The following results were observed in the aforedescribed lubricant life test:

Table III

LUBRICANT LIFE OF LUBRICATING BASE I PLUS SILICONE ADDITIVES

Silicone Additive	Additive Concentration [1]	Water Contact Rate (cc./min.)	Lubricant Life (hrs.)
A	0.1	0	186
A	0.1	15	50
A	0.1	30	29
A	0.5	30	30
A	1.0	30	26
B	2.0	30	15

[1] Weight percent, based on total composition.

It will be noted that Additive A was extremely effective in increasing the lubricant life of Lubricating Base I. More specifically, the addition of only 0.1% by weight of Additive A approximatley doubled the life of Lubricating Oil Base I when employing 30 cc. per minute water contact rate. It will also be noted that Additive A had even a greater effect in increasing lubricant life under dry operating conditions, the increase amounting to about 250%. On the other hand, Additive B had no effect on the lubricant life of Lubricating Base I even though utilized at a concentration of 2 wt. percent.

EXAMPLE 2

The viscous mineral lubricating base utilized in this example was essentially a South American (Tia Juana) reduced asphaltic crude oil having an S.U.S. viscosity of about 5,000 at 210° F. This viscous mineral lubricating base, hereinafter referred to as Lubricating Base II, was "cut back" with trichlorethylene to give a composition (Composition I) consisting essentially of about 90% by weight of Lubricating Base II and about 10% by weight of trichlorethylene. Composition I which had a Saybolt Furol viscosity at 130° F. of about 1000 seconds was evaluated in the aforedescribed lubricant life test. A second composition (Composition II) was also evaluated in this example. The second composition consisted of 89.9% by weight of Composition I (i.e. 90 wt. percent of Lubricating Base II and 10 wt. percent of trichlorethylene) blended with 0.1% by weight of Additive A (described in Example 1) and 10% by weight of colloidal graphite. The colloidal graphite had essentially no thickening power and hence did not interfere with the gear life.

The following results were obtained with these two lubricant compositions:

Table IV

LUBRICANT LIFE OF LUBRICATING BASE II WITH AND WITHOUT SILICONE ADDITIVES

Composition	Concentration of Additive A[1]	Water Contact Rate (cc./min.)	Lubricant Life (hrs.)
I	None	0	75
I	None	30	15
II	0.1	0	185
II	0.1	30	26

[1] Weight percent, based on total composition (including volatile solvent).

It will be noted that the addition of 0.1% of the silicone composition of this invention, Additive A, substantially increased both the wet and dry life of Lubricating Base II.

What is claimed is:

1. A gear lubricant composition having a grease consistency consisting essentially of a major proportion of a viscous mineral lubricating base having an S.U.S. viscosity at 210° F. in the range of about 1,000 to 6,000, and in the range of about 0.05 to 1.5% by weight, based on the total lubricant composition, of a silicone composition consisting essentially of (1) dimethyl silicone polymer having an S.U.S. viscosity at 210° F. in the range of about 300 to 3,000, and (2) in the range of about 4.0 to 20.0 wt. percent, based on the silicone composition, of finely-divided silica.

2. A gear lubricant composition according to claim 1 wherein said viscous mineral lubricating base is a reduced asphaltic petroleum crude oil.

3. A gear lubricant composition according to claim 1 admixed with a volatile solvent.

4. A gear lubricant composition having a grease consistency consisting essentially of a major proportion of a viscous asphaltic mineral lubricating base having an S.U.S. viscosity at 210° F. in the range of about 1,500 to 5,000 and in the range of about 0.08 to 1.5% by weight, based on the total composition, of a silicone composition consisting essentially of (1) dimethyl silicone polymer having an S.U.S. viscosity at 210° F. in the range of about 500 to 2,000, and (2) in the range of about 8.0 to 12.0 wt. percent, based on the silicone composition, of finely-divided silica.

5. A gear lubricant composition consisting essentially of a major proportion of a viscous mineral lubricating base having an S.U.S. viscosity at 210° F. in the range of about 1,000 to 6,000, in the range of about 5 to 30% by weight, based on the total solvent-free composition of a volatile solvent, and in the range of about 0.05 to 1.5% by weight based on the total solvent-free composition of a silicone composition consisting essentially of (1) dimethyl silicone polymer having an S.U.S. viscosity at 210° F. in the range of about 300 to 3,000 and (2) in the range of about 4.0 to 20.0% by weight, based on the silicone composition of finely-divided silica.

6. Gear lubricant composition according to claim 5 wherein said solvent is trichlorethylene.

7. Gear lubricant composition according to claim 5 which contains in the range of about 5 to 15% by weight, based on the total solvent-free composition, of colloidal graphite.

8. A gear lubricant composition having the following formulation:

Ingredient	Weight percent (based on the total solvent-free composition)
Viscous mineral lubricating base	About 80 to 95.
Trichlorethylene (solvent)	About 5 to 30.
Colloidal graphite	About 5 to 15.
Silicone composition	About 0.08 to 1.5.

wherein said lubricating base has an S.U.S. viscosity at 210° F. in the range of about 1500 to 5000 and said silicone composition consists essentially of (1) dimethyl silicone polymer having an S.U.S. viscosity at 210° F. in the range of about 500 to 2,000 and (2) in the range of about 8.0 to 12.0 wt. percent, based on said silicone composition, of finely-divided silica.

9. A gear lubricant composition consisting essentially of a reduced asphaltic petroleum crude oil having an S.U.S. viscosity at 210° F. in the range of about 1,500 to 5,000 and in the range of about 0.08 to 1.5% by weight based on the total composition of a silicone composition consisting essentially of (1) dimethyl silicone polymer having an S.U.S. viscosity at 210° F. in the range of about 500 to 2,000 and (2) in the range of about 8.0 to 12.0 wt. percent, based on said silicone composition, of finely-divided silica.

References Cited in the file of this patent

UNITED STATES PATENTS

2,428,608	Baas	Oct. 7, 1947
2,466,927	Burton	Apr. 12, 1949
2,508,596	Cox	May 23, 1950
2,645,588	Barry	July 14, 1953
2,677,658	Bidaud	May 4, 1954
2,756,212	Hotten	July 24, 1956

FOREIGN PATENTS

715,592	Great Britain	Sept. 15, 1954

OTHER REFERENCES

"Lubricating Greases," by Boner (1954), pp. 921–22 and 795–99, Reinhold Pub. Co., New York, N.Y.