

UNITED STATES PATENT OFFICE

2,446,177

SILICONE GREASE AND METHOD OF PREPARATION

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No Drawing. Application June 21, 1945,
Serial No. 600,777

10 Claims. (Cl. 252—42.1)

(Granted under the act of March 3, 1883, as
amended April 30, 1928; 370 O. G. 757)

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This invention relates to lubricating compositions and to a method for their preparation.

The trend in modern developments in machine manufacture, particularly electric motor manufacture, and the demands placed on power generating devices by military applications call for constant reduction in the size of the power unit. It is quite possible, by appropriately insulating the coils in an electric motor with high temperature insulating materials, to reduce the size of the frame of an electric motor by substantially 25 to 30 per cent for a given horsepower. However, such reduction of power unit size on a large scale has been impracticable largely because the demands placed upon bearings in such units have exceeded the performance capacity of available lubricants. That is, bearing in such units become extremely overheated by heavy loading and by conduction of heat from the motor frame and lubricants either decompose or fail with resultant galling and seizure within the bearings. Accordingly, it is an object of the invention to provide lubricating compositions of such stability and lubricating quality that they can be used in bearings operating at high loads and speeds and at temperatures considerably higher than those ordinarily encountered in bearings without danger of failure of the lubricants or seizure of the bearings.

High-speed high-temperature bearings not only place exacting requirements upon the performance of the lubricant, but are also subject to serious wear as a natural consequence of the severe operating conditions and load placed upon them. Hence, it is another object of our invention to provide special lubricating compositions stable at high temperature and especially well adapted to employment on heavy duty bearings which compositions will provide for operation of the bearings with a minimum amount of wear.

It is another object of our invention to provide novel corrosion-protective, stable grease compositions for employment as lubricants in bearings of electrical and mechanically driven power units.

It is still another object of our invention to provide a method of preparing the very stable lubricating composition of our invention.

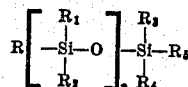
Other objects and advantages of the invention will be in part obvious and in part appear hereinafter in the detailed description of the compositions and the methods of their preparation.

The invention, accordingly, comprises the

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lubricating compositions or greases having the properties and the relation of components which will be exemplified in the compositions hereinafter described and the method of preparing such compositions which method of preparation involves the several steps and relation of one or more of such steps with respect to each of the others thereof and will be exemplified in detail in the processes hereinafter described and illustrated, and the scope of the invention will be indicated in the claims.

One class of organo-silicon compounds is illustrated by polymeric compositions of the following general structure:



in which, R, R₁, R₂, R₃, R₄ and R₅ represent any alkyl, aryl, alkaryl or aralkyl radicals, which compounds are available in the form of more or less viscous, oil-like fluids and, in general, are water-white and characterized by such properties as great thermal stability and high viscosity index. Relatively common compounds of this type are dimethyl silicone, diethyl silicone, ethyl methyl silicone, di-phenyl silicone, di-tolyl silicone, phenyl tolyl silicone, methyl phenyl silicone, ethyl phenyl silicone and alkyl aryl silicone polymers. Compounds like those named and methods for preparing them are described in United States Patents 2,352,974, 2,258,222, 2,258,219 and 2,371,068.

Although silicones seem to have physical properties possessed by our best mineral oils, they are not, in general, usable for purposes of lubrication because in many cases they do not protect the lubricated surfaces from corrosion and, in addition, frequently they simply do not lubricate sliding contacting metal surfaces. For example, dimethyl silicone polymer, having a viscosity of about 70 centistokes, one of the most common fluids corresponding to the above general formula, has no inherent rust inhibiting value such as that possessed by many hydrocarbon oils and it will be found that it is not a boundary lubricant and seizure will occur in bearings employing it as a lubricant.

The conversion of silicone fluids into grease compositions is hindered by the inherent incapacity of the fluids for dissolving the soap necessary to thicken the fluid to a grease consistency and it is practically impossible to form a grease merely by mechanically mixing the soap with the fluid. We have found that by dispersing a

suitable alkali-metal fatty acid soap such as lithium stearate, in a concentration of about 2 per cent in a hydrocarbon fraction of relatively low boiling range, for example, about 100° to 440° F., heating the mixture to the solvation temperature of the soap and maintaining it at substantially such temperature, a gel structure will result. At this point the silicone fluid, which must be one which is miscible with the hydrocarbon fraction, is added, agitated to form a homogeneous mix and the petroleum fraction removed to leave a grease. The general property which the hydrocarbon fraction should have is that it be relatively low-boiling and that it be miscible with the silicone fluid to be used as the grease base. The solvent should preferably have a boiling range well below that of the silicone oil, for example 100° below that of the oil and it should have no heavy ends. However, the boiling range of the solvent should be high enough to approach the solvation temperature of the soap which is generally close to its melting temperature. In general, hydrocarbon solvents having boiling ranges of about 300° to 400° F. are suitable at atmospheric pressure or low boiling point solvents can be used at 300-400° F. under pressure. Such hydrocarbon fractions or mutual solvents are benzene, petroleum ether, naphthas, deodorized kerosene and, generally, those hydrocarbon fractions used as insecticide carrying oils are useful in that they have the right boiling temperature range and are free of "heavy ends."

The last step comprising removal of the mutual solvent can be carried out conveniently at a temperature of about 200° F. at a reduced pressure. Using such a method, illustrated in greater detail in the examples forming part of this specification, a grease of extremely high stability having a silicone fluid as a base can be prepared with any desired soap concentration.

The nature, scope and method of accomplishing the objects and carrying out the method of the invention will be apparent from the following examples describing the compositions and the method of preparing them. In the following examples all proportions are given in parts or percentages by weight:

Example I

Lithium stearate, 2 parts, was dispersed in 100 parts by weight of a petroleum fraction having a boiling range of 300° to 400° F. (100° F. flash point naphtha) and the mixture heated to about 300° and maintained at that temperature until the lithium stearate had gone into solution to form a thick gel. Twenty-two (22) parts by weight of phenyl silicone (Dow-Corning 700 series) having a viscosity of about 100 centistokes at 25° C. was added to the lithium soap-petroleum fraction gel at the elevated temperature and the mass mixed to a uniform consistency. After the entire mass had been blended to a uniform consistency, it was held at an elevated temperature of about 200° to 250° F. in a vessel under a reduced pressure of 10 millimeters of mercury in order to assist in the removal of the petroleum fraction. The product, upon the removal of the petroleum fraction, was a stable grease of very smooth consistency having a soap concentration of about 9 per cent.

Balancing ingredients, such as corrosion inhibitors, can be blended with the composition in the amount of about a few tenths to about 2 parts by weight, depending upon the severity of the corrosive condition to which the grease will

be exposed. Suitable corrosion inhibitors are typified by the aryl fatty acids and polyvalent metallic soaps thereof as exemplified by the compounds disclosed in United States Patent application, Serial Number 531,241 filed April 15, 1944 by William A. Zisman and Hayward R. Baker.

Example II

Six (6) parts of lithium stearate were dispersed in 45 parts of petroleum ether and the dispersion heated to a temperature of 250° C. in a bomb. At this temperature, the pressure within the bomb was about 200 pounds per square inch. When solvation of the soap was complete, after about 30 minutes, 24 parts of dimethyl silicone polymer (Dow-Corning 500 series), having a viscosity of 70 centistokes at 25° C., were added to the bomb without permitting the temperature or pressure to drop. After a 20 minute period of agitation of the bomb to develop homogeneity of the mixture therein, the bomb was cooled rapidly and pressure released. Substantially all the petroleum ether evaporated off and a smooth homogeneous grease comprising about 20 per cent soap in dimethyl silicone was left.

It will be noted from this example and others to be given that very low boiling solvents such as petroleum ethers can be used for bringing the soap into a solvated condition. However, inasmuch as a relatively high temperature is necessary to bring the soap into such condition, it is necessary that the process be carried out in a pressure vessel of some type such as a bomb with attendant inconvenience.

Example III

Six (6) parts of lithium stearate and 1 part of polybutene (12,000 molecular weight) were dispersed in 45 parts of petroleum ether and the dispersion heated to a temperature of 200° C. in a bomb. At this temperature, the pressure in the bomb reached 200 pounds per square inch. Upon completion of the solvation of the soap and polymer, after 30 minutes at 200° C., 35 parts of dimethyl silicone (Dow-Corning 500 series) were added without permitting the temperature or pressure to drop appreciably. When homogeneity of the mixture in the bomb had developed, the pressure was released and the petroleum ether allowed to evaporate off to leave behind a smooth homogeneous grease comprising about 15 per cent of lithium soap in dimethyl silicone.

Example IV

Four (4) parts of lithium stearate were dispersed in 30 parts of Stoddard Solvent and the mixture heated to 150° C. in an open vessel to complete solvation of the soap. When a maximum thickening had been attained, approximately 10 minutes of heating needed, 22 parts of diethyl silicone fluid was added and the mixture agitated and held at 150° C. until a homogeneous mix was formed. The mix was poured into a shallow pan and held at a temperature of 125° C. in a current of air until all of the solvent had evaporated before being allowed to cool.

Example V

Three (3) parts of lithium stearate were dispersed in 35 parts of deodorized kerosene (boiling range 425-475° F.) and the mixture heated to 250° C. to effect solvation of the soap therein. Upon the formation of a thick gel, there were added 25 parts of a mixture of dimethyl and diphenyl silicone (1:1 by weight) and the total

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mixture held at 250° C. and agitated until a homogeneous mass was formed. The mass was then poured into a shallow pan, held at about 150° C. until substantially all of the solvent had evaporated off and allowed to cool.

Example VI

Four (4) parts of lithium stearate were dispersed in 30 parts of Stoddard Solvent and the mixture heated to 150° C. in an open vessel to effect solvation of the soap. Completion of the solvation of the soap was indicated by the attainment of a maximum amount of thickening of the ingredients in the vessel, that is the formation of a gel. The thickened mass was held at the temperature of about 150° C. and mixed in a current of warm air until a substantial portion of the solvent had been removed by evaporation with the result that a very thick gel was left in the vessel. At this stage 20 parts of diethyl and dimethyl silicone polymer (1-1 by weight) were added and the total mixture agitated and held at 150° C. until a completely homogeneous mix was formed. The mix was then poured into a shallow pan and held at a temperature of about 125° C. in a current of warm air until all of the solvent had been removed before it was allowed to cool.

Example VII

Lithium stearate, 6 parts, was dispersed in about 200 parts by weight of a petroleum fraction having a boiling range of 300° to 400° F. (100 flash naphtha) and the mixture heated to about 300° and maintained at that temperature until the lithium stearate had gone into solution to form a thickened gel. As pointed out in connection with other examples, the completion of this stage is identifiable as the point at which maximum thickening of the solution occurs. Forty-two (42) parts by weight of a mixture of phenyl silicone polymer and ethyl silicone polymer (approximately 1-1 by weight) were added to the gel after evaporating off from the gel part of the solvent used for solvating the soap. The residual soap gel and the silicone polymer were mixed at about 300° F. to a uniform consistency. The product, upon removal of the rest of the petroleum fraction, was a grease of very smooth consistency which had a soap concentration of about 12 per cent.

The compositions of these examples thus contemplate using more than 70 parts of the silicone polymer blended with less than 30 parts of the lithium stearate.

Thus the method of preparing silicone-base grease compositions in accordance with the invention comprises blending a soap with the silicone fluid by employing a relatively low-boiling petroleum or hydrocarbon fraction as a mutual solvent for bringing the soap into colloidal solution in the base fluid. From the above examples it can be seen that the mutual solvent used for solvating the soap in aid of bringing it into colloidal dispersion within the silicone fluid should be one which has a relatively narrow boiling range, preferably a moderately high boiling point which, however, is about 100° below that of the silicone fluid being used and further it should have no heavy ends. The examples also show that the solvent can be removed to a substantial extent prior to the addition of the silicone fluid. It is essential in carrying out the method that the silicone fluid used as a base for the grease be compatible with the petroleum fraction

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and that the latter has no heavy ends. Some specific compositions of silicone greases which can be prepared according to the method of our invention are suggested in the following table:

Soap	Silicone
Lithium stearate	diphenyl silicone
Do.	dimethyl silicone
Do.	diethyl silicone
Do.	halogenated alkyl or aryl silicone

All the grease compositions prepared according to the above examples were characterized by extreme stability. Tests showed that bleeding of the greases was, in all cases, less than 4 per cent. After 3 hours at ambient temperatures in a BEC grease testing machine running on a 5-gram sample the microcone penetration changed only from 89 to 93 (the test is described by Kaufman et al., in Ind. Eng. Chem., Anal. Ed., 11, 108 (1939)). Worked samples of this type showed no tendency to bleed after standing for four months.

Another test involving operation in a Fafnir 305W unshielded bearing at 1750 revolutions per minute under a load of 750 pounds per square inch for 123 hours showed that the greases developed no evidence of roughness and were adherent and stable to working loads.

Since certain changes may be made in the compositions of our invention and changes made in the method of their preparation and different embodiments of the invention can be made by varying the fluid used as the base, the soap and the balancing ingredients, it is intended that all matter contained in the above description and examples shall be interpreted as illustrative and descriptive of a few preferred embodiments and not in a limiting sense.

"Consisting essentially of" as used in the claims means that the composition is made up almost entirely of the ingredients recited and these ingredients are the main and characterizing ones, but this expression does not exclude the presence of minor amounts of other ingredients which are commonly employed in lubricating compositions or which are merely inert. Such added ingredients would not change the essential character of the composition.

The invention described herein may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefor.

Having described our invention what we claim as new and desire to secure by Letters Patent of the United States is:

1. A lubricant consisting essentially of, an organo-silicon polymer as a base fluid blended with a minor amount of lithium stearate to produce a stable grease.

2. A lubricating composition consisting essentially of, as a base fluid, more than 70 parts of an alkyl silicone polymer having a viscosity of about 100 centistokes at 25° C. blended with less than 30 parts of lithium stearate to produce a stable grease.

3. A lubricating composition consisting essentially of, as a base fluid, more than 70 parts of an aryl silicone polymer having a viscosity of about 100 centistokes at 25° C. blended with less than 30 parts of lithium stearate to produce a stable grease.

4. A lubricating composition consisting essen-

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tially of, as a base fluid, more than 70 parts of a mixed alkyl-aryl silicone polymer having a viscosity of about 100 centistokes at 25° C. blended with less than 30 parts of lithium stearate to produce a stable grease.

5. A lubricating composition consisting essentially of, as a base fluid, about 85 parts of dimethyl silicone having a viscosity of about 100 centistokes at 25° C. blended with about 15 parts of lithium stearate to produce a stable grease.

6. A lubricating composition consisting essentially of, as a base fluid, about 85 parts of diphenyl silicone polymer having a viscosity of about 100 centistokes at 25° C. blended with about 15 parts of lithium stearate to produce a stable grease.

7. The method of preparing a silicone-base grease consisting essentially of, dispersing lithium stearate in a relatively low-boiling petroleum fraction, heating said dispersion of lithium stearate to induce solvation, adding to the solution thus obtained a silicone fluid miscible with said petroleum fraction, maintaining said mixture at an elevated temperature until homogeneity of the mixture is attained, and removing the petroleum fraction by evaporation to leave a residue of a stable grease consisting essentially of the lithium stearate dispersed in the silicone base fluid.

8. The method of preparing a silicone-base grease, dispersing lithium stearate in a relatively large amount of a petroleum fraction, heating said dispersion to an elevated temperature to form a relatively dilute solution thereof, adding to said solution a silicone fluid miscible with said petroleum fraction, maintaining the mixture at an elevated temperature until homogeneity is attained, and removing the petroleum fraction to leave behind as a residue a stable grease consisting essentially of a gel of the lithium stearate in the silicone base fluid.

9. The method of preparing a silicone-base grease consisting essentially of, dispersing about 2 parts of lithium stearate in about 100 parts of a petroleum fraction which boils in the range of about 200° to 400° F., heating said dispersion of lithium stearate in said petroleum fraction to about 300° F. to produce solvation of the lithium stearate therein to form a solution of about 2 per cent concentration, adding to the solution thus obtained about 20 parts of a silicone fluid which is miscible with said petroleum fraction, maintaining said mixture at about solvation tem-

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perature until homogeneity is attained, and removing the petroleum fraction by evaporation to leave a residue of a stable grease containing about 12 per cent of lithium stearate dispersed in the silicone base fluid.

10. The method of preparing a silicone base grease consisting essentially of, dispersing lithium stearate in a relatively low-boiling petroleum fraction, heating said dispersion of lithium stearate in said petroleum fraction to produce solvation thereof to form a gel, removing a substantial part of the petroleum fraction by evaporation, adding to the gel thus obtained a silicone fluid which is miscible with said petroleum fraction, maintaining said mixture at an elevated temperature until homogeneity is attained, and evaporating the residue of petroleum fraction to leave a residue of a stable grease consisting essentially of the lithium stearate dispersed in the silicone.

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