

UNITED STATES PATENT OFFICE

2,553,423

LUBRICATING GREASE COMPOSITION

William W. Shepard, Cincinnati, Ohio, assignor
to Standard Oil Development Company, a corporation of Delaware

No Drawing. Application October 12, 1949,
Serial No. 121,049

9 Claims. (Cl. 252—33.2)

1

The present invention relates to lubricating grease compositions and in particular to grease compositions consisting mainly of lubricating oil of appropriate grade thickened to a grease consistency with a combination of a metal sulfonate with a salt of a low molecular weight acid. The invention also relates to a method of preparing greases of the character referred to.

In the patent to Zimmer and Duncan, Reissue Patent No. 23,082, issued January 25, 1949, there is described a lubricating grease composition, and a method of manufacturing such composition, wherein a lubricating oil is thickened with a complex thickener consisting of an oil-soluble metal sulfonate having a molecular weight in the sulfonate radical, of about 300 to 600, together with a salt of one of the low molecular weight organic acid, such as calcium acetate, lithium acetate, and the like. The present invention is related to that described in said patent, but involves various improvements which may be set forth in greater detail hereinafter. The patent indicates that lubricating oils may be thickened to satisfactory grease consistency by converting the oil soluble sulfonates to lyophilic but relatively oil-insoluble complex coordination compounds of the so-called Werner type. The conversion of oil-soluble sulfonates to the oil-insoluble complex is described as being accomplished by reacting the sulfonate with water-soluble salts of nitrogen oxide acid, acetic acid, formic acid, citric acid, lactic acid, and the like. The Zimmer and Duncan patent indicates that the formation of the oil-insoluble complex depends upon the presence in the reaction of a polyvalent cation. The patent suggests that either the sulfonate or the low molecular weight salt must contain a polyvalent metal component, and that appears to be true when the product is based on sulfonates of uniform or substantially uniform molecular weight.

A major weakness of greases of the sulfonate-low molecular weight salt complex type is their tendency toward a lack of mechanical stability. When subjected to mechanical shear both in the standard ASTM grease worker and in the BEC grease testing machine, some of the compositions of the general type described in the aforesaid Zimmer and Duncan patent broke down rather early in testing.

It has now been discovered that particular advantage lies in combining a mixture of sulfonates, at least one of which has a combining (sulfonate radical) weight below 400, preferably

2

about 350 to 380, and another above about 400, preferably 420 to 500. For example, a grease prepared from 50% oil-soluble sulfonates of 375 molecular weight sulfonic acid and 50% of 425 molecular weight sulfonic acid performed very satisfactorily in the standard BEC grease testing machine. By contrast, a grease made with sulfonates of 375 combining weight sulfonic acid alone stiffened to a rigid gel-like structure and was thrown out of the test bearing. On the other hand, a grease made with 425 combining weight sulfonic acid alone fluidized and flowed out of the bearing within a short time.

The sulfonates of at least two different molecular weights are preferably combined in approximately equal quantities, but 1 to 2 parts of the lower molecular weight salts may be combined with 1 to 2 parts by weight of those of higher molecular weight. The sodium sulfonates are preferred, the calcium sulfonates being second choice and strontium sulfonates third, but other metal sulfonates also may be used though not quite as satisfactory. The alkali and alkaline earth sulfonates are all reasonably satisfactory. They must, of course, be oil-soluble. The oil may be any suitable mineral base lubricating oil of 35 to 200 S. U. S. viscosity.

A grease was prepared having the following formulation:

Grease A

- 9.25% Sodium salt of mahogany acid (M. W. about 375)
- 9.25% Sodium salt of mahogany acid (M. W. about 425)
- 74.00% Mineral oil (Coastal oil, 40 S. U. S. at 210° F.)
- 7.5% Calcium acetate

The sulfonate salts were dissolved in the mineral oil and the calcium acetate was added as a 16.7% aqueous solution. After emulsification by stirring, this mixture was heated at a temperature of about 220° F. for one hour and then the temperature was gradually raised to a higher temperature between 250° and 300° F., specifically, 260° F. At this point, dehydration was substantially complete and firm, semi-transparent grease was obtained.

Two other greases of the following formulations were prepared by substantially the same method:

Grease B

- 18.4% Sodium salt of mahogany acid (M. W. about 375)

73.9% Mineral oil (as in Grease A)
7.7% Calcium acetate

Grease C

18.6% Sodium salt of mahogany acid (M. W. about 425)
74.7% Mineral oil (as in Grease A)
6.7% Calcium acetate

Test results on these three greases are shown below:

Grease	Dropping Point, °F.	BEC Test Results
A-----	371	Grease softened slightly.
B-----	410	Grease stiffened to a hard gel-like structure and was thrown out of test bearing.
C-----	368	Grease fluidized and flowed out of test bearing.

In general, the total quantity of high and low molecular weight sulfonates used should be between 5 and 25% of the weight of the total lubricant. The low molecular weight non-sulfonate salt should comprise 2 to 10 or 12% of the total weight. Hence, the total complex (sulfonates and low molecular weight salt) comprises about 7 to 35% of the lubricant.

The sulfonate salts are preferably derived from petroleum sulfonic acids which are formed by the drastic treatment of mineral oil compositions, especially viscous residuums and other heavy fractions, with strong or fuming sulfuric acid. The treated fractions comprise a lower acid sludge layer, containing the low molecular weight water-soluble sulfonic acids. An upper layer contains the oil-soluble sulfonic acids of approximately 350 molecular weight and higher. These higher sulfonic acids, separated in fractions of less than 400 and more than 400 molecular weight, respectively, are thereafter neutralized with appropriate metal bases to obtain the desired metal sulfonates for the present invention. The production of such sulfonates is well known in the art and forms no part of the present invention.

What is claimed is:

1. A lubricating grease composition comprising a lubricating oil and a sufficient quantity of a mixture of substantially equal parts of metal sulfonates of at least two sulfonic acids having, respectively, a molecular weight of 420 to 500 and a molecular weight of 350 to 380, combined in a complex with a salt of an acid selected from the group consisting of low molecular weight aliphatic carboxylic acids, to thicken said oil to a grease consistency.

2. A composition as in claim 1 containing 7 to 35% by weight of said complex.

3. A lubricating grease composition consisting essentially of mineral base lubricating oil thickened to a grease consistency with 5 to 25% by weight, based on the total composition, of a metal salt of a mixture of substantially equal parts of at least two sulfonic acids having re-

spectively, a molecular weight of 350 to 380 and a molecular weight of 420 to 500, with 2 to 10% of a low molecular weight salt selected from the group which consists of the alkali and alkaline earth metal salts of low molecular weight carboxylic acids.

4. A composition as in claim 3 wherein the salt of low molecular weight acid is an acetate.

5. A composition as in claim 3 wherein the salt of low molecular weight acid is calcium acetate.

6. A lubricating grease composition of high structural stability under conditions of high shear, which consists essentially of a mineral base lubricating oil of 35 to 200 S. U. S. viscosity at 210° F. thickened to a solid grease consistency with 5 to 25% by weight, based on the total composition, of a sulfonate mixture of 1 to 2 parts of the sodium salt of sulfonic acid having a molecular weight of 350 to 380 combined with 1 to 2 parts of the sodium salt of sulfonic acid having a 420 to 500 weight, together with 2 to 10% of a calcium salt of a low molecular weight aliphatic carboxylic acid.

7. Composition as in claim 6 wherein said calcium salt is calcium acetate.

8. A lubricating grease of approximate formula:

74.00% by weight of mineral oil of 35 to 200 S. U. S. viscosity at 210° F.

9.25% Sodium salt of sulfonic acid of molecular weight about 375

9.25% Sodium salt of sulfonic acid of molecular weight about 425

7.5% Calcium acetate

9. The process of preparing a lubricating grease of the sulfonate base type which comprises dissolving substantially equal portions of at least two sodium sulfonates having molecular weights, respectively, of from 350 to 380 and from 420 to 500 in mineral base lubricating oil of 35 to 200 S. U. S. viscosity at 210° F., adding an aqueous solution of a calcium salt of a low molecular weight aliphatic carboxylic acid, stirring to emulsify, heating to about 220° F. for a period of time sufficient to evaporate the water, and further heating to a temperature of about 250° to 300° F. for a period sufficient to form a firm grease structure.

WILLIAM W. SHEPARD.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
Re. 23,082	Zimmer et al. -----	Jan. 25, 1949
2,468,098	Morway et al. -----	Apr. 26, 1949
2,468,099	Morway -----	Apr. 26, 1949
2,483,800	Zimmer et al. -----	Oct. 4, 1949
2,487,080	Swenson -----	Nov. 8, 1949
2,487,081	Swenson -----	Nov. 8, 1949