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SULFONATE GREASE

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This invention relates to the production of lubricating greases and more particularly to the production of a lubricating grease thickened with a complex of a petroleum sulfonate and a low molecular weight carboxylic acid salt.

Various suggestions have been made in the prior art for the improvement of the high temperature properties and the modification of other properties of lubricating greases by modifying the characteristics of the material used to thicken lubricating oil to a grease consistency. The thickener commonly used is a soap or a mixture of soaps of the fatty acids. Soaps of calcium, barium, sodium, lithium, and the like, have long been used as thickeners for mineral base lubricating oils. The calcium soap greases are the most widely used of all greases but are not particularly suited for the lubrication of anti-friction bearings which operate at fairly high temperatures. Since there is a tendency in the industry largely to increase the operating temperature of anti-friction bearings as lubricants have become available to maintain such bearings, greases of superior performance at high temperature are desirable.

A number of suggestions have been made recently that greases of high dropping point can be made from petroleum sulfonates by incorporating therein a salt of a low molecular weight acid. In Reissue Patent No. 23,082 to Zimmer et al. on January 25, 1949 there is disclosed a grease composition made by thickening a mineral lubricating oil with a so-called Werner complex formed from a petroleum sulfonate and a salt of a low molecular weight carboxylic acid such as acetic acid. This patent teaches that the amount of thickener used may vary from about 0.5% to 50% of the total composition. According to the disclosure of the reissue patent, the petroleum sulfonate is blended with the desired mineral oil by agitation at the desired temperature. When the blend is prepared, the metallic salt of the lower molecular weight carboxylic acid, such as calcium acetate for example, is added and emulsification takes place immediately. Dehydration is brought about by raising the temperature of the mixture to about 250° F., and after the desired amount of dehydration the grease mixture is cooled and packaged.

According to the Zimmer et al. patent, the amount of the reactant salt employed, i. e., the metallic salt of the monocarboxylic acid is said to be dependent upon the approximate number of sulfonate ions present in the mixture, i. e., the amount of the reactant salt is calculated on an

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"anion per anion" basis. In other words, the molar proportion of the petroleum sulfonate and the reactant salt for the grease of Zimmer et al. is disclosed as being 1:1.

Although the grease compositions made in accordance with the disclosure of the Zimmer et al. patent have many desirable characteristics under some conditions of use, they are found to be lacking in certain other properties. For example, it has been found that under conditions of extreme shear, they do not perform satisfactorily. Also, their oxidation resistance is low and it is found that even the addition of oxidation inhibitors fail to improve this important characteristic.

It has now been found, and is the subject of this invention, that substantial improvement in the performance of the sulfonate greases, both in regard to oxidation resistance and other desirable characteristics, can be obtained by a very considerable increase in the relative amount of the metallic salt of the low molecular weight carboxylic acid present in the coordination complex preferably until it is substantially equal in weight proportion to the amount of the petroleum sulfonate. In other words, when the relative amount of the acid salt used in the preparation of the thickening complex is increased so that the mol ratio of the acid salt to the petroleum sulfonate is about 6 to 1, a very superior grease composition results.

It is, therefore, the object of this invention to prepare a superior grease composition by incorporating into a lubricating oil an alkaline earth metal sulfonate that is soluble in the oil, that is to say, one that has an acid radical weight of between 300 and 500 and in addition to the oil soluble sulfonate a salt of a low molecular weight aliphatic carboxylic acid.

The invention has a further object the production of a grease composition which contains a complex addition product of an oil soluble alkaline earth sulfonate and a salt of a low molecular weight carboxylic acid, the complex addition product being composed of from 28 to 55% of the salt in the acid solution and about 45 to 72% of the sulfonate.

A further object of this invention is to prepare a grease composition by incorporating in a lubricating oil base a complex addition product of an oil soluble alkaline earth sulfonate and an alkaline earth salt of an acid selected from the group consisting of low molecular weight aliphatic carboxylic acids, said complex addi-

tion product being composed of a molar proportion of the metal salt to the metal sulfonate within a range of from 2.5:1 to 7.5:1.

The exact structure of the present grease thickener, that is, the mixture of the metal sulfonate and the metal salt, is unknown. It is believed, however, that the two components form a complex addition product of the protective colloid type, the metallic salt being the bodying agent for the thickener and the oil soluble sulfonate acting as the protective colloid in dispersing and modifying the characteristics of the structure.

The preferred embodiment of this invention contemplates utilizing from 10 to 30% by weight

S. S. U. and a viscosity at 100° F. of 500 S. S. U. were blended together in a vessel and heated with stirring to a temperature of about 150° F. The desired amount of calcium acetate was added in a 20% aqueous solution and the batch slowly heated to dehydration, stirring being continued throughout the heating. The dehydration was carried out to a maximum temperature of 300° F. After dehydration, the grease was cooled to room temperature without stirring, and filtered through a 28 x 500 mesh screen.

The test results on the grease composition obtained by the standard test of methods are for convenience tabulated in the table below:

TABLE I

Sulfonate greases

[10% Strontium Sulfonate (450 M. Wt.), 2% of a Partially Esterified Polyhydric Alcohol Modifier, A Coastal Distillate (500 SSU at 100° F., 55 SSU at 210° F.) and Calcium Acetate.]

Mol Ratio (Calcium Acetate to Strontium Sulfonate)	1.25:1	2.50:1	3.75:1	6.25:1	7.50:1
Calcium Acetate, Per Cent by Weight	2	4	6	10	12
Penetration mm./10 Unworked	316	256	225	180	207
60 Strokes Worked	412	317	297	274	325
Water Wash, Per Cent Loss at 77° F.	70	60	17.5	20.0	-----
AXS-1574 Wheel Bearing Test ¹	Fail	Fail	Fail	Pass	Fail
Drop Point, ° F.	184	454	478	565+	402
ABEC-NLGI High Temp. Spindle Test Hrs. at 300° F. and 10,000 R. P. M.	-----	75	132	277	-----
Norma-Hoffmann Bomb Test ² Hrs. to 5 p. s. i. Drop	50	140	250+	250+	-----
ABEC Test, Appearance ³ After Test	Fluid	Fluid	Fluid	Channelled Good	-----

¹ Army Experimental Specification 1574 (430 R. P. M. for 6 hrs. at 220° F., spindle temp.).

² Government Specification AN-G-15 (100 hrs. minimum).

³ Annular Bearing Engineer Committee Bearing test (3,500 R. P. M. for 20 minutes at room temperature).

of the complex addition product. However, an amount of the complex addition product within a range of from 5 to 50% by weight results in a very satisfactory grease when blended with the desired oil. The lubricating oil, which is used as the base stock, may be any of the various lubricating oils known to the art either natural or synthetic.

Although the complex addition product comprises from 28 to 55% by weight of the acid salt to about 45 to 72% by weight of the sulfonate in the preferred embodiment, greases with excellent properties may be made by using from 17 to 62% by weight of the acid salt with 38 to 83% by weight of the sulfonate, that is to say, the molar ratio of the acid salt of the low molecular weight carboxylic acid to the alkaline earth metal sulfonate may vary from 1:1 to 10:1, the preferred molar ratio being from 2.5:1 to 7.5:1.

The petroleum sulfonic acid used to form the alkaline earth metal salt should have a molecular weight within a range of from about 350 to 600, preferably 400 to 500, and any of the alkaline earth metal sulfonates are operable. Examples of the low molecular weight carboxylic acids which may be used to react with the alkaline earth metal basic materials such as the alkaline earth metal oxides, carbonates or hydroxides to form the acid salts are acetic, oxalic, formic, tartaric, propionic, lactic, and the like, the salts of the alkaline earths and acetic acid being preferred.

In accordance with the process of this invention, several examples of sulfonate greases containing a complex composed of 10% strontium sulfonate and from 2 to 12% calcium acetate were prepared. These greases were all manufactured according to the following procedure.

The sulfonate concentrate, a modifier such as a partially esterified polyhydric alcohol, for example, sorbitan trioleate, and a mineral coastal distillate, having a viscosity at 210° F. of 55

In summation, the present invention comprises the grease composition formed by incorporating into a lubricating oil the desired amount of complex addition product of an oil soluble alkaline earth petroleum sulfonate and an alkaline earth metal salt of an acid selected from the group consisting of low molecular weight carboxylic acids in which the addition product is formed of varying percentages of the acid salt with about 10% of the sulfonate.

It is to be understood that conventional additives may be used in the grease compositions of this invention. These additive materials such as tackiness agents, viscosity index improvers, anti-corrosion agents, oxidation inhibitors, extreme pressure agents and the like may be incorporated by methods familiar to the art.

What is claimed is:

1. A grease composition comprising a lubricating oil and from 10 to 30% by weight of a complex addition product of an oil soluble alkaline earth metal petroleum sulfonate and an alkaline earth metal salt of an acid selected from the group consisting of low molecular weight carboxylic acids, said complex addition product being formed of from 28 to 55% by weight of said acid salt with about 45 to 72% by weight of the said oil soluble sulfonate.

2. A grease composition comprising a lubricating oil and from 10 to 30% by weight of a complex addition product of an oil soluble alkaline earth metal petroleum sulfonate and an alkaline earth metal salt of an acid selected from the group consisting of low molecular weight aliphatic carboxylic acids, said complex addition product being formed from substantially equal percentages of the said oil soluble metal sulfonate and the said acid salt.

3. A grease composition comprising a mineral lubricating oil and from 10 to 30% by weight of a complex addition product of an oil soluble alkaline earth metal petroleum sulfonate and an

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alkaline earth metal salt of an acid selected from the group consisting of low molecular weight aliphatic carboxylic acids, said complex addition product being composed of from 2.5 to 7.5 mols of said acid salt to 1 mol of said oil soluble metal sulfonate.

4. A grease composition comprising a mineral lubricating oil and 20% by weight of a complex addition product of an oil soluble alkaline earth petroleum sulfonate and an alkaline earth metal salt of an acid selected from the group consisting of low molecular weight carboxylic acids, said complex addition product having a molar ratio of 6.25 mols of said acid salt to 1 mol of said oil soluble metal sulfonate.

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5. A grease composition according to claim 1 in which the metal salt of the oil soluble sulfonate is strontium sulfonate and the acid salt is calcium acetate.

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REFERENCES CITED

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

UNITED STATES PATENTS

Number	Name	Date
Re. 23,082	Zimmer	Jan. 25, 1949

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