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EXTREME PRESSURE GREASE

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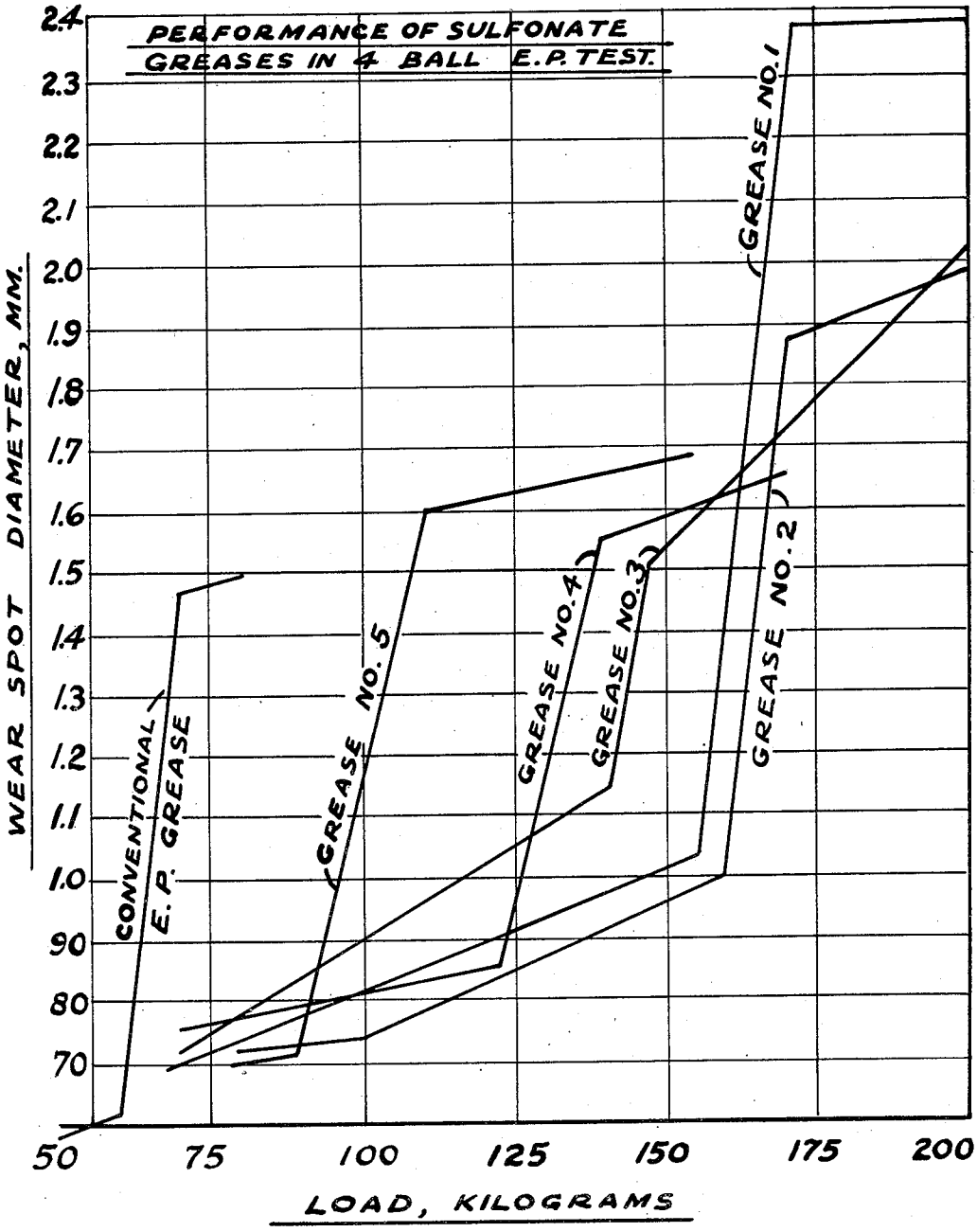

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EXTREME PRESSURE GREASE

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This invention relates to an improved extreme pressure lubricating grease composition and more particularly relates to an extreme pressure grease composition which contains incorporated therein the alkaline earth metal salt of a high molecular weight sulfonate and in conjunction therewith alkaline earth metal salts of a low molecular weight alkyl sulfonic acid and a low molecular weight carboxylic acid.

It has recently been found that the presence of the salts of low molecular weight carboxylic acids in combination with salts of high molecular weight petroleum sulfonates imparts considerable extreme pressure properties to the grease composition. This is apparently due to the activation at high loads of the sulfur present in the sulfonate. This activation allows the formation of a metallic sulfide film which is evidenced by a black film appearing upon the wear area with which the grease composition comes in contact.

It has now been found that a grease composition showing load carrying properties superior to the grease compositions described above can be prepared from sulfonates where a portion of the low molecular weight carboxylic acid salt used is replaced by the salt of a low molecular weight alkyl sulfonic acid, in order to increase the amount of sulfur present in the formulation. Apparently it is necessary to retain some of the carboxylic acid salt in the product since, when all the low molecular weight carboxylic acid salt is replaced, the product is not outstanding in extreme pressure properties. While the reason for this phenomenon is not apparent, it is believed that the presence of the low molecular weight carboxylic acid salt aids in the activation of the sulfur present and permits it to combine with the metal of the bearing surfaces forming a protective metallic sulfide film.

It is the object of this invention, therefore, to provide an improved extreme pressure lubricating grease composition which comprises admixing with a mineral oil base stock, a salt of a high molecular weight sulfonic acid, a salt of a low molecular weight alkyl sulfonic acid, and a salt of a low molecular weight aliphatic carboxylic acid.

Experience has established in grease making and in the use of greases that it is the best practice to select as the lubricating oil in the grease, an oil of the same type as would have been selected if a liquid lubricant could have been used. A wide range of lubricating oils is therefore permissible in grease production. In general, the base oil of the compositions of the present invention comprises an oil having a S. S. U. in the range of from about 30 to 180 Saybolt Universal seconds at 210° F. These lubricating oils may be either mineral lubricating oils or synthetic lubricating oils produced by methods familiar to the art.

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The high molecular weight sulfonic acids which are employed in the greases of this invention are suitably prepared by treating petroleum oils of the lubricating oil range with strong sulfuric acid. The sulfonic acids are then converted to the desired salts, which in the petroleum industry are well known by the name of petroleum sulfonates. The high molecular weight sulfonic acids which are employed have molecular weights in the range from about 300 to about 600, preferably from about 400 to about 500. The high molecular weight sulfonates can also be derived from relatively pure sulfonic acids having from about 10 to about 33 carbon atoms per mol. For example, sulfonated products of alkylated aromatics such as benzene, toluene, xylene, etc., alkylated with olefins or olefin polymers of the type of polypropylene, polyisobutylene, etc., can be used. Likewise, alkyl sulfonic acids of the 300–600 molecular weight range are suitable. The high molecular weight sulfonates of the alkaline earth metals calcium, strontium and barium are employed, the strontium sulfonates being especially preferred.

The low molecular weight alkyl sulfonic acids which are employed, for example, methane sulfonic acid, ethane sulfonic acid, propane sulfonic acid, etc., have molecular weights in the range of 96 to 166, corresponding to C_1 to C_6 sulfonic acids. The salts employed are those of alkaline earth metals, of which the calcium salt is preferred.

The low molecular weight carboxylic acid salt is preferably calcium acetate. However, in accordance with this invention, there may be used any of the alkaline earth metal salts of low molecular weight carboxylic acids, such as formic, acetic, propionic, butyric, oxalic, furoic, lactic, etc.

The greases of the invention contain from about 10% to about 30% by weight of total salts. The weight ratio of the high molecular weight sulfonate salt to the low molecular weight sulfonate plus carboxylic acid salt may be from about 1:0.4 to about 1:1.25. The low molecular weight constituents are employed in weight ratios of sulfonate to carboxylic salt of from about 0.8:1 to about 7.5:1. For example, good greases can be prepared which contain from 5 to 20%, preferably 10%, of high molecular weight sulfonate,

2 to 6%, preferably 4%, of low molecular weight sulfonate, and 2.5 to 7.5%, preferably 5%, of low molecular weight carboxylic acid salt, these percentages being by weight and based on the weight of the total composition.

EXAMPLE

A series of greases was prepared containing 10% of the strontium salt of petroleum sulfonic acid having a molecular weight of 450, and various quantities of calcium acetate and/or calcium ethane sulfonate. The following formulation is illustrative.

Composition

10% strontium sulfonate
5% calcium acetate, anhydrous
3.5% ethanesulfonic acid
1.5% hydrated lime
85.0% acid extracted coastal mineral oil, 55 S. U. S./210° F. viscosity

Procedure

The greases in the series were comparable, all being prepared in the same mineral oil base stock and using the same general procedure. In preparing the greases containing calcium ethane sulfonate, the free ethane sulfonic acid was dissolved in twice its weight of water and was neutralized with the hydrated lime. When the reaction appeared to be complete as evidenced by a drop in the temperature to about 100° F., the neutralized solution was added to a blend of the strontium sulfonate and the mineral oil and the batch was stirred at 180° F. to 200° F. until no solid particles were present. Then the calcium acetate was added as a 20% aqueous solution and the product dehydrated slowly, with stirring, until a temperature of 300° F. was reached. The product was cooled to room temperature without stirring, and was easily homogenized to a smooth grease structure.

The extreme pressure properties of the grease compositions of this invention containing mixed low molecular weight salts of sulfonic and carboxylic acids were compared with greases containing either one or the other of these constituents in an extreme pressure 4-ball testing machine at various loads. In this test machine, one steel ball is rotated against three stationary balls at difierent loads, thus producing wear spots on the stationary balls. The size of the wear spots is an indication of the anti-wear properties of the grease being tested. In comparison with a conventional extreme pressure grease, all of the sulfonate greases gave superior performance at high loads. At very high loads, however, the greases of the invention gave exceptional performances, as shown by the data in the following table:

TABLE.—SULFONATE GREASES CONTAINING 10 WEIGHT PER CENT STRONTIUM SALT OF 450 MOL. WEIGHT PETROLEUM SULFONIC ACID

Grease Number	Low Mol. Weight Salts, Weight Per Cent		4-Ball E. P. Tester Wear Spot Diameter, m. m. at 150 Kg. Load
	$(CH_3COO)_2Ca$	$(C_2H_5SO_3)_2Ca$	
1	7.5	2	1.01
2	5	4	0.96
3	2.5	6	1.54
4	0	8	1.60
5	10	0	1.67
Conventional E. P. Grease			Welded

The data in the table show that whereas the conventional extreme pressure grease is incapable of supporting high loads, the greases which contained both low molecular weight sulfonate and low molecular weight carboxylic acid salt gave low wear spot diameters in the range 0.96 to 1.54 m. m. at the high load of 150 kg.

The advantages of the greases prepared in accordance with the invention are shown graphically in the accompanying Figure 1. In the figure wear spot diameter in millimeters from the 4-ball tester is plotted against the load in kilograms. It is to be noted that the grease compositions of the invention, exemplified by greases No. 1, 2, and 3, carry extremely heavy loads in comparison with greases No. 4 and 5 which contain either low molecular weight sulfonate or carboxylic acid salt. It is also to be noted that although greases No. 3 and No. 4 are substantially equivalent at about 125 kg. and at about 160 kg., grease No. 3 is superior within this range. Also shown in the figure are the results obtained with the conventional extreme pressure grease comprising sulfurized fatty oil in a lithium base grease, which was capable of supporting only light loads.

To summarize briefly, it has been found that an improved extreme pressure grease composition may be made by incorporating into the desired lubricating oil base stock, the desired amount of alkaline earth metal salt of a high molecular weight sulfonic acid that is oil soluble and water insoluble, for example, a strontium salt of a sulfonic acid having a molecular weight within a range of 300 to 600 with an alkaline earth metal salt of a low molecular weight sulfonic acid that is oil insoluble and water soluble, for example, a calcium salt of an alkyl sulfonic acid having a molecular weight within a range of from 96 to 166, and an alkaline earth metal salt of a low molecular weight carboxylic acid, for example, calcium acetate. It is to be understood, of course, that the percentages of the various constituents may be varied to produce the particular grease composition desired. It is also to be understood that various addition agents may be added to these grease compositions to change their performance characteristics, for example, oxidation inhibitors, viscosity index improvers, tackiness agents, and the like may be incorporated in the grease to modify those characteristics.

What is claimed is:

1. An improved lubricating grease composition consisting essentially of a lubricating oil base stock having incorporated therein from about 5 to 20% of an alkaline earth metal sulfonate prepared from a sulfonic acid having a molecular weight within a range of from 300 to 600, from about 2% to 6% by weight of an alkaline earth metal sulfonate prepared from alkyl sulfonic acid having a molecular weight within a range of from 96 to 166 and about 2.5% to 7.5% of an alkaline earth metal salt of an aliphatic carboxylic acid having from 1 to 4 carbon atoms.

2. An improved lubricating grease composition consisting essentially of a lubricating oil base stock having incorporated therein about 5 to 20% by weight of an alkaline earth metal sulfonate prepared from a petroleum sulfonic acid having a molecular weight within the range of from 400 to 500, from 2 to 6% by weight of an alkaline earth metal sulfonate prepared from an alkyl sulfonic acid having a molecular weight within a range of from 96 to 166, and from 7.5% to 2.5% by weight of an alkaline earth metal salt of an

aliphatic carboxylic acid having from 1 to 4 carbon atoms.

3. An improved lubricating grease composition consisting essentially of a lubricating oil base stock having incorporated therein about 10% by weight of a calcium sulfonate prepared from a sulfonic acid having a molecular weight of 450, about 4% by weight of a calcium alkyl sulfonate prepared from a sulfonic acid having a molecular weight of 110, and about 5% by weight calcium acetate.

4. An improved extreme pressure lubricating grease composition which consists essentially of a lubricating oil base stock thickened to a grease consistency with a mixture of about 10% of water-insoluble, oil-soluble alkaline earth metal salt of a petroleum sulfonate, about 4% of a water-soluble, oil-insoluble alkaline earth metal salt of an alkyl sulfonate, and about 5% of an alkaline earth metal salt of an acid selected from the goup consisting of aliphatic carboxylic acids having from 1 to 4 carbon atoms.

5. An improved extreme pressure lubricating grease composition which consists essentially of a lubricating oil base stock thickened to a grease consistency with a mixture of about 5 to 20% by

weight of an oil-soluble, water-insoluble alkaline earth metal salt of a sulfonic acid, about 2 to 6% by weight of an oil-insoluble, water-soluble alkaline earth metal salt of an alkyl sulfonic acid, and about 7.5% to 2.5% by weight of an alkaline earth metal salt of an acid selected from the group consisting of aliphatic carboxylic acid having from 1 to 4 carbon atoms.

6. An improved extreme pressure lubricating grease composition which consists essentially of a lubricating oil base stock thickened to a grease consistency with a mixture of about 10% by weight of a strontium salt of a sulfonic acid having a molecular weight of 450, about 4% by weight of a calcium salt of ethane sulfonic acid, and about 5% by weight of calcium acetate.

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