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KETO-ACID SOAP GREASES

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The present invention relates to lubricating grease compositions and, particularly, to lubricating greases containing soaps of keto-acids of the long chain monocarboxylic type. The invention has special application to calcium soap greases based on such acids, but is not necessarily limited thereto.

Lime soap greases as a class are an important type of lubricants. They are widely used in industry because of their smooth texture and their structure stability at ordinary temperatures. They are economical and are easily made and for this reason they are used for a majority of applications where high temperatures are never encountered. They are somewhat deficient, however, where temperatures approaching or exceeding the boiling point of water are encountered, because lime greases rely upon the presence of a small amount of water to maintain their true colloidal grease structure. If this water is evaporated, as may occur at moderately high temperatures, the greases become grainy, the oil separates from the soap, and failure of lubrication promptly ensues.

It has previously been suggested that lime soap greases might be modified in various ways to improve somewhat their high temperature properties. Thus, it has been suggested that a complex soap be substituted for the conventional soap of straight fatty acids, for example, by introducing a metal acetate or other low molecular weight water-soluble salt which forms a complex with the normal fatty acid soap and improves high temperature stability. It has also been suggested by the present inventors in a previous application, Serial No. 81,206 filed March 12, 1949, that a calcium base grease containing 12-hydroxy stearic acid might have improved high temperature properties.

It has now been discovered that greases made from keto-acids of the long chain type, for example, monocarboxylic acids having not less than 12 nor more than 22 carbon atoms, have outstanding high temperature properties. The mono-keto-acids are specifically preferred. Apparently, the presence of the keto group stabilizes lime soap greases in the same manner that the presence of a small amount of water stabilizes the conventional prior art products. The keto group, however, is not affected by a rising temperature and the product remains stable where the conventional lime base grease would disintegrate upon evaporation of its normal water content. The proportions of soap to be used in such a grease tend to run somewhat higher than

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those of conventional lime soaps, but, in general, proportions from 5% to 30% of lime soap, based on the weight of the total composition, appear to be operable. Specifically, proportions of 10 to about 25% are preferred. The soap may contain other constituents but preferably is preponderantly the soap of one or more of the keto-acids coming within the description given above. Thus, commercially available products, or products which can readily be prepared from conventional raw materials may contain other acids such as hydroxy acids or normal fatty acids along with a major proportion of the keto-acid product. The keto-stearic acids are particularly suitable, e. g. 12-keto-stearic acid, 4-keto-stearic acid, and various other isomers.

The invention may more fully be understood by reference to the following specific example:

Example

A grease was prepared by heating 20 parts by weight of a 12-keto-stearic acid, 20 parts of mineral base lubricating oil, and 2.7 parts of hydrated lime. These ingredients were cooked to a temperature of 250° F. and thereafter additional lubricating oil of mineral base was added, together with 1 or 2% of water, until the soap content approximated about 22% of the weight of the finished grease. The product thereafter was cooled to about 180° F. and packaged. It showed the following inspection:

	Keto-Stearic Acid Grease	Commercial Lime Grease
Soap Content, Percent	22	13.
Worked Penetration 77° F.	300	280.
Free Acid, Percent	0.1	nil.
Free Alkali, Percent	0.16	0.1
Dropping Point, °F.	290	195.
Water Resistance[1]—Percent Water Absorbed at 150° F.	25	14.
Temperature at which Grease Separates[2].	265° F.—nil. 280° F.—trace 290° F.—1% 305° F.—14%	150° F.—nil. 165° F.—trace 180° F.—39%.

[1] 100 grams of grease are placed in a grease worker. 20 c. c. of water is added and worked for 300 strokes. If no free water is present, an additional 20 c. c. is added and worked again for 300 strokes. This procedure is repeated until the grease sheds further additions of water or breaks down to a thin emulsion.

[2] Two grams of grease are weighed into a flask and placed in an oven at the desired temperature for 45 minutes. It is then removed, allowed to cool and the separated oil drained and weighed.

The 12-keto-stearic acid described above was prepared by oxidizing 12-hydroxy stearic acid with potassium dichromate and sulfuric acid in benzene solution at approximately room temperature. The reaction product was recrystallized from glacial acetic acid, the final product hav-

ing an acid number of 186. The product was analyzed and found to contain 89% by weight of 12-keto stearic acid and 11% of 12-hydroxy stearic acid.

The table above shows comparisons between the product of the example and a standard commercial lime soap grease of about the same consistency. The soap used in the commercial grease was a mixture of calcium oleate and calcium stearate. It should be noted, as previously indicated, that the soap content of keto-stearic acid grease is higher than that of the commercial product, about 22% of 12-keto stearic acid soap producing a grease of the same consistency as 13% of standard lime soap. In larger batches, the difference is not quite as great, the soap content being more nearly equal, but it appears that somewhat more soap of the keto-acid type is required than of standard fatty acid soaps to produce a product of a given consistency.

The product of the example, it will be noted, had a melting or dropping point of 290° F., whereas the conventional lime grease melted at 195° F. Both showed excellent water resistance, but at 180° F. the conventional grease showed serious oil separation while the keto stearic acid grease showed only 1% separation at 290° F. Since oil separation quickly results in a failure of the lubricant, it is evident that this is an extremely important consideration where high temperature operation is involved. In this respect, greases prepared from 12-keto stearic acid are much superior to conventional lime-stearic acid greases.

While the above example is limited to a particular keto stearic acid, it will be understood that related products derived from palmitic, myristic, and lauric acids, or from the C_{20} and C_{22} fatty acids and the corresponding glycerides, may all be used. Mixtures of them may be used also, the choice of soap stock depending upon the degree of saturation required (which is dependent upon the desired resistance to oxidation, etc.) the softness or hardness of the soap, which is dependent upon the chain length to some extent, and the like as suggested above. Satisfactory soaps have been prepared from 4-keto stearic acid, derived from hydrogenated oiticica oil but various isomers and homologs may be used within the above stated limits.

While lime is the preferred saponifying agent, other metal bases may be used of the alkali metal and alkaline earth metal groups. The greases also may contain soaps of other acids, in minor proportions, and the usual modifiers, such as antioxidants, tackiness agents, extreme pressure additives, viscosity index improvers, and the like, may be included in conventional proportions, as will be evident to those skilled in the art.

In general, the products are prepared by combining about 1 to 2 parts of the aliphatic monoketo fatty material, which may be either the fatty acid or the corresponding glyceride ester, with 1 to 2 parts by weight of the mineral base lubricating oil. A sufficient quantity of the metal base saponifying agent is added to saponify and substantially neutralize the fatty material. These ingredients are heated to saponification temperature between about 200° and 300° F. Thereafter, further quantities of oil are added, from 2 to 10 parts by weight, depending upon the consistency desired in the finished product. This additional oil is preferably a mineral base oil, but synthetic oils, such as the conventional dibasic acid alkyl esters or the polyglycols, or mixtures thereof, with each other and/or with mineral base oil, may be employed. As is well-known in the art, the esters are not very suitable for forming the original soap in situ, and when an ester base grease is desired the soap should be preformed independently or should be formed in mineral oil which does not hydrolyze during the saponification.

Soaps of the keto-acids may be preformed in much the same manner as conventional soaps and by the use of suitable homogenizing equipment these may be incorporated into lubricating oils, like other preformed soaps, to prepare the greases. The formation of the soap in situ, as described in detail above, is usually preferred.

What is claimed is:

1. A lubricating grease composition comprising a major proportion of lubricating oil thickened to a grease consistency with a minor proportion of a metal soap of 12-keto stearic acid.

2. A lubricating grease composition comprising a mineral base lubricating oil thickened to grease consistency with a soap of a metal selected from the alkali and alkaline earth metals, said soap being predominantly of 12-keto stearic acid.

3. Composition according to claim 2 wherein the metal soap is a lime soap.

4. Composition according to claim 1 wherein the metal soap is a lime soap.

5. The process of preparing a lubricating grease which comprises combining about 20 parts by weight of mineral base lubricating oil, about 20 parts of an aliphatic monocarboxylic acid which is predominantly 12-keto stearic acid, and about 2.7 parts of hydrated lime, heating the mixture to a temperature of about 250° F. for a time sufficient to substantially complete saponification to form a grease thickening soap, and thereafter adding additional lubricating oil and cooling to form a grease of 5 to 30% soap content by weight.

6. A lubricating grease composition consisting essentially of mineral base lubricating oil thickened to a grease consistency with 5 to 30% by weight, based on the total composition, of the lime soap of an acid composed substantially of 11% by weight of 12-hydroxy stearic acid and 89% 12-keto stearic acid.

7. Composition according to claim 6 having a soap content of about 22% by weight.

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