

UNITED STATES PATENT OFFICE

2,289,748

GREASE COMPOSITION FOR LUBRICATION

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No Drawing. Application February 18, 1939,
Serial No. 257,206

1 Claim. (Cl. 252—42)

The present invention relates to an improved grease composition for lubrication and to methods for preparing the same.

Grease compositions are prepared by adding metallic soaps to lubricating oils, especially the soaps of those metals which cause the oil to gel. Of these metals, those of the first and second groups of the periodic table of elements are the most important, particularly sodium, potassium, calcium and barium. These may be used together or separately or they may be mixed with other metal compounds, particularly aluminum.

The various soaps are used for different purposes as is well known in the arts; for example, soda or potash soaps are preferred for high temperature service and form the so-called fibrous greases, while lime and barium soaps are used for producing water resistant greases. The quality of the greases is determined in the first instance by the particular soaps employed, but a considerable variation may be obtained by varying the amount of the soap and, for certain purposes, it has been proposed to provide a slight excess of free alkali; for other purposes, a slight excess of free acid is preferred in the final composition. The excess in either case is generally quite small, usually below about 1% and often only the smallest traces are sufficient to produce a considerable change in the structure of the grease, making it suitable for one or another type of lubrication.

Lubricating greases, like oils, are subject to deterioration by oxidation. The deterioration is slow but it results in the formation of partial oxidation products which appear to accelerate oxidation and deterioration until crusts which are of no lubricating value are formed. The accumulation of the crusty material gradually prevents proper lubrication by the unoxidized portion of the grease. This deterioration is also catalyzed by various metallic surfaces and metallic ingredients, and particularly is it affected by copper containing metals such as brass. Greases of all of the types mentioned above, whether containing traces of free acid or free alkali, are subject to this type of decomposition and, like lubricating oils, the deterioration may to some extent be checked by the addition of inhibiting agents of which the best known are the aromatic hydroxy and amine compounds.

It has been found that the choice of the inhibitor is particularly affected by whether the grease is one containing a trace of free acid or free alkali. Briefly, it has been found that when using greases containing free acid, the phenolic inhibitors are as a class markedly superior to

the amino compounds and, on the other hand, when using alkaline greases, that is to say those containing free alkali, the amino compounds are greatly to be preferred to the phenolic materials. No reason is known for this particular effect, but it is believed that the free acid present reacts quite rapidly with the free amine and in the same way, a free alkali reacts quite rapidly with phenol and in this way the inhibitors are rapidly removed if they are chosen in this way. On the other hand, no reaction takes place between the amine and the alkali, and any reaction which might possibly take place between the phenol and the acid is so slow as to be negligible. However this may be, the fundamental effect on which the present applicants rely is their discovery that phenolic inhibitors are much more effective in greases which contain free acid and, on the other hand, amino compounds are likewise much more effective in greases containing free alkali.

The grease may be made up of various fats or fatty oils, such as tallow, stearine, stearic acid, vegetable oils, marine oils and the various mixed fats obtained therefrom, and also from rosin, naphthenic acids or synthetic acids, for example those obtained by the oxidation of highly purified oils, waxes, deoiled petrolatum and the like. The amount of the soap or soap mixtures used in the oil may, as indicated before, vary considerably say from 5 to 10% to as much as 40 to 50% of the total mixture. As is well known in the art, relatively small amounts of the soap produce low melting greases whereas larger amounts, up to 30, 40 or 50%, produce increased hardness and raise the melting point.

Among the phenolic inhibitors which are used in connection with the present grease compositions, containing an excess of free acid, the most important ones are alpha and beta naphthols, various cresols and other alkylated phenolic materials, dihydroxy and polyhydroxy aromatics containing single or condensed rings, and also other compounds such as nitro phenols, halo phenols, guaiacol, eugenol and the like. The phenolic inhibitors specifically mentioned are the ones preferred, but it may be stated as a general rule that the phenolic materials as a class are quite effective for the present purposes and they are particularly effective in the presence of free acids, whereas they are much less effective in the presence of free alkali.

Among the amino compounds which are found to be particularly effective in grease compositions may be included alpha and beta naphthylamines, the substituted naphthylamines such as phenyl alpha and phenyl beta naphthylamine, various cresyl amines, halo and nitro amines, various di-

amines such as phenylene diamines or naphthalene diamine, diphenyl amine and the like. Here again it can be stated as a general rule that the amines as a class are effective inhibitors in checking the oxidation of grease compositions and the amino compounds are much more effective in the presence of alkali than in the presence of free acid.

The amounts of the inhibitors of the two classes mentioned above may be as little as .10% or even less, and are used in less than 1% concentrations in the present case. In the specific choice of an inhibitor for a grease, many factors must be taken into consideration. Some inhibitors are much more effective than others. The amount and the kind of soaps also have a profound effect in the life of the grease and the oil also has a minor effect so that all of these factors must be taken into account. Nevertheless, for the same basic grease formula, i. e., greases made from the same oil and soap in the same proportions, it will be found that the amino inhibitors as a class are superior to the phenolic inhibitors where free alkali is present and vice versa where free acid is present.

The present greases may contain in addition to the ingredients mentioned above, the various others which are employed in the grease making art for specific purposes. It is usually the case to prepare soda and potash grease in the absence of water whereas lime or barium greases usually contain about 1% of water. Glycerine or glycols may be present or absent depending on the particular quality desired and among the other materials organic thickeners such as polybutylene waxes, for example, petrolatum, inorganic fillers, and extreme pressure agents such as chlorine or sulfur compounds may be added. These materials are found not to interfere with the operation of the inhibiting compounds.

EXAMPLE I

The test used to establish the oxidation stability of our greases is that developed by Wright and Lutz, described in Product Engineering, vol. 7, pp. 210-13 (1936). The apparatus consists of a pressure-tight bomb fitted with an accurate gauge, and five small glass dishes of standard size and shape. These dishes are loaded with the grease to be tested and a catalyst, which may be any metal desired in the form of a sheet or a fine powder. This catalyst serves the double purpose of imitating conditions in a bearing and decreasing the time required for the test. We use a thin sheet of brass cut to fit the dish, or, no catalyst for very unstable greases. The filled dishes are placed in a suitable rack, and the whole sealed into the bomb. The bomb is then flushed thoroughly with oxygen, filled to a suitable pressure, and placed in an oil bath at 175° F. After temperature equilibrium is established, the pressure is adjusted to 110 lbs./sq. in. Readings are taken of the pressure at two hour intervals.

The pressure is found to drop slowly at first, eight to twenty hours being required for one pound loss, but suddenly the rate increases to several pounds every two hours. The time elapsed prior to the rapid increase in oxygen absorption is called the life of the grease.

A ball bearing grease made from a Mid-Continent oil of 200 Saybolt Universal seconds at 100° F., 14% of sodium stearate, and ½% of aluminum stearate, with 0.1% free stearic acid, was tested with 0.1% each of the following antioxidants:

Susceptibility of soda-aluminum grease

Antioxidant	Life
	Hours
None.....	50
Phenyl-α-naphthylamine.....	70
Phenyl-B-naphthylamine.....	70
Guaiacol.....	288
P-cresol.....	95

These data show that the amino inhibitors in a grease containing free acid give little improvements but the use of phenolic inhibitors, especially guaiacol, is much superior.

EXAMPLE II

Another grease, made with the same oil, but containing 21.25% sodium stearate, 2.75% barium stearate and 0.14% free sodium hydroxide gave the following results:

Susceptibility of soda-barium grease

Antioxidant	Life
	Hours
None.....	45
Phenyl-α-naphthylamine.....	170
Guaiacol.....	150

In this case the amine is superior although the guaiacol is fairly good.

EXAMPLE III

A third grease prepared with a similar oil and containing 15.5% sodium soap and 3.0% calcium soap of tallow, with 0.60% free sodium hydroxide, gave the following results with 0.1% of antioxidants:

Susceptibility of soda-calcium grease

Antioxidant	Life
	Hours
None.....	136
Phenyl-α-naphthylamine.....	312
Guaiacol.....	174

Here again the amine is superior to the phenol in the alkaline grease.

EXAMPLE IV

A low temperature cup grease made from transformer oil and 10% of calcium soap of lard oil, containing excess free lime was tested with 0.1% of inhibitors of both types.

Susceptibility of cup grease

Antioxidant	Life
	Hours
None.....	4
Guaiacol.....	54
Phenyl-α-naphthylamine.....	75

The present invention is not limited to any theory of the action of the inhibiting compounds in the various greases, but only to the following claim in which it is desired to claim all novelty inherent in the invention.

We claim:

An improved grease composition comprising 5 to 50% of soda soap in mineral lubricating oil, a slight excess of acid and as an inhibitor of oxidation a small amount of guaiacol.

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