Patented Dec. 29, 1936 2,065,857

UNITED STATES PATENT OFFICE

2,065,857

ANTILEAK AGENT FOR GREASES

William P. Hilliker, Hammond, Ind., assignor to Standard Oil Company, Chicago, Ill., a corporation of Indiana

No Drawing. Application October 27, 1933, Serial No. 695,510

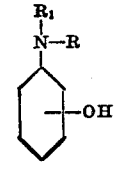
7 Claims. (Cl. 87—9)

This invention relates to a new grease composition containing soaps and method for preparing the same. My invention is particularly concerned with the method of preparing calcium soap and aluminum soap greases so that they do not leak or separate during storage or use into a predominately oil phase and a phase higher in soap than the original composition.

Heretofore cup greases have been prepared by mixing water, oil, and lime, in a steam-jacketed grease mixer. The fatty acid is then added and heat is applied. After an interval of about 40 to 60 minutes, when the temperature has reached 240–260° F., the soap is ready for mixing. About 2% to 3% of water is added, and when the batch foams up it is driven down by the addition of oil, the heat being abstracted by the addition of cold oil and by the vaporization of water so that the temperature drops to about 230° F. Oil is added until the batch contains about 25% soap, and during this interval the temperature drops gradually until it reaches about 205–210° F. If desired, the soap percentage may be reduced to about 15, and the batch should reach a temperature of about 180–190° F. at this point. This process is about the same for fats as for fatty acids except that it requires 12 to 20 hours at 240–260° F. to effect the saponification of the fats.

When calcium soap greases are stored for a period of six months or more or used, the oils oftentimes tend to ooze out of the composition and rise to the top of said composition. This phenomenon is called "leakage." The separated oil has the appearance of an oil which has been sweated out of the grease and collects in pools or pockets in any depression which forms on the surface of the grease. This differs from "separation" at high temperatures in that the oil which leaks may be stirred back into the grease to form a homogeneous mass. When complete separation of the grease occurs, agitation or stirring will not effect a homogeneous mass.

I have discovered that certain classes of organic compounds, when added to greases containing calcium or aluminum soap, prevent leaking or the breaking down of the grease into an oil phase and a thick semi-solid residue. The classes of organic compounds or anti-leak agents which may be used to prevent this particular type of deterioration are the aminophenols, substituted aminophenols, alkyl aminophenols, aryl aminophenols, aralkyl aminophenols, phenolic aromatic compounds such as the hydroxy benzene compounds and polyhydroxy benzene compounds, alkyl amines and aryl amines. Some of the amino phenols may be represented by the general formula:



wherein R represents a hydrogen atom, alkyl, aryl or aralkyl radical and R_1 represents a hydrogen atom, alkyl, aryl or aralkyl radical.

My invention is concerned, therefore, with the preparation of grease compositions containing calcium or aluminum soap which do not leak during storage or use. These calcium or aluminum soap greases are generally referred to as cup greases. The greases may be prepared by any of the conventional methods, for example, calcium or aluminum soap of an animal or vegetable fatty acid or oil is mixed with a mineral oil of 90 to 500 Saybolt viscosity at 100° F., and a small amount of water. The mixture is agitated and heated in a steam jacketed kettle to a temperature of about 200–212° F. until a homogeneous mass is obtained. After the grease has been prepared, the anti-leak agent should be mixed therewith in amounts of 0.1% to 0.4%. A grease of any desired consistency can be prepared by varying the soap content of the composition and/or using a low or high viscosity mineral oil. Generally, the soap content of the cup grease varies from 5% to 25%. Other methods for preparing cup greases will be found in the book by Klemgard on Lubricating Greases and my invention may be used in any cup grease. For example, the anti-leak agents may be used in the cup greases disclosed in my U. S. Patent No. 1,989,197, application for which was filed August 9, 1932 which describes a new cup grease comprising hydrogenated fatty oils or hydrogenated fatty acids.

The following is an example of a formula for

cup grease in which my stabilizing agents may be employed:

	Lbs.
Hydrated lime	1.44
Water	0.55
Caustic soda	0.17
Mixed fatty acids	11.66
Mineral oil (Saybolt viscosity 300 at 100° F)	86.50
Anti-leak agent (paramethyl aminophenol)	0.15

The viscosity of the mineral oil used in the general formula may vary from 85 to 500 Saybolt at 100° F., however, the cup greases which give considerable trouble with leakage are those which contain mineral oils having a viscosity from 85 to about 400 Saybolt viscosity at 100° F. and from 5% to 20% of calcium or aluminum soaps. Instead of using mixed fatty acids in the above formula, other high molecular weight fatty acids, saponifiable fats and fatty oils and mixtures of the same may be used, for example, tallow, lard oil, cocoanut oil, castor oil, corn oil, palm oil, soy bean oil, oleic acid, and the like and mixtures thereof.

As another example of my invention the following formula for cup greases may be used:

	Lbs.
Tallow	12.55
Cottonseed fatty acids	3.10
Hydrated lime	1.97
Caustic soda	0.13
Paraffin oil (Saybolt viscosity 80 at 100° F)	80.90
Water	1.70
Anti-leak agent (dimethyl amine)	0.2

As stated hereinbefore, my anti-leak agents can be used in cup greases containing aluminum soaps of fatty acids. These greases can be prepared by adding an aluminum soap to an oil, stirring until smooth, heating with steam to 200° to 400° F. until the soap dissolves. The composition can be cooled slowly or rapidly in thin films, or stirred down to a lower temperature, or water can be added, depending upon the type of grease desired. The anti-leak agent should be added after dissolving the soap.

Samples of cup greases of a consistency of about 316 as taken by the A. S. T. M. penetrometer at 77° F., containing about 13% of calcium-mixed fatty acid soaps in mineral oils of 300 Saybolt viscosity at 100° F., were stabilized with about 0.15% of various different anti-leak agents and stored for 17 months at room temperatures and then given an additional storage of 12 months at 150° F. The following table gives the results obtained with the respective anti-leak agents:

Anti-leak agent	17 months storage at room temperature	Additional storage of 12 months at 150° F.
Control (no anti-leak agent)	Leakage	Leakage.
Paramethyl aminophenol	No leakage	No leakage.
Para aminophenol	do	Do.
Parabenzyl aminophenol	do	Do.
Hydroquinone	do	Very slight leakage.
4-hydroxyl diphenyl	do	Do.
Ortho cresol	do	Do.
Alpha naphthol	do	Do.
Diphenyl amine	do	Do.
Dibenzyl amine	do	Do.
Dimethyl amine	do	Do.

The term "leakage" is used in the above table to describe the condition of the cup grease at the end of the storage period, and indicates that a portion of the oil in the grease composition has risen to the top of the grease or separated from the grease.

Other compounds which can be used for the purpose of my invention are ethyl aminophenols, para diethyl and dipropyl aminophenols, phenyl aminophenols, tolyl aminophenols, para phenyl aminophenols, dibenzyl aminophenols, 2-methyl amino 1-naphthol, aminonaphthols, alkylated polyhydroxy benzene compounds, tertiary butyl catechol, catechol, para cresol, amino cresols, dibutyl amine, alkylene polyamines, propylene polyamine, and the like. The anti-leak agents should not contain any thio groups. However, sulphur per se can be used with reasonable success as an anti-leak agent.

I am aware of the use of anti-oxidants in compositions which tend to oxidize on storage, however, the action of the anti-leak agents cannot be explained upon the theory of oxidation.

Although my invention has been illustrated by the use of specific grease compositions and anti-leak agents, it should be understood that other modifications may be used without departing from the scope of the following claims.

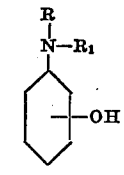
I claim:

1. An improved grease composition comprising a homogeneous mixture of a mineral oil, about 5% to about 20% of a calcium soap of a high molecular weight fatty acid, and a small proportion of an anti-leak agent not exceeding 0.4%, containing no thio groups, which prevents the separation of oil from said grease, said anti-leak agent being an organic aryl compound having an unneutralized hydroxyl group attached to the aryl nucleus.

2. A cup grease composition comprising a homogeneous mixture of oil, calcium soaps of high molecular weight organic acids and a small proportion, not exceeding about 0.4%, of an anti-leak agent, containing no thio groups, which prevents the separation of oil from said composition, said anti-leak agent being an unneutralized phenolic compound.

3. An improved cup grease comprising oil, calcium soaps and not more than about 0.4% of an anti-leak agent, containing no thio groups, which prevents the separation of the oil from said grease, said anti-leak agent being an aminophenol.

4. An improved grease composition comprising a substantially homogeneous mixture of an oil, calcium soaps, and a small proportion of an anti-leak agent, containing no thio groups, which prevents the separation of oil from said grease, said anti-leak agent being an aminophenol of the general formula



wherein R represents a hydrogen atom, alkyl radical, aryl radical or aralkyl radical, and R_1 represents a hydrogen atom, alkyl radical, aryl radical or aralkyl radical.

5. An improved grease composition comprising a homogeneous mixture of a mineral oil, from about 5% to about 20% of a calcium soap of a high molecular weight fatty acid, and a quantity of an anti-leak agent not exceeding about 0.4%, said anti-leak agent being a substi-

tuted aminophenol and containing no thio groups.

6. An improved grease composition comprising a substantially homogeneous mixture of a mineral oil, calcium soaps of high molecular weight fatty acids, and a small proportion of an anti-leak agent not exceeding about 0.4%, containing no thio groups, which prevents the separation of oil from said grease, said anti-leak agent being an aminophenol wherein one amino hydrogen has been substituted with an organic radical.

7. The process of preventing a cup grease containing calcium soaps from separating into a predominately oil phase and a predominately soap phase during storage or use which comprises incorporating into said grease not more than 0.4% of an aminophenol, containing no thio groups, which prevents said phase separation.

WILLIAM P. HILLIKER.