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ALKALI METAL SOAP THICKENED LUBRICATING OIL

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The present invention relates to improved lubricating greases, in particular to lubricating greases which are stable at high temperatures and which are resistant to mechanical stress and to oxidation and processes for preparing them.

Greases used at high temperature under difficult working conditions, at high speed and under heavy loads, must possess good resistance to mechanical stress, stability at high temperature and good resistance to oxidation to ensure satisfactory lubrication.

It is well known that greases having an alkaline soap base, notably sodium soaps, dispersed in a highly refined mineral oil having a good viscosity index, are in general best suited for such purposes. The use of certain additional compounds with such greases, particularly polyglycols and polyalkylene amines, act as peptizing agents, resulting in the effective dispersal of the alkaline soaps in the base oils, and which at the same time give the greases an excellent resistance to mechanical stress and a good stability at high temperatures. Aromatic amines such as phenylalpha naphthylamine, act as anti-oxidants when incorporated in such greases.

The present invention relates to greases which have a good stability at high temperature and under mechanical stress, a remarkable resistance to oxidation as well as other advantageous properties, which contain a stabilizing agent obtained by condensing an alkylene oxide with a phenolic aminated resin.

The proportion of stabilising agent incorporated in the grease is, according to the invention, between 0.1 and 5%, particularly between 0.5 and 2% by weight based on the total grease composition.

The phenolic-amino-resins may be obtained by condensation of an alkylphenol with, for example, formaldehyde or trioxymethylene in the presence of ammonia.

The alkylene oxides or the polyoxyalkylenes capable of being condensed with the phenolic-amino-resins preferably contain from 2 to 6 carbon atoms per molecule, and include polyoxides. They are preferably ethylene oxide, propylene oxide, or butylene oxide.

The lubricating oils used as base oils in the greases of the present invention may be mineral or synthetic oils having a viscosity at 100° F., preferably between 15 and 433 centistokes and particularly between 18 and 217 centistokes, and having a viscosity index within the range 0 to 160, and preferably greater than 60.

The metal soaps employed in the greases of the present invention are preferably alkali or alkaline-earth metal soaps, particularly sodium or lithium soaps having the ability to form greases having a sufficiently high drop point, and are present in the greases in grease-making proportions, preferably between 3 to 30% by weight based on the total composition, and according to the type of grease which it is desired to obtain.

The acids used to obtain the grease-making soaps are preferably long chain fatty acids which may be saturated

or unsaturated and may be obtained, for example, from animal, marine or vegetable hydrogenated oils and greases or by the oxidation of hydrocarbons, or they may be triglycerides.

Other addition compounds may be incorporated in the greases of the present invention, such as agents improving adhesion, anti-rust agents, and extreme pressure agents.

The greases in the invention may be prepared by the ordinary mixing, heating and cooling processes. Thus the fatty acid and the stabilizing agent described above may be added to all or part of the selected lubricating oil, and after heating the mixture to a temperature sufficient to obtain a homogeneous mixture, the saponification agent is added and further heating takes place until homogenization and saponification takes place. The grease is then poured on to trays and after cooling, the blocks of grease so formed are worked up to the desired consistency. The stabilizing agent may be added, if desired, at the end of the heating, before the pouring, instead of with the fatty acid.

The use of stabilizing agents according to the present invention is particularly efficacious for greases obtained from substantially naphthenic oils, or from synthetic oils of an aromatic character.

The present invention therefore comprises improved greases comprising a lubricating oil, a grease-making proportion of a metal soap, and a minor proportion of a stabilizing agent consisting of the reaction product of a phenolic-amino-resin with an alkylene oxide.

The following examples show the advantages obtained by the composition of the present invention.

Two phenolic-amino-resins A and B were prepared by heating the following mixtures for 6 hours at 130°–150° F.

	Resin A	Resin B
Octyl phenol	100	100
Dilution oil (viscosity of 50° C. 29 to 38 centistokes)	110	50
Trioxymethylene	20	20

15 parts of ammonia were injected during 6 hours, then heated for 2 hours at 212°–220° F. and finally for 3 hours at 300° F. In this way the resins A and B having the following characteristics were obtained:

	Resin A	Resin B
Viscosity at 210° F. in centistokes	183	
Percent of N	1.4	2.2
Percent active product	48	67

From the resin A the stabilizing agent C was prepared by reacting with 15% of ethylene oxide at a temperature of 320°–356° F. for 15 hours.

In the same way the stabilizing agent D was obtained by reacting 25% of ethylene oxide with resin B at a temperature of about 320° F. for 20 hours.

The mode of operation used for the preparation of the greases in the examples was derived from the recognised grease-making process called "pan cooling." It consists of putting all the ingredients in a tank, eliminating the water, heating until the melting of the mass in the neighbourhood of 390° F. The mass is then poured into trays and, after re-cooling, the pans of grease are worked and homogenized.

A grease having the following composition was pre-

pared as hereinbefore described, and labelled "Grease A."

Stearic acid, percent ---------- 15
Pure soda, percent ---------- 2.45
Mineral oil, percent ---------- 82.55

(Viscosity at 100° F.=32 centistokes. Viscosity index=115.)

1.2% in weight of resin B was incorporated in grease A, the mixture being labelled "Grease B."

EXAMPLE I

1% in weight of stabilizing agent C was incorporated in grease A.

EXAMPLE II

1.5% in weight of the stabilizing agent D was incorporated in grease A.

Table I shows the results of the tests for the greases described above.

Table I

Results		Greases prepared as in—			
		Grease A	Grease B	Example I	Example II
Characteristics	Drop point, ° F.	173	174	178	183
Flow test	100 g. of grease are placed on a wire gauze cone having a mesh aperture of 149 microns (100 mesh) placed above a beaker and held in position for 50 hours. The percent in weight of the oil separated is ascertained.	9	8.7	0.9	0.8
Stability as regards mechanical working	Penetration ASTM: Worked 50 strokes	350	352	287	283
	Worked 1,000 strokes	370	374	320	308
	Worked 100,000 strokes	Fluid	Fluid	345	325
Oxydization test (Norma-Hoffman)	A sample of 20 g. of grease is placed in a bomb under an oxygen pressure of 7 kg./cm.² at 99° C. The time necessary to obtain a pressure drop of 0.35 kg./cm.² is measured.	[1] 10	[1] 600	[1] 400	[1] 600

[1] Hours.

Table I shows that by incorporating stabilizing agents according to the invention, improved greases are obtained, and which are superior to even greases containing a phenolic-amino-resin.

What we claim is:

1. A lubricating grease composition comprising a major proportion of a mineral lubricating oil, in the range of 3 to 30 weight percent of an alkali metal soap of a long chain fatty acid, and in the range of 0.1 to 5 weight percent of a stabilizing agent consisting essentially of a reaction product formed by the condensation of a higher alkyl phenol, a formaldehyde and ammonia in an oil diluent at a temperature in the range of 130° to 300° F. to obtain a phenolic-amino-resin containing in the range of 1.4 to 2.2 weight percent combined nitrogen and then reacting said resin within the range of 15 to 25 weight percent, based on said resin, of an alkylene oxide having in the range of 2 to 6 carbon atoms at a temperature in the range of 320° to 356° F.

2. A lubricating grease composition comprising a major proportion of a mineral lubricating oil, in the range of 3 to 30 weight percent of an alkali metal soap of a long chain fatty acid, and in the range of 0.1 to 5 weight percent of a stabilizing agent consisting essentially of a reaction product formed by the condensation of a higher alkyl phenol, a trioxymethylene, and ammonia at a temperature in the range of 130° to 300° F. to obtain a phenolic-amino-resin containing in the range of 1.4 to 2.2 percent combined nitrogen, and then reacting said resin within the range of 15 to 25 weight percent of ethylene oxide at a temperature in the range of 320° to 356° F.

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