United States Patent Office 2,854,410 Patented Sept. 30, 1958

2,854,410

SOAP-THICKENED LUBRICATING GREASES CONTAINING AN ALCOHOL-SUBSTITUTED AMIDE

Philippe Gaston Louis Bidault, Seine Maritime, France, assignor to Esso Standard Societe Anonyme Française, Paris, France, a body corporate

No Drawing. Application August 3, 1953
Serial No. 372,179

Claims priority, application France December 12, 1952

7 Claims. (Cl. 252—42.1)

The present invention relates to improved lubricating greases. More particularly, the invention refers to lubricating greases of improved stability at high temperatures and under the influence of mechanical working. In brief compass, the invention pertains to lubricating greases containing a small stabilizing proportion of the condensation product of an alkylene oxide with an amine or amide of a fatty acid.

It is well known that in certain uses of greases, for instance in the lubrication of ball or roller bearings operating at high speed or under very high stresses, few greases retain their consistency and their structure. Indeed, the poor lubricating properties of the oils generally used cause rapid heating of the bearings, which results in a destruction of the structure of the grease; the oil then separates from the soap and runs off the bearings under the action of the centrifugal force.

It is well accepted that the lubricating properties of a grease depend mainly on the properties of the oil entering into its composition. The heating of a bearing, for instance, will be less if the oil possesses a suitable viscosity at the temperature at which it is used and a high viscosity index. It is, therefore, desirable in many cases to use lubricating greases containing oils having a high viscosity index.

However, it is well known that when using paraffinic oils in the preparation of greases with an alkali soap base, and more specifically of sodium greases, it is very difficult to avoid the separation of the soap from the oil and to obtain stable greases even with very slow cooling. In the case of rapid cooling, for example by pouring the liquefied grease at a temperature above its pour point in a thin layer on plates, a considerable degree of sweating of the oil and a separation of practically all the soap from the oil will be observed. This phenomenon is less pronounced if the mineral oils used are of naphthenic composition.

In the manufacture of alkali soap base greases, it is possible to prevent the sweating of the oil during storage or working by adding certain compounds, such as naphthenates or arylalkylsulphonates of sodium.

The present invention relates to a process for preparing greases which possess good stability at high temperatures and under the influence of mechanical stresses and which will remain perfectly stable and homogeneous even when undergoing violent cooling. These and other results are obtained in accordance with the invention by adding, particularly to alkali soap greases, certain stabilizing and dispersing agents formed mainly by condensation of amines and amides with alkylene oxides.

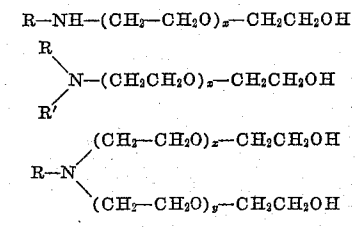
According to another characteristic, the proportion of stabilizers is in the region of 0.1–5%, preferably 0.1–2%, and highly refined oils with a high viscosity index are generally used.

Other advantages and characteristics of the invention will appear more clearly from the description hereinafter in which a number of examples for obtaining greases will

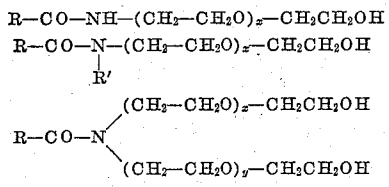
be given for purposes of illustration without limiting the invention thereto.

The stabilizers suitable for the purposes of the invention, which are the product of the condensation of amines and amides with alkylene oxides, may be illustrated by the following formulae, if for instance, ethylene oxide is taken as the basis.

(1) *Hydroxyaminopolyalkylene compounds:*



(2) *Hydroxyamide-polyalkylene compounds:*



In the above formulae, R and R′ are aliphatic or aromatic hydrocarbon radicals and x and y are whole numbers.

The amines and amides used for preparing these products can be aliphatic or aromatic, primary or secondary amines and amides, and their number of carbon atoms may range from 10 to 24. Particularly advantageous are the fatty amines and amides derived from corresponding fatty materials containing more than 10 carbon atoms, such as stearine, tallow, coconut, soya, etc.

The alkylene oxides or polyoxyalkylenes to be reacted with the amines and amides are oxides of ethylene, propylene, butylene, etc., having preferably 2–6 carbon atoms, or the corresponding polyoxides.

Thus, it will be seen from the formulae given above that the bracketed alkylene group for the given example of ethylene oxide only contains 2 carbon atoms. In the same way, 3 carbon atoms would be obtained with propylene oxide, 4 atoms with butylene oxide, etc.

Moreover it will be noted that the substances resulting from the condensation of amines with alkylene oxides always retaining their basic function can react with acids to form salts. In the case of secondary amines, the resultant products will be quaternary ammonium salts. The molecular weight of these compounds is likely to vary between about 300 and 7000. Finally, the polyalkylene groups have hydroxylated terminal groups which are capable of being esterified or etherified by conventional methods.

Furthermore, the lubricating oils used according to the invention may be of mineral or synthetic origin, having a Saybolt viscosity at 37.8° C. of from 80–2000 seconds, preferably of 90–1000 seconds and a viscosity index ranging from 0–160, but preferably above 60.

The soaps to be dispersed in an oil having lubricating properties adequate for the contemplated use are preferably alkali metal soaps, and particularly those of sodium and lithium which tend to form greases with a sufficiently high pour point. They may be present in the grease in proportions of 3–30% by weight of total grease, depending on the final characteristics it is desired to obtain. The fatty materials to be saponified are preferably saturated or unsaturated long-chain fatty acids. The fats themselves, such as triglycerides, may likewise

be used, as well as the fatty acids obtained from hydrogenated animal, marine or vegetable oils or fats or those obtained by the oxidation of hydrocarbons, these fatty acids having a number of carbon atoms ranging from 8–40.

Finally, it is advantageous, though not indispensable, to use an anti-oxidant such as phenyl alpha-naphthylamine or other well known oxidation inhibitors, generally in proportions of 0.1–2%. It is, of course, obvious that other addition products, such as tackiness agents, anti-rust agents, extreme pressure agents and others may likewise be incorporated in suitable proportions, as is well known in the art.

The greases composed in accordance with the invention may be prepared by the usual processes of mixing, heating and cooling. Thus, the fatty acid and stabilizing agents described above are added to all or part of the selected mineral oil. After heating to a temperature sufficient to obtain a homogeneous mixture, a saponifying agent is added and heating is continued until the mixture is completely dehydrated and melted. The grease is then poured in pans and, after cooling, the cakes of grease formed are worked to the desired consistency.

The stabilizing agent may also be added, if desired, at the end of cooking and before pouring of the grease.

It should also be noted that such stabilizers may likewise be suitable for improving the stability properties of greases obtained from naphthenic oils or synthetic oils of an aromatic character.

In the following examples basically the same grease manufacturing and testing methods were used as follows. The fatty acid is saponified with caustic soda, in the presence or absence of the stabilizer, at about 180° C.; cooking is continued to 225° C.; at this temperature, the molten grease is poured into pans and then worked cold to the desired consistency. The greases obtained were tested for their A. S. T. M. consistency characteristics and pour points as follows.

(1) The percentage of oil separted from the grease after remaining for 50 hours in a drying cabinet at 100° C. In this test, 100 g. grease are placed in a fine mesh, conical metal sieve placed above a calibrated container.

(2) Resistance to mechanical working by measurement of the consistency after 100,000 strokes in the mechanical "worker."

Example I

The stabilizer is the condensation product of amines obtained from soya fatty acids with 5 molecules ethylene oxide (mean molecular weight 494).

Ingredients	Percent By Weight	
	a	*b*
Stearic Acid	18	18
Pure Caustic Soda	3	3
Phenyl alpha-Naphthylamine	1	1
Mineral Oil (Saybolt Viscosity at 37.8° C.=300, Viscosity Index=9)	78	77.8
Stabilizer		0.2
Properties:		
Pour Point, ° C	180	180
A. S. T. M. Consistency—		
After 60 Strokes	300	255
After 100,000 Strokes	390	310
Oil Separated at 100° C. After 50 Hours, Percent	9	0.2

In test (*a*) during cooling in the pans, a large portion of the oil separate dfrom the grease containing no stabilizer.

Example II

The stabilizer is the same as that used for Example I, but the mineral oil has a viscosity index of 75 and is more viscous.

Ingredients	Percent By Weight	
	a	*b*
Stearic Acid	23	18
Pure Caustic Soda	4	3
Phenyl alpha-Naphthylamine	1	1
Mineral Oil (Saybolt Viscosity at 37.8° C.=550, Viscosity Index=75)	72	77.8
Stabilizer		0.2
Properties:		
Pour Point, ° C	203	190
A. S. T. M. Consistency—		
60 Strokes	345	260
100,000 Strokes	Liquid	320
Oil Separated at 100° C. After 50 Hrs., Percent	12	0.5

Example III

The stabilizer is the condensation product of amides obtained from coconut fatty acids with 50 molecules ethylene oxide (mean molecular weight 2400).

Ingredients	Percent By Weight
Stearic Acid	20
Caustic Soda	3.5
Phenyl alpha-Naphthylamine	1
Mineral Oil (Saybolt Viscosity at 37.8° C.=550, Viscosity Index=102)	75.1
Stabilizer	0.4
Properties:	
Pour Point, ° C	200
A. S. T. M. Consistency—	
60 Strokes	265
100,000 Strokes	321
Oil Separated at 100° C. After 50 Hrs., Percent	0

Example IV

The stabilizer is the condensation product of amines obtained from tallow fatty acids and 15 molecules ethylene oxide (mean molecular weight 930).

Ingredients	Percent By Weight	
	a	*b*
Stearic Acid	15	15
Caustic Soda	2.5	2.5
Phenyl alpha-Naphthylamine	1	1
Mineral Oil (Saybolt Viscosity at 37.8° C.=150, Viscosity Index=105)	81.5	81.25
Stabilizer		0.25
Properties:		
Pour Point, ° C		188
A. S. T. M. Consistency—		
60 Strokes		265
100,000 Strokes		315
Oil Separated at 100° C. After 50 Hrs, Percent		0.37

It is impossible to manufacture a grease from the ingredients of Example IV*a* using an oil of low viscosity and high viscosity index and the recommended method (i. e. pouring the compound into pans). When the grease is poured into the pans it will be found, upon cooling, that the soap and oil have separated entirely.

In Example IV*b* on the other hand, it will be found that owing to the incorporation into the grease of 0.25% stabilizer, there is obtained upon cooling a prefectly homogeneous cake of grease, which does not sweat oil and which, after working, gives a grease having all the desired properties.

It will be seen from the above examples that by using suitable stabilizing agents as defined, it is possible to make from mineral or synthetic oils of highly varying viscosity and high viscosity index, relatively soft lubricating greases characterized by a high pour point, good stability of consistency under the action of mechanical stress and practically no "sweating" at a temperature of 100° C.

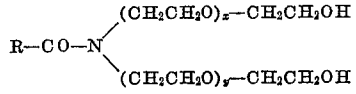
During cooking of the grease, small quantities of various soaps may be introduced, such as aluminum, calcium, barium soap, etc. or very small proportions of a neutraliz-

ing base (lime, baryta, etc.) to alter the properties of the grease in the desired direction.

It is, of course, obvious that the present invention has been described purely for the purpose of illustration and in no sense restrictively, and minor changes may be made without departing from its scope.

What is claimed is:

1. A lubricating grease comprising a major proportion of a lubricating oil having a viscosity in the range of 90–1000 S. S. U. at 37.8° C. and a viscosity index of 60–160, in the range of 3–30 wt. percent of a grease-making alkali metal soap of a high molecular weight fatty acid and in the range of 0.1–5 wt. percent of an amide having the general formula:



wherein R is an aliphatic hydrocarbon radical having from 10 to 24 carbon atoms and wherein x and y are whole numbers, the molecular weight of said amide being between 300 and 7000.

2. The grease of claim 1 in which R is the hydrocarbon radical of coconut acid, and $x+y=50$, said molecular weight being about 2400.

3. A lubricating grease comprising a major portion of a lubricating oil, a minor grease making proportion of an alkali metal soap base thickener and containing between

0.1 and 5.0 weight percent of an aliphatic substituted amide having a molecular weight between about 300 and 7,000 derived from a carboxylic acid having from 10 to 24 carbon atoms and having each of the nitrogen valencies linked to an ether alcohol having a terminal hydroxyl group.

4. The lubricating grease defined in claim 3 wherein said lubricating oil has a Saybolt viscosity between about 90 and 1,000 seconds at 37.5° C. and a viscosity index between about 60 and 160.

5. The lubricating grease as defined in claim 3 wherein said substituted amide is a product formed by condensing the amide with an alkylene oxide having from 2 to 6 carbon atoms in the molecule.

6. The product defined in claim 5 wherein the alkylene oxide is ethylene oxide.

7. The product defined in claim 6 wherein the amide is condensed with from 5 to 50 molecules of ethylene oxide.

References Cited in the file of this patent

UNITED STATES PATENTS

1,970,578	Schoeller et al.	Aug. 21, 1934
2,002,613	Orthner	May 28, 1935
2,085,706	Schoeller et al.	June 29, 1937
2,445,935	Bondi	July 27, 1948
2,475,589	Bondi	July 12, 1949
2,545,190	Bondi	Mar. 13, 1951