LUBRICATING OILS THICKENED WITH METAL SALTS OF CYANURIC ACID

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This invention relates to improved grease compositions containing salts of cyanuric acid and more particularly is concerned with greases made from natural and synthetic oils or oleanugous materials to which have been added salts of cyanuric acid as thickening agents.

Greases are normally semi-solid to semi-solid lubricants and are generally grouped into three classes: (1) ad-mixtures of oils and solid lubricants such as graphite, mica, talc, etc.; (2) blends of residuum, waxes, uncombined fats, rosin oils, etc.; and (3) soap-thickened oils normally comprising oils and added thickening agents, such as the sodium, calcium, aluminum or lead soaps of fatty acids. This latter class of soap-thickened oils is commercially very important and very widely used industrially inasmuch as a large variety of greases of different consistencies, properties and characteristics can be produced by selecting a particular metal, a particular mixture of fatty or similar acids and a particular oil stock.

It has been found that salts of cyanuric acid are excellent thickening agents and, when added to oils, will thicken the same to greases capable of use not only as general-purpose lubricants for bearings, journals, line-shafting and the like but also applicable under a wide range of varying temperature conditions and of particular use at elevated temperatures. These salts further find utility as thickening agents in general for materials such as alcohols and other inflammables in the manufacture of "Sterno" or like products.

Illustrative of the salts of cyanuric acid which have been found applicable as grease thickening agents are the alkaline metal salts notably sodium and potassium, the alkaline earth metal salts notably calcium and magnesium, the heavy metal salts notably zinc and aluminum, the ammonium salts, and the amine salts such as ethylamine, diethylamine, triethylamine, propylamine, ethanolamine, amine, aniline, morpholine, pyridine, cyclohexylamine, etc. It is also to be appreciated that the term "salts" includes the mono-, di- and tri-salts of cyanuric acid, as well as mixtures thereof, resulting from the reaction of the cyanuric acid and the particular base salt-forming material used and dependent upon the molar proportions employed.

The materials to which these salts of cyanuric acid are added have been referred to as "oils" but it is to be appreciated that this term is not limited to the narrow interpretation thereof but is intended to cover all oily or oleaginous materials capable of use as lubricants or as greases when compounded with the salts of cyanuric acid.

These oleaginous materials would consequently include: (1) the petroleum hydrocarbon mineral lubricating oils; (2) the synthetic hydrocarbon lubricants including the various ethylene, propylene and butylene (and sometimes higher members of the series) having molecular weights ranging from about 250 to about 50,000 or more; (3) the polyglycol synthetic lubricants such as polyethylene glycol (polyethylene oxide), polypropylene glycol (polypropylene oxide), their esters and esters as well as the higher polyalkylene oxides, polyglycidyl ethers, polyglycolic acids, etc.; (4) the ester synthetic lubricants such as diesters of glycol, diesters of aliphatic and aromatic dibasic acids notably bis(2-ethylhexyl)sebacate and bis(2-ethylhexyl phthalate), triesters of phosphoric acid, etc.; (5) the silicone oils (sioxanes) having viscosities of from about 40 to about 1000 centistokes at 100° F., such as methyl silicones, methyl phenyl silicones, phenyl silicones, etc.; and (6) the fluorine-derivative synthetic lubricants (fluorocarbons) such as tertiary amines, ethers and esters, the alkyl or aryl groups of which have been fluorinated.

The amounts or concentrations of the salts of cyanuric acid to be included in the oleaginous materials for thickening purposes will naturally vary as to the specific nature of the salt and the oil themselves, the nature and intended purpose and use of the resulting grease, the properties and characteristics desired, etc. It has been found that from about 3 percent to about 20 percent by weight is an effective range, with from about 7 percent to about 12 percent by weight being the commercially preferred range.

The salts may be compounded with the oils by a simple addition thereto with stirring at room or elevated temperature. If a smoother grease is desired, milling of the salt may be resorted to, prior to its incorporation into the oil. The resulting greases range from semi-soft and buttery in texture to smooth, thick, viscous, firm and semi-solid.

The invention will be further illustrated in more specific detail by the following tests and related examples. It should be understood, however, that although these tests and examples may describe in more particular detail some of the very specific features of the invention, they are given primarily for purposes of illustration and the invention in its broader aspects is not to be construed as limited thereto.

EXAMPLE 1

Slurry 1290 grams of cyanuric acid in 6 liters of water and add to this slurry a solution of 800 grams of sodium hydroxide in 6.5 liters of water. Heat this mixture to about 80° C. and then cool to room temperature and filter. Heat the filtrate to about 80° C. and then, with constant stirring, add a solution of 7.5 liters of methanol and 22.5 liters of n-propanol and filter through a Buchner funnel. Slurry the filter cake in about 25 liters of n-propanol and distill off liquid (azeotrope) until the volume of the liquid in the distillation flask is reduced to about 12.5 liters. (Final temperature about 90° C.) Filter this slurry remaining in the distillation flask through a Buchner funnel and then dry the product at 80-90° C. for 4-5 hours in an oven which provides for safe removal of the evaporated liquid.

Evaluation No. 1

The thickening properties of the sodium cyanurate prepared in Example 1 were evaluated in a silicone fluid as follows: 10 grams of sodium cyanurate was dispersed, with stirring, in 100 cc. of SF-96 (200) silicone fluid having a viscosity of 200 centistokes or 912 S. S. U. at 100° F. This fluid originally had a density of 8 lbs. per gallon, a pour point of about —60° F. and a flash point of about 600° F.

The addition of the sodium cyanurate thickened the silicone oil to a semi-solid grease-like consistency which was then tested according to ASTM specifications, D217-52T, as described in part 5, ASTM standards, 1952, pages 119-128. The results of the penetrometer tests were as follows:

Penetration (unworked) ———— 320 mm./10
Penetration (worked 60 strokes) ———— 333 mm./10
Penetration (worked 120 strokes) ———— 339 mm./10
The penetration which was obtained after heating with stirring to a temperature of about 220° C. (428° F.) with the penetration measurements taken also at about 220° C. (428° F.), was 345 mm./10.

The resulting grease was suitable for general-purpose use in bearings, journals, line-shafting and the like and was especially adapted for high temperature applications.

**Evaluation No. 2**

A grease was prepared by incorporating 6 grams of sodium cyranurate (Example 1) in SF-96 (1000) silicone fluid having a viscosity of 1000 centistokes or 4620 S. S. U. at 100° F. The density of this fluid originally was 8 lbs. per gallon, its pour point was below -57° F. and its flash point was above 600° F. The addition of the sodium cyranurate thickened the silicone fluid into a semi-solid grease which could be inverted when in an open container without pouring or flowing. This consistency of the composition was sufficiently thick and viscous as to render it suitable for grease purposes.

**Evaluation No. 3**

A grease was prepared by incorporating 3 grams of sodium cyranurate (Example 1) in SF-96 (1000) silicone fluid having a viscosity of 1000 centistokes or 4620 S. S. U. at 100° F. The density of this fluid was 8 lbs. per gallon, its pour point was below -57° F. and its flash point was above 600° F. The addition of the sodium cyranurate thickened the silicone fluid into a firm grease which was sufficiently thick and viscous as to render it suitable for grease purposes.

**Evaluation No. 4**

20 grams of sodium cyranurate (Example 1) was added, with stirring, to 100 cc. of an ester synthetic lubricant consisting of diocyle sebacate. The diocyle sebacate was very thin originally and had almost a watery consistency. The addition of the sodium cyranurate thickened the diocyle sebacate so that it had the consistency of a light cold cream and was suitable for use as a grease.

**Evaluation No. 5**

18 grams of sodium cyranurate (Example 1) was added, with stirring, to 100 cc. of a SAE 30 (Sun Oil) petroleum hydrocarbon mineral lubricating oil. The addition of the sodium cyranurate thickened the oil to a consistency of a heavy cold cream and rendered it suitable for use as a grease.

**Evaluation No. 6**

16 grams of sodium cyranurate was added to a polyglycol synthetic lubricant (polyethylene oxide, UCC Ucon Lubricant LB-300X, water insoluble) and thickened the same so that it was semi-solid and would not flow. Its consistency was sufficiently heavy and viscous as to render it useful as a grease.

**EXAMPLE 2**

Lithium cyranurate was prepared by dispersing 28.4 grams of 91% cyranic acid in 150 ml. of water, adding 16.8 grams of LiOH·2H₂O in 100 ml. of water in a steady stream with agitation, and heating. The mixture was heated to 100° C., allowed to cool to room temperature and vacuum filtered. The filtrate was again heated to 100° C. under a reflux condenser, the heat was turned off, and 500 ml. of ethanol preheated to 75° C. was added slowly. The resulting precipitate was vacuum filtered and the filter cake slurried in 600 ml. of ethanol and again filtered. The lithium cyranurate filter cake was again slurried in 600 ml. of ethanol and dehydrated by distilling with agitation to a high temperature of 78° C. It was vacuum filtered, air-dried, and further dried in a hot-air oven at 75° F. for 12 hours.

A grease having a penetration range of 325-340 mm./10 was prepared by mixing 10 parts by weight of the dried lithium cyranurate with 90 parts of the SF-96 (200) silicone oil described in Evaluation No. 1.

**EXAMPLE 3**

A solution of 35.3 grams of BaCl₂·2H₂O in 100 ml. of water was added during 25 minutes and at about 30° C. to 25 grams of sodium cyranurate dissolved in 500 ml. of water. The resulting slurry was vacuum filtered and washed with water and then with two 100 ml. portions of ethanol. The resulting barium cyranurate was air-dried and then heated for 12 hours at 75° T. in a hot-air oven.

Upon admixture of 10 grams of the dried barium cyranurate with 40 grams of SF-96 (200) silicone oil a grease having a penetration range of 320-330 mm./10 was obtained.

**EXAMPLE 4**

Calcium cyranurate was prepared by adding 18 grams of 98% CaCl₂ dissolved in 100 ml. of water, to a solution of 25 grams of sodium cyranurate in 500 ml. of water and recovering and drying the precipitate as described in Example 3. When 10 grams of the product were mixed with 40 grams of SF-96 (200) silicone oil a grease having a penetration range of 317-332 mm./10 was obtained.

**EXAMPLE 5**

A solution of 20.8 grams of 95% zinc chloride in 100 ml. of water was prepared and added to a solution of 25 grams of sodium cyranurate in 500 ml. of water. The resulting precipitate of zinc cyranurate was recovered and dried by the procedure described in Example 3. When a 10 gram sample of the zinc cyranurate was mixed uniformly with 40 grams of SF-96 (200) silicone oil a thickened grease was formed which had a penetration range of 330-345 mm./10.

Although several specific examples of the inventive concept have been described, the same should not be construed as limited thereby nor to the specific substances mentioned therein but to include various other compounds of equivalent constitution as set forth in the claims appended hereto. It is understood that any suitable changes, modifications and variations may be made without departing from the spirit and scope of the invention.

This is a continuation-in-part of our copending application Serial No. 359,107 filed January 16, 1956, now abandoned.

What we claim is:

1. A grease composition comprising a major proportion of an oil and, as a thickening agent therein, a minor quantity within the range of from about 3 to about 20 percent by weight of a salt of cyranic acid.

2. A grease composition as defined in claim 1 wherein the oil is a petroleum hydrocarbon mineral oil.

3. A grease composition as defined in claim 1 wherein the oil is a polyglycol synthetic lubricant.

4. A grease composition as defined in claim 1 wherein the oil is an ester synthetic lubricant.

5. A grease composition as defined in claim 1 wherein the oil is a silicone oil.

6. A grease composition as defined in claim 1 wherein the salt of cyranic acid is an alkali metal salt.

7. A grease composition as defined in claim 1 wherein the salt of cyanuric acid is a sodium salt.

8. A grease composition as defined in claim 1 wherein the salt of cyanuric acid is a zinc salt.

9. A grease composition as defined in claim 1 wherein the salt of cyranic acid is an alkali earth metal salt.

10. A grease composition comprising a major proportion of an oil and, as a thickening agent therein, a minor quantity within the range of from about 7 to about 12 percent by weight of a salt of cyranic acid.

No references cited.