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(54) **Phosphazene containing lubricating grease compositions.**

(57) A lubricating grease composition is provided which comprises a lubricating fluid, an alkali metal soap and a cyclic phosphazene.

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PHOSPHAZENE CONTAINING LUBRICATING GREASE COMPOSITIONS

Description

The present invention relates to lubricating grease compositions, and to alkali metal soap lubricating grease compositions in particular.

Lubricating greases have been defined by the American Society for Testing and Materials (ASTM) to be solid to semi-fluid lubricants which include a thickening agent dispersed in a liquid lubricant. Although this definition is of relatively recent origin, samples of fat and lime taken from the axle of Pharaoh Tutankamen's chariot from about 1400 BC indicate that lubricating greases have been known since antiquity.

Over the centuries many different lubricating grease compositions have been used. It appears the earliest known greases were made using vegetable oils, such as linseed oil, castor oil or cottonseed oil. For example, in about 200 AD the physician Galen reported the coagulation of linseed oil by lead oxides. The utility of animal oils and fish oils in greases were also discovered. After the first oil well came into production in Titusville in 1859, this new lubricant and its components, such as naphthenic and paraffinic oils, was adopted for general use in lubricant compositions. Although in recent years greases employing synthetic materials such as silicone oils, alkylbenzenes, phosphate esters, fluorinated oils, polyglycols and diesters have also gained acceptance, these lubricants were developed largely following World War II.

The thickening agents used in greases may be those, such as soaps, which have been known for many years, or may be more recently developed materials, such as polyureas. These thickening agents are generally classified as soap or non-soap thickening agents, and the greases resulting from their combination with a lubricating fluid are usually classified accordingly.

Soap thickening agents generally are metal salts of relatively large organic acids such as myristate, linoleate, linolenate, laurate, stearate, oleate, palmitate, benzoate, and azelate, although some salts of relatively small organic acids, such as acetates or propionates, may be included. Mixtures of different acids may also be used. For example, many soaps are derived from tallow, which contains a mixture of aliphatic molecules such as stearin, palmitin and olein. The number of carbon atoms in the organic portion of the soap and the degree of its saturation affect the thickening properties of the soap, with soaps which include organic moieties having 12 to about 18 carbon atoms usually having thickening properties appropriate for most grease applications.

Soap thickeners may also be complex soaps. Complex soaps may be defined as soaps wherein the soap crystal or fiber is formed by the co-crystallization of two compounds, the normal soap and a complexing agent, such as wherein a single metal ion is complexed with two or more dissimilar organic moieties. For example, a long chain moiety, such as stearic acid, and a relatively short chain moiety, such as acetic acid, may be complexed with the same metal ion, such as in calcium stearate acetate. In the alternative, a di- or tri- basic moiety, such as azelaic acid, may be complexed with a soap, such as lithium 12-hydroxy stearate, or with more than one metal ion, such as in dilithium azelate. Mixtures of different soaps may also be used in the same lubricating grease composition.

A variety of different metal ions may be used in making soap thickening agents, such as aluminum, cadmium, cobalt, stontium, nickel, mercury, molybdenum, lead, barium, calcium, sodium and lithium ions. The properties of these metal ions also affect the properties of the thickening agent and the lubricating greases in which they are used, with these properties in turn determining the applications for which a particular grease is appropriate. For example, sodium greases may be used under conditions of high temperature and isolation from water, such as in gear cases. Lithium greases are useful over a wide range of temperatures, are water resistant, and are frequently used as general purpose automotive greases, such as to lubricate ball joints, universal joints and wheel bearings. Aluminum complex soap greases may be used in situations, such as oven door hinges, roller bearings and hoists in steel mills, where resistance to oxidation and to oil separation at high temperatures and pressures is important.

Non-soap thickening agents are generally of two types. These types are (1) inorganic gelling agents, and (2) organic thickeners. Inorganic gelling agents are usually substances such as carbon black and modified, oleophilic clays such as Bentonite, with clay type greases usually being non-melting and having good heat resistance. Organic thickeners are organic compounds, such as ureas or polyureas, Teflon, polyethylene and terephthalamic acid, and may result in a grease having a high melting point and an extended grease life at elevated temperatures.

In many mechanisms wherein a lubricant is required, the load on adjoining parts is supported entirely by a lubricant film between the part surfaces. However, failure of the lubricant may permit metal/metal contact through the lubricant film. This contact frequently results in spalling and microscopic tearing of the metal, thereby resulting in premature part failure. This problem is particularly acute in automotive applications, such a differentials, universal joints, constant velocity joints, roller bearings in wheel bearing applications, clutches and fan drive bearings, as these situations may present severe sliding conditions in combination with high contact stress. Corrosion of the metal in the presence of the grease may also play a role in part failure.

Lubricating grease compositions typically contain one or more additives to retard part corrosion and lubricant degradation in order to prolong the mechanism's useful life. Typical grease additives include antioxidants, extreme pressure agents, antiwear agents, and corrosion inhibitors. Examples of such additives include zinc oxide, 2,6-di-t-butyl phenol, alkyl succinic acids, dinonyl naphthane sulfonates, tricresyl phosphate, and molybdenum disulfide.

Among the substances known as additives and lubricant components are substituted cyclic phosphazenes. U.S. Patent No. 3,280,222 and No. 3,291,865 to Kober, et al. respectively disclose oxyalkylated aminophenoxy sustituted cyclic phosphazenes, and cyclic phosphazenes having phenoxy type substituents. These substituted phosphazenes are disclosed as being useful as hydraulic fluids, lubricants and additives.

U.S. Patent No. 3,234,304 and No. 3,316,330 to Nichols disclose cyclic phosphazenes having phenoxy and fluoroalkyl phenoxy substituents as being useful as working fluids, lubricants, chemical intermediates and plasticizers. U.S. Patent No. 3,136,727, also to Nichols, discloses a penta-phenoxy-mono (phenoxy phenoxy) substituted cyclic phosphazene. This phosphazene is also alleged to be useful as a working fluid, lubricant, chemical intermediate and plasticizer.

U.S. Patent No. 2,109,491; No. 2,192,921; and No. 2,214,769 to Lipkin also disclose various substituted phosphazenes as additives to lubricant compositions. These phosphazenes may be substituted by a variety of organic compounds, such as alcohols, phenols, mercaptans and amines. Lipkin discloses these phosphazenes may be added to lubricants such as mineral oils containing fatty oils, fatty acids, metallic soaps, sulfur or combined chlorine.

Although various additives have been found to enhance the antiwear properties of certain lubricant compositions, these additives may interact with other compounds present in the lubricant or may become oxidized under the conditions of use. Further, an additive which functions well in one type of grease may be ineffective or even detrimental in another type of grease composition. As alkali metal soap greases - and lithium soap and lithium complex soap greases in particular - are used in many applications such as in motorized vehicles where inadequate lubricant performance may have serious consequences, an alkali metal soap grease composition having improved antiwear properties may offer significant practical advantages.

The present invention is a lubricating grease composition which comprises a lubricating fluid, an alkali metal soap, and a cyclic phosphazene. Alkali metal soaps of sodium and lithium are preferred, with lithium and lithium complex soaps being most preferred. It is preferred that the cyclic phosphazene be one described by the general formula:

$$(P=N)_nR_{2n}$$

wherein R is selected from a group consisting of substituted and unsubstituted phenoxy moieties, substituted and unsubstituted alkoxy moieites, the corresponding thio moieties, amino moieties and mixtures thereof, and n is an integer equal to 3 to about 4. Phosphazenes wherein R is selected from the group consisting of phenoxy, alkyl substituted phenoxy, aryl substituted phenoxy, and C_{11}-C_{30}

alkyl moieties and mixtures thereof are particularly preferred.

The present invention is a lubricating grease composition which comprises a lubricating fluid, an alkali metal soap, and a cyclic phosphazene.

The lubricating fluid may be a vegetable, animal, synthetic or mineral oil, or a mixture thereof, such as linseed oil, cottonseed oil, silicone oil, alkyl benzenes, phosphate esters, fluorinated oils, polyglycols and di-esters. However, mineral oils such as naphthenic, paraffinic, aromatic, and naphthenic/paraffinic mixed base oils, and synthetic materials such as di-isooctyl azelate, di-2-ethylhexyl sebacate, di-2-ethylhexyl azelate, dibasic polyesters, neopentyl polyol esters, polyalphaolefins and alkyl benzenes are preferred. Mineral oils, and especially naphthenic, paraffinic and naphthenic/paraffinic mixed base oils are particularly preferred.

The cyclic phosphazene used in the composition of the present invention may be described by the general formula:

$$(P{=}N)_n\ R_{2n}$$

According to the invention, n may be equal to 3 to about 4, so that the phosphazene is a cyclic trimeric, tetrameric or pentameric phosphazene. Trimeric and tetrameric phosphazenes are preferred, with trimeric phospha-zenes being most preferred. Mixtures of different phosphazenes may also be used. For example, many commercially available phosphazene compositions contain a mixture of trimeric and tetrameric phosphazenes.

Consistent with the invention, R is selected from the group consisting of substituted and unsubstituted alkyl moieties such as ethyl, methyl, propyl, butyl, oleyl, dodecyl, nonyl, and 2-chloroethyl, substituted and unsubstituted alkoxy moieties such as methoxy, propoxy, ethoxy, and tri-fluoro ethoxy, substituted and unsubstituted phenoxy moieites such as phenoxy, m-methylphenoxy, p-t-butylphenoxy, m,p-dimethoxyphenoxy, m-methoxyphenoxy, 2,4,6-trichlorophenoxy, m-ethoxyphenoxy, m,p-di(trifluoromethyl)phenoxy, m-chlorophenoxy, 2,4,6-trimethylphenoxy, (methylphenyl)phenoxy, phenylcarbinoxy, naphthoxy, thionaphthoxy, and amino such as ethylamino, propylamino, benzylamino, and amylamino. In addition, R may be selected from the mercaptan equivalents of phenoxy and alkoxy moieties. Preferably, the alkyl moieties are those having about 6 to about 30 carbon atoms, such as nonyl and dodecyl, and the alkylphenoxy and alkylphenylthio moieties are those having about 7 to about 30 carbon atoms such as p-dodecylphenoxy. R may also be selected from mixtures of alkyl, phenoxy, alkoxy, phenylthio, alkylthio and amino moieties, so that the phosphazene may be substituted by two, three, four or more different moieties.

It is further preferred that R be selected from the group consistently of phenoxy, alkyl-substituted phenoxy, aryl-substituted phenoxy and C_{11}-C_{30} alkyl moieties and mixtures thereof, such as nonyl, dodecyl, phenoxy, p-methylphenoxy, m-methylphenoxy, p-ethylphenoxy, m,p-dimethylphenoxy, phenylphenoxy, and phenoxyphenoxy. Phenoxy, alkyl-substituted phenoxy moieties and mixtures thereof are most preferred.

Each phosphorus atom in the phosphazene preferably is substituted by two organic substituents, so that the cyclic phosphazene is completely substituted with one or more organic moieties. The lubricating grease may contain small amounts of phosphazene which are incompletely substituted. However, as organic substituted phosphazenes are usually made from phosphazenes which are substituted by chlorine, chlorine is usually present in those phosphazenes wherein organic substitution has been incomplete. As residual chlorine appears to contribute to phosphazene hydrolysis, these incompletely substituted phosphazenes preferably are present, if at all, in relatively small amounts such as less than 10% by weight and more preferably less than 5% by weight of the total phosphazene content of the lubricating grease.

Cyclic phosphazenes suitable for use in the lubricating grease of the present invention are available commercially or may be prepared by processes known in the art. Examples of such processes may be found in those references cited above. In the alternative, suitable phosphazenes may be prepared by the phase transfer process described in U.S. Patent No. 4,600,791, issued July 15, 1986.

The amount of cyclic phosphazene present in the lubricating grease composition of the present invention will depend, among other possible factors, on the amount of antiwear enhancement desired. In most compositions the cyclic phosphazene will be present in an amount equal to about 0.1% to about 20% by weight of the lubricant composition. More preferably, however, the cyclic phosphazene will be present in an amount equal to about 1% to about 10% by weight. Amounts of about 2% to about 6% by weight are usually most preferred.

The lubricant composition of the present invention also includes an alkali metal soap thickener. The organic portion of the alkali metal soap usually includes a relatively large organic acid group, such as lignoceryl, valeryl, lauryl, myristyl, palmityl, stearyl, oleyl, linoleyl, azelyl, linolenyl, benzoyl, o-, m- or p-toluyl and palmitoleyl. The grease usually includes a soap with one or more fatty moieties having at least 10 carbon atoms, as such soaps usually have reduced solubility in water in comparison to soaps of shorter chain moieties. Fatty moieties having about 12 to about 20 carbon atoms, such as lauryl, palmityl, stearyl, oleyl or linolenyl are most preferred. Stearyl and 12-hydroxy stearyl are most preferred. Mixtures of different organic moieties may also be used. For example, when the soap is derived from tallow the organic moieties may include stearyl, palmityl and oleyl. Relatively small organic moieties, such as acetyl and propionyl, may also be included.

It is critical to the invention that the soap include an alkali metal. Sodium and lithium are preferred, with lithium being most preferred. Other soaps or non-soap thickeners may be included, such as calcium stearate included with sodium stearate. However, it is preferred that non-alkali metal soaps and non-soap thickeners, when present, constitute less than 50% by weight of the total thickener content of the grease, and more preferably less than 25% by weight of the total thickener content.

It is further preferred that the alkali metal soap be a complex soap, wherein the soap component includes a complexing agent in addition to the groups described above. For alkali metal soaps this complexing agent usually is a di or tri-basic moiety, such as one or more of azelaic acid (1,2-heptanedicarboxylic acid), sebacic acid, 1,5-pentanedicarboxylic acid, 1,9-nonanedicarboxylic acid, and lithium borates such as lithium metaborate and lithium tetraborate. Complex lithium 12-hydroxy stearate soap greases such as Unirex N-2 grease, available from the Exxon company, are most preferred.

The amount of alkali metal soap present in the grease composition will depend on a variety of factors, such as the identity of the lubricating fluid, whether the grease should be more or less viscous for the intended application, and whether other thickeners such as calcium stearate or non-soap thickeners are present. For most greases, however, it is preferred that the alkali metal soap be present in an amount equal to about 2% to about 30% by weight of the lubricating grease composition. Amounts of alkali metal soap of about 5 to about 20% by weight are usually more preferred.

Additionally, the lubricating grease composition may contain other components to enhance desirable grease properties. Examples of such additives include anti-oxidants and corrosion inhibitors, such as phenothiazine, p-nonylphenoxy acetic acids, 2,6-di-t-butyl phenol, phenyl-alpha-naphthyl amine, dinonyl naphthane sulfonates, and alkyl succinic acids, as well as other additives known in the art.

The components of the lubricating grease composition of the present invention may be combined and compounded into a grease by means known in the art, such as by mixing the alkali metal soap with the lubricating fluid and heating the mixture moderately to dissolve the soap in the lubricating fluid. The phosphazene may then be incorporated by simple mixing techniques.

Several grease compositions were made and tested to provide a means for comparing embodiments of the lubricating grease of the present invention with greases not embodying the invention. All greases were prepared using a base oil blend of 45% wt Citgo 150 Neutral Oil and 55% wt Citgo 150 Brightstock.

Greases were prepared by placing a fatty acid, a metal salt and base oil in a container and heating the mixture to 100-170°C with stirring to dissolve the fatty acid and metal salt in the oil. After a period of time - usually about 2 hours - the mixture was cooled to about 100°C and additional base oil blended in to yield a grease. Additional components, when desired, were then added by simple mixing techniques.

These greases were tested for coefficient of static friction, coefficient of dynamic friction, and volume loss of the test block due to block wear from testing performed using a Falex I Oscillating Grease Tester at room temperature, 87.5 rpm, 5000 cycles at 90° oscillation. The block wear loss is indicated in the tables below in $mm^3 \times 10^{-3}$.

Examples 1 - 4

Alkali Metal Soaps

Lubricating grease compositions which include an alkali metal soap were tested as discussed above. Some of these greases also contained a trimeric phosphazene completely substituted by phenoxy and ethylphenoxy moieties. This phosphazene was made by esterifying trimeric dichloro-phosphazene with phenol and ethylphenol in a medium comprising a mixture of water, a base, a water immiscible solvent and a phase-transfer catalyst, with the ratio of phenol to ethylphenol in the medium being approximate 1:1. This type of reaction process is explained in U. S. Patent No. 4,600,791, issued July 15, 1986.

For Example 1(a), more than one sample was tested. The amount of alkali metal soap, the amount of phosphazene, as well as the results of testing of these compositions are indicated below in Table I.

Examples 5 - 7

Non-Alkali Metal Soap

Lubricating greases thickened by non-alkali metal soaps were tested as discussed above. Phosphazene - containing greases included the same phosphazene as Examples 1-4. Calcium complex greases were those thickened by calcium

12-hydroxy stearate/acetate complex soaps. The results of this testing are indicated below in Table II.

Examples 8 - 13

A grease thickened by 12% lithium 12-hydroxy stearate in base oil with 5% of an additive (except for Example 8 which did not contain an additive) was tested as discussed above. The greases of Examples 9 and 10, wherein the grease included a phosphazene, were consistent with the present invention. The greases of Examples 8, 11 - 13 did not embody the invention. The results of this testing are indicated below in Table III.

The consistency of the greases used in Examples 2(a), 2(c), 4(a), 4(c), 6(a) and 6(c) was also tested. The worked and unworked penetration were tested according to ASTM D 1403. The dropping point of these greases was tested according to ASTM D 2265. The results of this testing are indicated below in Table IV, with the higher numbers for worked and unworked pentration indicating softer greases. The dropping point of the greases for Examples 4(a), 4(c), 6(a) and 6(c) could not be measured because these greases flowed at room temperature. The greases for Example 7 where of even poorer quality and resembled thick oils rather than greases. This may be a disadvantage, as a fluid-like consistency may make a grease undesireable for many applications as the grease may be too fluid to maintain its position where it is needed in an apparatus, thereby defeating the purpose for which the grease is used.

TABLE I

FALEX I OSCILLATING GREASE TESTING

EX.	Grease	Static	Dynamic	Block Wear
1.	7.5% Li 12-hydroxy Stearate			
(a)	0% phosphazene	0.11/0.14	0.09/0.09	4.1/5.1
(b)	2.5% "	0.22	0.11	3.7
(c)	5.0% "	0.16	0.11	8.8
2.	12.0% Li 12-hydroxy Stearate			
(a)	0% phosphazene	0.14	0.09	8.4
(b)	2.5% "	0.15	0.10	2.2
(c)	5.0% "	0.11	0.10	1.2
3.	20.0% Li 12-hydroxy Stearate			
(a)	0% phosphazene	0.16	0.10	4.3
(b)	2.5% "	0.17	0.08	4.0
(c)	5.0% "	0.16	0.09	3.0
4.	7.5% Na 12-hydroxy Stearate			
(a)	0% phosphazene	0.13	0.10	10.1
(b)	2.5% "	0.17	0.11	5.1
(c)	5.0% "	0.16	0.12	7.6

TABLE II

FALEX I OSCILLATING GREASE TESTING

EX.	Grease	Static	Dynamic	Block Wear
5.	15.0% Ca Complex			
(a)	0% phosphazene	0.11	0.10	2.0
(b)	2.5% "	0.24	0.10	4.1
(c)	5.0% "	0.12	0.11	3.2
6.	23.0% Ca Complex			
(a)	0% phosphazene	0.12	0.10	0.9
(b)	2.5% "	0.18	0.11	2.2
(c)	5.0% "	0.18	0.12	3.1
7.	30.0% Ca Stearate			
(a)	0% phosphazene	0.12/0.14	0.10/0.10	18.6/9.3
(b)	2.5% "	0.18	0.11	28.9
(c)	5.0% "	0.18	0.10	11.8

TABLE III

FALEX I OSCILLATING GREASE TESTING

EX.	Additive	Static	Dynamic	Block Wear
8	---	0.14	0.09	8.4
9	Hexaphenoxy phosphazene	0.16	0.11	2.9
10	Ex. 1-7 phosphazene	0.12	0.10	1.2
11	Dithiodiphosphoamidate	0.12	0.10	2.6
12	Trixylyl phenyl phosphate	0.13	0.10	5.0
13	Dixylyl phenyl phosphate	0.13	0.10	4.3

TABLE IV

GREASE CONSISTENCY

EX.	Grease	Unworked	Worked	Dropping Point (°F)
	12% Li 12-hydroxy stearate			
2(a)	0% phosphazene	312	314	397
(c)	5% phosphazene	318	320	401
	7.5% Na 12-hydroxy stearate			
4(a)	0% phosphazene	> 400	> 400	none
(c)	5% phosphazene	> 400	> 400	none
	23% Ca Complex			
6(a)	0% phosphazene	> 400	> 400	none
(c)	5% phosphazene	> 400	> 400	none

CLAIMS

1. A lubricating grease composition comprising a lubricating fluid, an alkali metal soap, and a cyclic phosphazene.

2. The lubricating grease of claim 1 wherein the alkali metal soap includes an alkali metal selected from the group consisting of sodium, lithium, and mixtures thereof.

3. The lubricating grease of claim 2 wherein the alkali metal is lithium.

4. The lubricating grease of claim 3 wherein said grease includes a lithium soap and a complexing agent.

5. The lubricating grease of claim 4 wherein said complexing agent is a di- or tri-basic acid.

6. The lubricating grease of claim 4 wherein said complexing agent is a lithium borate.

7. The lubricating grease of claim 5 wherein said complexing agent is an azelaic acid.

8. The lubricating grease of claim 1 wherein said lubricating fluid is a mineral oil.

9. The lubricating grease of claim 2 wherein said cyclic phosphazene is described by the general formula:

$$(P=N)_nR_{2n}$$

wherein: R is selected from the group consisting of substituted and unsubstituted alkyl moieties, substituted and unsubstituted alkoxy moieties, substituted and unsubstituted alkylthio moieties, substituted and unsubstituted phenoxy moieties, substituted and unsubstituted phenylthio moieties, and amino moieties and mixtures thereof; and

n is an integer equal to 3 to about 4.

10. The lubricating grease of claim 9 wherein R is selected from the group consisting of phenoxy, alkyl-substituted phenoxy, aryl-substituted phenoxy and C_{11}-C_{30} alkyl moieties and mixtures thereof.

11. The lubricating grease of claim 10 wherein R is selected from the group consisting of phenoxy and alkyl-substituted phenoxy moieties and mixtures thereof.

12. The lubricating grease of claim 9 wherein n is equal to 3.

13. The lubricating grease of claim 1 wherein the alkali metal soap is an alkali metal stearate.

14. The lubricating grease of claim 13 wherein the alkali metal soap is an alkali metal 12-hydroxy stearate.

15. The lubricating grease of claim 1 wherein said alkali metal soap is present in an amount equal to about 2% to about 30% by weight of said lubricating grease.

16. The lubricating grease of claim 15 wherein said alkali metal soap is present in an amount equal to about 5 to about 20% weight of said lubricating grease.

17. The lubricating grease of claim 1 wherein said cyclic phosphazene is present in an amount equal to about 0.1% to about 20% by weight of said lubricating grease.

18. The lubricating grease of claim 17 wherein said cyclic phosphazene is present in an amount equal to about 1% to about 10% by weight of said lubricating grease.

19. A lubricating grease comprising a mineral oil, a lithium complex soap and a cyclic phosphazene described by the general formula:

$$(P{=}N)_nR_{2n}$$

wherein R is selected from the group consisting of substituted and unsubstituted phenoxy moieties and mixtures thereof;

n is an integer equal to 3 to about 4; and

said lithium soap being present in an amount equal to about 5% to about 20% by weight and said phosphazene being present in an amount equal to about 1% to about 10% by weight of said lubricating grease.