This invention pertains to a grease composition prepared from a polysiloxane (e.g., methyl phenyl polysiloxane) and a complex basic aluminum soap; that is, this invention pertains to the use of a complex basic aluminum soap as a grease thickening agent for a polysiloxane base oil.

Not too long ago, the lubrication art was confronted with the problem of preparing grease compositions which could be used at both low and high temperatures. It was the inherent desire of the petroleum industry to prepare grease compositions with petroleum base oils, the petroleum oils being as highly refined as possible to increase the viscosity index to its maximum. However, even with the most highly refined petroleum base oils, it has been difficult to prepare greases which give adequate lubrication at extremely low and high temperatures.

Greases which are plastic at high temperatures as well as low temperatures are useful in such severe variations of weather conditions as are encountered in airplane operations, in the operation of military equipment in extreme cold weather and hot weather, etc. It is extremely advantageous to be able to use one grease composition which is plastic at low and high temperatures, and is further characterized by a high resistance to emulsification in water.

Numerous synthetic oils have been placed upon the market in recent years to satisfy certain requirements in the lubrication field. For example, a polysiloxane oil is normally useful under extremely warm or cold conditions. The thickening agents normally used to thicken petroleum hydrocarbon oils to form greases are dispersible in polysiloxane oils only with great difficulty. A most successful method found to disperse the normally used soaps in polysiloxanes to form greases involves the dispersal of the soap in a volatile hydrocarbon oil, then slowly working this soap dispersion into a polysiloxane, after which the volatile hydrocarbon is removed by distillation. This method, which is normally used in dispersing lithium soaps in polysiloxane oils, is cumbersome and unnecessarily costly. Furthermore, the oxidation resistance of the lithium soap thickened polysiloxane grease is not as great as that which could be desired.

It is an object of this invention to prepare a grease composition which is serviceable at low and high temperatures.

It is another object of this invention to prepare a polysiloxane base oil grease composition with considerably greater ease than has been done heretofore.

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It is a still further object of this invention to prepare a highly oxidation resistant polysiloxane base oil grease composition which can be prepared directly without any intermediate steps involving additional solvents. These and further objects of this invention will be apparent from the following description and the appended claims.

It has been discovered that a grease composition resistant to emulsification in water and useful at low and high temperatures can be obtained by incorporating in a polysiloxane oil a complex basic aluminum soap containing at least two anions substantially organic in character, the aluminum soaps of said organic anions being water-insoluble and different appreciably in the extent of their solubilities in hydrocarbon oils.

The grease composition of this invention comprises a polysiloxane base oil and a complex basic aluminum soap.

By "complex basic aluminum soap" is meant that the complex aluminum soap molecule contains at least one hydroxyl anion for each aluminum cation, and at least two dissimilar anions substantially hydrocarbonaceous in character.

The term "essentially hydrocarbon (substantially hydrocarbonaceous)" means those radicals which are composed mainly of hydrogen and carbon, and include such radicals which contain, in addition, minor amounts of substituents such as chlorine, bromine, oxygen, nitrogen, etc.

The polysiloxane base oils can be represented by the formula indicative of the structure of a monosiloxane:

\[
\begin{align*}
\text{R} & \quad \text{Si-O-Si} & \quad \text{R'}
\end{align*}
\]

wherein \( \text{R} \) represents an alkyl radical containing no more than 5 carbon atoms, \( \text{R'} \) represents an aromatic radical, an alkaryl radical or an araloyl radical, and \( \eta \), having a value of at least 10, represents the number of monomeric units in the polymer. When \( \text{R'} \) is an alkaryl radical or an araloyl radical, the alkyl groups attached to the aromatic nucleus contain no more than 3 carbon atoms. It is particularly preferred that the polysiloxane base oil be a methyl phenyl-polysiloxane; that is, in the above formula, \( \text{R} \) represents a methyl radical, and \( \text{R'} \) represents a phenyl radical.

In the actual formation of a polysiloxane, all of the monomeric units may not have the same formula as that represented hereinabove. For example, in some of the monomeric units, there...
may be two R radicals attached to the silicon atom; in other monomeric units, there may be two R radicals attached to the silicon atom. However, the majority of the monomeric units are as represented in the above formula.

Examples of R radicals include the radicals derived from methane, ethane, propane, propene, butane, butene, pentane, hexene, etc. Examples of R radicals include the radicals derived from benzene, anthracene, phenyl methane, phenyl ethane, phenyl propane, methyl benzene, ethyl benzene, propyl benzene, etc.

The polysiloxane base oil is used as the major component of the composition of the present invention.

The organo anions or groups in the aluminum soaps of this invention are generally oleophilic (i.e., groups derived from or residues of acids, which are oil-soluble); however, one of the organo anions has a greater solubility in lubricating oil than another organo anion. For purposes of simplification of the discussion of the characteristics of the organo anions of the complex aluminum soap, the organo anions of greater oil solubility will be designated as “relatively oleophilic” anions, and the organo anions of lesser oil solubility will be designated as “relatively oleophobic” anions. That is, the organo acids of the relatively oleophilic anions are relatively oil-soluble, while the organo acids of the relatively oleophobic anions are relatively oil-insoluble, i.e., less oil-soluble as compared to the oleophilic organo acids.

In order to characterize further the organo anions of the aluminum soaps of this invention, the characteristics properties of each of the organo anions are noted as follows:

The aluminum di-soaps of each of the organo anions (i.e., the aluminum di-soaps of the oleophilic anions and the aluminum di-soaps of the oleophobic anion) are insoluble in water. For example, in the aluminum-benzoate-stearate example of this invention, the aluminum di-soap of the benzoate anion (i.e., aluminum di-benzoate) and the aluminum di-soap of the stearate anion (i.e., aluminum di-stearate) are insoluble in water.

The aluminum di-soaps of the more soluble organo anions (i.e., the relatively oleophilic anions) are soluble in a petroleum hydrocarbon lubricating oil (e.g., a California solvency-refined paraffinic oil having a viscosity of 485 SUS at 100°F) in an amount of at least 5% at 400°F. That is, at 400°F, 5% of the aluminum soap of the oleopholic organo anion will form a true solution in a petroleum hydrocarbon lubricating oil. On the other hand, the aluminum soaps of the less soluble organo anions (i.e., the relatively oleophobic anions) are soluble in a petroleum hydrocarbon lubricating oil in an amount of less than 1% at 400°F. That is, at 400°F, less than 1% (from 0% to about 1%) of the aluminum soap containing the oleophobic anions will dissolve forming a true solution.

Furthermore, the aluminum soaps of the relatively oleophobic anions melt at a temperature above 400°F, and the aluminum soaps of the relatively oleophilic anions melt at a temperature less than 350°F.

The complex aluminum soaps of this invention are polymeric in structure, that is, the complex aluminum soaps have more than one aluminum atom and at least two dissimilar organo anions throughout the polymeric structure. It is possible for the complex aluminum soaps to contain as many as 1,000 or more monomeric units, each monomeric unit containing one aluminum atom having all of its valences satisfied by at least one hydroxyl group and two organo anions. Thus, it is readily understood that although aluminum can have a valence of +3, it is not meant herein to limit the complex aluminum soap of this invention to one containing only three specific anions. In the over-all average, the valence bonds of the aluminum atoms can be directed to more than three specific anions, that is, to more than one hydroxy anion and more than two organo anions. The average molecule in the soap may contain a plurality of relatively oleophilic anions or a plurality of relatively oleophobic anions per aluminum atom. For example, it may be advantageous in some instances to use a complex aluminum soap as exemplified by aluminum benzoate-stearate-caprate.

Suitable relatively oleophilic anions are anions of aliphatic (saturated and unsaturated) aromatic, aralkyl, and cycloaliphatic carboxylic acids. The acids must be sufficiently hydrocarbonaceous in character to impart the desired oil solubility. Thus, the aliphatic (saturated and unsaturated) carboxylic acids may contain from 8 to about 30 carbon atoms, preferably from 12 to 18 carbon atoms. The aliphatic substituent in the various cyclic carboxylic acids may contain at least 4 carbon atoms on the aliphatic group attached to the ring. The aralkyl, alkaryl and cycloaliphatic carboxylic acids preferably contain a total of about 16 carbon atoms. The relatively oleophobic anion may be an alkyl phenol containing at least 4 carbon atoms in the alkyl group, preferably 18 carbon atoms in the alkyl group; e.g., cetyl phenol.

It is preferred that the organo-substituted acids of sulfur and phosphorus contain at least 14 carbon atoms, and more especially at least 20 carbon atoms, in the organo substituent. The aliphatic acid anions may contain various substituents, such as hydroxy, amino, alkoxy, e.g., methoxy, and like radicals, so long as the anion remains substantially hydrocarbonaceous in character.

Examples of the carboxylic acids from which the oleophilic anions are derived are: caprylic acid, capric acid, laurie acid, myristic acid, palmitic acid, stearic acid, 12-hydroxy stearic acid, arachidic acid, melissic acid, oleic acid, linoleic acid, butyric acid, hexyl benzoic acid, cetyl benzoic acid, dodecyl benzoic acid, phenyl butyric acid, phenyl hexanoic acid, phenyl decanoic acid, cetyl benzene sulfonic acid, a di-dodecyl benzene sulfonic acid (e.g. a dipropypropylene benzene sulfonic acid), an alkane phosphonic acid having at least 24 carbon atoms in the alkane group, cetyl thio phosphoric acid, naphthenic acids, etc.

Of these, stearic acid, hydroxy stearic acids, naphthenic acids of molecular weight above about 350, and allyl benzene sulfonic acids having at least 30 carbon atoms in the alkyl substituents are preferred.

The relatively oleophobic anions are substantially hydrocarbon in structure and may be selected from anions of aliphatic (saturated and unsaturated), aromatic, aralkyl, alkaryl and cycloaliphatic mono- and polycarboxylic acids. Acids having up to two carboxyl groups are preferred, the monocarboxylic acids being especially preferred. For the desired properties, aliphatic monocarboxylic acids of 4 to 7 carbon atoms are employed. When the carboxylic acid contains
2,654,710

2,654,710

2 carboxyl groups, the acid contains at least 2 carbon atoms, preferably from 8 to 11 carbon atoms, and in some cases up to 20 carbon atoms, so long as the anion resulting therefrom is relatively oleopholic as compared to the oleophilic anion employed. The alkyl groups of the aralkyl and alkaryl carboxylic acids contain no more than 3 carbon atoms. Thus, the alkaryl and the aralkyl carboxylic acids contain a total of not more than 9 carbon atoms, preferably a total of 7 carbon atoms.

Suitable oleopholic anions are derived from benzoic acid, methyl benzoic acid, ethyl benzoic acid, toluid acid, phenyl acetic acid, phenyl propanionic acid, oxalic acid, malonic acid, isocoumaric acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, phthalic acid, succinic acid, carboxy methyl cellulose, polyacrylic acid, etc. Of these, the benzoic, azelaic and toluid acids are preferred.

Because of the increased effectiveness in obtaining a high melting, high water resistant grease, it is preferred that the oleopholic anion of the aluminum soap of this invention be an anion of an aliphatic carboxylic acid (e.g., stearic acid), and that the oleopholic anion be an anion of an aromatic carboxylic acid (e.g., benzoic acid).

It is essential to the success of this invention that the more oil-soluble organo anion (i.e., the relatively oleophobic group) and the less oil-soluble organo anion (i.e., the relatively oleopholic group) be present in such proportions to each other that the complex aluminum soap of this invention will have the desired dispersibility in the base oil to bring about the formation of a grease structure.

When petroleum hydrocarbon oils are used as the base oils for greases thickened with the complex basic aluminum soaps of this invention, those greases become more gelatinous (i.e., increase in consistency) as the oleopholic-oleophobic anion ratio is decreased. Conversely, the grease compositions of this invention wherein polysiloxanes are used as the base oils increase in consistency (i.e., become more gelatinous) as the oleopholic-oleophobic anion ratio increases.

The ratio of oleophobic to oleophilic anions in the average molecule of the soap may range from 1.0 to 10.0.

The ratio of oleophobic anion to oleophilic anion (on an average molecule basis) can be altered so that the desired grease structure may be obtained in polysiloxane oils of varying solency characteristics. That is, it is generally desirable when using a polysiloxane oil of high solen cy capacity to use a basic aluminum soap which is less oil-soluble than the basic aluminum soap preferred when the polysiloxane oil is of a low solen cy capacity. For example, when using a polysiloxane lubricating oil which is almost devoid of aromatic hydrocarbons, it is usually desirable to use a basic aluminum soap of this invention wherein the oleophobic anion is considerably more oil-soluble than the oleophilic anion. On the other hand, when using a polysiloxane lubricating oil having considerable quantities of aromatic hydrocarbons present, it is desirable to use a basic aluminum soap of this invention wherein the oleophilic anion is not too highly oil-soluble.

The number of free hydroxyl groups present in the basic aluminum soap of this invention may vary from 1.0 to 1.5 hydroxyl groups for each aluminum atom in the soap. When the number of free hydroxyl groups present is less than about 1, the resulting aluminum soap is of a higher acidic character than normally is desired in greases. If the number of free hydroxyl groups reaches as high as 2 hydroxyl groups per aluminum atom, the grease prepared from such an aluminum soap is somewhat granular. Thus, it is preferred to use a basic aluminum soap of this invention wherein the free hydroxyl groups range from about 1.0 to about 1.5 hydroxyl groups per aluminum cation.

Although it is preferred to use oleopholic anions derived from carboxylic acids because of the improved texture of the greases prepared therefrom, the relatively oleopholic anion may on the average be partly an anion of an inorganic acid of phosphorus, e.g., phosphoric acid, an inorganic acid of boron, e.g., boric acid, or in some cases an anion of an inorganic acid of silicon, e.g., silicic acid. For example, the oleopholic anion portion of the average molecule may contain, in part, a phosphate (—PO₃) anion. Furthermore, the relatively oleopholic anion may be derived from phenol; that is, the relatively oleopholic anion may be an alkyl phenol containing no more than 3 carbon atoms in the alkyl group.

Examples of complex basic aluminum soaps of this invention are: aluminum benzoate stearate, aluminum benzoate oleate, aluminum benzoate, 12-hydroxy stearate, aluminum toluate stearate, aluminum benzoate naphthenate, aluminum benzolate hydrogenated rosin, aluminum benzoate stearate, aluminum azelate stearate, aluminum phosphite benzolate stearate, aluminum benzoate hydroxy stearate, etc. Of these, aluminum azelate stearate, aluminum toluate stearate, and aluminum benzoate stearate are preferred, the last being especially preferred.

The aluminum soaps of this invention can be prepared according to methods involving co-precipitation. For example, aqueous solutions of mixtures of the water-soluble soaps (e.g., sodium soaps) in the desired proportion of relatively oleopholic and relatively oleophobic anions are admixed with an aqueous solution of an aluminum salt (e.g., aluminum sulfate). The resulting precipitate of the basic aluminum complex soap is then preferably purified to remove the salts such as sodium sulfate. Basic aluminum complex soaps yielding greases of high water resistance, high melting point and excellent chemical and physical stability may also be prepared in situ according to the method described in Jones Patent No. 2,469,041, wherein a fatty acid (e.g., stearic acid), is added to a mineral oil solution of an aluminum alcoholate (e.g., aluminum butoxide), to form aluminum stearate.

The following examples illustrate the preparation of the complex aluminum soaps described herein:

Example 1.—The preparation of aluminum azelate stearate

A mixture of 13.5 parts by weight of commercial stearic acid (approximately 60% stearic acid and the remainder mainly palmitic acid) and 10 parts by weight of azellic acid was dissolved in 400 parts by weight of water at approximately 180° F. This solution was slowly added, with stirring, to a solution of 24.9 parts by weight of aluminum sulfate octadecahydrate in 300 parts by weight of water at this same temperature. The resulting reaction mixture was filtered. The white precipitate which was formed during the reaction was washed with water by adding the
white precipitate to a large amount of water and then stirring vigorously. The precipitate was washed in this manner three separate times until only a faint positive sulfite ion test was attained in the filtrate from the third wash. The resulting mixed soap was then dried at a temperature ranging from 210° F. to 250° F., was powdered and passed through a No. 60 mesh sieve.

**Example 2.—Preparation of aluminum toluate stearate**

A mixture of 28.6 parts by weight of toluate acid containing a 2:1 mixture of the meta and para isomers, 27 parts by weight of commercial stearic acid, and 18 parts by weight of sodium hydroxide was dissolved in 400 parts by weight of water at about 180° F. This solution was slowly added, with stirring, to 50 parts by weight of aluminum sulfate octadecahydrate in 300 parts by weight of water at the same temperature. The resulting complex polyvalent metal soap was filtered, washed and dried as described in Example 1. On ashing, the soap left 13.9% Al₂O₃ (theoretical for the aluminum toluate stearate is 13.6%).

**Example 3.—Preparation of aluminum benzoate stearate**

A mixture of 12.2 parts by weight of benzoic acid, 27.1 parts by weight of commercial stearic acid and 16.8 parts by weight of potassium hydroxide was dissolved in about 500 parts by weight of water at 150° F., forming a clear solution. To this solution was added 33.3 parts by weight of aluminum sulfate octadecahydrate dissolved in about 300 parts by weight of water. The resulting aluminum benzoate stearate was obtained by filtering, washing and drying the precipitate as above in Example 1. On ashing, the soap left 11.5% Al₂O₃ (theoretical for Al₂C₅H₁₅O₃N=11.4%).

While the true basic aluminum complex soaps of the present invention produce greases having high water resistance and high melting points as well as excellent texture, physical properties of aluminum soaps from the relatively oleophilic acids, e.g., stearic acid, with aluminum soaps or the relatively oleophobic acids, e.g., benzoic acid, do not yield satisfactory greases, even though such mixture of soaps is exposed to prolonged heating.

The complex basic aluminum soaps of this invention can be used in polysiloxanes to form grease compositions in amounts of from 15% to 40%; however, it is preferred to use from 25% to 35% by weight based on the finished composition.

The grease compositions of this invention can be prepared according to numerous processes. An advantageous process includes the addition of the complex basic aluminum soap to a polysiloxane at room temperatures and heating, with agitation, to temperatures ranging from 400° F. to 600° F., then cooling the mixture to room temperature.

The following Table I presents data to illustrate the effect of (1) complex basic aluminum soap concentration, and (2) oleophobic anion/oleophilic anion ratio on the texture and consistency of grease compositions of this invention.

The polysiloxane used as the base oil was a polymethylphenyl silicone, wherein the R group in the above formula was a methyl radical and the R' group was a phenyl radical. This particular polymethylphenyl silicone is known as Dow Corning—550 and has a boiling point of 250° C. at an absolute pressure of less than 15 mm. of mercury, a viscosity in the range of 300-400 SSU at 100° F., a specific gravity of 1.08 (25° C./25° C.), a freezing point of —54° F., and a refractive index of 1.487. The complex basic aluminum soap was aluminum benzoate stearate. The soap concentration in the greases was 34% by weight based on the final composition. The greases were prepared by dispersing the aluminum benzoate stearate in the polysiloxane at temperatures ranging from 500° F. to 525° F., then cooling to room temperature.

<table>
<thead>
<tr>
<th>Table I</th>
<th>Benzate/ Stearate Ratio</th>
<th>Consistency (ASTM Worked Penetration)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>0.33</td>
<td>34</td>
</tr>
<tr>
<td>2.</td>
<td>0.5</td>
<td>35</td>
</tr>
<tr>
<td>3.</td>
<td>1.0</td>
<td>36</td>
</tr>
<tr>
<td>4.</td>
<td>3.0</td>
<td>36</td>
</tr>
</tbody>
</table>

In the following Table II, data are presented to show the effect of the change in the relatively oleophobic anion. The complex basic aluminum soap used in this grease preparation was a basic aluminum toluate stearate having a toluate/stearate ratio of 3.0. The base oil used was the same base oil as used in Table I, and the soap concentration was 32% by weight of the final composition. The grease was prepared by dispersing the soap in the polysiloxane at a temperature of 525° F., then cooling to room temperature.

<table>
<thead>
<tr>
<th>Table II</th>
<th>Soap Used</th>
<th>Consistency (ASTM Worked Penetration)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>o-toluate-stearate</td>
<td>293</td>
</tr>
<tr>
<td>2.</td>
<td>p-toluate-stearate</td>
<td>333</td>
</tr>
</tbody>
</table>

In the preparation of greases used in obtaining the data of the following Table III, the base oil was a polymethylphenyl silicone known as Dow Corning—710. This base oil had a viscosity of 1060 SSU at 100° F., a boiling point of 250° F. at an absolute pressure of less than 15 mm. of mercury, a specific gravity of 1.11 (25° C./25° C.), a freezing point of —10° F., and a refractive index of 1.532. The greases were prepared by dispersing the soap in the oil at temperatures ranging from 490° F. to 500° F.

<table>
<thead>
<tr>
<th>Table III</th>
<th>Soap Used</th>
<th>Oleophobic/Oleophilic Ratio</th>
<th>Soap Cons.</th>
<th>Consistency (ASTM Worked Penetration)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Aluminum benzate stearate</td>
<td>1.0</td>
<td>35</td>
<td>298</td>
</tr>
<tr>
<td>2.</td>
<td>Aluminum p-toluate stearate</td>
<td>3.0</td>
<td>30</td>
<td>206</td>
</tr>
</tbody>
</table>

In addition to the complex basic aluminum soaps, the polysiloxane base oils may contain other agents which increase the oxidation resistance still further, improve further the extreme pressure characteristics, etc.

I claim:

1. A grease composition comprising a major proportion of a polysiloxane base oil and a complex basic aluminum soap in an amount sufficient to thicken the polysiloxane base oil to the consistency of a grease, said complex basic aluminum
soap having at least two unlike organo-anions, one organo-anion derived from an organic acid, the aluminum di-soap of which acid is characterized by an oil solubility of at least 5% at 400°F, and another organo-anion derived from an organic acid, the aluminum di-soap of which acid is characterized by an oil solubility of less than 1% at 400°F.

2. A grease composition comprising a major proportion of a polysiloxane oil and a complex basic aluminum soap in an amount sufficient to thicken the polysiloxane oil to the consistency of a grease, said polysiloxane oil being derived from monosiloxanes containing alkyl radicals and aromatic radicals, said complex basic aluminum soap having at least two unlike organo-anions, one organo-anion derived from an organic acid, the aluminum di-soap of which acid is characterized by an oil solubility of at least 5% at 400°F, and another organo-anion derived from an organic acid, the aluminum di-soap of which acid is characterized by an oil solubility of less than 1% at 400°F.

3. A grease composition comprising a major proportion of a polysiloxane oil and a complex basic aluminum soap in an amount sufficient to thicken the polysiloxane oil to the consistency of a grease, said polysiloxane oil being derived from monosiloxanes, the major proportion of which contains an alkyl radical and an aromatic radical, said complex basic aluminum soap having at least two unlike organo-anions, one organo-anion derived from an organic acid, the aluminum di-soap of which acid is characterized by an oil solubility of at least 5% at 400°F, and another organo-anion derived from an organic acid, the aluminum di-soap of which acid is characterized by an oil solubility of less than 1% at 400°F.

4. The composition of claim 3, wherein the alkyl radical is a methyl radical, and the aromatic radical is a phenyl radical.

5. A grease composition comprising a major proportion of a polysiloxane base oil of the formula

\[
\begin{array}{c}
\text{R} \\
\text{Si-O-} \\
\text{R'}
\end{array}
\]

wherein R represents an alkyl radical containing no more than 5 carbon atoms, R' represents a radical containing an aromatic nucleus, and n represents the number of monosiloxane units present in the polysiloxane, and a small amount, sufficient to thicken the polysiloxane base oil to the consistency of a grease, of a complex basic aluminum soap, said complex basic aluminum soap having at least two unlike organo-anions, one organo-anion derived from an organic acid, the aluminum di-soap of which acid is characterized by an oil solubility of at least 5% at 400°F, and another organo-anion derived from an organic acid, the aluminum di-soap of which acid is characterized by an oil solubility of less than 1% at 400°F.

6. A grease composition comprising a major proportion of a polysiloxane base oil of the formula

\[
\begin{array}{c}
\text{CH}_3 \\
\text{Si-O-} \\
\text{Phenyl}
\end{array}
\]

wherein n represents the number of monosiloxane units present in the polysiloxane base oil, and a small amount, sufficient to thicken the polysiloxane base oil to the consistency of a grease, of a complex basic aluminum soap having at least two unlike organo-anions, one organo-anion derived from an organic acid, the aluminum di-soap of which acid is characterized by an oil solubility of at least 5% at 400°F, and another organo-anion derived from an organic acid, the aluminum di-soap of which acid is characterized by an oil solubility of less than 1% at 400°F.

7. A grease composition comprising a major proportion of a methyl, phenyl-polysiloxane lubricating oil and about 15% to about 40% of a complex basic aluminum soap containing at least two unlike organo-anions, one organo-anion derived from an organic acid, the aluminum di-soap of which acid is characterized by a white oil solubility of at least 5% at 400°F, and another organo-anion derived from an organic acid, the aluminum di-soap of which acid is characterized by a white oil solubility of less than 1% at 400°F, substantially organic in character and differing appreciably in their solubilities in petroleum hydrocarbon oils, at least one anion being relatively oleophilic and another anion being relatively oleophobic.

8. A grease composition comprising a major proportion of a methyl, phenyl-polysiloxane lubricating oil and about 25% to about 35% of a complex basic aluminum soap containing at least two unlike organo-anions, one organo-anion derived from an organic acid, the aluminum di-soap of which acid is characterized by a white oil solubility of at least 5% at 400°F, and another organo-anion derived from an organic acid, the aluminum di-soap of which acid is characterized by a white oil solubility of less than 1% at 400°F, substantially organic in character and differing appreciably in their solubilities in petroleum hydrocarbon oils, at least one anion being relatively oleophilic and another anion being relatively oleophobic.

9. A grease composition comprising a major proportion of a methyl, phenyl-polysiloxane lubricating oil and about 15% to about 40% of an aluminum benzoate stearate.

10. A grease composition comprising a major proportion of a methyl, phenyl-polysiloxane lubricating oil and about 25% to about 35% of an aluminum benzoate stearate.

BRUCE W. HOTTEN.

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