United States Patent Office 3,223,627 Patented Dec. 14, 1965

3,223,627

LUBRICATING COMPOSITIONS CONTAINING METAL SALT OF BENZOIC ACID

Hans G. Vesterdal, Elizabeth, N.J., assignor to Esso Research and Engineering Company, a corporation of Delaware

No Drawing. Filed Mar. 22, 1963, Ser. No. 267,352
3 Claims. (Cl. 252—25)

This invention relates to lubricating compositions. More particularly, the present invention relates to lubricating compositions which comprise lubricating oils thickened to a grease consistency with metal salt of benzoic acid and which may preferably contain metal salt of other acids. In a particular embodiment, this invention relates to lubricating grease compositions which are suitable for high temperature lubrication.

BACKGROUND

The trend in the design of modern aircraft has accentuated the need for lubricating greases which will effectively lubricate anti-friction bearings operating at high rotational speeds and high temperatures. While considerable progress has been made in recent years in producing improved aircraft greases, some difficulty has been encountered in producing a grease which will effectively lubricate bearings operating at high rotational speeds and high temperatures for prolonged periods of time. Conventional aircraft greases currently available to the consumer have generally failed to meet the stringent requirements placed on such a lubricant.

DISCOVERY

It has now been discovered, and this discovery forms the basis of the present invention, that lubricating oils and, in particular, the silicon-containing lubricating oils (i.e., silicones and silanes) may be effectively thickened to a grease consistency with metal salt of benzoic acid, e.g., calcium benzoate, alone, or more preferably, with a mixture of metal sale of benzoic acid and metal salt of C_1 to C_6 fatty acid, e.g., acetic acid. Lubricating compositions prepared according to the preferred form of the present invention are especially adapted for use within the wide temperature range of −65° F. to +600° F. and are particularly efficacious in lubricating bearings operating at high rotational speeds, e.g., 10,000 r.p.m., and at temperatures of up to 450° F. for prolonged periods of time. Moreover, the more preferred lubricating compositions (made from silicone fluids) may be effectively used for shorter periods of time at temperatures up to 700° F. The term "silicon-containing lubricating oils" as used herein is intended to apply to those oils containing silicon atoms as part of their molecular structure (silicones and silanes), and does not include lubricating oils, e.g., mineral oils, to which silicon, e.g., sand, has been mechanically added.

Lubricating compositions made by thickening a silicon-containing oil with metal salt of benzoic acid, alone, have long spindle lubrication lives at 450° F., particularly when they are stabilized with a substantial amount of amine inhibitors. However, such greases have rather poor anti-wear and extreme pressure properties. The presence of well dispersed, finely divided particles of a salt of a low molecular weight fatty acid, e.g., acetic acid, overcomes many of these deficiencies. When benzoic acid salt is used as the sole thickener, it will be present in amounts of from 20 to 48 wt., e.g., 28 to 38 wt. percent (based on a total formulation of 100%).

Preferably, lubricating greases prepared according to the present invention will comprise a lubricating oil and from about 34 to 68 wt. percent (based on a total grease formulation weight of 100 wt. percent), preferably from about 41 to 58 wt. percent, of a mixture of benzoic acid salt and the metal salt of a C_1 to C_6 fatty acid, e.g., acetic acid. The salt mixture will, in turn, contain from 6 to 28 wt. percent, preferably from 11 to 22 wt. percent, of salt of the low molecular weight fatty acid, e.g., calcium acetate. The remainder of the salt mixture will be metal salt of benzoic acid, e.g., calcium benzoate.

METAL COMPONENTS OF THE SALTS

The metal components of the salts will generally be alkali metals or alkaline earth metals although it may sometimes be desirable to use other metals, e.g., aluminum. It is preferred to use the alkaline earth metals since the use of alkali metals, e.g., Na, tends to produce water soluble greases which are undesiderable in certain applications. Of the alkaline earth metals, calcium is the most preferred. It is not meant to imply that all of these metals serve with equal efficiency. For example, magnesium salts may be used, but some difficulty has been encountered in dispersing the resulting salts in the oils used. These various metals may be used in such forms as their hydroxides.

ACID COMPONENTS OF THE SALTS

Benzoic acid is the acid component of the primary thickener of this invention. While it would be expected that related acids would be similarly effective, the contrary has been found to be true. For example, laboratory attempts to produce a satisfactory solid lubricating grease from metal salt of salicyclic acid have proven unsuccessful. Moreover, if the esters of benzoic acid are used rather than metal salt, solid greases do not result. For example, it has been reported (M. C. Agens in U.S. No. 2,837,482) that up to 25% or more of octyl tetrachlorobenzoate or dioctyl tetrachlorophthalate can be added to silicone fluids (polyorgano siloxanes) to produce *fluid* lubricants having enhanced lubricating properties. Thus, it would appear that benzoic acid, when combined with a suitable metal, is unique. Calcium benzoate is the most preferred primary thickener.

The acid components of the other salts which can be advantageously combined with benzoic acid salt are the C_1 to C_6 fatty acids, e.g., formic acid, acetic acid, hexanoic acid, etc. Of these acids, acetic acid is preferred. The use of the metal salt of these acids, e.g., calcium acetate, increases the extreme pressure and anti-wear properties of the ultimate lubricating grease compositions.

PREPARATION OF THE SALTS

The salts of the present invention may be formed separately and added, in a preformed state, to the lubricating oil or one or more of the salts may be formed in situ. Preferably, salt of benzoic acid will be formed in situ while salt of C_1 to C_6 acid will be preformed. The metal salt of C_1 to C_6 fatty acid, particularly calcium acetate, is known to be extremely desirable in the preparation of lubricating compositions, since lubricants containing substantial amounts of such a salt possess both extreme pressure (EP) and anti-wear properties. Because of the insolubility of these salts, e.g., calcium acetate, in mineral lubricating oil, it has generally been the practice of industry to use either surfactants and/or the salts or soaps of higher aliphatic acids, e.g., C_7 to C_{30} fatty acids, as suspending agents to maintain the calcium acetate dispersed throughout the oil. The present invention involves a departure from prior practice in that it has been found that metal salt of benzoic acid, i.e., an aryl acid salt, is particularly effective as a suspending agent to maintain metal salt of C_1 to C_6 fatty acid, e.g., calcium acetate, uniformly dispersed throughout a lubricating oil. The unique combination of calcium acetate and benzoic acid salt is particularly desirable and advantageous in

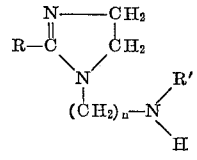
the preparation of lubricating compositions prepared from silicon-containing lubricating oils where calcium acetate has been infrequently used. The ultimate qualities of the lubricating greases prepared according to the present invention can be enhanced by reducing the particle size of the metal salt of C_1 to C_6 fatty acid, e.g., calcium acetate, to as small a size as is possible. Reduction of, for example, calcium acetate to a particle size below 5 microns, e.g., 1 micron or less, produces a lubricating grease which is much smoother and has clearly enhanced wearing properties when compared with a grease prepared from commercially available calcium acetate which has a particle size of from 25 to 200 microns. It is possible to prepare metal salt of acids, e.g., calcium acetate, in extremely finely divided form by various techniques.

For example, the in situ preparation of salts wherein the salts, e.g., calcium acetate, are formed in the presence of a suspending agent, produces small salt particles. Alternate techniques for preparing finely divided calcium acetate include the use of milling, grinding, etc., as well as the use of surfactants. A particularly preferred technique which has been recently developed involves the preparation of, for example, calcium acetate in the presence of a surfactant prepared from an amino imidazoline.

Briefly described with reference to calcium acetate, this technique for obtaining a small particle size is as follows: Calcium acetate is mixed with water to form a saturated solution, or more preferably a concentrated calcium acetate/water slurry, e.g., 35 to 80 wt. percent, of calcium acetate in water. This solution is then added to a small amount of lubricating oil, e.g., mineral lubricating oil, in which a surface active agent of the amino imidazoline salt type has been previously dispersed. This resulting mixture is heated to drive off sufficient water to form an aqueous emulsion, e.g., 50 wt. percent of water based on the total weight of the mixture. The emulsion is then cooled to form an oatmeal-like mass, into which is mixed a relatively large amount, e.g., about 4 times the volume of said oatmeal-like mass, of a volatile hydrocarbon solvent such as heptane, etc. Solvents, such as heptane, form a partial azeotrope with water and thus aid in its removal. The solvent also removes water mechanically (entrainment). Heating is initiated and the solvent refluxed while simultaneously drawing off the water. When substantially all of the water has been removed, e.g., the water content of the remaining mixture is less than 1.0 wt. percent based on the total weight of the remaining mixture, lubricating oil, e.g., 2 parts of a mineral lubricating oil, is added to the emulsion residue and the solvent, e.g., heptane, removed by distillation. The resulting gel contains finely dispersed calcium acetate crystal needles less than 1 micron e.g., about 0.5 micron, in length. Alternatively and more preferably, after the aqueous emulsion has been reduced to a water content of about 1.0 wt. percent based on the total weight of the remaining mixture, it can be reduced to dryness (without adding any more oil) by distilling both heptane and water from the mixture. As the mixture containing heptane and calcium acetate becomes sufficiently concentrated, calcium acetate particles will precipitate out. Calcium acetate in the form of a soft, fluffy powder can then be recovered by drying the precipitate.

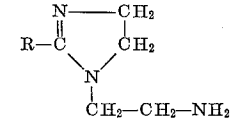
It is believed that the acid salt of the imidazoline acts as a crystal growth inhibitor by coating the calcium acetate particles as they form, thus preventing their aggregation into larger particles.

The amino imidazolines used in forming the imidazoline salts of the type just described include those having the general structure:



In the above formula, n represents an integer of from about 2 to 6, preferably 2 to 3; R represents a C_{12} to C_{22}, preferably a C_{14} to C_{18}, hydrocarbon group, either saturated or unsaturated, and preferably aliphatic; while R' is either hydrogen or a C_2 to C_{18} alkyl group. Preferably, R' is hydrogen and n is a small integer, e.g., 2, in order that the effectiveness of the imidazoline can be as great as possible per pound of material. In other words, the apparent effectiveness of the imidazoline in the present invention seems to depend on the ring structure and the terminal amino group, while the number of carbon atoms in the branch merely dilutes the apparent effectiveness.

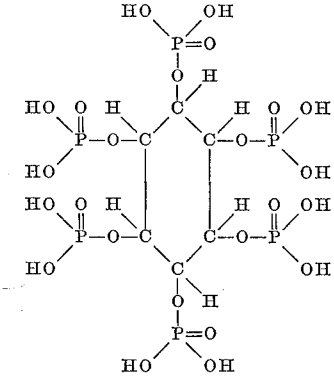
A specific example of an imidazoline of the above formula, which has been used with considerable success, is a commercially available imidazoline; 1-(2-amino ethyl)-2-(n-alkyl)-2-imidazoline, which has the general formula:



wherein R represents heptadecenyl and heptadecadienyl chains in a mole ratio of about 1:1. This product is commercially available under the trade name Nalcamine G–39M and is sold by Nalco Chemical Company, Chicago, Illinois.

The acids, which can be reacted with imidazoline to form the salts, include inorganic mineral acids such as ortho, pyro and meta phosphoric acids, hydrochloric acid, sulfuric acid, nitric acid, and also phytic acid which is closely related to phosphoric acid. Phytic acid is the preferred acid.

Phytic acid is the hexaphosphoric acid ester of inositol. It is a strong acid containing twelve acidic hydrogen groups. Its structural formula is believed to be as follows:



This material, having a molecular weight of 666 with 12 reactive hydrogen groups, has a combining weight (mole equivalent weight) of 55.

Phytic acid is derived from grain and is a by-product from waste corn steep liquor. A description of phytic acid and its preparation is given in Chemical Engineering, January 27, 1958, under the title "Ion Exchange Now Yields Phytic Acid," published by McGraw-Hill Publishing Co., Inc., New York, New York.

Another surfactant which is particularly useful in preparing the salts in finely divided form are materials of the type exemplified by barium dinonyl naphthalene sulfonate. These materials are merely added to the lubricating oil and the metal salt of acetic and benzoic acids are formed in situ, e.g., by adding lime. The presence of small amounts of materials such as barium dinonyl naphthalene sulfonate in the ultimate lubricating composition are not detrimental and impart other advantages to the lubricant, e.g., rust inhibition. The nonyl groups in barium dinonyl naphthalene sulfonate as it is available commercially are a mixture of straight and branched chains. They are usually added by way of alkylation with tripropylene.

SUITABLE LUBRICATING OILS

The lubricating oil in which the acid salts are incorporated is preferably a lubricant of the type which is, per se, best suited for the particular use for which the ultimate lubricating composition is designed. Where temperatures on the order of 400° F. and above are expected, synthetic oils form a preferred class of lubricating oils because of their high thermal stability. The synthetic oil may be an organic ester, e.g., di-2-ethylhexyl sebactate, etc. Alternatively, polymerized olefins, copolymers, polyalkylene glycols, polyalkylene oxides, polyphenylethers, polyorgano siloxanes, etc. may be used. These lubricating oils will usually have a viscosity of about 1 to 500, e.g., 20 to 400, centistokes at 100° F.

The preferred lubricating oils for use according to the present invention include, in general, any silicon-containing lubricating oil. These are particularly preferred because of their high thermal stability and compatibility with the salts of the present invention. These silicon-containing oils are generally of two types; those known as siloxanes (or silicones) and those known as silanes. The latter (silanes) can be represented by the general formula:

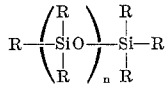


wherein R represents an alkyl group, e.g., C_6–C_{20}, or an aryl group such as phenyl, naphthyl, biphenyl, etc., or substituted aryl groups. The R's may be the same or different. Examples of such oils include heptyl triphenyl silane, diheptyl diphenyl silane, sinonyl dinaphthyl silane, etc. A silane of particular import is a dilauryl diphenyl silane that is commercially available from the Dow Corning Corporation of Midland, Michigan, under the trade designation of QF–6–7009. The characteristics of that silane oil are shown in Table I.

Table I

Color	Clear, straw
Viscosity, centistokes at:	
0° F.	1500
77	70
100	38
210	6.3
400	1.7
700	0.65
Viscosity Index (100–210° F.)	125
Relative Density (77° F.)	0.90
Freeze Point, ° F.	−30
Flash Point, ° F.	500
4-Ball Wear Test mm. Scar Diam. (1,800 r.p.m., 10 Kg., 75° C., 1 hour)	0.40

Even more preferred as the lubricating oil base are those materials generally defined as siloxanes (silicones). Siloxanes are made up of recurring units of silicon and oxygen atoms as indicated by the general formula below:



wherein R represents alkyl, aryl, alkaryl, aralkyl, or cycloalkyl radicals and n is an integer of from about 2 to 50 or more, e.g., 6 to 35. The particular value of n is unimportant, however, so long as the polymer possesses a suitable viscosity, etc. Examples of such compounds are the dimethyl siloxanes, diethyl siloxanes, methylphenyl siloxanes, ethyl phenyl siloxanes, etc. Two exemplary siloxanes, both extremely desirable, are commercially available from the Dow Corning Corporation, Midland, Michigan, under the trade designations QF–6–7012 and QF–6–7024 and are both polymethyl phenyl siloxanes. The properties of these materials are shown in Table II.

Table II

	QF–6–7012	QF–6–7024
Viscosity, centistokes at 100° F.	64.3	225.4
Viscosity, centistokes at 210° F.	21.2	20.40
Viscosity Index	162	109
Pour Point, ° F.	<100	+25

A particularly useful reference to those who are interested is a book entitled "Synthetic Lubricants" edited by R. C. Gunderson and A. W. Hart, copyright 1962 by the Reinhold Publishing Corporation. In particular, Chapter 7 is devoted to silicones and has been written by several employees of the Dow Corning Corporation. That book is incorporated herein by reference.

If desired, a blend of oils of suitable viscosity may be employed as the lubricating oil instead of employing a single oil.

The preferred lubricating compositions of the present invention may contain other lubricant additives, if desired, to improve specific properties of the lubricant without departing from the scope of the present invention. Thus, the lubricating compositions of the present invention may contain corrosion and rust inhibitors, extreme pressure agents, anti-oxidants, dyes, etc. Whether or not such additives are employed and the amounts thereof depend to a large extent upon the severity of the conditions to which the composition is subjected and upon the stability of the original lubricating oil. For example, siloxanes are, in general, more stable than mineral lubricating oils and thus require the addition of little, if any, oxidation inhibitors to duplicate the low temperature performance of mineral oils. However, high temperature operations, of necessity, require the use of anti-oxidants in silicone fluids. When such conventional additives are used, they are generally added in amounts between about 0.01 and 10 wt. percent based on the weight of the total composition. Preferred anti-oxidants for the greases of the present invention include phenyl alpha naphthylamine, p,p'-dioctyl diphenyl amine and dipyridyl amine.

It has been found that a mixture of oxidation inhibitors is more efficacious than the same total concentration of a single inhibitor alone. For example, a grease prepared from a siloxane (Dow Corning QF–6–7012) had an uninhibited lubrication life at 450° F. and 10,000 r.p.m. of 42 hours. With 5 wt. percent of p,p'-dioctyl diphenyl amine it has a similar lubrication life of 188 hours. With 5 wt. percent of phenyl α naphthylamine it had a lubrication life of 222 hours. It was surprising to note that when 3 wt. percent of phenyl α naphthylamine and 2 wt. percent of p.p'-dioctyl diphenylamine were employed, the lubrication life rose to 238 hours. When this unique combination of additives is employed, from 2 to 5 wt. percent of phenyl α naphthylamine will generally be used and from 1 to 3 wt. percent of p,p'-dioctyl diphenyl amine will generally be employed, although occasionally other amounts may be desirable.

EXAMPLES

The present invention will be more clearly understood by reference to the following examples which include a preferred embodiment. Unless otherwise indicated, all parts are by weight.

EXAMPLE 1

Two hundred forty parts of benzoic acid were intimately mixed with 264 parts of a silicone fluid (QF–6–7012), 66 parts of hydrated lime were added and the mixture heated to 250° F. with stirring. At 250° F. the mixture was quite hard and an additional 285 parts of silicone fluid were added in increments of 15–30 gms. until a more manageable mass was obtained, i.e., softer. The resulting composition was heated to 300° F. and maintained at that temperature for 2 hours. The grease composition was divided into two equal parts, noted as 1(A)

and 1(B). Part 1(B) was cooled and milled. Part 1(A) was inhibited with 25 parts phenyl α-naphthylamine and 17 parts p,p'-dioctyl diphenyl amine, cooled to 110° F., and milled. The approximate composition and properties of those greases are shown in Table III.

EXAMPLE 2

One hundred sixty-eight parts of benzoic acid, 84 parts of acetic acid and 12 parts of barium dinonyl naphthalene sulfonate (in the form of a 50 wt. percent solution in a mineral lubricating oil) were intimately mixed with 238 parts of a silicone fluid (QF–6–7012). One hundred eight parts of hydrated lime were added to the mixture which was then heated at 320° F. for about 2 hours. The composition was cooled to 250° F. where 18 parts of phenyl naphthylamine and 12 parts of p,p'-dioctyl diphenyl amine were added. The resulting composition was cooled to 110° F. and milled. The approximate composition and properties of that grease are shown in Table III.

EXAMPLE 3

One hundred eighty parts of benzoic acid, 90 parts of acetic acid and 12 parts of barium dinonyl naphthalene sulfonate were intimately mixed in a grease kettle with 198 parts of a dimethyl diphenyl siloxane (Dow Corning QF–6–7012). One hundred twenty parts of hydrated lime were added and the resulting mixture was heated, with stirring, to 280° F. At this stage the reaction mixture was dry, i.e., the water of reaction had evaporated. Twenty additional parts of silicone fluid were then added and the mixture heated with stirring to 330° F. Heating was terminated and the mixture was cooled to about 250° F. at which point 18 parts of phenyl alpha naphthylene amino and 12 parts of p,p'-dioctyl diphenyl amine were added. The resulting mixture was cooled to 80° F. and milled in a Morehouse Mill. The approximate composition and properties of the resulting grease are shown in Table III.

Table III

	1(A)	1(B)	2	3
Composition:				
Calcium Acetate			18.75	19.86
Calcium Benzoate	32.3	34.0	33	34.90
Lubricating Oil	62.7	66.0	40.30	36.50
Phenyl Naphthylamine	3		3.06	3.02
p,p'-Dioctyl diphenyl amine	2		2.02	2.02
Excess Hydrated Lime [1]			0.85	1.68
Barium Dinonyl Naphthylene Sulphonate			2.02	2.02
Properties:				
Appearance	[2]	[2]	[3]	[3]
ASTM Penetration at 77° F., mm./10—				
Unworked			333	267
Worked 60 strokesg	218	210	320	304
Neutralizatiln No. (percent as Oleic Acid)	5.6	5.9		
Alkalinity (percent as NaOH)			2.92	0.39
Dropping Point, ° F	500+	500+		500+
Lubrication Life [4]—				
450° F. and 10,000 r.p.m. (hrs.)	[5]	105	41	318
400° F and 10,000 r.p.m. (hrs.)				1,590
4-Ball Wear Test,[6] Scar Dia. (mm.)	1.37	1.52	0.50	0.33
Mean Hertz Load, E.P. Value			69+	48
			[7]	[7]

[1] Excess hydrated lime represents that lime in excess of the amount theoretically required to form the metal salt.
[2] Excellent, smooth, light gray.
[3] Excellent, smooth.
[4] ABEC–NLGI Spindle Test.
[5] Still running at 585 hours.
[6] 75° C., 70 kg., 1 hr., 1,800 r.p.m., steel on steel.
[7] No weld at 398 kg.

From Table III it can be seen that the addition of the amine inhibitors greatly enhanced the lubrication life of grease 1(A) as compared to grease 1(B). Grease 1(A) was particularly effective at lubricating bearings operating at high rotational speeds (10,000 r.p.m.) at 450° F. Its lubrication life is in excess of 585 hours. It can also be noted that the extreme pressure properties of Examples 1(A) and 1(B) are rather poor. These greases did not contain any fatty acid salt, e.g., calcium acetate. The grease shown in Example 2 has excellent wear characteristics but a very short lubrication life (41 hours). This short lubrication life is apparently due to the high alkalinity of the grease (2.92% calculated as NaOH). It is believed that the polyphenyl methyl siloxanes used are not stable in a strongly alkaline media. It has been noted that the lubrication life rapidly rises as the percent alkalinity of the grease is decreased. The grease shown in Example 3, which was only slightly alkaline (0.39% calculated as NaOH), had an excellent lubrication life at 450° F. as well as excellent extreme pressure properties. The grease of Example 1(A), which was acidic (5.6% calculated as oleic acid) had an even longer lubrication life. It is preferred that the grease be no more alkaline than about 0.50% calculated as NaOH. Preferably, the grease should be substantially neutral. Even more preferably, the grease should be slightly acidic. The importance of the acidity seems to include the apparent formation of amine salts by the amine inhibitors. These salts are much less volatile at the high temperature of spindle operation and hence a longer bearing life results (normally the amine inhibitors tend to volatilize at elevated temperatures).

The terms "alkalinity" and "acidity" as used herein refer to those phenomenon which are determined as follows: ten ml. of 0.5 N HCl are added to a 10 gm. charge of grease sample which is slurried in 100 ml. naphtha and 50 ml. ethanol. This is then back titrated with 0.5 N NaOH to a phenolphthalein end point. The results are then calculated and reported as percent NaOH or oleic acid.

The grease of Example 3 is a particularly desirable material. It has been compared with a commercially available premium grease (herein identified as product S). They were both tested in the same manner in an English rig (a high speed bearing test) at 392° F. and 100 lb. thrust load. The grease of Example 3 had a test life of 380 hours as contrasted with only 200 hours for product S.

Having thus described the invention with a certain degree of particularity, it will be realized that numerous modifications and adaptations may be made within the spirit and scope of the invention as hereinafter claimed.

I claim:

1. A soap-free lubricating grease composition ranging from an alkalinity of about 0.50% calculated as NaOH to an acidity of about 5.6% calculated as oleic acid, comprising:

(a) as the base oil, a lubricating oil selected from the group consisting of silicone and silane oils, and

(b) about 34 to 68 wt. percent of a grease thickener, said grease thickener consisting essentially of a mixture of alkaline earth metal salt of benzoic acid and alkaline earth metal salt of C_1 to C_6 fatty acid, and wherein said alkaline earth metal salt of said C_1 to C_6 fatty acid represents about 6 to 28 wt. percent of said mixture.

2. A lubricating grease composition as defined in claim 1, wherein said alkaline earth metal is calcium and wherein said base oil is a silicone oil.

3. A soap-free lubricating grease composition ranging from an alkalinity of about 0.50% calculated as NaOH to an acidity of about 5.6% calculated as oleic acid, consisting essentially of:

(a) a polymethyl phenyl siloxane as the base oil having a viscosity at 100° F. of from 20 to 400 cs.,
(b) from 19 to 47 wt. percent of calcium benzoate,
(c) from 11 to 22 wt. percent of calcium acetate,
(d) from 2 to 5 wt. percent of phenyl alpha naphthylamine, and

(e) from 1 to 3 wt. percent of p,p'-dioctyl diphenyl amine.

References Cited by the Examiner

UNITED STATES PATENTS

2,182,137	12/1939	Ricketts	252—41
2,671,758	3/1954	Vinograd et al.	252—39
2,976,242	3/1961	Morway	252—39
3,113,849	12/1963	McCoy	44—7

FOREIGN PATENTS

608,879	11/1960	Canada.

OTHER REFERENCES

"Manufacture and Application of Lubricating Greases," by Boner, Reinhold Pub. Corp., 1954, New York, pages 46–49 and 52–53.

DANIEL E. WYMAN, *Primary Examiner.*