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2,921,899

OXIDATION-RESISTANT LUBRICATING GREASES CONTAINING INORGANIC ALKALI METAL COMPOUNDS OF HIGH ALKALINITY

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The present invention relates to improved oxidation-resistant lubricating greases and to methods of preparing the same. The invention also relates to substantially anhydrous organic oleaginous materials, the oxidation-resistance of which is improved by the addition of the greases of the present invention.

In brief compass, the invention pertains to greases thickened to a grease consistency with a thickener containing a high proportion, preferably a major proportion, based on thickener, of a substantially oil-insoluble basic inorganic oxidation-inhibiting alkali metal compound having an original alkalinity of at least 21.5% Na_2O (as determined by the A.S.T.M. D-501-49 test). The poly-alkali metal salts of inorganic polybasic oxygen acids of silicon and phosphorus are preferred for the purposes of the invention.

Prior to the present invention it has been found that the poly-alkali metal phosphates and silicates of which trisodium phosphate is an outstanding example, have excellent oxidation-inhibiting characteristics when added to various organic materials, such as lubricating oils of mineral and/or synthetic origin, fatty oils, greases, asphalts, etc. Also, lithium hydroxide has been recommended as an oxidation-inhibitor for lithium stearate- and hydroxy stearate-greases. One of the prerequisites of the effectiveness of these oxidation-inhibitors is, of course, a substantial chemical inertness of the inhibitor toward the material to be protected against oxidation, so that no undesirable reaction between these materials will destroy or detrimentally change the one or the other.

For example, it has been found that the alkaline inhibitors mentioned above may be added to mineral oil base soda and lithium soap greases with good success either during grease manufacture or to the finished greases. However, when it was attempted to add lithium hydroxide or trisodium phosphate to lime soap bases, or even to mixed lithium-lime soap greases during the manufacturing stage, calcium metal was replaced in the soap by lithium, completely changing the character of the soap. Complicated milling procedures had to be employed in these and similar cases to incorporate the inhibitor into the finished grease.

Difficulties of equal magnitude may arise when the basic inorganic oxidation inhibitors are incorporated into certain synthetic oil base lubricating greases, independent of the type of thickener used. Many of the synthetic lubricating oils, particularly those of the dibasic acid ester and complex ester type, enter into undesirable side reactions with the alkaline inhibitors particularly at elevated temperatures and/or in the presence of appreciable amounts of water.

Also, in many cases it is difficult, if not impossible, to dissolve or adequately to disperse the inorganic inhibitor in mineral, fatty or synthetic oils as such. Thorough milling must be employed in these cases frequently com-

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bined with the use of special solubilizing or dispersing agents.

The present invention overcomes the aforementioned difficulties and affords various additional advantages. It is, therefore, an object of the present invention to provide for an improved method of increasing the oxidation resistance of organic oleaginous materials. Another object of the invention is to provide improved compositions containing basic inorganic compounds as oxidation inhibitors. Other and more specific objects and advantages of the invention will appear from the description given hereinafter.

The present invention is based on the discovery that oil-insoluble inorganic base alkali metal compounds having oxidation-inhibiting characteristics may be readily incorporated into metal soap-thickened mineral oil base greases in large proportions of as much as 50% by weight or more in the course of the manufacturing stage of the grease. More specifically, the mole ratio of inorganic inhibitor to soap should be at least 1 and may be as high as 10-20. The proportions of soap and inorganic compounds may be so chosen that a grease results which is thickened with an essentially inorganic type of grease thickener containing a small quantity of metal soap acting as a dispersing agent for the inorganic alkali metal compound in the dispersant oil.

Greases so prepared have good structure, smooth appearance and excellent high temperature stability in addition to exceptionally high oxidation resistance. They may be used as such for purposes normally requiring high temperature greases. However, these greases find their greatest utility as oxidation-inhibiting additives for other greases or various oleaginous organic materials in which the inorganic alkali metal compounds cannot be readily dispersed as such for the reasons set forth above. This utility is based on the surprising discovery that the greases of the invention are fully compatible with and readily dispersible in most organic oleaginous media without the need of arduous milling procedures.

It has also been found that the greases of the invention may be incorporated into oleaginous materials without detrimental side effects at conditions at which addition of the inorganic alkali metal compound inhibitor as such was found to be prohibitive as the result of undesirable side reactions. For example, the greases of the invention may be added in oxidation-inhibiting proportions to virtually all conventional grease stocks in any stage of the grease-making process. Also, the full benefit of the oxidation-inhibiting quality of the inorganic alkali metal compounds may be secured when the greases of the invention are added in oxidation-inhibiting proportions to such fatty or synthetic oils or compositions containing the same, as normally are reactive with the inorganic alkali metal compounds at conditions of preparation or use. Furthermore, the greases of the invention may serve to introduce oxidation-inhibiting proportions of the inorganic alkali metal compounds into liquid oleaginous materials without the use of additional solubilizer or dispersing aids.

The invention is broadly applicable to all inorganic alkali metal compounds of the proper alkalinity to exert an oxidation inhibiting effect on organic oleaginous materials which form carboxylic acids when undergoing oxidation by air. It has been found that an original alkalinity of the alkali metal compound of at least about 21.5% Na_2O is desirable for a prolonged oxidation-inhibiting effect. The compounds of most universal utility and consistently highest efficiency comprise the polyalkali metal salts of inorganic polybasic oxygen acids of silicon and phosphorus and, more particularly, the sodium and potassium salts of this type. By poly-metal

salts is meant those having at least two alkali metal atoms attached to a polybasic acid group. These salts are substantially oil-insoluble. Examples of suitable alkali metal salts are the ortho- and pyro-phosphates and the ortho-, sesqui-, di- and meta-silicates.

While the exact mechanism of this inhibition is not clearly understood, it appears that ordinary oxidation in air, of most oleaginous materials, e.g. of lubricating oils and greases, proceeds in at least two stages. These are (1) the formation of peroxides and (2) the decomposition of the peroxides to form sludge-producing organic acids. Evidently, the basic salts neutralize these acids as rapidly as they are formed. This prevents both the accumulation of sludge and the acceleration of the primary oxidation stage (peroxide formation) which usually occurs in the presence of such acids and sludge. The neutralization reaction proceeds in preference to any reaction of the basic salts with carbon dioxide present in the reaction medium or in the surrounding air.

The choice of salts varies somewhat with the temperature range for which stabilization is most important. In general, the alkali metal phosphates and silicates are most suitable for high temperature service, e.g. for temperatures from about 200° to about 400° F. or higher, because they are capable of decomposing stepwise in the presence of mineral oils to salts of lower metal content and alkalinity. Bases which react with CO₂ in preference to any reaction with other organic acids, such as most alkali and alkaline earth metal hydroxides, are much less satisfactory because they are converted to carbonates before they can react with the other organic acids, and carbonates are less efficient oxidation inhibitors. However, lithium hydroxide has been found particularly effective as an inhibitor for lithium greases. In many applications, the use of trisodium phosphate is critical for optimum results.

The liquid dispersant used in the grease-inhibitor concentrates of the invention should be an oil of lubricating grade which is chemically substantially inert toward the inhibitor compound used. Mineral oil base lubricating oils having a viscosity of at least 30 SSU at 100° F. are most suitable for this purpose. Certain synthetic hydrocarbon materials of lubricating oil grade derived from various conventional polymerization and/or alkylation processes or from the products of the distillation and/or hydrogenation of carbonaceous solids, the catalytic synthesis of hydrocarbons from carbon monoxide and hydrogen, etc. may likewise be used. Substituted hydrocarbonaceous materials, such as oxygen, halogen, nitrogen, sulfur and/or phosphorus derivatives of the above-mentioned materials, including fatty oils and fats, such as lard oil, sperm oil, etc. and their sulfur-containing derivatives, may form all or part of the grease dispersant.

A class of oleaginous materials which may serve as the dispersant of the greases of the invention comprises many synthetic oils of lubricating grade and viscosity (>30 SSU at 100° F.), particularly when combined with varying proportions of the mineral oils described above. Examples of synthetic oils include ether alcohols, such as those corresponding to the formula $RO(C_nH_{2n}O)_xH$ wherein R is an alkyl group, for example butyl, *n* is an integer from 1-5, for example 3, and *x* is greater than 1, for example 14; esters of monobasic carboxylic acids; totaling 20-130 carbon atoms, such as those of C₄-C₁₈ aliphatic acids with C₄-C₁₈ aliphatic alcohols, the C₄-C₁₈ radicals including the butyl, isobutyl, hexyl, octyl, iso-octyl, 2-ethyl hexyl, nonyl, decyl, lauryl, stearyl and similar radicals; di-esters of dibasic acids, such as adipic or sebacic acid with monohydric alcohols, such as hexyl, octyl, 2-ethyl hexyl or higher alcohols; esters of polyethylene glycols with C₈-C₁₃ branched-chain carboxylic acids; complex esters of polybasic carboxylic acids; polyhydric alcohols, monobasic acids and/or monohydric alcohols, such as the well known glycol-centered or dibasic

acid-centered complex esters of suitable viscosity; phosphoric acid esters or thioesters of aliphatic alcohols or mercaptans of up to about 18 carbon atoms; halocarbon oils, such as polymers of chlorofluoro alkylenes, e.g. chlorotrifluoroethylene; organo-siloxanes; sulfite esters; organic carbonates; mercaptals; formals; etc. All these synthetic oils are well known in the art and require no further identification for those skilled in the art.

Grease thickeners useful for the preparation of grease-inhibitor concentrates in accordance with the present invention include the metal soaps conventional for grease-making, and more particularly the alkali metal, alkaline earth metal and mixed alkali metal-alkaline earth metal soaps, e.g. the sodium, potassium, lithium and/or calcium soaps, of fatty acids, such as stearic, oleic, palmitic or other fatty acids having 12-30 carbon atoms per molecule, as well as substituted acids, such as the hydroxy, poly-hydroxy, keto, alkyl and alkoxy derivatives of such fatty acids. Various metal salts, particularly alkali metal and/or alkaline earth metal salts, of low molecular weight carboxylic acids having 1-5 carbon atoms, such as sodium or calcium acetate or crotonate may be combined with these soaps in various proportions to form complex soap-salt thickeners in a manner known per se. Preferably, the alkali metal of the soap is the same as that of the alkali metal compound inhibitor. For example, a soda-soap is normally used in conjunction with trisodium phosphate and a lithium soap in conjunction with lithium hydroxide, etc.

As indicated above, the grease-inhibitor concentrates of the invention may contain as much as 50% by weight or more of the inorganic alkali metal compound inhibitor. More specifically, the grease should contain about 15-80% by weight of this compound, the preferred range being about 20-50% by weight of the grease, depending on the intended use of the latter. The soap-thickener proportion of the grease may likewise vary over a wide range but should, in general, not exceed equimolar proportions as compared to the inhibitor compounds. Mole ratios of inorganic inhibitor to soap of about 10-20 are preferred. Quantities of about 1-25 wt. percent of soap thickener are suitable in most cases, soap concentrations of about 5-20 wt. percent, based on total grease being preferred. The remainder of the grease is a lubricating oil of the type specified above and may contain small amounts of other conventional additives which are not incompatible with the inorganic inhibitor compound, such as other oxidation inhibitors, extreme pressure agents, oiliness additives, pigments, etc.

The grease-inhibitor concentrates of the invention may be prepared by essentially conventional grease-making procedures. For example, the fatty acid or fat used to supply the soap constituent of the grease may be heated in a lubricating oil, preferably a mineral lubricating oil, to about 180°-220° F. in a conventional grease kettle. Caustic soda or other alkali metal hydroxide may be added, preferably as an approximately 50° Bé. aqueous solution to the hot solution to neutralize the acid or saponify the fat. This soap stock may then be partially dehydrated by further heating to about 300°-350° F. while stirring. Thereafter, the inorganic inhibitor compound in aqueous solution is added to the hot soap concentrate, dehydrated at 300°-400° F. and the remainder of the lubricating oil added. Stirring is continued while the grease is allowed to cool to about 200° F., and the grease may then be poured into pans or commercial package containers. Alternatively, the method outlined above may be varied by heating the grease until molten (400°-500° F.) and then casting the melt into pans. The grease-inhibitor concentrate after cooling may be homogenized, such as by stirring in a conventional grease kettle.

As pointed out before, the grease-inhibitor concentrates of the invention may be used as lubricating greases with-

out further additions. They may also be used as inhibitor concentrates to be added to other organic oleaginous materials which form organic acids when oxidized by air. When used for the latter purpose, the greases of the invention should be added in amounts sufficient to introduce about 0.1–15 wt. percent, preferably about 0.5–10 wt. percent, of the alkali metal compound inhibitor into the oleaginous material to be protected against oxidation. Organic oleaginous materials which may be so improved include the hydrocarbon oils of mineral or synthetic origin, their oxygen, halogen, nitrogen, sulfur and/or phosphorus derivatives, the fatty oils and fats, as well as the synthetic lubricating oils mentioned above as dispersants of the grease-inhibitor concentrate of the invention, as well as asphalts, waxes, vegetable oils, etc. No additional solubilizer or dispersing aid is required for an adequate and substantially permanent oxidation-inhibiting effect.

An even more important application of the grease-inhibitor concentrates of the invention is their use as inhibitor concentrates for other lubricating greases, one or more constituents of which tend to react with the alkali metal compound inhibitors as such or which are in some other respect detrimentally affected by the direct addition of the inorganic alkali metal compound inhibitor. For example, these concentrates may be used to introduce about 0.1–15 wt. percent, or more, of lithium hydroxide or sodium phosphate or silicate into all types of synthetic oil base greases and all types of greases thickened with non-soap type organic thickeners, such as carbon black, cellulose derivatives, etc. or inorganic thickeners, such as silica gel, amorphous silica, bentonitic and Attapulgis clays and conventional modifications thereof, such as bentonites modified by quaternary ammonium bases, Attapulgis clay modified by small amounts of surface active agents; calcium silicate, etc. These inorganic thickeners may contain waterproofing agents, such as various silicone oils, sodium methyl silicate; chloromethyl silane; resins, such as alkyl resins; C_{2-18} primary or secondary alcohols etc.

Regarding suitable methods of incorporating the grease-inhibitor concentrates of the invention into non-compounded organic materials of the type mentioned above, it is merely necessary to add the grease of the invention to the liquid or liquefied organic material while stirring. Heating is necessary only where required to liquefy the base material. The concentrate is added to the grease which is to be inhibited and the mixture thus obtained may be homogenized by stirring in a grease kettle or by passing through a colloid mill. The addition is preferably carried out at room temperature of, say, 60°–80° F., but elevated temperature of up to about 300° F. may be used.

The invention will be best understood from the following description of specific embodiments and working examples thereof.

EXAMPLE I

A soda-soap grease containing a large quantity of sodium phosphate was prepared having the following composition:

	Percent by weight
12-Hydroxystearic acid	7.0
Candelilla wax	2.8
Sodium hydroxide, anhydrous	2.8
Trisodium phosphate, anhydrous	37.0
Mineral oil (V./210=110 SUS; V.I.=90)	49.0
Phenyl alpha-naphthylamine	1.4

Preparation of the grease

Twenty grams of 12-hydroxystearic acid and 8 grams of candelilla wax were dissolved in 64 grams of mineral oil at 180°–200° F. Eight grams of sodium hydroxide dissolved in 20 grams of water were added to neutralize the fatty acid. The free alkali on the soap was approxi-

mately 0.5%. Two hundred and forty grams of trisodium phosphate dodecahydrate were dissolved in an equal weight of water and then added to the soap stock. The soap-salt complex was then partially dehydrated by heating to 300° F., whence the remainder of the mineral oil was added (75 grams). The phenyl alpha-naphthylamine was added at this stage. Dehydration was then completed by heating the grease to 300° F. In some cases, traces of water may be required to maintain the grease-like consistency. The candelilla wax serves merely as a dispersing agent.

Inspections on the base grease

15	Micropenetration, mm./10	38
	Dropping point, ° F.	450+
	Grease hardening life at 300° F., hours. ¹	3200+

¹ Grease micropenetration was only 25 when test discontinued.

EXAMPLE II

A grease-inhibitor concentrate was prepared containing sodium phosphate and calcium stearate soap. The composition of this grease was as follows:

	Grams
25. Calcium stearate -----	57
Mineral oil -----	30
Na ₃ PO ₄ ·9H ₂ O -----	20

The grease was prepared by adding the sodium phosphate in aqueous solution to the calcium stearate-oil mixture and heating to 300° F. When dehydration was complete, a hard grease was obtained which is useful as an anti-oxidant.

EXAMPLE III

A lithium stearate grease having the following composition was prepared by heating the ingredients to 400° F. and casting:

	LiOH.H ₂ O	Percent
	-----	28.4
40	Stearic acid	6.8

	Mineral oil (V./100:300 S.U.S.)	64.8

This grease titrated 27.5% free LiOH.

To show the effectiveness of this grease as an anti-oxidant, 4% of the grease was added to a grease containing 9% of a mixed calcium-lithium (80:20) hydroxystearate soap. This grease was evaluated at 250° F. by the dish oxidation test.¹ After 600 hours at this temperature, it is still a light-colored product and shows no oil separation. The same grease containing no inhibitor concentrate is of brown-black color after 480 hours, showing advanced oxidation, and bleeds a large amount of oil.

EXAMPLE IV

Finely divided Attapulgit clay was mixed with mineral oil of 500 S.S.U. viscosity at 100° F. and 90 V.I. and a small amount of commercial surface active materials containing hydrophilic groups (OH or NH₂ groups) and oleophilic groups (alkyl groups). The mixture was heated under pressure to about 300° F. and milled while preventing water losses (grease A). To one portion of this grease A about 2.7 wt. percent of the grease of Example I was added by mixing at room temperature (grease B). To another portion of grease A about 9.3 wt. percent of the grease of Example I was added by mixing at room temperature (grease C). Into a third portion of grease A there was incorporated about 3.8 wt. percent of anhydrous trisodium phosphate which was dispersed in oil by ball-milling (grease D). Comparative test results obtained with these greases are summarized in Table I below.

¹ Dish oxidation test: 30 grams of grease are placed in a 2'' x 2'' x 1/2'' stainless steel dish so that none of the grease touches the edge of the dish. The pans are placed in an oven at 250° F. Periodically color degradation, oil separation, grease consistency and structure are measured. The life of the grease is arbitrarily taken when the grease becomes fluid.

EXAMPLE V

A modified bentonitic clay powder prepared by treating bentonite with a quaternary ammonium alkyl salt in a conventional manner was added to mineral oil of 1200 S.S.U. at 100° F. and 90° V.I. along with about 1.3 wt. percent of acetone based on total composition. After stirring at room temperature for ½ hour, the grease was milled (grease E). One portion of this grease was mixed at room temperature with about 2.8 wt. percent of the grease of Example I (grease F). These greases were tested with the results shown in Table I below.

EXAMPLE VI

Two lubricating greases were prepared by mixing finely divided amorphous silica with mineral oil and passing through a colloid mill. The mineral oil was a solvent refined distillate having a viscosity of about 500 S.S.U. at 100° F. and a V.I. of 90. The amorphous silica was finely divided water-resistant substantially anhydrous silica (containing 87% SiO₂ and 13% waterproofing agent) manufactured and sold by DuPont under the trade name "Du Pont Hydrophobic Silica" and described in U.S. Patent Nos. 2,657,149 and 2,676,148.

For a proper understanding of the data summarized in Table I it should be kept in mind that a grease which has hardened to a micro penetration of about 5 as the result of oil oxidation and evaporation, will permit bearing failure in the high speed spindle test. It will be noted that grease A which did not contain the grease-inhibitor concentrate of the invention reached a micro-penetration of 5 after about 700–800 hours at 300° F. while grease B which contained a small amount of the grease-inhibitor concentrate was still soft enough to lubricate (micro penetration of 12) after about 1200 hours. Similar comparisons favorable to the greases of the invention may be made for the other greases listed.

EXAMPLE VIII

In order to determine the relationship between inorganic inhibitor concentration and grease characteristics, a number of greases were prepared by adding varying proportions of trisodium phosphate to a sodium hydroxystearate grease, substantially in the manner described in Example I. The composition and properties of these greases are summarized in Table II below.

TABLE II.—EFFECT OF TRISODIUM PHOSPHATE ON DROPPING POINT AND GREASE HARDENING LIFE AT 400° F.

Designation.....	1	2	3	4	5	6	7	8	9
Formula, Percent by weight:									
12-hydroxystearic acid.....	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0
Candelilla wax.....	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0
Sodium hydroxide.....	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2
Trisodium phosphate.....		3.2	6.4	9.6	12.8	16.0	19.2	25.6	38.4
Mineral oil, V./210=110, V.I.=90.....	67.8	65.6	61.4	59.2	55.0	52.8	49.6	42.2	29.4
Phenyl alpha-naphthylamine.....	1.0		1.0		1.0			1.0	1.0
Inspections:									
Worked penetration.....	236	215	270	270	214	270	265	207	80
Dropping point, ° F.....	380	400	500	530+	498	500+	500+	500+	500+
Grease hardening life at 400° F. (hours to a micro penetration of 5).....	<1	20	215	210	230	170	100	120	25
Mole ratio of trisodium phosphate to fatty acid.....		¼	½	¾	1	1¼	1½	2	3

One of the greases was mixed at room temperature with about 19.0 wt. percent of the grease of Example I and contained about 9.8 wt. percent of silica (grease G). The other grease contained about 12 wt. percent of silica and no organic alkali metal compound inhibitor (grease H). These greases were likewise tested as tabulated below in Table I.

EXAMPLE VII

An acetylene carbon black was stirred at room temperature into mineral oil of 300 S.S.U. at 100° F. and 75 V.I. This grease was mixed at room temperature with about 2.8 wt. percent of the grease of Example I (grease I). The grease was tested as reported in Table I below.

EXAMPLE IX

Further experiments were carried out to determine the influence of oil viscosity on grease stability. For this purpose, several greases were prepared substantially by the method outlined in Example I using base oils of

TABLE I

Grease.....	A ¹	B ¹	C ¹	D ¹	E	F	G	H	I
Composition:					6.5	5.8	9.8	12	
Bentonite.....									
Amorphous silica.....	11.40	11.40	11.40	11.40					7.0
Attapulgit.....									
Carbon black.....							70.2	87.0	90.2
Mineral oil—									
V./100:300.....	83.11	80.49	73.81	79.31	91.7	90.1			
V./100:500.....						2.8	19.0		2.8
V./100:1,200.....		2.7	9.3	3.8 (ball-milled Na ₂ PO ₄)	1.5	1.3			
Grease of Example I (37%Na ₂ PO ₄).....							1.0	1.0	
Acetone ²	1.0	1.0	1.0	1.0					
Phenyl alpha-naphthylamine.....					310	305	310		
Inspections:					87	90	94	75	
Worked penetration.....	310	290	308	308					
Micro penetration.....	92	80	90	90					
Grease hardening tests:					500-700	2,160	2,100	500	1,400
Hours to a micro-penetration of 5.....	700-800	12	61 at 900 hrs.	43 at 900 hrs.					
Micro-penetration at 1,200 hours.....									

¹ Balance of these compositions (4.49%) consists of surface active agents which assist the dispersion of the Attapulgit in the oil.

² As a dispersion aid.

³ 5 grams of grease are packed into a 204 bearing, which is then stored at 300° F. in air. At intervals, the grease is removed from the bearing and its micro-penetration is measured. In general, when the grease reaches a micro-penetration of 5 the bearing, in which it is the lubricant, will fail.

varying viscosity. The composition and properties of these greases are summarized in Table III below.

cent of a material selected from the group consisting of trisodium phosphate and lithium hydroxide and about

TABLE III.—EFFECT OF MINERAL OIL VISCOSITY ON GREASE HARDENING LIFE OF GREASES CONTAINING TRISODIUM PHOSPHATE¹

Designation.....	10	11	12	13	14
Formula, percent by weight:					
Sodium hydroxide.....	3.0	2.8	3.0	3.0	2.5
12-hydroxystearic acid.....	20	22		20	
Double pressed stearic acid.....			20		16.7
Trisodium phosphate.....	11.5	11.7	11.5	11.5	9.6
Mineral oil—					
V./210=65, V.I.=90 ²	63.5				
V./210=110, V.I.=90 ²		61.5	63.5		
V./210=140, V.I.=100 ³				63.5	
V./210=190, V.I.=100 ⁴					69.5
Sodium Sulfonate (63%).....	2.0	2.0	2.0	2.0	1.7
Inspections:					
Mineral oil viscosity at 210° F., S.U.S.....	65	110	110	140	190
Micro penetration at 77° F.....	40	69	55	85	69
Dropping point, ° F.....	498	470+	448	500+	500+
Grease hardening life at 400° F., hours.....	115	212	230	245	250

¹ Mol ratio of fatty acid to trisodium phosphate 1:1.

² Solvent extracted and dewaxed.

³ Solvent extracted.

⁴ Steam reduced residue.

It will be noted that the grease hardening life increased with the viscosity of the base oil up to about 140 S.S.U. at 210° F. Further increases in viscosity had little effect. It is desirable, therefore, to use base oils having viscosities of about 130–150 S.S.U. at 210° F. for the greases of the present invention.

It will be understood that the improved oleaginous materials prepared in accordance with the invention may contain other conventional modifiers, such as other antioxidants, e.g. phenyl alpha-naphthylamine; corrosion inhibitors, e.g. sorbitan esters of fatty acids; tackiness agents, e.g. polyisobutylene of about 1,000 mol. wt.; viscosity index improvers, e.g. polyisobutylene of about 15,000–20,000 mol. wt.; detergents, e.g. calcium sulfonate, sodium sulfonate, etc.; extreme pressure agents, e.g. sulfurized sperm oil or chlorinated wax; etc.

The invention is not limited to the specific figures of the foregoing examples. The relative proportions of the materials used and the reaction conditions may be varied within the limits indicated in the specification to obtain products of varying characteristics.

What is claimed is:

1. A method of inhibiting a lubricating grease against oxidation wherein said lubricating grease comprises a major proportion of a lubricating oil and a minor grease thickening amount of a calcium soap thickener, which method consists of mixing into said grease an amount sufficient to inhibit oxidation of said grease of a grease-inhibitor concentrate comprising about 15 to 80 wt. per-

1 to 25 wt. percent of an alkali metal soap of C₁₂ to C₃₀ fatty acid and a dispersant proportion of a mineral lubricating oil.

2. A method according to claim 1, wherein said lubricating grease contains a mixed calcium-lithium soap thickener.

3. A method according to claim 1, wherein said material is trisodium phosphate.

4. A method according to claim 1, wherein said material is lithium hydroxide.

References Cited in the file of this patent

UNITED STATES PATENTS

2,079,051	Sullivan et al.	May 4, 1937
2,434,539	Beerbower et al.	Jan. 13, 1948
2,455,659	Duncan et al.	Dec. 7, 1948
2,605,225	Armstrong	July 29, 1952
2,626,896	Dilworth	Jan. 27, 1953
2,626,899	Abrams	Jan. 27, 1953
2,648,633	Peterson et al.	Aug. 11, 1953
2,684,944	Zajac	July 27, 1954
2,738,329	Parry	Mar. 13, 1956
2,748,081	Peterson et al.	May 29, 1956

FOREIGN PATENTS

160,330	Australia	Aug. 7, 1952
850,051	Germany	Sept. 22, 1952
706,555	Great Britain	Mar. 31, 1954