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LITHIUM SOAP GREASE CONTAINING BASIC ALKALINE EARTH METAL SULFONATE

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This invention relates to a lithium base lubricating grease composition, and particularly to such a composition which is inhibited against copper corrosion.

Lithium base lubricating grease compositions have found substantial use in aircraft controls and for other purposes where operation over a wide temperature range, and particularly operation at extremely low temperatures, is encountered. U. S. Patent No. 2,450,221, Ashburn, Barnett and Puryear, is typical of a superior lithium base grease of this type prepared from a lithium soap of a hydroxy fatty acid or the glyceride thereof, such as hydrogenated castor oil, and containing as the major proportion of the liquid lubricating base an oil soluble high molecular weight high boiling liquid aliphatic dicarboxylic acid ester within the lubricating oil viscosity range and possessing lubricating properties. As disclosed therein, the lithium soap may be formed from a major proportion of the hydroxy fatty acid or glyceride thereof, and a minor proportion of a saturated fatty acid, the grease containing a small excess of free fatty acid. Such greases have exceptional shear and texture stability over a wide temperature range and excellent low temperature properties.

While the lithium base greases of the aforesaid type have proved eminently satisfactory in service, the increased use of copper and copper alloys in certain applications, particularly in aircraft controls, has introduced an additional problem of rendering the grease non-corrosive to copper in long time service. For this purpose, U. S. Army specification 2–134 has prescribed a rigorous copper corrosion test for qualification under this specification. In attempting to meet this specification, it has been found that corrosion inhibitors heretofore employed in greases are ineffective.

One of the principal objects of the present invention is to provide a lithium base grease which satisfactorily meets the copper corrosion test of U. S. Army specification 2–134.

Another object of the present invention is to

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provide a lithium base grease which retains all the desirable properties of the previously known greases of this type, including shear and texture stability over a wide temperature range and excellent low temperature properties, and at the same time is effectively inhibited against corrosion of copper and copper alloys in long time service.

In accordance with the present invention, the foregoing objects have been attained by incorporation in the lithium base grease about 0.25 to 3% by weight of an oil soluble basic alkaline earth metal salt of a sulfonated hydrocarbon wherein the weight ratio of alkaline earth metal to sulfur content is from 1.1 to 2 times that represented by the normal salt $M(SO_3R)_2$, where M is the alkaline earth metal and R is the hydrocarbon residue. The foregoing salt is to be distinguished from the previously used normal alkaline earth metal sulfonates, such as calcium mahogany sulfonate, which latter have been found to be completely ineffective for purposes of the present invention.

The basic alkaline earth metal sulfonates which are effective copper corrosion inhibitors for purposes of the present invention, are known compounds which have heretofore been suggested along with the normal mahogany sulfonates as detergent additives for liquid lubricants, such as motor oils. However, it was not to be anticipated that the basic alkaline earth metal sulfonates would function as copper corrosion inhibitors in this different environment of a lithium base lubricating grease composition such as to pass the rigorous copper corrosion test of the said 2–134 specification, particularly when the better known and more generally used mahogany sulfonates proved completely ineffective for this purpose.

The basic sulfonates employed in accordance with this invention may be prepared by sulfonation of a lubricating oil stock or other mineral hydrocarbon oil, by contacting with sulfuric acid of high concentration at an elevated temperature of the order of 150–180° F., to produce substantial

sulfonation without excessive loss to acid sludge. The resulting sulfonated oil, after separation of the acid sludge, may be reacted directly with powdered hydrated alkaline earth metal oxide in an amount substantially in excess of that required to neutralize the oil. Preferably, however, the basic sulfonate is formed by first purifying the sulfonated oil by neutralizing with sodium hydroxide, extracting the sodium sulfonate with an aqueous alcohol such as isopropyl alcohol, separating the aqueous alcoholic extract from the residual oil, precipitating the sulfonic acids by acidifying with sulfuric acid, filtering and forming a lubricating oil solution of the sulfonic acids, and finally reacting with an aqueous solution of a water soluble alkaline earth metal salt. Thus, in preparing a basic barium sulfonate, an aqueous solution of barium chloride with the latter in substantial excess of that required for neutralization of the sulfonic acids, is added to the oil solution. The reaction mix is subjected to steaming with agitation for a substantial period of time, such as about 1–4 hours, to form the basic barium sulfonate, which is generally a material of intermediate chemical composition between the truly basic barium sulfonate $BaOHSO_3R$ and the normal barium sulfonate $Ba(SO_3R)_2$. Following separation of the aqueous phase, the lubricating oil solution of the basic barium sulfonate is washed and then heated to remove residual moisture to obtain a lubricating oil concentrate of the desired additive in about 25–50% concentration. The basic salt of sulfonic acid may also be made by reacting an oil solution of the sulfonic acid with a methanol solution of the base (e. g. barium hydroxide) and then removing the methanol. In general, the weight ratio of alkaline earth metal to sulfur content in the desired material for purposes of the present invention will be in excess of 1.1 times that represented by the normal salt $M(SO_3R)_2$ and up to two times that represented by the said normal salt.

While any of the alkaline earth metal basic salts of a sulfonated hydrocarbon, including those of calcium, strontium and magnesium, can be employed, the basic barium sulfonate is preferred. Moreover, while the hydrocarbon selected for sulfonation is generally a mineral oil fraction, particularly a lubricating oil distillate or solvent extract thereof, this is not essential. Other oil soluble hydrocarbon sufonic acids suitable for preparing basic sulfonates of the present invention may be produced by sulfonating alkyl aromatic hydrocarbons, such as alkyl benzenes and alkyl naphthalenes, wherein the alkyl group or groups in the side chains have at least about 8 carbon atoms and preferably more to provide oil solubility. Thus an alkyl aromatic hydrocarbon suitable for the production of the desired oil soluble sulfonic acids may be formed by condensation of aromatic hydrocarbons with chlorinated paraffin wax, alkyl chlorides such as chlorinated kerosene, polymerized olefins such as polypropylene, fatty alcohols etc. Suitable condensing agents are the Friedel-Crafts catalysts, sulfuric acid and the like.

The above-described basic alkaline earth metal sulfonates may be employed in lithium base greases formed from any saturated fatty material, such as the conventional saturated fatty acids or glycerides thereof, to provide copper corrosion inhibition.

Preferably, the lithium base greases formed from hydroxy fatty acids or their corresponding glycerides, such as hydrogenated castor oil, are employed to obtain the improved shear and texture stability. Suitable types of soap-forming hydroxy fatty acids and glycerides thereof are disclosed in said Patent No. 2,450,221. While the soap-forming hydroxy fatty acids or their glycerides may be used as the sole acidic component of the lithium soap, they may be blended or combined with the conventional saturated fatty acids or fats in such proportions that at least 50% of the total acidic component is comprised of the hydroxy fatty acids or glycerides thereof. In general, any of the recognized fatty acid materials normally used in grease manufacture may be employed providing they are essentially saturated in character. These fats and fatty acids include mixtures of fatty acid glycerides found in naturally occurring fats and oils, together with fractionated components thereof. The fatty acids may be a mixture of acids split off from these fats or prepared from hydrogenation of fish oils, or the individual acids themselves. Very satisfactory results are secured by employing as the saturated fatty material for the formation of the lithium soap a mixture of about 60–80% of hydrogenated castor oil or 12-hydroxy stearic acid with 40–20% of a saturated fatty acid such as stearic acid. The grease may contain about 10–30% by weight of the lithium soap.

In order to obtain the improved wide temperature range and low temperature properties, the liquid lubricating base of the lithium grease composition is preferably formed of a major proportion of a synthetic oleaginous lubricating compound or condensation product, many types of which are now known in the art. Very satisfactory synthetic lubricants of this character are represented by the high molecular weight high boiling liquid aliphatic dicarboxylic acid esters which are within the lubricating oil viscosity range and possess lubricating properties. The compounds within this particular class are the esters of such acids as sebacic, adipic, pimelic, azelaeic, alkenyl succinic, alkylmaleic, etc. The esters thereof are preferably the aliphatic esters and particularly the branched chain aliphatic diesters. Specific examples of the preferred oleaginous compounds are di-2-ethyl hexylsebacate, di-2-ethyl hexyl azelate, di-2-ethyl hexyl adipate, di-sec-amyl sebacate, di-2-ethyl hexyl alkenyl succinate, di-2-ethoxyethyl sebacate, di-2-(2'-methoxyethoxy) ethyl sebacate, di-2-(2'-ethyl butoxy) ethyl sebacate, di-2-butoxy ethyl azelate, di-2-(2'-butoxy ethoxy) ethyl alkenyl succinate, etc.

These oleaginous compounds may be used as the sole oil component of the grease or they may be blended with a mineral lubricating oil. Where a blend is employed, and low temperature properties are required, the mineral lubricating oil is preferably a light refined distillate mineral lubricating oil, such as a naphthene or paraffin base distillate, having an SUS viscosity at 100° F. of about 50–130 and preferably about 100 secs. The mineral lubricating oil will generally constitute less than 50% of the blend, and ordinarily about 40–20% thereof. The mineral lubricating oil blend is advantageous where the lithium soap is formed in situ. In such case, the saponification of the fatty material with the lithium hydroxide and dehydration of the resulting soap are conveniently carried out in the presence of a portion of the mineral lubricating oil, and the synthetic lubricant together with the additives employed in the grease are then added following dehydration and as the agitated soap mix cools.

The following examples are given to illustrate the present invention:

EXAMPLE 1

A lithium base grease was prepared from a fatty material consisting of about 75% by weight of hydrogenated castor oil and 25% by weight of stearic acid. The lubricating base used was a mixture of about 75% by weight of di-2-ethyl hexyl sebacate with about 25% by weight of a naphthene base mineral lubricating oil having an SUS viscosity at 100° F. of about 50–60. The method of preparation involved charging a steam-heated kettle with the required amount of 10.3% lithium hydroxide solution together with a small amount of water and heating to about 180° F. The hydrogenated castor oil was then added together with a small proportion of the naphthene base mineral lubricating oil, the latter being less than the amount of hydrogenated castor oil. The kettle contents were held at 170–190° F. for about 4 hours while agitated, and then the stearic acid was added and the temperature maintained with stirring for another 1–2 hours to complete saponification. The saponified mix was then heated with stirring at about 290–330° F. for about 4 hours to effect dehydration. The balance of the mineral lubricating oil was then added with stirring as the kettle contents cooled down to about 270° F. Finally, the di-2-ethyl hexyl sebacate was slowly added with stirring during further cooling, and any additives were then introduced into the grease before the latter was drawn at a temperature below 200° F.

A series of greases were prepared in the foregoing manner, to each of which was added about 0.5% by weight of phenyl alpha naphthylamine as an oxidation inhibitor, together with various additives of the corrosion inhibiting type, in an effort to produce a grease meeting the copper corrosion test of the 2–134 specification. The consistencies of the various greases were adjusted within the ball and roller bearing grease range, varying from a soap content of about 12.5% to about 18% by weight. As pointed out above, each of the greases had an approximately 3:1 weight ratio of lithium soap of hydrogenated castor oil to lithium stearate, and had an approximately 3:1 weight ratio of di-2-ethyl hexyl sebacate to mineral lubricating oil. The following Table I shows the results obtained on these greases in the listed 2–134 corrosion test employing the various additives in the base grease and in the weight percent as listed in the table.

Table I

Additive Weight Percent	2-134 Corrosion Test		
	Copper	Grease	Result
1. None	Light brown	Slightly darker	Fail.
2. Tallow (4.1%)+Ca mahogany sulfonate (6.6%).	Greenish brown	Darker	Do.
3. Lauryl acid orthophosphate (1.0%)+Ca mahogany sulfonate (3.0%).	Dark brown	Black near copper.	Do.
4. Na mahogany sulfonate (2.7%)	do	Slight discoloration.	Do.

The 2–134 corrosion test of the foregoing table was run by placing a copper strip in a Norma-Hoffman bomb so as to be partially immersed in a sample of the grease under test, and then maintaining the bomb under oxygen pressure (110 pounds per square inch initial pressure) at 210° F. for 20 hours. During that period, no pressure drop due to oxygen absorption must occur. Then at the completion of the 20-hour period, both the sample of grease and copper strip are inspected. In addition to the requirement for no pressure drop during the test, there must be no more than a very faint stain on the copper strip and no more than a slight stain on the grease in order to be rated as passing. Slightly inferior products which do not merit a full "pass" rating are given a rating of "borderline to pass" where there is no more than a light stain on the grease and no pressure drop; and a rating of "borderline to fail" where there is a stain on the copper with or without a stain on the grease and no pressure drop. All other results are rated as "fail."

Since the foregoing greases of Table I, including the base grease of sample 1 which contained no corrosion inhibitor, all were compounded with about 0.5% by weight of the effective oxidation inhibitor phenyl alphanaphthylamine, they all satisfactorily met the requirement for no pressure drop during the test, and this item is not separately listed in the table. It is to be noted that the base grease of sample 1 produced a light brown stain on the copper strip while the grease was also slightly discolored, and thus failed the test. Sample 2 containing a combination of 4.1% of tallow with 6.6% of conventional calcium mahogany sulfonate, which combination is recognized to be an effective corrosion inhibitor in other relationships, also failed the test. Sample 3 containing 1% of lauryl acid orthophosphate with 3% of the conventional calcium mahogany sulfonate, both of which are recognized corrosion inhibitors in other relationships, also failed the test. Sample 4 containing 2.7% of sodium mahogany sulfonate was a clear failure in this test.

SAMPLE 2

A base grease was prepared in the foregoing manner of Example 1 and had the following calculated composition:

	Weight percent
Lithium soap of hydrogenated castor oil	16.75
Lithium stearate	5.58
Excess LiOH	0.05
Paraffin base mineral lubricating oil having SUS at 100° F. of 102.4	18.87
Di-2-ethyl hexyl sebacate	56.31
Phenyl alpha naphthylamine	0.72
Glycerin (from the fat)	1.68
Dye	0.0003

To the foregoing base grease, various additives were added in the percentages as shown in the

following Table II, and the resulting grease samples tested in the 2–134 corrosion test with the indicated results:

Table II

Additive Weight Percent	2-134 Corrosion Test		
	Copper	Grease	Result
1. None	Light brown	Faint lavender around copper.	Fail.
2. Mercaptobenzothiazole (0.25%).	do	No change	Do.
3. N,N'-disaliculidene-1,2 diaminopropane (0.25%).	do	Dark gray	Do.
4. Alkyl Maleic Acid (0.25%)	Dark brown	Purple	Do.
5. Sulfonated Tallow (0.5%)	Non-uniform stain.	Lavender	Do.
6. 4-methyl 2,6-ditertiary butyl phenol (0.5%).	Dark brown	Slightly darker	Do.
7. Ca phenol sulfonate (0.5%)	Unchanged	Light lavender	Borderline to fail.
8. Sorbitan mono-oleate (0.5%)	Light stain	Unchanged	Do.
9. "Santolube 203A" (1.23%)	Unchanged	do	Pass.
10. "Santolube 203A" (0.5%)	Very slight stain.	Very slight stain.	Borderline to pass.
11. "Santolube 203A" (0.25%)	do	Very slight darkening.	Borderline to fail.

In the foregoing Table II, "Santolube 203A" indicates a commercial product manufactured by Monsanto Chemical Company and is a lubricating oil concentrate containing approximately 50% by weight of a basic barium derivative of a sulfonated hydrocarbon which is representative of the satisfactory types of basic alkaline earth metal sulfonates of the present invention. A typical sample of "Santolube 203A" gave the following tests:

Viscosity SUS at 210° F	55.5
Color Lovibond ½ in. cell	660.0
Chlorine, weight per cent	0.13
Sulfur, weight per cent	1.03
Barium, weight per cent	4.15

As will be noted, the weight ratio of barium metal to sulfur content calculates to 4.03, as against a theoretical ratio for the normal barium sulfonate of 2.14 and for the completely basic barium sulfonate of 4.28. Other typical basic barium sulfonates tested, and which have been effective for purposes of the present invention have had weight ratios of barium metal to sulfur contents of 3.27 and 2.69 respectively. In general, it can be stated that the weight ratio of alkaline earth metal to sulfur content in the effective materials may vary between about 1.1 up to two times the theoretical weight ratio for the normal salt. It thus appears that the truly basic compound is not necessary, but a complex material which is basic in character so as to have a weight ratio of the alkaline earth metal to sulfur content which is in excess of 1.1 times the theoretical ratio for the normal salt, is satisfactory for purposes of the present invention. These satisfactory materials are termed herein "basic alkaline earth metal sulfonates."

Referring again to the foregoing Table II, sample 1 shows that the base grease fails the 2–134 corrosion test. Samples 2–8 show that various additives which are recognized as highly effective corrosion inhibitors in other environments, when added to the said base grease, failed to produce compositions which passed this rigorous test. On the other hand, samples 9–11 show that the basic barium sulfonate was unexpectedly effective in a proportion of about 0.25% and over in enabling the grease to pass this rigorous test.

EXAMPLE 3

In order to illustrate the effect of free acidity or free alkalinity in a lithium base grease containing a basic alkaline earth metal sulfonate of the present invention, the following Table III is listed. This shows the results obtained on 3:1 lithium soap of hydrogenated castor oil-lithium stearate greases containing 3:1 mixture of di-2-ethyl hexyl sebacate and paraffin base mineral lubricating oil, together with 0.5% phenyl alpha naphthylamine and 1% "Santolube 203A" (except that sample 5 contained 1.5% "Santolube 203A").

Table III

	Percent Free Fatty Acid as Oleic	Percent Free Alkalinity as LiOH	2-134 Corrosion Test
1	0.23		Fail.
2		0.05	Pass.
3			Fail.
4		0.1	Borderline.
5 [1]		0.1	Do.
6		0.2	Pass.

[1] Contains 1.5% of "Santolube 203A."

Sample 1, which is a grease typical of that shown in Patent No. 2,450,221 in that excess stearic acid was added to provide a free fatty acid content of 0.23%, except that the said grease also contained basic barium sulfonate of the present invention, failed the 2–134 corrosion test. Sample 2 which is the base grease of Table II above containing 1% of the basic barium sulfonate and having a free alkalinity of 0.05%, passed this test. On the other hand, sample 3 which was prepared to strict neutrality failed the test, and samples 4 and 5 which contained 0.1% free alkalinity were borderline in this test. Sample 6, which was prepared to 0.2% alkalinity readily passed the 2–134 corrosion test. On the basis of numerous tests, it can be stated that the grease of the present invention should be at least neutral instead of acid, and preferably should have a free alkalinity of at least 0.2% and varying up to about 1%. In the range from neutral to less than 0.2% free alkalinity, satisfactory greases prepared in accordance with the present invention and meeting the 2–134 corrosion test can be produced, although the results may vary from batch to batch. On the other hand, by providing a free alkalinity of at least 0.2% in conjunction with the basic alkaline earth sulfonate additive, satisfactory lithium base greases passing the 2–134 corrosion test can be produced with uniformity and ease.

EXAMPLE 4

A satisfactory basic barium sulfonate additive for purposes of the present invention was prepared as follows: A petroleum mahogany sulfonic acid was prepared in conventional manner by the sulfonation of a distillate lubricating oil fraction followed by separation of the sulfonic acid product. The latter analyzed 30.7% sulfonic acid, 24.3% mineral lubricating oil and 45.0% volatile material. 2314 grams of the foregoing petroleum sulfonic acid product were charged to a 3-neck flask fitted with a stirrer, and 2,000 grams of naphthene base lubricating oil distillate having an SUS viscosity at 100° F. of about 300 were added together with 500 cc. of anhydrous methyl alcohol. The mixture was stirred and heated to 113° F., and then a solution of 376 grams of barium hydroxide in 3,000 cc. of anhydrous methyl alcohol was added in three portions. The quantity of barium hydroxide was 10% in excess of the calculated amount to produce the basic barium sulfonate salt. The resulting mixture was then heated further to distill off methyl alcohol and water, and the temperature was held at 338–365° F. for 1½ hours. Any small amount of remaining water in the reaction mixture was then removed by sweeping with nitrogen. The dried mixture was then filtered through a body of solid filter aid material while hot; and 195 grams additional of the naphthene base lubricating oil were added in order to bring the active material to 25% concentration. Analysis of the resulting lubricating oil concentrate of the basic barium sulfonate by ashing gave the following results:

	Theory	Found
Barium Sulfonate	11.48	11.16–11.13
Barium	6.7	6.57–6.54

The weight ratio of barium to sulfur in the active material calculated to 4.27, which is substantially theoretical for the true basic barium sulfonate. In the following Table IV, the figures given for weight per cent of the basic barium sulfonate are those for the concentrate; consequently, the weight per cent of the active basic sulfonate is ¼ of the listed figures.

In order to illustrate the results obtained with other types of synthetic lubricant bases, the following Table IV sets forth the results secured with the foregoing basic barium sulfonate additive and also the commercial "Santolube 203A" in a 3:1 lithium soap of hydrogenated castor oil-lithium stearate grease using a 3:1 mixture of di-2-ethyl hexyl azelate-paraffin base mineral lubricating oil as the lubricant base, and containing 0.5% phenyl alphanaphthylamine.

Table IV

Additive Weight Percent	2-134 Corrosion Test		
	Copper	Grease	Result
1. Basic Ba Sulfonate (1.0%)	Slight stain	Slight pink	Pass.
2. Basic Ba Sulfonate (3.0%)	Unchanged	Unchanged	Do.
3. "Santolube 203A" (1%)	Very slight stain.	Slight pink	Do.
4. "Santolube 203A" (2%)	do	do	Do.
5. "Santolube 203A" (3%)	Unchanged	Unchanged	Do.

EXAMPLE 5

The following example illustrates the preparation of an approximately 6,000 pound plant batch of lithium base grease in accordance with the present invention. The ingredients employed were as follows:

Ingredients	Weight in pounds	Percent by Weight
Hydrogenated Castor Oil	786	12.68
Triple Pressed Stearic Acid	262	4.21
Lithium Hydroxide Monohydrate	199.8	3.23
Water (Distilled or Condensed Steam)	1,779.2	
Paraffin Base Mineral Lubricating Oil with an SUS viscosity at 100° F. of 101	1,218	19.61
Di-2-ethyl hexyl sebacate	3,654	58.83
Phenyl alphanaphthylamine	309	0.50
Dye	0.211	
"Santolube 203A"	61.8	1.00

The hydrogenated castor oil in molten form was added to the kettle with stirring together with about 483 pounds (61.5% of the hydrogenated castor oil charge) of the mineral lubricating oil. The oil is maintained less than the amount of hydrogenated castor oil at this stage in order to promote proper conditioning of the soap base during later stages of the manufacturing procedure. With the kettle contents being stirred at a temperature of about 175–200° F., a quantity of water which was less than the quantity of hydrogenated castor oil charge (in this case about 56.5% of the hydrogenated castor oil) was added, and then the lithium hydroxide in the form of a 10% aqueous solution was introduced. The amount of lithium hydroxide was calculated to be sufficient for saponification of all the fatty material and to provide a calculated excess of about 0.46% lithium hydroxide figured on the basis of finished grease at 18% soap content. Saponification was effected during a 4-hour period of stirring at a temperature of 180–200° F., with shots of water being added from time to time to assist the reaction. The stearic acid was then introduced and stirring continued at the foregoing temperature for about 1 hour with additional shots of water being added to prevent the soap base from balling up and becoming gummy. The temperature was then raised as quickly as possible to 320–330° F. and maintained at the elevated temperature for a period of 5–9 hours for dehydration and until the soap base became translucent and had a tendency to fracture when cold. The remaining mineral lubricating oil was then introduced slowly as the temperature dropped to 300° F. The di-2-ethyl hexyl sebacate introduction was then begun and continued slowly over a period of about 8 hours as the temperature dropped to about 220° F. and the desired grease consistency was attained. The phenyl alphanaphthylamine, the dye, and finally the "Santolube 203A" were added with continued stirring, and the grease was finally drawn at a temperature of 180–190° F.

Typical tests obtained on the foregoing grease were as follows:

Penetration, ASTM at 77° F. worked	294
Dropping point, °F	362
Lithium soap, per cent	18.19
Free alkali as LiOH, per cent	0.21
Norma-Hoffman oxidation 210° F., 100 hours pressure, drop pounds	4
2–134 corrosion test	Pass
Pressure drop during corrosion test	0
Water resistance, grease washed from bearing, per cent	1.2
Low temperature torque —67° F., 2,000 gram centimeter torque, secs. per revolution	2.4

As shown by the foregoing tests and others not listed, the said grease satisfactorily met all of the requirements of U. S. 2–134 and AN–G–25 specifications.

It will be understood that the grease may also contain other additives in small proportions which are compatible with the essential ingredients and do not interfere with the desirable properties thereof. Such additional additives include extreme pressure or lubricity agents, such as dibenzyl disulfide, viscosity index improving and pour depressant type materials such as acrylic acid ester polymers, materials for improving resistance in the salt spray humidity cabinet test such as sorbitan mono-oleate, and the like.

Obviously many modifications and variations of the invention, as hereinbefore set forth, may be made without departing from the spirit and scope thereof and, therefore, only such limitations should be imposed as are indicated in the appended claims.

I claim:

1. A lubricating grease composition comprising as the essential constituents an oleaginous liquid lubricating base as the major constituent, about 10–30% by weight of a lithium soap of a saturated soap-forming fatty material, and from 0.25 to 3% by weight of an oil-soluble basic alkaline earth metal salt of a sulfonated hydrocarbon wherein the weight ratio of alkaline earth metal to sulfur content is from 1.1 to 2 times that represented by the normal salt $M(SO_3R)_2$, where M is the alkaline earth metal and R is the hydrocarbon residue, said basic salt being in sufficient proportion to enable said grease composition to pass the copper corrosion test of U. S. Army specification 2–134.

2. A lubricating grease composition according to claim 1, wherein said basic salt is a basic barium sulfonate.

3. A lubricating grease composition comprising as the essential and at least major proportion of the lubricating base an oil-soluble high molecular weight high-boiling liquid aliphatic dicarboxylic acid ester within the lubricating viscosity range and possessing lubricating properties, sufficient lithium soap of soap-forming fatty material to thicken said lubricant base, said soap-forming fatty material consisting of more than 50% by weight of a hydroxy soap-forming fatty material selected from the group consisting of hydroxy fatty acids and hydroxy fatty acid glycerides, and from 0.25 to 3% by weight based on said composition of an oil-soluble basic alkaline earth metal salt of a sulfonated hydrocarbon wherein the weight ratio of alkaline earth metal to sulfur content is from 1.1 to 2 times that represented by the normal salt $M(SO_3R)_2$, where M is the alkaline earth metal and R is the hydrocarbon residue, said basic salt being in sufficient amount to enable said grease composition to pass the copper corrosion test of U. S. Army specification 2–134.

4. A lubricating grease composition according to claim 3, wherein said basic salt is a basic barium sulfonate.

5. A lubricating grease composition according to claim 4, which contains about 0.05–1.0% free alkalinity calculated as lithium hydroxide.

6. A lubricating grease composition according to claim 5, wherein the said basic salt is a basic barium sulfonate present in an amount of about 0.7–2% by weight.

7. A lubricating grease composition comprising as the lubricant base a minor proportion of mineral lubricating oil and a major proportion of a branched chain aliphatic diester of a dicarboxylic acid selected from the group consisting of sebacic, azelaic and adipic acids, about 10–30% by weight based on the composition of lithium soap of a mixture of a major proportion of hydrogenated castor oil and a minor proportion of stearic acid, and from 0.25 to 3% by weight based on said composition of an oil-soluble basic alkaline earth metal salt of a sulfonated hydrocarbon wherein the weight ratio of alkaline earth metal to sulfur content is from 1.1 to 2 times that represented by the normal salt $M(SO_3R)_2$, where M is the alkaline earth metal and R is the hydrocarbon residue, said basic salt being in sufficient amount to enable said grease composition to pass the copper corrosion test of U. S. Army specification 2-134.

8. A lubricating grease composition according to claim 7 which contains about 0.15–1.0% free alkalinity calculated as lithium hydroxide.

9. A lubricating grease composition according to claim 8, wherein the said basic salt is a basic barium sulfonate present in an amount of about 0.7–2% by weight.

10. A lubricating grease composition according to claim 9, wherein the branched chain aliphatic di-ester is the ethyl hexyl di-ester.

11. A lubricating grease composition consisting essentially of the following ingredients in per cent by weight:

Di-2-ethyl hexyl sebacate	40–80
Mineral lubricating oil	8–35
Lithium soap of hydrogenated castor oil	8–22
Lithium stearate	3–10
Glycerin	0.7–2.5
Free alkali as LiOH	0.05–1.0
Basic barium sulfonate	0.5–3.0
Oxidation inhibitor	0.2–2.0

12. A lubricating grease composition consisting essentially of the following ingredients in per cent of weight:

An approximately 3:1 mixture of di-2-ethyl hexyl sebacate and mineral lubricating oil	60–86
Lithium soap of an approximately 3:1 mixture of hydrogenated castor oil and stearic acid	12–30
Glycerine	0.7–2.5
Free alkali as LiOH	0.2–0.6
Basic barium sulfonate	0.7–2.0
Phenyl alphanaphthylamine	0.2–1.0

GEORGE W. ECKERT.

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