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HIGH-TEMPERATURE GREASE

Richard A. Butcosk, Jackson Heights, N. Y., assignor to Socony-Vacuum Oil Company, Incorporated, a corporation of New York

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This invention has to do with greases characterized by a high order of effectiveness under severe operating conditions, particularly for service at temperatures ranging from 400° F. to 500° F. and higher.

It is well known that greases lose some or all of their effectiveness when subjected to high temperature operations. In lubricating machine parts, for example, it is essential that a grease retain its gel structure during use; failure to do so results in high consumption of the grease and frequent servicing. In general, available greases suffer from a marked tendency to change in character when used over a wide range of temperature, particularly at high temperatures of the order of 400–500° F. and higher. Some conventional greases are characterized by excessive softening when exposed to such high temperature operation, thereby being extruded too rapidly from the area being lubricated to provide efficient lubrication.

It is an object of this invention, therefore, to provide a grease effective for high temperature use, such as at 400–500° F. and higher. A further object is to provide a grease which retains its original character over a wide range of operating conditions. Other objects of the invention will be apparent from the following description.

It has now been discovered that lithium base greases containing an ester of a phosphoric or phosphorous acid in certain proportions, are substantially free of the shortcomings referred to above.

The greases contemplated herein have a lithium soap content of the order of 5–25 per cent on the basis of the finished grease. Fats and fatty acids which can be used are those generally found in soap type grease. Such fatty materials have from about 12 to about 22 carbon atoms per molecule. Representative of such materials are vegetable, animal and fish fatty oils, and hydrogenated fatty materials thereof. Stearin, stearic acid, cottonseed oil acids, oleic acid, palmitic acid, myristic acid, hydrogenated fish oils such as "Hydrogenated Fish Oil, Iodine No. 77" and "Hydrofol," are typical. A preferred soap herein is lithium stearate. Methods of preparation of lithium soaps are well known in the art and discussion of the same is believed to be unnecessary.

The mineral oil constituent of the greases of this invention are those suitable for use at high temperatures. The oil can vary considerably in character; for example, it can be a residual or distilled oil. The oil constituent, however, is characterized by a viscosity (S. U. V.) of greater

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than 100 seconds at 100° F., and generally from about 100 to about 2,000 seconds at 100° F.

Serving as a lubricant vehicle with the mineral oil constituent, and cooperating therewith, is an ester of a phosphoric or phosphorous acid, i. e., an oxy-acid of phosphorus. The ester can have as organic constituents alkyl, cycloalkyl and/or aryl groups. Esters found to be effective include: tributyl phosphate, triphenyl phosphate, tricresyl phosphate and triphenyl phosphite. Another ester suitable for use here is tri-tertiary-amyl phenyl phosphite.

As indicated above, the proportions of lithium soap and phosphorus ester are balanced in order to provide greases of the desired character. The lithium soap or soaps comprise from about 5–25 per cent (by weight) of the finished grease, and preferably from about 8 per cent to a maximum of 18 per cent. The phosphorus ester or esters present in the grease will range from about 10 to about 50 per cent (by weight) of the grease product, and preferably from about 17 per cent to about 30 per cent. Increasing the ester content tends to make the grease firmer at temperatures of the order of 400–500° F., in addition to the firm consistency at lower temperatures. With more than about 50 per cent by weight of a phosphorus ester in the grease, however, there is a marked tendency for the grease to become hard during service, thereby having lower lubricating value. Moreover, when 50 per cent or more of a phosphorus ester is used with the aforementioned quantities of lithium soap, little if any improvement in dropping point is realized. Greases with less than about 5% of lithium soap seem to have very little mechanical stability and are usually semi-fluid in nature. Greases with more than 25% of lithium soap are quite expensive, and are too firm or are block form and not satisfactory for use as ball bearing lubricants. The balance of the grease is mineral oil of the character described above. Accordingly, the various constituents (on a dry basis) of the new greases can vary within the following limits:

	Per cent by weight
Lithium soap	5–25
Phosphorus ester	10–50
Ratio of lithium soap to ester	1:1 to 1:3
Mineral oil, less than 100 secs. S. U. V. @ 210° F	Balance

It is to be understood that the greases of this invention can also contain other characterizing materials and fillers, so long as the foregoing balance between the lithium soap and phosphorus ester is maintained. For example, the greases

can contain grease antioxidants such as amines, phenols, sulfides, etc.; fillers such as asbestos, graphite, mica, talc, etc.; and lubricity improving agents such as free fat, free fatty acids, esters of alkyl and/or aryl acids, sulfurized fats, lead soaps, etc.

The new greases are prepared by dissolving a lithium soap in mineral oil at a temperature above about 230° F. The mixture foams as the soap dissolves, indicating evaporation of water. When all foaming has ceased, a phosphorus ester is added and the resulting mixture is stirred and heated at 430° F. for 15 or 30 minutes. The product is then withdrawn from the vessel in which it is prepared and run into a suitable mold. The grease is allowed to cool whereupon it solidifies. The solid grease can then be cut into cakes of the desired size, and milled to desired consistency.

The greases of this invention are illustrated by the following examples tabulated in the table below. Provided also in the table are comparable examples in which different metal soaps are used instead of a lithium soap; however, all soaps are stearates. Another comparable example involves a thiophosphate ester in place of a phosphate or phosphite ester. Each of the greases shown in the table was prepared according to the procedure described above.

Table

Grease No.	1	2	3	4	5	6	7	8	9	10
Composition:										
Mineral Oil [1]	50	50	50	50	50	50	50	50	50	50.
Phosphorus Compound.	30 tricresyl phosphate.	30 triphenyl phosphate.	30 tributyl phosphate.	30 triphenyl phosphite.	30 tricresyl phosphate	30 tricresyl phosphate.	30 tricresyl phosphate	30 tricresyl phosphate.	30 tricresyl phosphate.	30. triphenyl thiophosphite.
Soap	20	20	20	20	20	20	20	20	20	20.
Metal Base	Lithium	Lithium	Lithium	Lithium	Aluminum	Calcium	Magnesium	Lead	Sodium	Lithium.
Properties:										
ASTM Dropping Point, ° F.	500+	487	459	500+	240	210	liquid	500+	Liquid	403.
Approximate Penetration (Unworked).	150	200	230	220	semi-fluid	semifluid	liquid			220.
Structure	Short Fibre.	Short Fibre.	Short Fibre.	Short Fibre.				Grainy and mushy.		Buttery.
Neutralization No. mgms KOH/g (ASTM).	0.3	0.14	0.3	Alkalinity 1.4.						0.14.

[1] 100 Sec. S. U. V. at 100° F.

It will be noted from greases 1–4 that the lithium soaps and the phosphate and phosphite esters cooperate to provide a high dropping point and excellent penetration value. In this connection, it appears that aryl phosphates are more advantageous than alkyl phosphates, as shown by a comparison of greases 1, 2 and 3. In sharp contrast with these greases are those identified as 5 through 9, in which lithium has been replaced by aluminum, calcium, magnesium, lead and sodium, respectively. All of the latter greases failed to retain their character at high temperatures, that is, the soaps and liquid phase did not hold together and did not produce the desired gel structure. Although grease 8, involving a lead soap, is characterized by an excellent dropping point value, it is of poor structure.

Grease 10 in which the ester is a thiophosphite, is shown for comparison with grease 4 in which the ester is the corresponding phosphite. The structure of grease 10 is not retained at high temperature, and the dropping point is substantially less than that which characterizes grease 4.

An additional desirable feature is manifested by grease 4, namely, alkaline character. This is of value inasmuch as the grease is resistant to oxidation.

I claim:

1. A high temperature grease consisting essentially of: mineral oil, a lithium soap content of from about 5 to about 25 per cent by weight, and about 10 to about 50 per cent by weight of an ester of an oxy-acid of phosphorus, said grease retaining its original gel structure at temperatures ranging from 400° F. to 500° F.

2. A high temperature grease consisting essentially of: mineral oil having an S. U. V. from about 100 to about 2,000 seconds at 100° F., a lithium soap content of from about 8 to about 18 per cent by weight, and about 17 to about 30 per cent by weight of an ester of an oxy-acid of phosphorus.

3. A high temperature grease consisting essentially of: about 50 per cent by weight of a mineral oil having an S. U. V. of 1,000 seconds at 100° F., about 20 per cent by weight of lithium soaps, and about 30 per cent by weight of tricresyl phosphate.

4. A grease as defined by claim 1 wherein the ester is a phosphoric acid ester.

5. A grease as defined by claim 1 wherein the ester is a phosphorous acid ester.

6. A grease as defined by claim 2 wherein the ester is tricresyl phosphate.

7. A grease as defined by claim 2 wherein the ester is triphenyl phosphate.

8. A grease as defined by claim 2 wherein the ester is tributyl phosphate.

9. A grease as defined by claim 2 wherein the ester is triphenyl phosphite.

RICHARD A. BUTCOSK.

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