Patented Feb. 16, 1937

2,070,781

UNITED STATES PATENT OFFICE

2,070,781

GREASE

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No Drawing. Application February 13, 1935, Serial No. 6,384

10 Claims. (Cl. 87—9)

This invention relates to improvements in greases and in particular to greases of the so called cup grease type adapted to use at high temperatures.

The conventional cup greases (i. e. calcium soap greases) are of great utility and importance. They have a structure which permits easy dispensing and handling and as a rule they have a rather clear and transparent appearance. Furthermore, they vary very little in consistency with temperature up to about 140° F. and are therefore very satisfactory for use up to this temperature. Above 140° F., however, they begin to soften and lose body and at a temperature varying from about 160° F. to about 200° F., depending on the soap content, they commence to separate. The material which separates out is a hard soapy mass which greatly interferes with lubrication.

Various methods have been devised for combating this tendency of cup greases to separate at elevated temperatures. One of the best methods is the addition of certain esters, particularly the partial esterification products of the polyhydroxy alcohols. One of the most suitable of these esters is commercial diglycol stearate which can be added to a conventional cup grease in concentrations of a few percent and which will render the grease non-separating at temperatures much higher than those at which it would otherwise commence to separate.

Neither this nor any of the other methods which have been devised for combating high temperature separation in cup greases has entirely succeeded in solving the concomitant problem of low melting point and loss of consistency at high temperatures. As was pointed out above, conventional cup greases begin to soften or "melt" at about 140° F. and they are therefore unsatisfactory for some types of use even though they are modified so as not to separate. We have found that this problem can be solved by the use of barium soaps in conjunction with the calcium soaps in greases, the effect being particularly marked in anhydrous greases which have been rendered non-separating by the use of esters of the type above mentioned.

We have made up greases of the conventional cup grease type substituting barium soap for all of the usual calcium soap and we have found that these greases have very peculiar properties. In every case they begin to stiffen as they are heated above 250° F. instead of separating as do the calcium soap greases. We are not aware just what the reason for this is but we believe it may be due at least in part to the fact that barium soaps are less oil soluble than calcium soaps at high temperatures. This theory is confirmed by the fact that on continued heating the straight barium soap greases begin to "leak" or lose oil, even though as much as 4% of commercial diglycol stearate is added. Furthermore, on cooling from high temperatures, the latter greases become soft and murky.

Thus, the straight barium soap greases are not satisfactory for most purposes. We have found that much more satisfactory greases can be produced by using a mixture of calcium and barium soaps, preferably a mixture in which the relative amounts of the two soaps are carefully controlled. The calcium soap present in the mixture gives it a good body or consistency at low temperatures while the barium soap gives similar results at the higher temperatures. Furthermore, the use of calcium soap gives the desirable clear appearance which is typical of anhydrous cup grease. If too little calcium soap is used this clear appearance is not obtained. If an excess of calcium soap is used, the high temperature consistency and particularly the consistency in the temperature range 200–250° F. is lowered. In other words the penetration is increased. By consideration of these and other factors, we have determined that the most desirable proportion of calcium soap, based on the total soap present, is from 15% to 40% and preferably from 20% to 25% by weight. It should be understood, however, that desirable results are obtained by using as little as 5% calcium soap with the barium soap or as little as 5% barium soap with the calcium soap.

The amount of total soap used in a grease may vary throughout the conventional cup grease range, for instance, from 15% to 30% by weight. The amount of soap used will depend in large measure on the desired consistency of the grease. Expressing the consistency in terms of the penetration at 77° F. as determined by method No. D217—33T of the American Society for Testing Materials, the penetration (P) will vary with the soap content (S) expressed as a weight percentage of soap in the total grease, approximately in accordance with the following formula:

$$P=540-12.5\,S$$

This formula will be understood to be only approximate since it will vary with the ratio of calcium to barium soap, the viscosity, kind and amount of oil used, the amount and kind of ester or other non-separation agent present, the

method of manufacture, the amount of working, etc.

It is highly desirable to use some ester or other non-separation agent in our grease since the effect of barium soaps on the non-separating type of cup grease is much more marked than their effect on the conventional type of cup grease. This is particularly true in the case of anhydrous greases. The quantity of non-separation agent required appears to be slightly less in the case of the mixed soap greases than in the case of the conventional cup greases. Furthermore, the optimum quantity is less critical in the case of the mixed soap greases than in the case of the straight calcium soap greases. In addition to commercial diglycol stearate, other esters which can be used are the mono-stearates, mono-oleates, and mono-palmitates of glycol and glycerine and the di-stearates, di-oleates and di-palmitates of glycerine.

The following may be taken as a typical weight composition range for my new greases:

	Per cent
Soap	15 to 30
Ester	2 to 5
Oil	65 to 83

It is to be understood that other ranges of soap content, e. g. from 2 to 40% (and corresponding oil percentages) can be used since our invention in its broadest form is not limited to any particular amounts of total soap, oil, ester, etc. As previously pointed out, from 15 to 40% of the total soap should preferably be calcium soap and the remainder barium soap.

The grease is preferably anhydrous, since the unique properties of calcium-barium soap greases are brought out best in the anhydrous greases, but it will be understood that a small amount of water, e. g. from a trace to 3%, may be present.

The usual ester content is from 2 to 3½% but when the desired grease is to be stable at high temperatures, for instance over 270° F., the amount of ester should be increased somewhat over that shown above. A suitable range is 3½ to 4½% and as much as 5% may be desirable. The necessity for increased amounts of ester for extreme high temperature greases is apparently due to the decreasing oil solubility of barium soaps at high temperatures. It is also to be understood that the ester can be replaced in some cases by other non-separation agents and can even be omitted altogether, particularly in the hydrous type of grease.

The oil used in our grease can, of course, be of any desired type and viscosity. It may, for instance, range from S. A. E. 10 to S. A. E. 50. Lighter oils such as straw oils are still more suitable, e. g. oils having viscosities in the neighborhood of 80 seconds Saybolt at 100° F.

The exact optimum composition of any particular grease will depend on various factors, such as the desired high and low temperature consistencies, the importance of leakage and separation, the desired appearance and other factors. This optimum composition can be readily determined by experiment in the light of the remarks contained herein. One typical preferred composition is as follows:

	Per cent
Barium soap	15
Calcium soap	5
Commercial diglycol stearate	3
Oil	77

The soaps used in our new greases may be made from any desired type of fats or fatty acids. They are, however, preferably and most expeditiously made from fatty acids rather than fats. Commercial stearic, palmitic and oleic acids can also be used, as can fatty acids split from hydrogenated fats. Commercial stearic acid is not recommended for the hydrous type of grease.

The soaps may be made in any desired way but are preferably made by reacting the lime and baryta in the form of their hydroxides separately or together with the fatty acids in the presence of a little oil as in conventional grease-making practice.

As an example of the commercial manufacture of our new greases, the composition last above mentioned can suitably be made by mixing the hydrated lime and hydrated barium hydroxide stoichiometrically equivalent to the desired amount of soap with the stoichiometrical equivalent amount of fatty acid and an amount of oil equal to the amount of fatty acid. This mixture is then heated as rapidly as possible to about 300° F. Rapid heating avoids excessive foaming of the soap. When the foaming has subsided the batch is adjusted to the neutral point by adding a little hydroxide or fatty acid whichever is required, the diglycol stearate is then added and the oil is gradually graded in. The resulting grease should be cooled rather rapidly, for instance in layers one-half to five-eighths inch thick. However, rapid cooling does not seem to be as essential as it is in some other types of greases.

As has been pointed out above, one of the factors which makes our mixed calcium and barium soap greases highly desirable is the fact that each soap lends a high consistency within a particular temperature range.

While we have described our invention in connection with certain specific embodiments thereof and in connection with certain theories of operation, it is to be understood that these are by way of illustration only and not by way of limitation and we do not mean to be bound thereby but only to the liberal interpretation of the appended claims in which we have attempted to set forth the novel features inherent in our invention.

We claim:

1. A grease of the cup grease type having a low penetration at high temperatures comprising lubricating oil, calcium soap and barium soap, in which the amount of calcium soap present is from about 15% to about 40% of the total calcium and barium soaps present.

2. A grease according to claim 1 containing, in addition to the constituents mentioned in claim 1, a partial esterification product of a polyhydroxy alcohol and a high molecular weight, soap forming fatty acid.

3. A grease according to claim 1 containing, in addition to the constituents mentioned in claim 1, commercial diglycol stearate.

4. A grease of the cup grease type having a low penetration at high temperatures comprising lubricating oil, calcium soap and barium soap, in which the amount of calcium soap present is from about 20% to about 25% of the total calcium and barium soaps present.

5. A grease according to claim 4 containing, in addition to the constituents mentioned in claim 4, a partial esterification product of a polyhydroxy alcohol and a high molecular weight, soap forming fatty acid.

6. A grease according to claim 4 containing, in addition to the constituents mentioned in claim 4, commercial diglycol stearate.

7. A cup grease having approximately the following percentage composition by weight:

	Per cent
Mixed calcium and barium soaps	15 to 30
Partial esterification product of a polyhydroxy alcohol	2 to 5
Oil	65 to 83

8. A grease according to claim 7 in which the amount of calcium soap present is from about 15% to about 40% of the total calcium and barium soaps present.

9. A grease according to claim 7 in which the amount of calcium soap present is from about 20% to about 25% of the total calcium and barium soaps present.

10. A cup grease having approximately the following percentage composition by weight:

	Per cent
Barium soap	15
Calcium soap	5
Commercial diglycol stearate	3
Oil	77

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