

## UNITED STATES PATENT OFFICE

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## LUBRICANT

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This invention relates to novel compositions, as well as to methods of preparing and using the same, and it relates more particularly to the preparation of high quality lubricating greases.

It is well known in the grease making art that the preparation of calcium soap greases containing high viscosity mineral oils is practically impossible. It is also true that the calcium soap greases are prepared containing water, which is necessarily present to give the lubricating grease a solid structure. When the water is removed, by heating the greases above the boiling point of water or prolonged heating at slightly lower temperatures than 212° F., the grease body is destroyed, with resulting soap precipitation which soap will cause abrasion of bearing parts or other working parts.

One object of this invention is to provide a solid grease lubricant, having a high viscosity mineral oil base at 100° F. which will impart a much greater adherent lubricant to metal surfaces than that prepared with the conventional grease making oils of relatively much lower viscosity.

Another object of this invention is to provide a solid calcium grease lubricant, that when heated above 212° F. will retain its body only thinning out slightly and which when again cooled will reset back to its original grease structure.

It has now been discovered that these and other objects of the invention can be accomplished by dispersing calcium soaps of conventional types, either soaps of fatty acids or the calcium soaps made from glycerides, in phenol extracts of mineral lubricating oil.

More broadly, the invention comprises incorporating a soap into a mineral lubricating oil fraction which is soluble in solvents having a selective solvent action on aromatic and unsaturated hydrocarbons. Solvents of this type such as phenol, furfural, nitro benzene, beta, beta'-dichlor ethyl ether, cresol, etc., have the characteristic property of being able to extract from mineral lubricating oil stocks, a fraction consisting essentially of non-paraffinic hydrocarbons. These extracts usually consist mostly of aromatic and unsaturated aliphatic hydrocarbons or mixed derivatives thereof.

The types of crude oils from which the extracts should be used according to the present invention should be obtained, are preferably the so-called naphthenic base or asphaltic base oil, such as Colombia, Gulf Coastal, Venezuela, West Texas, etc. The stock to be extracted is so selected as to boiling range and viscosity that a good yield

will be obtained of an extract fraction having a viscosity of about 1000-10,000 seconds Saybolt at 100° F. The solvent used in making this extract is, of course, preferably completely removed in order to leave a solvent-free, (a completely de-phenolized) extract. The viscosity index of such extract may range from about 0 to -450. These extracts may also be treated with acids and alkali to give practically any desired color ranging from almost water-white to a light red.

As the soap constituents of the lubricating greases of this invention, it is preferred to use a calcium or lime soap because the results obtained with lime soap are so remarkable compared to those obtained in the prior art in this particular field. However, the invention, in its broader aspects, also applies to other types of soaps, such as alkali metal soaps, e. g. of sodium and potassium; other alkaline earth metal soaps, such as those of barium, magnesium and the like, as well as to aluminum soaps. In preparing these various soaps, one may use not only the common fatty acids, such as stearic, oleic acid, and the like, but also the animal and vegetable fats and oils and various fatty acid derivatives thereof, as well as other types of organic carboxylic acids, such as naphthenic acids, particularly those derived from petroleum stock. In preparing lubricating greases, according to this invention, usually about 5 to 50% of soap should be used, calculated on the total weight of the grease, the balance, namely 95 to 50%, consisting substantially entirely of the non-paraffinic hydrocarbon extract, together, if desired, with a small amount of other addition agents, such as oil thickeners, e. g., polyisobutylene having a molecular weight of over 1000, or other polymers, graphite, water, dyes, oxidation inhibitors, etc., the total amount of these various other addition agents usually not exceeding 5%.

In carrying out this invention, various compounding methods may be used, for instance, the soap may be prepared separately and then dissolved directly in the oil stocks at elevated temperatures. However, in many cases it is preferred to form the soap in situ in the lubricating oil base stock, as for example, by dissolving the fatty acids to be used in the oil and then adding the basic substances, such as the lime or soda to effect saponification of the fatty acid directly in the oil, thereby making a grease stock. Another alternative is to dissolve the fatty acids in part of the oil base stock and to dissolve the base in another part of the oil stock and then to mix the two oil solutions, with such heating and stir-

ring as necessary to effect the desired saponification. A still further alternative is to dissolve the acid in part of the oil, then add the base, with heating and stirring until saponification is completed and then add the remainder of the oil. Usually the heating is first carried out just sufficiently to effect saponification, a suitable modifying temperature usually being 150–180° F., and then later, if desired, increasing the heating to some temperature above 212° F. in order to dehydrate the grease product (e. g. about 250–350° F.). Any substantially non-volatile or only slightly volatile addition agent to be added may be incorporated even at the elevated temperature but if water is to be added, the product should be cooled to at least below 230° F., preferably below 212° F.

The invention will be better understood from a consideration of the following examples:

#### EXAMPLE 1

A grease was prepared from the following materials, the proportions being by weight:

|                                                                     | Per cent |
|---------------------------------------------------------------------|----------|
| Animal fatty acid.....                                              | 6.76     |
| Cottonseed fatty acid.....                                          | 6.72     |
| Soya bean fatty acid.....                                           | 6.72     |
| Hydrated lime.....                                                  | 3.20     |
| Dephenolized phenol extract of Colombian lubricating oil stock..... | 76.60    |

The fatty acids are dissolved in  $\frac{1}{2}$  of the volume of the phenol extract and heated to 180° F. At this temperature the calcium hydrate, dispersed as a slurry in the balance of the oil, is added with the stirring. The stirring is preferably continued at this temperature for an hour or longer to complete the reaction, or if desired, the temperature may be raised to 250–300° F. in order to dehydrate the mass. In either case the lubricant is then allowed to cool or may be drawn into containers.

The dehydrated product has an A. S. T. M. penetration of 150 at 77° F. This product may be repeatedly heated above 212° F. without affecting the reforming properties of the grease structure on cooling.

#### EXAMPLE 2

Another lubricating grease prepared from the same dephenolized phenol extract was compounded from the following amounts of materials:

|                                  | Per cent |
|----------------------------------|----------|
| Animal fat.....                  | 10.0     |
| Hydrated lime.....               | 1.1      |
| Dephenolized phenol extract..... | 88.4     |
| Water.....                       | 0.5      |

The animal fat is dissolved in  $\frac{1}{2}$  of the phenol extract and heated to 150° F. where the hydrated lime is then added while stirring, and then the mass is heated to 300–350° F. After complete reaction has occurred, the oil-soap solution is cooled to 200° F. and the water added. After the water is completely dispersed, the balance of the oil is added and worked thoroughly into the grease to form a smooth, homogeneous product.

The grease thus produced has very satisfactory lubricating properties and has an A. S. T. M. penetration of 275–300 at 77° F.

The phenol extract used in Examples 1 and 2 have the following viscosity characteristics:

|                                              |       |
|----------------------------------------------|-------|
| Viscosity at 100° F. (Saybolt).....seconds.. | 4281  |
| Viscosity at 210° F. (Saybolt).....do.....   | 115.2 |
| Viscosity index.....                         | —45   |

Other greases equally suitable as high quality lubricants, may be prepared from sodium soaps, barium soaps, aluminum soaps, and various mixtures or combinations of these soaps as illustrated by the following example:

#### EXAMPLE 3

##### Driving journal compound

|                                   | Per cent |
|-----------------------------------|----------|
| 10 Stearic acid.....              | 42.3     |
| NaOH.....                         | 6.5      |
| Dephenolized phenol extracts..... | 51.2     |

#### EXAMPLE 4

##### Ball bearing grease

|                                     | Per cent |
|-------------------------------------|----------|
| 15 Stearic acid.....                | 13.0     |
| Aluminum stearate.....              | 0.50     |
| NaOH.....                           | 2.00     |
| 20 Dephenolized phenol extract..... | 84.50    |

#### EXAMPLE 5

##### Ball bearing grease

|                       | Per cent |
|-----------------------|----------|
| 25 Stearic acid.....  | 21.25    |
| Barium hydroxide..... | 1.25     |
| NaOH.....             | 3.05     |
| Phenol extract.....   | 74.35    |

#### EXAMPLE 6

##### Chassis lubricant

|                                                                   | Per cent |
|-------------------------------------------------------------------|----------|
| Aluminum stearate.....                                            | 5.0      |
| Aluminum naphthenate.....                                         | 0.5      |
| 35 Polyisobutylene (molecular weight about 60,000 to 80,000)..... | 1.0      |
| Phenol extract.....                                               | 93.5     |

It is not intended that this invention be limited to the specific examples on the materials used or to the percentages thereof given in the above examples, which were cited only for the sake of illustration, nor to any theory as to the mechanism of the operation of the invention, but only by the appended claims in which it is intended to claim all novelty inherent in the invention as broadly as the prior art permits.

We claim:

1. A lubricating grease substantially stable when repeatedly heated above 212° F. and cooled, comprising a minor proportion of lime soap and a major proportion of a phenol extract of a naphthenic base mineral lubricating oil, said extract having a viscosity of about 1000–10,000 seconds Saybolt at 100° F. and a viscosity index of about 0 to —450.

2. The method of manufacturing lubricating greases which comprises extracting a mineral lubricating oil base stock with a solvent having a selective solvent action on aromatic and unsaturated hydrocarbons, recovering a solvent-free extract having a viscosity of about 1000–10,000 seconds Saybolt at 100° F. and a viscosity index of about 0 to —450, dissolving fatty acids in said extract, heating the resultant solution to a saponifying temperature, adding hydrated lime thereto while stirring, and heating and stirring the mass until the desired saponification has been completed.

3. Lubricating grease according to claim 1, in which the phenol extract has a viscosity index at least as low as —45 and in which the phenol extract comprises at least about 70% by weight of the total grease.

4. A lubricating grease substantially stable when repeatedly heated above 212° F. and cooled,

comprising a minor proportion of soap selected from the group consisting of alkaline earth metals and aluminum soaps of fatty acids and naphthenic acids, and a major proportion of a mineral lubricating oil fraction having a viscosity index substantially below zero, extracted from a petroleum oil selected from the class consisting of naphthenic and asphaltic base petroleum lubricating oil stocks by a solvent having a selective solvent action on aromatic and unsaturated hydrocarbons, said extract having a viscosity of about 1000-10,000 seconds Saybolt at 100° F.

5. Lubricant according to claim 4 in which the soap is an alkaline earth metal soap of fatty acids.

6. A lubricating grease substantially stable when repeatedly heated above 212° F. and cooled, comprising a minor proportion of a lime soap of fatty acid and at least 50% of a mineral lubricating oil fraction consisting substantially entirely of non-paraffinic hydrocarbons, extracted from a mineral oil selected from the class consisting of naphthenic and asphaltic base petroleum lubricating oil stocks by a solvent having a selective solvent action on aromatic and unsaturated hydrocarbons, said extract having a viscosity index substantially below zero and having a viscosity of about 1,000-10,000 seconds Saybolt at 100° F.

7. A lubricating grease substantially stable when repeatedly heated above 212° F. and cooled, comprising a minor amount of an alkaline earth

metal soap, a major amount of a mineral lubricating oil fraction extracted from a petroleum oil selected from the class consisting of naphthenic and asphaltic base petroleum lubricating oil stocks by a solvent having a selective solvent action on aromatic and unsaturated hydrocarbons, said extract consisting substantially entirely of non-paraffinic hydrocarbons, and having a viscosity index substantially below zero and a viscosity of about 1,000-10,000 seconds Saybolt at 100° F., said lubricant also containing a minor amount, less than 5%, of polyisobutylene having a molecular weight of over 1000.

8. A lubricating grease substantially stable when repeatedly heated above 212° F. and cooled, comprising a minor proportion of soap selected from the group consisting of alkaline earth metals and aluminum soaps of fatty acids and naphthenic acids, and a major proportion of a mineral lubricating oil fraction having a viscosity index substantially below zero and consisting essentially of aromatic and unsaturated aliphatic hydrocarbons, extracted from a petroleum lubricating oil stock by a solvent having a selective solvent action on aromatic and unsaturated hydrocarbons, said extract having a viscosity of about 1,000-10,000 seconds Saybolt at 100° F.

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