

UNITED STATES PATENT OFFICE

2,197,433

STABILIZED GREASE LUBRICANT

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No Drawing. Application February 3, 1939,
Serial No. 254,479

5 Claims. (Cl. 87-9)

This invention relates to a stabilized grease lubricant; and it comprises as a new manufacture particularly useful as a lubricant for air brake cylinders a grease composed of a calcium soap in homogeneously blended admixture or solution with a highly refined paraffinic petroleum fraction having a viscosity index of the order of 100, being substantially freed of poly-naphthene constituents by refinement of the oil and having a Saybolt viscosity of at least 500 at 100° F., said grease being mechanically stable against bleeding of oil, or low absorbability in rubber parts requiring lubrication and possessed of considerable resistance against oxidation-deterioration, which resistance can be multiplied by incorporating in the stable mixture infinitesimal proportions of diphenyl-amine or an equivalent oxidation inhibitor; all as more fully hereinafter set forth and as claimed.

Lubricating greases are commonly made of soap mixed with ordinary lubricating oils. Ordinary cup grease as used in automobiles, for chassis lubrication, is a well known example. These soap greases have the undesirable property of "leaking" or "bleeding" oil. They lack mechanical stability. They are also subject to deterioration by oxidation with atmospheric air. Another disadvantage is the tendency of the oil constituent of the grease to be absorbed in rubber parts with which the grease comes in contact in use, causing swelling of the rubber. In various devices, particularly in air brakes used on railway cars, the rubber packings and similar parts become impregnated with oil present in the grease and are deleteriously affected. The grease gives up oil to rubber.

The three characteristics desired in lubricating greases, mechanical stability, resistance to oxidation, and non-absorbability in rubber, present problems to the grease manufacturer. Mechanical stability has been increased in some cases by adding to ordinary greases constituents such as phenol compounds in small quantity. Various oxidation inhibitors have been used. The tendency of oil-soap greases to give up oil to rubber parts requiring lubrication has remained an unsolved problem. Meeting specifications of the three properties particularly desired in air-brake grease has been difficult.

An object achieved in the present invention is

the production of a soap grease having substantially complete mechanical stability, high resistance to oxidation and an extremely low absorbability in rubber.

I have discovered that a maximum of mechanical stability with freedom from oil leakage is accomplished by incorporating a calcium soap of one of the higher fatty acids, most advantageously calcium stearate or palmitate, in highly viscous paraffinic oil freed of usual naphthenic or asphaltic constituents. I have found that highly refined paraffinic lubricating oils, as distinguished from ordinary oils containing appreciable contents of poly-naphthenes, and of high viscosity, form with calcium soaps homogeneous mixtures or solutions having permanent stability as an outstanding characteristic. The highly refined paraffinic oil holds a relatively large proportion of calcium stearate soap, from 15 to 25 per cent of the grease, without separation of the oil from the soap and the blended mixture or solution is remarkably resistant to oxidation deterioration without addition of antioxidants. This oxygen resistance is susceptible of multiplication to a high degree, when desired, by addition of an extremely small amount of diphenylamine, or similar antioxidant. In addition to freedom from leakage, and actual and potential resistance against oxidation, the grease also shows an extremely small tendency to lose oil to rubber with which the grease may come into contact in use. The grease is of great utility in lubricating the cylinders of air brakes. The solution-mixture of viscous paraffinic oil with calcium soap of good quality combines in one all the three properties particularly desired for air brake lubrication.

In air brake lubrication, the cylinder carrying a piston with rubber parts is dosed with grease which is expected to persist for the overhaul life of the equipment, in general a matter of three years. The importance of the three main characteristics desired in air brake grease is apparent.

In making the new grease, I have used paraffinic oil of high viscosity obtained in refining high grade paraffin base oils by solvent extraction. I have also used a lubricating oil obtained in refining paraffinic lubricating stocks by the aluminum chlorid method. This method is described in U. S. Patent 1,127,465. It removes naphthenic constituents. These highly refined paraffinic oils

extremely low in naphthene content or substantially freed of poly-naphthenes to a viscosity index of the order of 100 and when of a Saybolt viscosity above 500 at 100° F. form with calcium soap greases having the three characteristics desired in a lubricating grease. As stated, the resistance to oxidation is readily increased to a high degree by a small addition of diphenylamine. A quantity of the order of 0.1 per cent is usually sufficient. Quantities from 0.05 per cent to 0.3 per cent diphenylamine are suitable.

In making the grease, the relative quantities of soap and oil are, as I have found, a factor in controlling the desired properties of the grease. At least 15 per cent calcium soap to 85 per cent refined oil, by weight, should be used. And the proportion of soap may run to 25 per cent with 75 per cent oil. Greater proportions of soap are not advisable. Calcium stearate is superior to the other soaps. Calcium palmitate is next in order. Other soaps may be used but are not particularly recommended. Calcium stearate and palmitate permit incorporation of small proportions of sodium soaps.

The high viscosity of the refined paraffinic oil has been found to be an important factor in controlling the three desired grease characteristics. While oils of lower viscosity may be used, I have found that a viscosity of at least 500 Saybolt at 100° F. is required for meeting usual quantitative specifications as to non-bleeding, oxidation-resistance and non-swelling of rubber in air-brake lubricants, for example. All three of these specifications, as a matter of fact, have been raised as a result of the present invention.

In addition to the three characteristics which have presented problems in the past, the grease has otherwise suitable properties, including a melting point of 220-225° F.; worked penetration, A. S. T. M., 280 to 300 at 77° F.; and a solidifying temperature between -40° and -50° F.

In making the grease, the best method is to mix the three main ingredients, refined paraffinic oil, pure hydrated lime, and double pressed stearic acid, together at a temperature of 120 to 160° F. in a kettle and then to gradually raise the temperature to 180° to 200° F., continuing the heating within this temperature range with continued agitation for 6 to 12 hours. Under these conditions the fatty acid combines with the hydrated lime with formation of water, the saponification being complete when the grease assumes the proper consistency. Full saponification can be determined by chemical analysis. The grease is then drawn from the kettle into containers. The oxidation inhibitor, most advantageously diphenylamine, may be added before drawing off the grease. The mixture so made becomes homogeneously blended as a permanent solution or emulsion.

The grease can also be made from pure fats such as tallow, lard oil, horse fat, etc. In this case the fat, hydrated lime and a portion of the oil, about 25 per cent of the total oil ingredient or less, are cooked together in a pressure kettle to effect saponification. Temperatures of 250° to 350° F. are suitable. When saponification is substantially complete, the soap base may be run into an open kettle and the balance of the oil ingredient with chemical stabilizers, when desired, are incorporated by agitation, together with a little water where necessary to a proper consistency. The blended mixture so made may be drawn off into containers for the grease.

In a specific example of the invention, a grease made up for the purpose of lubricating air brake cylinders, the following composition was found to give the best results:

	Per cent
Calcium stearate soap made from double pressed stearic acid.....	19.0
Aluminum chloride treated Pennsylvania lubricating oil having a viscosity index of 100 and a Saybolt viscosity of 677 at 100° F.....	79.7
Water by analysis.....	0.3
Glycerol.....	1.0

A blended homogeneous mixture of this composition showed complete stability against bleeding of liquid oil when subjected to a standardized test. In this test a 30 gram sample of the grease was spread in a wire-gauze conical basket and maintained at a temperature of 160° F. for a period of 21 days. The above grease showed no loss of oil. Other greases of conventional composition, made for the same service, when subjected to the same test, lose from 30 to 50 per cent of their total weight in 21 days standing at 140° F.

The grease showed, without addition of any oxidation inhibiting agent, an oxidation induction period of 10 hours, which is about twice as long as the induction period shown by the usual grease of this type. Incorporation in the grease of 0.1 per cent of diphenylamine produced an oxidation induction period exceeding 500 hours. Thus the oxidation stability was multiplied 50 times. The susceptibility of the grease to inhibition of oxidation by diphenylamine is an outstanding property.

In a standard rubber absorption test applied by immersing a 5 gram sample of a rubber packing material used in airbrakes in a beaker containing 200 cc. of the grease and maintaining the system at a temperature of 165° F. for 500 hours, the rubber showed an absorption of oil amounting to 15 per cent of the weight of the rubber. This compares with a gain in weight of similar rubber from ordinary grease amounting to 25 to 45 per cent in 500 hours.

What I claim is:

1. A stable oxygen-resistant, non-bleeding lubricating grease having a low absorbability in rubber, which consists essentially of a homogeneous blended mixture of 15 to 25 per cent calcium stearate and 85 to 75 per cent of a paraffinic petroleum oil of a viscosity exceeding 500 Saybolt at 100° F. and refined to a viscosity index around 100, said oil being substantially free of poly-naphthenic constituents.

2. An oil-soap grease adapter for use in lubricating air-brake cylinders and which has a low absorbability in rubber and a good resistance to oxidation and is substantially non-bleeding, said grease consisting essentially of a blended mixture of about 15 to 25 per cent of a purified calcium soap chosen from the group consisting of calcium stearate and calcium palmitate and a paraffinic petroleum lubricating oil refined to a viscosity index about 100 or above and of a viscosity exceeding 500 Saybolt at 100° F., said oil being substantially free of poly-naphthenic constituents.

3. An oil-soap grease adapted for use in lubricating air-brake cylinders having rubber parts, which is composed of about 19 per cent calcium stearate soap made from double pressed stearic acid, and about 79 per cent Pennsylvania

lubricating oil which has a Saybolt viscosity above 600 at 100° F., and which has been refined by aluminum chloride treatment so as to have a viscosity index of about 100 and so as to be substantially free from poly-naphthenic constituents, the balance of said grease being chiefly water and glycerol.

4. An oil-soap grease adapted for use in lubricating air-brake cylinders having rubber parts, which is composed of about 19 per cent calcium stearate soap made from double pressed stearic acid, about 79 per cent Pennsylvania lubricating oil which has a Saybolt viscosity above 600 at 100° F. and which has been refined by aluminum chloride treatment so as to have a viscosity index of about 100 and so as to be substantially free from poly-naphthenic constituents,

and about 0.1 per cent diphenyl amine, the balance being chiefly water and glycerol.

5. An oil-soap grease adapted for use in lubricating air-brake cylinders and which has a low absorbability in rubber and a good resistance to oxidation and is substantially non-bleeding, said grease consisting essentially of a blended mixture of about 15 to 25 per cent of a purified calcium soap chosen from the group consisting of calcium stearate and calcium palmitate, a paraffinic lubricating oil which has a viscosity exceeding 500 Saybolt at 100° F. and which has been refined so as to have a viscosity index of about 100 and so as to be substantially free from poly-naphthenic constituents, and from 0.05 to 0.3 per cent of diphenyl amine.

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