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GREASES THICKENED WITH PHENYLENE DIAMIDES

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This invention pertains to the use of phenylene diamides as thickening agents in grease compositions. N,N'-acyl derivatives of phenylene diamines (e. g., N,N'-acetyl stearoyl-p-phenylene diamines) represent a particularly effective class of thickening agents for grease compositions.

Metal soaps, such as calcium and lithium stearates, are examples of well-known agents which are used to thicken lubricating oils to the consistency of a grease. Because the metal soaps are pro-oxidative, that is, increase the susceptibility of grease compositions to oxidation, it has been necessary to incorporate great amounts of oxidation inhibitors in these greases.

The grease compositions of this invention are more resistant to oxidation than those thickened with metal soaps, and also have increased melting points. The grease compositions described herein are more resistant to heat than soap-thickened greases, that is, at increased temperatures the thickening agents of grease compositions of this invention do not lose their thickening action as rapidly as soaps do.

It is a tremendous advantage in lubricating systems which are not readily accessible to use grease compositions which withstand high temperatures and the effects of oxidation for long periods of time. Greases which are used to lubricate such parts as found in sealed bearings of electric motors, sealed bearings of compressors, rocker arms of airplane motors, numerous pieces of high speed equipment, such as high speed motors, must be resistant to oxidation and have high temperature stability.

Grease compositions have certain fundamental desirable properties, and it is an object of this invention to prepare grease compositions in which the properties of the grease may be retained throughout wide ranges of temperatures and pressures over long periods of time.

It is another object of this invention to prepare grease compositions which are not susceptible to the pro-oxidation action of metal soaps.

It is a further object of this invention to prepare grease compositions which maintain their grease structure throughout wide changes in temperature.

These and further objects of this invention will be apparent from the following description and the appended claims.

It has been discovered that greases can be prepared by the thickening action of acyl derivatives of phenylene diamine in an oil of lubricating viscosity. That is, an oil of lubricating viscosity can be thickened to the consistency of a grease by incorporating therein an acyl derivative of phenylene diamine.

The grease compositions of this invention comprise mainly oils of lubricating viscosity and acyl derivatives of phenylene diamines.

As used herein, the term "acyl derivatives of phenylene diamines" means (1) phenylene diamines containing two acyl radicals, one acyl radical being attached to each of the nitrogen atoms, (2) phenylene diamines containing one acyl radical and one aliphatic radical, that is, phenylene

diamines containing one acyl radical attached to a nitrogen atom and an aliphatic radical attached to the other nitrogen atom, and (3) phenylene diamines containing one acyl radical attached to one nitrogen atom, leaving one amino group as such, that is, the acyl derivative of phenylene diamine will have one acyl radical attached to one nitrogen atom, and two hydrogen atoms attached to the other nitrogen atom. Because of their stability to oxidation and their effectiveness as thickening agents, the phenylene diamines containing two acyl radicals, one acyl radical being attached to each of the nitrogen atoms, are the preferred acyl derivatives of phenylene diamines for the purposes of this invention. Furthermore, it is preferred that the nitrogen atoms of the phenylene diamines be in the para position to each other. However, the ortho and meta acyl derivatives of phenylene diamines also function as thickening agents in lubricating oils according to this invention.

The acyl derivatives of phenylene diamines of this invention contain a total of from 18 to 30 carbon atoms, 18 to 24 carbon atoms being preferred. That is, in addition to the 6 carbon atoms of the phenylene nucleus, the acyl derivatives of phenylene diamines contain from 12 to 24 carbon atoms, 12 to 18 being preferred.

As used herein, the term "aliphatic radicals" means those radicals which are composed mainly of hydrogen and carbon, and include such radicals which contain, in addition, minor amounts of substituents, such as oxygen, nitrogen, etc.

Examples of acyl radicals include those derived from the following fatty acids: formic acid, acetic acid, propionic acid, butanoic acid, caproic acid, caprylic acid, capric acid, lauric acid, tridecanoic acid, myristic acid, palmitic acid, stearic acid, arachidic acid, mixtures of fish oil fatty acids, etc.

Examples of aliphatic radicals include the radicals derived from methane, ethane, ethene, propane, propene, butane, butene, pentane, hexane, isohexane, heptane, isoheptane, octane, isooctane, decane, dodecane, tetradecane, hexadecane, octadecane, isosane, docosane, etc.

Examples of acyl derivatives of phenylene diamines which can be used according to this invention to thicken lubricating oil compositions to the consistency of a grease include the ortho, meta and para isomers of the following: N-formyl-N'-stearoyl phenylene diamine, N-acetyl-N'-stearoyl phenylene diamine, N,N'-dilauroyl phenylene diamine, N-valeryl-N'-caprylyl phenylene diamine, N-acetyl-N'-capryl phenylene diamine, N-butyryl-N'-arachidyl phenylene diamine, N,N'-dicaprylyl phenylene diamine, N,N'-dicapryl phenylene diamine, N-acetyl-N'-oleoyl phenylene diamine, N-acetyl-N'-stearoyl phenylene diamine, N-butyryl-N'-lauryl phenylene diamine, etc.

Suitable lubricating oils useful as base oils in the preparation of the grease thickened with acyl derivatives of phenylene diamines of this invention include a wide variety of oils, such as naphthenic base, paraffin base and mixed base mineral oils, synthetic oils, e. g., alkylene oxide polymers such as polypropylene oxide polymers, and other hydrocarbon lubricants, e. g., lubricating oils derived from coal products. Other synthetic oils include esters of alkylene oxide type polymers, e. g., acetylated propene oxide polymers prepared by acetylating propene oxide polymers containing hydroxyl groups, dicarboxylic acid esters, poly esters, esters of polyhydric alcohols and liquid esters of acids of phosphorus and silicon.

The acyl derivatives of phenylene diamines are used in amounts sufficient to thicken the base oil to the consistency of a grease. Amounts of about 5% to about 40% by weight, based on the final composition, normally thicken base oils to the consistency of a grease; however, with the greater proportion of the base oils, grease compositions are obtained when from about 10% to about 25%

by weight of acyl derivatives of phenylene diamines are incorporated in the base oils.

When it is desired to thicken lubricating oils only slightly (e. g., in the preparation of thickened lubricating oils for use as air cleaner oils) only about 2% of the acyl derivatives of phenylene diamine need be incorporated in the lubricating oil.

The grease compositions of this invention are prepared quite similarly to other greases using other thickening agents; that is, the acyl derivatives of phenylene diamines and the base oils are brought together in a mixing vessel, and with constant agitation, the mixture is heated up to temperatures ranging from 200° F. to 500° F., then cooled to room temperature. For the most part, the lubricating oils thickened according to this invention to the consistency of a grease are easier to prepare than greases containing metal soaps as thickening agents. The acyl derivatives of phenylene diamines thickened greases normally do not pass through the various phases which are noticed in the preparation of metal soap-thickened greases.

The following examples illustrate the method of preparing greases which are thickened with acyl derivatives of phenylene diamines according to this invention, using N-acetyl-N'-stearoyl-p-phenylene diamine as the thickening agent.

Example 1

A mixture of 10 grams of N-acetyl-N'-stearoyl-p-phenylene diamine and 30 grams of a California solvent-refined paraffin base oil having a viscosity of 450 SSU at 210° F. was heated, with agitation, to 340° F., then cooled to room temperature.

The resulting grease composition was unctious and had a melting point of 320° F.

Example 2

A mixture of 5 parts by weight of N-acetyl-N'-stearoyl-p-phenylene diamine and 25 parts by weight of a California solvent-refined paraffin base oil having a viscosity of 185 SSU at 100° F. was heated to a temperature of 340° F., then cooled to room temperature.

The resulting grease composition was unctious and had a melting point of 360° F.

I claim:

1. A grease composition comprising a major proportion of an oil of lubricating viscosity and a small amount, sufficient to thicken the lubricating oil to the consistency of a grease, of aliphatic acyl phenylene diamine containing a total of from 18 to 30 carbon atoms and not more than one acyl group attached to a nitrogen atom.

2. A grease composition comprising a major proportion of an oil of lubricating viscosity and a small amount, sufficient to thicken the lubricating oil to the consistency of a grease, of aliphatic acyl phenylene diamine containing a total of from 18 to 24 carbon atoms and not more than one acyl group attached to a nitrogen atom.

3. A grease composition comprising a major proportion of an oil of lubricating viscosity and a minor amount, sufficient to thicken the oil to the consistency of a grease, of N,N'-di(aliphatic acyl) phenylene diamine containing a total of from 18 to 30 carbon atoms.

4. A grease composition comprising a major proportion of an oil of lubricating viscosity and from about 5% to about 40% by weight of N,N'-di(aliphatic acyl) phenylene diamine containing a total of from 18 to 30 carbon atoms.

5. A grease composition comprising a major proportion of an oil of lubricating viscosity and from about 10% to about 25% by weight of N-acetyl-N'-stearoyl-p-phenylene diamine.

6. A grease composition comprising a major proportion of an oil of lubricating viscosity and from about 10% to about 25% by weight of N-acetyl-N'-stearoyl-p-phenylene diamine.

7. A grease composition comprising a major proportion of a petroleum base lubricating oil and from about 10% to about 25% of N,N'-di(aliphatic acyl)phenylene diamine containing a total of from 18 to 30 carbon atoms.

8. A grease composition comprising a major proportion of a petroleum base lubricating oil and from about 10% to about 25% of N-acetyl-N'-stearoyl-p-phenylene diamine.

9. A grease composition comprising a major proportion of a petroleum base lubricating oil and a small amount, sufficient to thicken said lubricating oil to the consistency of a grease, of aliphatic acyl phenylene diamine containing a total of from 18 to 30 carbon atoms and not more than one acyl group attached to a nitrogen atom.

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