ASHLESS LUBRICANT COMPOSITION

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ABSTRACT
A lubricant composition with improved antioxidant capability is provided by way of an additive composition containing a metal free sulfur-containing compound, an aromatic amine, and a hindered amine. Particularly effective metal-free sulfur-containing compounds include ashless dithiocarbamates, such as methylenebis(dibutyldithiocarbamate), and sulfurized fatty acids.
ASHLESS LUBRICANT COMPOSITION

[0001] The present invention relates to lubricant compositions that are stabilized from oxidation by the presence of (a) a metal-free sulfur-containing compound, (b) a hindered amine, and (c) an aromatic amine.

BACKGROUND OF THE INVENTION

[0002] 1. Field of the Invention

[0003] This invention relates to lubricating oil compositions, their method of preparation and use. Specifically, this invention relates to lubricating compositions that contain an antioxidant additive comprising a metal-free sulfur-containing compound, a hindered amine and an aromatic amine.

[0004] 2. Description of the Related Art

[0005] Oxidation is a major cause of the breakdown of lubricants. This results in a shortened lifespan of the lubricant, requiring more frequent changes, especially in demanding environments such as internal combustion engines.

[0006] Antioxidants have therefore played an important role as additives in lubricants in order to extend their useful life. Aryl amines (also called aromatic amines), especially secondary diarylamines, e.g., alkylated diphenylamines, phenothiazines, and alkylated N-naphthyl-N-phenylamines have been important additives to lubricating compositions. Also important have been phenolic compounds in retarding oxidation.

[0007] Other combinations of antioxidants have also been used. U.S. Pat. Nos. 5,073,278 and 5,273,669 to Schumacher et al. disclose the synergistic combination of aromatic amines and hindered amines in a lubricating oil. U.S. Pat. No. 5,268,113 to Evans et al. discloses the combination of a hindered amine with phenolic compounds.

[0008] Sulfurized organic compounds have also been shown to have antioxidant activity. U.S. Pat. Nos. 4,880,551 to Doe and 6,743,759 to Stunkel disclose the synergies between an ashless dithiocarbamate with triazole compounds. U.S. Pat. No. 6,806,241 discloses the synergy between an ashless dithiocarbamate, a molybdenum compound, and an alkylated diphenylamine.

SUMMARY OF THE INVENTION

[0009] We have discovered that a lubricant composition containing an additive comprising a metal free sulfur-containing compound, an aromatic amine, and a hindered amine can synergistically give antioxidant protection. More specifically, the invention provides for a lubricant composition which comprises a mineral or a synthetic base oil, a mixture of such oils, or a grease, and an antioxidant additive composition comprising (as weight percent of the total lubricant composition):

[0010] at least one, metal-free sulfur-containing compound at between about 0.001 and 10%, preferably between about 0.1 and 1.0%, and most preferably at between about 0.25 and 0.5%;

[0011] at least one hindered amine at between 0.001 and 10%, preferably between about 0.05 and 1.0%, and most preferably between about 0.1 and 0.5%; and

[0012] at least one aromatic amine at between 0.001 and 10%, preferably between about 0.1 to 1.0%, and most preferably between about 0.25 and 0.5%.

[0013] Particularly effective metal-free sulfur-containing compounds include ashless dithiocarbamates, such as methylenbis(dibutylthiocarbamate), and sulfured fatty acids. In addition, the highest degree of synergy was noted with a relatively small amount of hindered amine in the additive, such as a preferred composition comprising about 0.1% hindered amine, about 0.4% aromatic amine, and about 0.5% metal-free sulfur-containing compound.

DETAILED DESCRIPTION OF THE INVENTION

Lubricant Basestocks

[0014] Typical lubricant basestocks that can be used in this invention can include both mineral and synthetic oils. Included are polyalphaolefins, (also known as PAOS), esters, diesters and polyol esters or mixtures thereof. The basestock comprises at least 90%, preferably at least 95% of the total lubricant composition.

Grease

[0015] Base grease compositions consist of a lubricating oil and a thickener system. Generally, the base oil and thickener system will comprise 65 to 95, and 3 to 10 mass percent of the final grease respectively. The base oils most commonly used are petroleum oils, bio-based oils or synthetic base oils. The most common thickener systems known in the art are lithium soaps, and lithium-complex soaps, which are produced by the neutralization of fatty carboxylic acids or the saponification of fatty carboxylic acid esters with lithium hydroxide typically directly in the base fluids. Lithium-complex greases differ from simple lithium greases by incorporation of a complexing agent, which usually consists of di-carboxylic acids.

[0016] Other thickener systems that can be used in this invention include aluminum, aluminum complex, sodium, calcium, calcium complex, organo-clay, sulfonate and polyurea.

Metal-Free Sulfur-Containing Compounds

[0017] Sulfur-containing compounds used in this invention are of many types. Typically, the sulfur-containing compound is oil-soluble and contains a readily oxidizable sulfur atom or atoms. Examples of such compounds are sulfured olefins, alkyl sulfides and sulfides, dialkyl dithiocarbamates, dialkyl dithiocarbamate esters, ashless dithiocarbamates, thioam disulfides, sulfured fatty acids, sulfurred fatty acid derivatives, and thiadiazole compounds.

[0018] 1. Sulfured Fatty Acids and Derivatives

[0019] Sulfured fatty acids can be prepared by the reaction with unsaturated fatty acids with the sulfur sources mentioned above. Examples of unsaturated fatty acids include but are not limited to; linoleic acid, oleic acid, arachidonic acid, linolenic acid, and myristoleic acid.

[0020] Derivatives of sulfured fatty acids include but are not limited to sulfured fatty acid esters and sulfurred fatty acid amides.

[0021] 2. Other Sulfur-Containing Compounds

[0022] Ashless dithiocarbamates, tetraalkylthiuram disulfides, and thiadiazole compounds that are suitable for use in this invention include, but are not limited to; methylenebis(dialkyldithiocarbamate), ethylenebis(dialkyldithiocarbamate), and tetraalkylthiuram disulfide where the alkyl groups have preferentially have between 1 and 20 carbon atoms. Examples of preferred ashless
dithiocarbamates are methylenebis(dibutylthiocarbamate) and ethylenedibutylthiocarbamate). Examples of preferred thiuram disulfides include tetraalkylthiuram disulfide and hexaalkylthiuram disulfide. Examples of thiazole compounds include dialkylthiophenoxides.

[0023] It is found that sulfurfured olefins do not provide a noticeable synergy when used in the proposed three-component system of the invention. Sulfurfured olefins are usually derived from alpha olefins, isomerized alpha olefins, cyclic olefins, branched olefins, and polymeric olefins that are reacted with a sulfur source. Specific examples of olefins include but are not limited to: 1-butene, isobutylene, diisobutylene, 1-pentene, 1-hexene, 1-heptene, 1-octene, and more with longer carbon chains up to C10 and beyond to polymeric olefins. Examples of sulfur sources include sulfur, hydrogen sulfide, sodium hydrogen sulfide, sodium sulfide, sulfur chloride, and sulfur dichloride.

Hindered Amines

[0024] The hindered amines used in this invention are of many types, with three types predominating: pyrimidines, piperidines and stable nitroxide compounds. Many more are described in the book "Nitrones, Nitrosoamines, and Nitroso Compounds", E. Breuer, et al., 1989, John Wiley & Sons. The hindered amines are also known as HALS (hindered amine light stabilizers) and are a special type of amine that are capable of antioxidant behavior. They are used extensively in the plastics industry to retard photochemical degradation.

[0025] 1. Pyrimidine Compounds

[0026] Pyrimidine compounds are of the substituted tetrahydro type and include the general structure of a 2,3, 4,5-tetrahydropyrimidine as given below (I), and described by Volodarsky, et al. in U.S. Pat. No. 5,847, 035, and by Alink in U.S. Pat. No. 4,085,104.

\[
\begin{align*}
\text{(I)} & \\
\end{align*}
\]

[0027] R1 is H, O, or a hydrocarbon from 1 to 25 carbon atoms, or an alkoxyl radical with the oxygen bound to the nitrogen with the alkyl portion containing 1 to 25 carbon atoms. R2, R3, R4, R5, R6, and R7 are hydrocarbons with 1 to 25 carbon atoms each. Most preferably, R2, R3, R6, and R7 are methyls.

[0028] Other pyrimidine compounds that can are of the hexahydro type, (II)

\[
\begin{align*}
\text{(II)} & \\
\end{align*}
\]

[0029] R8 and R11 are H, O, or a hydrocarbon from 1 to 25 carbon atoms, or an alkoxyl radical with the oxygen bound to the nitrogen with the alkyl portion containing 1 to 25 carbon atoms. R9, R10, R11, R12, R13, R14, and R15 are hydrocarbons with 1 to 25 carbon atoms each. Most preferably, R9, R10, R14, and R15 are methyls.

[0030] 2. Piperidine Compounds

[0031] The piperidine compounds used in this invention are described by Schumacher, et al., U.S. Pat. No. 5,073, 278 and by Evans in U.S. Pat. No. 5,268,113. These compounds have the general formula (III):

\[
\begin{align*}
\text{(III)} & \\
\end{align*}
\]

[0032] where R16 is H, O, or a hydrocarbon from 1 to 25 carbon atoms, or an alkoxyl radical with the oxygen bound to the nitrogen with the alkyl portion containing 1 to 25 carbon atoms. R17, R18, R22, and R23 are preferably methyl groups. R20 is either OH, H, O, NH₂, an ester group O₂CR where R is a hydrocarbon with 1 to 25 carbon atoms or a succinimide group.

[0033] Examples of hindered amines based upon piperidine include 4-hydroxy-2,2,6,6-tetramethylpiperidine, 1-allyl-4-hydroxy-2,2,6,6-tetramethylpiperidine, 1-benzyl-4-hydroxy-2,2,6,6-tetramethylpiperidine, 1-(4-tert-butyl-2-enyl)-4-hydroxy-2,2,6,6-tetramethylpiperidine, 4- stearyl oxyloxy-2,2,6,6-tetramethylpiperidine, 1-ethy1-4-salicyloyloxy-2,2,6,6-tetramethylpiperidine, 4-methacryloyoxy-1,2,6,6-pentamethylpiperidine, 1,2,6,6-pentamethylpiperidine-4-yl-(3,5-di-tert-butyl-4- hydroxyphenyl)propionate, di(1-benzyl-2,2,6,6-tetramethylpiperidine-4-yl) maleate, di(2,2,6,6- tetramethylpiperidine-4-yl) succinate, di(2,2,6,6- tetramethylpiperidine-4-yl) glutarate, di(2,2,6,6- tetramethylpiperidine-4-yl) adipate, di(2,2,6,6- tetramethylpiperidine-4-yl) sebacate, di(1,2,6,6- pentamethylpiperidine-4-yl) sebacate, di(1,2,6,6- tetramethyl-2,6-diphenylpiperidine-4-yl) sebacate, di(1-ally1-2,2,6,6-tetramethylpiperidine-4-yl) phthalate, 1-hydroxy-4 beta-cyanoethoxy-2,2,6,6-tetramethylpiperidine, 1-acetyl-2,2,6,6-tetramethylpiperidine-4-yl) acetate, tri(2,2,6,6-tetramethylpiperidine-4-yl) trimellitate, 1-acryloyl-4-benzoyloxym-2,2,6,6-tetramethylpiperidine, di(2,2,6,6-tetramethylpiperidine-4-yl) diethyl malonate, di(1,2,6,6-pentamethylpiperidine-4-yl) butyl (3,5-di-tert-butyl-4-hydroxybenzyl) malonate, di(1-octyl oxyloxy-2,2,6,6-tetramethylpiperidine-4-yl) sebacate, di(1-cyclohexoxyloxy-2,2,6,6-tetramethylpiperidine-4-yl) sebacate, hexane-1,6-bis(4-carbamoyloxym-1-nbutyl)-2,2,6,6-tetramethylpiperidine, tolulene-2,4-bis (4-carbamoyloxym-1-n-propyl)-2,2,6,6-tetramethylpiperidine, dimethyl-bis(2,2,6,6-tetramethylpiperidine-4- oxy)silane, phenyl-tris(2,2,6,6-tetramethylpiperidine-4- oxy)silane, tris(1-propyl-2,2,6,6-tetramethylpiperidine-4-yl) phosphate, tris(1-propyl-2,2,6,6-tetramethylpiperidine-4-yl) phosphate, phenyl-bis(1,2,6,6-pentamethylpiperidine-4-yl) phosphate, 4-hydroxy-1,2,6,6-pentamethylpiperidine, 4-hydroxy-N-hydroxyethyl-2,2,6,6-tetramethylpiperidine, 4-hydroxy-N-(2-hydroxypropyl)-2,2,6,6-tetramethyl piperidine, 1-glycidyl-4-hydroxy-2,2,6,6-tetramethyl piperidine, 4-decyl-N-(2,2,6,6-tetramethylpiperidinyl) succinate.
Most useful in this invention are the 2,2,6,6-tetramethylpipiperidines, 1,2,2,6,6-pentamethylpipiperidines, 1-oxo-2,2,6,6-tetramethylpipiperidines, and 1-alkoxy-2,2,6,6-tetramethylpipiperidines.

3. Polymers Containing Hindered Amines

Polymeric 2,2,6,6-tetraalkylpipiperidines and 1,2,2,6,6-pentamethylpipiperidines are also prevalent and may be used in this formulation. The polymeric compounds used in this invention are described by Schumacher et al., U.S. Pat. No. 5,073,278, by Evans et al. in U.S. Pat. No. 5,268,113, and by Kazmierczak et al. in U.S. Pat. No. 4,857,595. There are several kinds of polymeric piperidine compounds available. Commercially available examples include Tinuvin® 622 from Ciba and Songlight® 9440 from Songwon.

Another type of hindered amine has been disclosed in U.S. Pat. No. 5,098,944 and describes hindered amines of the type shown in general formula (IV).

wherein PSP represents a substituent derived from a cyclic amine represented by a structure selected from the group in general formulae (V)

wherein PSP represents a substituent derived from a cyclic amine represented by a structure selected from the group consisting of wherein R24 represents C1-C24 alkyl, C2-C20 cycloalkyl C3-C20 aralkyl or alkaryl, C7-C24 alkoxyalkyl, or C1-C20 aminoalkyl, or C1-C20 aminoacycloalkyl; R25, R26, R27, and R28 independently represent C1-C24 alkyl, and R25 with R26, or R27 with R28 are cyclizable to C3-C22 cycloalkyl including the C1 and C2 atoms respectively, of the piperazin-2-one ring; R29 and R30 independently represent C1-C24 alkyl, and polymethylene having from 4 to 7 carbon atoms which are cyclizable; R31 represents H, C1-C6 alkyl, and phenyl; R32 represents C1-C25 alkyl, H, or O, or alkoxyl with a hydrocarbon chain between 1 and 25 carbon atoms; and, p represents an integer in the range from 2 to about 10.

Diarylamines

The diarylamines used in this invention are of the type Ar2NR. Since these are well known antioxidants in the art, there is no restriction on the type of diarylamines used in this invention, although there is the requirement of solubility in the lubricating composition.

The alkylated diphenylamines are well known antioxidants and there is no particular restriction on the type of secondary diarylamine used in the invention. Preferably, the secondary diarylamine antioxidant has the general formula (X) where R33 and R34 each independently represents a substituted or unsubstituted aryl group having from 6 to 30 carbon atoms. R35 represents either a H atom or an alkyl group containing from 1 to 30 carbon atoms. Illustrative of substituents for the aryl there can be mentioned aliphatic hydrocarbon groups such as alkyl having from about 1 to 20 carbon atoms, hydroxy, carboxyl or nitro, e.g., an aryl group having from 7 to 20 carbon atoms in the alkyl group. The aryl is preferably substituted or unsubstituted phenyl or naphthyl, particularly wherein one or both of the aryl groups are substituted with an alkyl such as one having from 4 to 18 carbon atoms. R35 can be either H or alkyl from 1 to 30 carbon atoms. The alkylated diphenylamines used in this invention can be of a structure other than that shown in the above formula which shows but one nitrogen atom in the molecule. Thus, the alkylated diphenylamine can be of a different structure provided that at least one nitrogen has 2 aryl groups attached thereto, e.g., as in the case of various diamines having a secondary nitrogen atom as well as two aryls on one of the nitrogens. The alkylated diphenylamines used in this invention preferably have antioxidant properties in lubricating oils, even in the absence of the molybdenum compound.

Examples of some alkylated diphenylamines that may be used in this invention include: diphenyl amine, 3-hydroxydiphenylamine; N-phenyl-1,2,2,6,6-pentamethylpipiperidines; N-phenyl-1,4-phenylenediamine; dibutylphenylamine; diocetylphenylamine; dinonylphenylamine; phenyl-alpha-naphthylamine; phenyl-beta-naphthylamine; diheptyldiphenylamine; and p-oriented styrenated diphenylamine.

Phenothiazines

Phenothiazines are another class of diarylamines with the general structure (VII),

Where R36 is H, or an alkyl from 1 to 30 carbon atoms, and R37 and R38 are alkyl from 1 to 30 carbon atoms

Lubricating Oil Compositions

The lubricating oil compositions of this invention can be prepared by adding the sulfur-containing compound,
the hindered amine, and the aromatic amine to a basestock. Combinations can contain from 0.001 to 10 weight percent of each of the three additives in the lubricating oil.

Grease Compositions

[0046] In another embodiment, a grease composition of this invention can be prepared by adding sulfur-containing compound, the hindered amine, and the aromatic amine to a base grease. Combinations can contain from 0.001 to 10 weight percent of each of the three additives in the lubricating oil.

Other Additives

[0047] In addition, other additives can be added to the lubricating compositions described above. These include the following components:

[0048] Other antioxidants, including phenols, hindered phenols, hindered terephens, sulfurarized phenols, zinc dihydrocarbyl dithiophosphates, zinc dithiocarbamates, organophosphites. A more complete list of useful phenols can be found in U.S. Pat. No. 5,073,278 to Schumacher et al.

[0049] Antiwear additives, including zinc dihydrocarbyl dithiophosphates, tricresol phosphate, dilauryl phosphate.

[0050] Dispersants, including polymethacrylates, styrene maleic ester copolymers, substituted succinimides, polyamine succinimides, polyhydroxy succinic esters, substituted Mannich bases, and substituted triazoles.

[0051] Detergents, including neutral and overbased alkali and alkaline earth metal sulfonates, neutral and overbased alkali and alkaline earth metal phosphates, sulfurarized phenates, overbased phosphonates, and thiophosphonates.

[0052] Viscosity index improvers, including polyacrylates, polymethacrylates, vinylpyrrolidone/methacrylate copolymers, polyvinylpyrrolidones, polyybutenes, olefin copolymers, styrene/acylate copolymers.

[0053] Pour point depressants, including polymethacrylate and alkylated naphtalene derivatives.

Example 1

Lubricant Compositions Containing a Hindered Amine, Diaryl Amine and a Sulfurarized Fatty Acid

[0055] Lubricant compositions prepared as above in Example 1, containing the combination of Cyasorb® UV-3853 and Vanlube® 961, and as sulfur-containing compound Arkema® VPS 15, a sulfurarized fatty acid mixture containing approximately 15% sulfur (w/w) from Arkema. PDSC (ASTM D1686) was performed as in Example 1, and data is reported in Table II.

TABLE II

<table>
<thead>
<tr>
<th>PDSC Induction Times for Motor Oil Blends</th>
<th>% Additive</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cyasorb UV 3853</td>
<td>0.25</td>
</tr>
<tr>
<td>VL 961</td>
<td>0.50</td>
</tr>
<tr>
<td>Inflameum C9268</td>
<td>3.90</td>
</tr>
<tr>
<td>Durasyn 166</td>
<td>95.10</td>
</tr>
<tr>
<td>PDSC @ 180° C, 500 lbs O2, 100 ml/min flow</td>
<td>Minutes to induction 69.1 173.3 76.6 8.3 13.4 185.2 213.5</td>
</tr>
</tbody>
</table>
Comparative Example 3
Lubricant Compositions Containing a Hindered Amine, Diaryl Amine and a Sulfurized Olefin

[0056] Lubricant compositions were prepared according to Example 1 above, containing the combination of Cyasorb UV3853 and Vanlube® 961, with sulfur-containing compound VANLUBE® SB, a sulfurized olefin containing approximately 45% sulfur from R.T. Vanderbilt Company, Inc. PDSC (ASTM D1686) was performed as in Example 1, and data is reported in Table III.

<table>
<thead>
<tr>
<th>PDSC Induction Times for Motor Oil Blends</th>
</tr>
</thead>
<tbody>
<tr>
<td>% Additive</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>Cyasorb UV 3853</td>
</tr>
<tr>
<td>VL 961</td>
</tr>
<tr>
<td>VL SB</td>
</tr>
<tr>
<td>Infremin 9268</td>
</tr>
<tr>
<td>Danfyn 166</td>
</tr>
<tr>
<td>500 lbs O₂</td>
</tr>
<tr>
<td>500 ml/min flow</td>
</tr>
<tr>
<td>Minutes to induction</td>
</tr>
<tr>
<td></td>
</tr>
</tbody>
</table>

What is claimed is:
1. A lubricating composition comprising at least 90% by weight of a lubricant base stock, and an antioxidant additive composition comprising:
   (a) a metal-free sulfur-containing compound at between about 0.001 and 10%, chosen from the group consisting of sulfurized fatty acids, ashless dithiocarbamates, tetramethylthiuram disulfides, and thiadiazoles,
   (b) a hindered amine at about 0.001 and 10%, and
   (c) a diaryl amine at about 0.001 and 10%.
2. The lubricant composition according to claim 1, wherein the additive composition comprises:
   (a) the metal-free sulfur-containing compound at between about 0.1 and 1.0%,
   (b) the hindered amine at about 0.05 and 1.0%.
   (c) the diaryl amine at about 0.1 and 1.0%.
3. The lubricant composition according to claim 2, wherein the additive composition comprises:
   (a) the metal-free sulfur-containing compound at between about 0.25 and 0.5%,
   (b) the hindered amine at about 0.1 and 0.5%.
   (c) the diaryl amine at between about 0.25 and 0.5%.
4. The lubricant composition according to claim 3, wherein the additive composition comprises:
   (a) the metal-free sulfur-containing compound at about 0.5%,
   (b) the hindered amine at about 0.1%,
   (c) the diaryl amine at about 0.4%.
5. The lubricant composition according to claim 1, wherein the metal-free sulfur-containing compound is chosen from the group consisting of ashless dithiocarbamates and sulfurized fatty acids.
6. The lubricant composition according to claim 5, wherein the ashless dithiocarbamate is methylenebis(dibutylthiocarbamate) or sulfurized fatty acids.
7. The lubricant composition according to claim 1, wherein the sulfur-containing compound is methylenebis(dibutylthiocarbamate) and the hindered amine is 4-piperidol-2,2,6,6-tetramethyl-RPW stearin (fatty acids mixture).
8. The lubricant composition according to claim 2, wherein the sulfur-containing compound is methylenebis(dibutylthiocarbamate).
9. The lubricant composition according to claim 3, wherein the sulfur-containing compound is methylenebis(dibutylthiocarbamate).
10. The lubricant composition according to claim 4, wherein the sulfur-containing compound is methylenebis(dibutylthiocarbamate).
11. The lubricant composition according to claim 1, wherein the additive composition comprises:
   (a) methylenebis(dibutylthiocarbamate) at about 0.5%,
   (b) 4-piperidol-2,2,6,6-tetramethyl-RPW stearin (fatty acids mixture) at about 0.1%, and
   (c) the diaryl amine at about 0.4%.
12. The lubricant composition according to claim 1, wherein the sulfur-containing compound is sulfurized fatty acid.
13. The lubricant composition according to claim 1, wherein the sulfur-containing compound is sulfurized fatty acid and the hindered amine is 4-piperidol-2,2,6,6-tetramethyl-RPW stearin (fatty acids mixture).
14. The lubricant composition according to claim 2, wherein the sulfur-containing compound is sulfurized fatty acid.
15. The lubricant composition according to claim 3, wherein the sulfur-containing compound is sulfurized fatty acid.
16. The lubricant composition according to claim 4, wherein the additive composition comprises:
   (a) sulfurized fatty acid at about 0.5%,
   (b) 4-piperidol-2,2,6,6-tetramethyl-RPW stearin (fatty acids mixture) at about 0.1%, and
   (c) the diaryl amine at about 0.4%.

* * * * *