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#### (54) ADDITIVE CONCENTRATE AND A METHOD OF LUBRICATING TRANSMISSIONS

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#### **Related U.S. Application Data**

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### (57) ABSTRACT

The present invention relates to an additive concentrate comprising a friction modifier, a corrosion inhibitor, and an oil of lubricating viscosity. The invention further provides a method for lubricating a transmission using the additive concentrate.

#### ADDITIVE CONCENTRATE AND A METHOD OF LUBRICATING TRANSMISSIONS

#### FIELD OF INVENTION

**[0001]** The present invention relates to an additive concentrate comprising a friction modifier, a corrosion inhibitor, and an oil of lubricating viscosity. The invention further provides a method for lubricating a transmission using the additive concentrate.

#### BACKGROUND OF THE INVENTION

[0002] Driveline transmissions especially automatic transmission fluids (ATFs), present highly challenging technological problems and solutions for satisfying the multiple and often conflicting lubricating and power transmitting requirements of modern automatic transmissions (including continuously variable transmissions of various types). Many additive components are typically included in an ATF, providing such performance characteristics as lubrication, dispersancy, friction control (for clutches), antiwear performance, anti-shudder performance, anti-corrosion and antioxidation performance. However, over periods of use, the additive components are consumed which can detrimentally damage transmissions. For example a transmission may experience increased shudder, chattering, or squawking (a vibration oscillation of a clutch that may be audible). Reducing the impact of shudder, chattering, or squawking has been achieved by employing friction modifiers such as those defined in U.S. Pat. Nos. 3,156,653, 3,070,546, 3,275,559, and 5,750,476; and U.S. Patent Applications 60/725,360 (now WO 2007/044820), 2005/0250655, and 2003/0220208. [0003] U.S. Pat. Nos. 3,156,653; and 3,070,546 both disclose lubricating compositions containing a carboxy amide. The lubricating compositions are suitable for reducing squawking or chattering.

**[0004]** U.S. Pat. No. 3,275,559 discloses an automatic transmission with suppressed squawking by employing a lubricating composition containing an N-alkyl alkylene diamine.

[0005] U.S. Pat. No. 5,750,476 discloses improving antishudder durability of a power transmission by employing a lubricating composition containing an anti-shudder improving effective amount of (a) an isomerised alkenyl succinimide, and (b) an oil-soluble phosphorus-containing compound. [0006] US Patent Application 2003/0220208 discloses a lubricating composition containing an additive combination of (i) an oil-soluble fatty acid ester of a polyhydric alcohol and (ii) an oil-soluble fatty acid amide. The lubricating composition is useful for the reduction of brake and clutch noise. [0007] US Patent Application 2005/0250655 discloses a lubricating composition containing a reaction product of a carboxylic acid or a reactive equivalent thereof with an aminoalcohol, wherein the friction modifier contains at least two hydrocarbyl groups, each containing at least 6 carbon atoms. [0008] U.S. Patent Application 60/725,360 (filed on Oct. 11, 2005, inventors Bartley, Lahiri, Baker and Tipton), now WO 2007/044820, discloses a reaction product of a hydroxy-

alkyl compound with an acylating agent or an amine. The resultant product may be an amide represented by the formula  $R^1R^2N$ —C(O) $R^3$ , wherein  $R^1$  and  $R^2$  are each independently hydrocarbyl groups of at least 6 carbon atoms and  $R^3$  is a hydroxyalkyl group of 1 to 6 carbon atoms or a group formed

by the condensation of said hydroxyalkyl group, through a hydroxyl group thereof, with an acylating agent.

**[0009]** Overcoming shudder, chattering, or squawking is typically achieved by replacing the transmissions' fluid (typically a factory fill fluid) with a replacement fluid. However, this results in excessive repair cost due to the difficulty of changing the fluid and increased waste lubricant. Thus it would be desirable to lubricate a transmission lubricant capable of minimising noise, shudder, chattering, or squawking, whilst also not adversely affecting the frictional balance and clutch holding capacity, nor increasing lubricant waste. The present invention provides a means of overcoming shudder, chattering, or squawking whilst at the same time minimising at least one of the cost, time, and waste involved in a conventional fluid replacement.

#### SUMMARY OF THE INVENTION

**[0010]** In one embodiment the invention provides an additive concentrate comprising:

- **[0011]** (a) 4 wt % to 67 wt % of a static friction reducing friction modifier;
- **[0012]** (b) 0 wt % to 10 wt %, or 0.1 wt % to 5 wt % of a corrosion inhibitor;
- **[0013]** (c) 3 wt % to 96 wt % of an oil of lubricating viscosity; and
- [0014] (d) 0 wt % to 20 wt % of other performance additives.

**[0015]** In one embodiment the invention provides a method of lubricating a transmission by supplying to the transmission a lubricating composition further treated with an additive concentrate comprising:

- **[0016]** (a) 4 wt % to 67 wt % of a static friction reducing friction modifier;
- **[0017]** (b) 0 wt % to 10 wt %, or 0.1 wt % to 5 wt % of a corrosion inhibitor;
- **[0018]** (c) 3 wt % to 96 wt % of an oil of lubricating viscosity; and
- [0019] (d) 0 wt % to 20 wt % of other performance additives.

**[0020]** In one embodiment the invention provides a method of lubricating a transmission containing a lubricant comprising supplying to the lubricant within the transmission an additive concentrate comprising:

- **[0021]** (a) 4 wt % to 67 wt % of a static friction reducing friction modifier;
- **[0022]** (b) 0 wt % to 10 wt %, or 0.1 wt % to 5 wt % of a corrosion inhibitor;
- **[0023]** (c) 3 wt % to 96 wt % of an oil of lubricating viscosity; and
- [0024] (d) 0 wt % to 20 wt % of other performance additives.

**[0025]** In one embodiment the invention provides an additive concentrate comprising:

- **[0026]** (a) 4 wt % to 67 wt % of a friction modifier with a co-efficient of friction of greater than 0.09;
- **[0027]** (b) 0 wt % to 10 wt %, or 0.1 wt % to 5 wt % of a corrosion inhibitor;
- **[0028]** (c) 3 wt % to 96 wt % of an oil of lubricating viscosity; and
- **[0029]** (d) 0 wt % to 20 wt % of other performance additives.

**[0030]** In one embodiment the invention provides a method of lubricating a transmission by supplying to the transmission a lubricating composition further treated with an additive concentrate comprising:

**[0031]** (a) 4 wt % to 67 wt % of a friction modifier with a co-efficient of friction of greater than 0.09;

- **[0032]** (b) 0 wt % to 10 wt %, or 0.1 wt % to 5 wt % of a corrosion inhibitor;
- **[0033]** (c) 3 wt % to 96 wt % of an oil of lubricating viscosity; and
- [0034] (d) 0 wt % to 20 wt % of other performance additives.

**[0035]** In one embodiment the invention provides a method of lubricating a transmission containing a lubricant comprising supplying to the lubricant within the transmission an additive concentrate comprising:

**[0036]** (a) 4 wt % to 67 wt % of a friction modifier with a co-efficient of friction of greater than 0.09;

- [0037] (b) 0 wt % to 10 wt  $\sqrt[6]{}$ , or 0.1 wt % to 5 wt % of a corrosion inhibitor;
- **[0038]** (c) 3 wt % to 96 wt % of an oil of lubricating viscosity; and
- [0039] (d) 0 wt % to 20 wt % of other performance additives.

**[0040]** In one embodiment the invention provides for the use of the additive concentrate described herein as a top treat package to diminish squawk in a transmission.

#### DETAILED DESCRIPTION OF THE INVENTION

**[0041]** The present invention provides an additive concentrate and a method for lubricating a transmission as disclosed above.

**[0042]** The additive concentrate is typically added as a top treat. The top treat may be added to the transmission as a service-fill or factory-fill. Typically the top treat may be added to the transmission as a service-fill.

**[0043]** The additive concentrate may contain the static friction reducing friction modifier at 4 wt % to 67 wt %, or 10 wt % to 50 wt %, or 20 wt % to 40 wt %.

**[0044]** The additive concentrate may contain the corrosion inhibitor at 0 wt % to 10 wt %, or 0.1 wt % to 5 wt %, or 0.2 wt % to 3 wt %.

**[0045]** The additive concentrate may contain the oil of lubricating viscosity at 3 wt % to 96 wt %, or 33 wt % to 89 wt %, or 47 wt % to 77.8 wt %.

**[0046]** The additive concentrate may contain the total of other performance additives at 0 wt % to 20 wt %, or 1 wt % to 12 wt %, or 2 wt % to 10 wt %.

**[0047]** The additive concentrate may be added to a transmission at a treat rate of 0.1 wt % to 30 wt %, or 1 wt % to 15 wt %.

Static Friction Reducing Friction Modifiers

**[0048]** The static friction reducing friction modifier may also be described as a friction modifier with a co-efficient of friction of greater than 0.09. The static friction is expressed in term of  $\mu$ T or the stabilized static coefficient from the SAE#2 test procedure. The test procedure is described in the Japanese Automobile Standard, JASO M-348-95, "Test method for friction property of automatic transmission fluids".

**[0049]** The static friction reducing friction modifier may also be described as a friction modifier of the type described in PCT Publication WO 2004/007652 A1. Typically static

friction reducing friction modifiers are characterised by their ability to provide a positive slope in a friction versus speed curve whilst maintaining a high static coefficient of friction. [0050] In one embodiment the static friction reducing friction modifier may be formed by the condensation of the hydroxyalkyl compound with an acylating agent or an amine. A more detailed description of the hydroxyalkyl compound is described in U.S. Patent Application 60/725,360 (filed on Oct. 11, 2005, inventors Bartley, Lahiri, Baker and Tipton) in paragraphs 8, and 19-21, now WO 2007/044820. The friction modifier disclosed in U.S. Patent Application 60/725,360 may be an amide represented by the formula  $R^{1}R^{2}N$ —C(O)  $R^3$ , wherein  $R^1$  and  $R^2$  are each independently hydrocarbyl groups of at least 6 carbon atoms and R<sup>3</sup> is a hydroxyalkyl group of 1 to 6 carbon atoms or a group formed by the condensation of said hydroxyalkyl group, through a hydroxyl group thereof, with an acylating agent. Preparative Examples are disclosed in Examples 1 and 2 (paragraphs 68 and 69 of U.S. Patent Application 60/725,360). In one embodiment the amide of a hydroxylalkyl compound is prepared by reacting glycolic acid, that is, hydroxyacetic acid, HO-CH2-COOH with an amine.

**[0051]** In one embodiment the static friction reducing friction modifier may be derived from the reaction product of a carboxylic acid or a reactive equivalent thereof with an aminoalcohol, wherein the friction modifier contains at least two hydrocarbyl groups, each containing at least 6 carbon atoms. The number of carbon atoms may range from 6 to 30, or 8 to 20. An example of such a friction modifier includes the reaction product of isostearic acid or an alkyl succinic anhydride with tris-hydroxymethylaminomethane. A more detailed description of such a friction modifier is disclosed in US Patent Application 2003/22000 (or International Publication WO04/007652) in paragraphs 8 and 9 to 14.

**[0052]** In one embodiment the static friction reducing friction modifier may be a polyol ester as described in U.S. Pat. No. 5,750,476 column 8, line 40 to column 9, line 28.

**[0053]** In one embodiment the static friction reducing friction modifier may be a low potency friction modifier as described in U.S. Pat. No. 5,840,662 in column 2, line 28 to column 3, line 26. U.S. Pat. No. 5,840,662 further discloses in column 3, line 48 to column 6, line 25 specific materials and methods of preparing the low potency friction modifier.

**[0054]** In one embodiment the friction modifier may be a hydroxylamine compound as defined in column 37, line 19, to column 39, line 38 of U.S. Pat. No. 5,534,170. Optionally the hydroxylamine may be borated as such products are described in column 39, line 39 to column 40 line 8 of U.S. Pat. No. 5,534,170.

**[0055]** In one embodiment the static friction reducing friction modifier comprises one or more of a hydroxy-amide, a hydroxy amide mono- or di-ester, a hydroxy oxazoline, a hydroxy oxazoline ester, an oxazoline diester, a tri-ester amide, or mixtures thereof.

**[0056]** In one embodiment the static friction reducing modifier is an amide represented by the formula  $R^1R^2N$ —C (O) $R^3$ , wherein  $R^1$  and  $R^2$  are each independently hydrocarbyl groups of at least 6 carbon atoms and  $R^3$  is a hydroxyalkyl group of 1 to 6 carbon atoms or a group formed by the condensation of said hydroxyalkyl group, through a hydroxyl group thereof, with an acylating agent.

**[0057]** In one embodiment the static friction reducing modifier is other than an amide represented by the formula  $R^1$ ,  $R^2N$ —C(O) $R^3$ , wherein  $R^1$  and  $R^2$  are each indepen-

dently hydrocarbyl groups of at least 6 carbon atoms and R<sup>3</sup> is a hydroxyalkyl group of 1 to 6 carbon atoms or a group formed by the condensation of said hydroxyalkyl group, through a hydroxyl group thereof, with an acylating agent. **[0058]** In one embodiment the static friction reducing modifier is a tertiary amine. The amine will contain three substituent hydrocarbyl groups, two of which are alkyl groups. The amine is represented by the formula

#### $R^4 R^5 N R^6$

wherein  $R^4$  and  $R^5$  are each independently an alkyl group of at least 6 carbon atoms (e.g., 8 to 20 carbon atoms or 10 to 18 or 12 to 16) and  $R^6$  is a polyhydroxyl-containing alkyl group or a polyhydroxyl-containing alkoxyalkyl group.

**[0059]** In one embodiment the amine comprises a product of di-cocoalkyl amine or homologous amines. Di-cocoalkyl amine (or di-cocoamine) is a secondary amine in which two of the R groups in the above formula are predominantly  $C_{12}$  groups, derived from coconut oil and the remaining R group is H.

**[0060]** In one embodiment,  $R^6$  is a polyol-containing alkyl group (that is, a group containing 2 or more hydroxy groups) or a group containing one or more hydroxy groups and one or more amine groups. For instance,  $R^6$  may be  $-CH_2-CHOH-CH_2OH$  or a homologue thereof, containing, for example, 3 to 8 carbon atoms or 3 to 6 carbon atoms or 3 to 4 carbon atoms, and 2, 3, 4 or more hydroxy groups (normally no more than one hydroxy group per carbon atom). A typical resulting product may thus be represented by

 $R^4R^5N - \!\!\!\!- CH_2 - \!\!\!\!- CHOH - \!\!\!- CH_2OH$ 

[0061] or homologues thereof, where  $R^4$  and  $R^5$  are, as described above, independently alkyl groups of 8 to 20 carbon atoms. Such products may be obtained by the reaction of a dialkyl amine with an epoxide or chlorohydroxy compound. In particular, reaction of a secondary amine with glycidol (2,3-epoxy-1-propanol) or "chloroglycerine" (that is, 3-chloropropane-1,2-diol) may be effective under conditions as described above. Such materials based on the reaction of dicocoamine with one or more moles of glycidol or chloroglycerine are particularly useful in providing friction-modifying performance. If reaction is with multiple moles of glycidol or chloroglycerine, or other epoxyalkanols or chlorodiols, a dimeric or oligomeric ether-containing group, that is, a hydroxyl-substituted alkoxyalkyl group, may result.

**[0062]** In one embodiment the static friction reducing modifier may be described as a compound comprising a core portion comprising 3 to 8 carbon atoms, (e.g., 3 to 6, or 3 carbon atoms), said core portion being substituted by:

(i) at least two hydroxy groups, or at least one hydroxy group and at least one alkoxy group of 1 to 4 carbon atoms wherein said alkoxy group is further substituted by at least one hydroxy group or another such alkoxy group; and

(ii) at least one amino group, the nitrogen atom thereof bearing two hydrocarbyl groups, each such hydrocarbyl group independently having 6 to 30 carbon atoms.

**[0063]** In one embodiment the static friction reducing modifier is a secondary or tertiary amine. The amine will contain at least two substituent hydrocarbyl groups, for example, alkyl groups. The amine is represented by the formula

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wherein  $R^7$  and  $R^8$  are each independently an alkyl group of at least 6 carbon atoms (e.g., 8 to 20 carbon atoms or 10 to 18 or 12 to 16) and  $R^9$  is a hydroxyl-containing alkyl group, a hydroxyl-containing alkoxyalkyl group, an amine-containing alkyl group, a hydrocarbyl group, or hydrogen, provided that when  $R^9$  is H, then at least one of  $R^7$  and  $R^8$  is an alkyl group of 8 to 16 carbon atoms such as, for instance, 10 to 16 carbon atoms or 12 to 14 carbon atoms. In certain embodiments both of  $R^7$  and  $R^8$  are alkyl groups of 8 to 16, or 10 to 16, or 12 to 14 carbon atoms. A commercial example of such an amine is Armeen  $2C^{TM}$ , which is a secondary amine where the two alkyl groups are believed to be predominately  $C_{12}$  to  $C_{14}$ .

**[0064]** In one embodiment the amine comprises di-cocoalkyl amine or homologous amines. Di-cocoalkyl amine (or di-cocoamine) is a secondary amine in which two of the R groups in the above formula are predominantly  $C_{12}$  groups, derived from coconut oil and the remaining R group is H. In another embodiment the amine may be Armeen HTL8<sup>TM</sup>, which is believed to be a secondary amine with mixed alkyl groups comprising 2-ethylhexyl groups (C8) and hydrogenated tallow groups (largely C16-18).

**[0065]** In another embodiment,  $\mathbb{R}^9$  in the above structure is  $-CH_2$ —CHOH— $\mathbb{R}^{10}$ , where  $\mathbb{R}^{10}$  is hydrogen or an alkyl group, e.g., a methyl group or an alkyl group of 1 to 26 carbon atoms, or 6 to 20 carbon atoms, or 12 to 18 carbon atoms, or 14 to 16 carbon atoms, or 14 carbon atoms. Such materials can be prepared by reaction of a secondary amine such as di-coccoalkylamine with an epoxide, such as propylene oxide (in the case where  $\mathbb{R}^{10}$  is methyl). The resulting products can thus, more specifically, be represented by the structure

R<sup>7</sup>R<sup>8</sup>N—CH<sub>2</sub>—CHOH—CH<sub>3</sub>

where  $R^7$  and  $R^8$  are, as described above, independently alkyl groups of 8 to 20 carbon atoms. The reaction of the dialkyl amine and the epoxide can be effected by reaction under pressure in the presence of a basic catalyst. In another embodiment,  $R^9$  is an aminopropyl groups such as —CH<sub>2</sub>— CH<sub>2</sub>—CH<sub>2</sub>—NH<sub>2</sub>, and the resulting product can be prepared by reaction of a secondary amine such as dicocoalkyl amine with acrylonitrile followed by a reduction.

**[0066]** In another embodiment,  $\mathbb{R}^9$  can be a polyol-containing alkyl group (that is, a group containing 2 or more hydroxy groups) or a group containing one or more hydroxy groups and one or more amine groups. For instance,  $\mathbb{R}^9$  may be  $-CH_2$ -CHOH--CH<sub>2</sub>OH or a homologue thereof, containing, for example, 3 to 8 carbon atoms or 3 to 6 carbon atoms or 3 to 4 carbon atoms, and 2, 3, 4 or more hydroxy groups (normally no more than one hydroxy group per carbon atom). A typical resulting product may thus be represented by

#### R<sup>7</sup>R<sup>8</sup>N—CH<sub>2</sub>—CHOH—CH<sub>2</sub>OH

or homologues thereof, where  $R^7$  and  $R^8$  are, as described above, independently alkyl groups of 8 to 20 carbon atoms. Such products may be obtained by the reaction of a dialkyl amine with an epoxide or chlorohydroxy compound. In particular, reaction of a secondary amine with glycidol (2,3epoxy-1-propanol) or "chloroglycerine" (that is, 3-chloropropane-1,2-diol) may be effective under conditions as described above. Such materials based on the reaction of dicocoamine with one or more moles of glycidol or chloroglycerine are particularly useful in providing friction-modifying performance. If reaction is with multiple moles of glycidol or chloroglycerine, or other epoxyalkanols or chlorodiols, a dimeric or oligomeric ether-containing group, that is, a hydroxyl-substituted alkoxyalkyl group, may result. **[0067]** In certain embodiments, thus, the amine may be a tertiary amine, that is, in which  $R^9$  is other than hydrogen. If all three groups  $R^7$ ,  $R^8$ , and  $R^9$  are alkyl groups, then  $R^9$  may also be an alkyl group of at least 6 carbon atoms (e.g., 8 to 20 carbon atoms or 10 to 18 or 12 to 16).

#### Corrosion Inhibitor

**[0068]** The lubricating composition may further comprise a corrosion inhibitor or mixtures thereof. In one embodiment the corrosion inhibitor also exhibits antiwear properties.

**[0069]** The corrosion inhibitors of the invention include benzotriazoles (typically tolyltriazole), 2-alkyldithiobenzimidazoles or 2-alkyldithio benzothiazoles, 1,2,4-triazoles, benzimidazoles, octylamine octanoate, condensation products of dodecenyl succinic acid or anhydride, dimercaptothiadiazoles and reactive equivalents thereof, or mixtures thereof. **[0070]** The corrosion inhibitor may comprise at least one of a dimercaptothiadiazole, 5-dimercapto-[1,3,4]-thiadiazole, 3,5-dimercapto-[1,2,4]-thiadiazole, 3,4-dimercapto-[1,2,5]thiadiazole, or 4-5-dimercapto-[1,2,3]-thiadiazole. Typically readily available materials such as 2,5-dimercapto-1,3-4thiadiazole or a hydrocarbyl-substituted 2,5-dimercapto-1,3-4thiadiazole are commonly utilised, with 2,5-dimercapto-[1,2,4]-thiadiazole.

3,4]-thiadiazole most commonly utilised due to availability. In several embodiments the number of carbon atoms on the hydrocarbyl-substituent group includes 1 to 30, 2 to 25, 4 to 20, 6 to 16, or 8 to 10.

[0071] In one embodiment, the thiazole compound may be the reaction product of a phenol with an aldehyde and a dimercaptothiadiazole. The phenol may be an alkyl phenol wherein the alkyl group contains at least about 6, e.g., 6 to 24, or 6, or 7, to 12 carbon atoms. The aldehyde may be an aldehyde containing 1 to 7 carbon atoms or an aldehyde synthon, such as formaldehyde. In one embodiment, the aldehyde is formaldehyde or paraformaldehyde. The aldehyde, phenol and dimercaptothiadiazole are typically reacted by mixing them at a temperature up to about 150° C. such as 50° C. to 130° C., in molar ratios of 0.5 to 2 moles of phenol and 0.5 to 2 moles of aldehyde per mole of dimercaptothiadiazole. In one embodiment, the three reagents are reacted in equal molar amounts. The product may be described as an alkylhydroxyphenylmethylthio-substituted [1,3,4]-thiadiazole; the alkyl moiety may be, among others, hexyl, heptyl, octyl, or nonyl.

**[0072]** Useful thiadiazole compounds thus may include 2-alkyldithio-5-mercapto-[1,3,4]-thiadiazoles, 2,5-bis(alkyldithio)-[1,3,4]-thiadiazoles, 2-alkylhydroxyphenylmethylthio-5-mercapto-[1,3,4]-thiadiazoles, and mixtures thereof.

[0073] Examples of suitable thiadiazole compounds include 2-octyldithio-5-mercapto-1,3,4-thiadiazole, 2-nonyldithio-5-mercapto-1,3,4-thiadiazole, 2-dodecydithio-5mercapto-1,3,4-thiadiazole, 2,5-dimercapto-1,3-4-thiadiazole (e.g., 2,5-bis(alkyl-dithio)-1,3,4-thiadiazoles) include 2,5-bis(tert-octyldithio)-1,3,4-thiadiazole 2,5-bis(tert-nonyldithio)-1,3,4-thiadiazole, 2,5-bis(tert-decyldithio)-1,3,4thiadiazole, 2,5-bis(tert-undecyldithio)-1,3,4-thiadiazole, 2,5-bis(tert-dodecyldithio)-1,3,4-thiadiazole, 2,5-bis(terttridecyldithio)-1,3,4-thiadiazole, 2,5-bis(tert-tetradecyldithio)-1,3,4-thiadiazole, 2,5-bis(tert-pentadecyldithio)-1.3.4-thiadiazole, 2,5-bis(tert-hexadecyldithio)-1,3,4thiadiazole, 2,5-bis(tert-heptadecyldithio)-1,3,4-thiadiazole, 2,5-bis(tert-octadecyldithio)-1,3,4-thiadiazole, 2,5-bis(tertnonadecyldithio)-1,3,4-thiadiazole or 2,5-bis(tert-eicosyldithio)-1,3,4-thiadiazole, or oligomers thereof. In one embodiment the hydrocarbyl-substituted 2,5-dimercapto-1, 3-4-thiadiazole comprises at least one of 2,5-bis(tert-octyldithio)-1,3,4-thiadiazole 2,5-bis(tert-nonyldithio)-1,3,4thiadiazole, or 2,5-bis(tert-decyldithio)-1,3,4-thiadiazole.

#### Oils of Lubricating Viscosity

**[0074]** The lubricating composition comprises an oil of lubricating viscosity. Such oils include natural and synthetic oils, oil derived from hydrocracking, hydrogenation, and hydrofinishing, unrefined, refined and re-refined oils and mixtures thereof.

**[0075]** Unrefined oils are those obtained directly from a natural or synthetic source generally without (or with little) further purification treatment.

**[0076]** Refined oils are similar to the unrefined oils except they have been further treated in one or more purification steps to improve one or more properties. Purification techniques are known in the art and include solvent extraction, secondary distillation, acid or base extraction, filtration, and percolation.

**[0077]** Re-refined oils are also known as reclaimed or reprocessed oils, and are obtained by processes similar to those used to obtain refined oils and often are additionally processed by techniques directed to removal of spent additives and oil breakdown products.

**[0078]** Natural oils useful in making the inventive lubricants include animal oils (e.g., lard oil), vegetable oils (e.g., castor oil), mineral lubricating oils such as liquid petroleum oils and solvent-treated or acid-treated mineral lubricating oils of the paraffinic, naphthenic or mixed paraffinic-naphthenic types and oils derived from coal or shale or mixtures thereof.

**[0079]** Synthetic lubricating oils are useful and include hydrocarbon oils such as polymerised and interpolymerised olefins (e.g., polybutylenes, polypropylenes, propyleneisobutylene copolymers); poly(1-hexenes), poly(1-octenes), poly(1-decenes), and mixtures thereof; alkyl-benzenes (e.g. dodecylbenzenes, tetradecylbenzenes, dinonylbenzenes, di-(2-ethylhexyl)-benzenes); polyphenyls (e.g., biphenyls, terphenyls, alkylated polyphenyls); alkylated diphenyl ethers and alkylated diphenyl sulphides and the derivatives, analogs and homologs thereof or mixtures thereof.

**[0080]** Other synthetic lubricating oils include polyol esters (such as Prolube®3970), diesters, liquid esters of phosphorus-containing acids (e.g., tricresyl phosphate, trioctyl phosphate, and the diethyl ester of decane phosphonic acid), or polymeric tetrahydrofurans. Synthetic oils may be produced by Fischer-Tropsch reactions and typically may be hydroisomerised Fischer-Tropsch hydrocarbons or waxes. In one embodiment, oils may be prepared by a Fischer-Tropsch gas-to-liquid synthetic procedure as well as other gas-to-liquid oils.

**[0081]** Oils of lubricating viscosity may also be defined as specified in the American Petroleum Institute (API) Base Oil Interchangeability Guidelines. The five base oil groups are as follows: Group I (sulphur content >0.03 wt %, and/or <90 wt % saturates, viscosity index 80-120); Group II (sulphur content  $\leq 0.03$  wt %, and  $\geq 90$  wt % saturates, viscosity index 80-120); Group III (sulphur content  $\leq 0.03$  wt %, and  $\geq 90$  wt % saturates, viscosity index  $\approx 120$ ; Group IV (all polyalphaolefins (PAOs)); and Group V (all others not included in Groups I, II, III, or IV). The oil of lubricating viscosity com-

prises an API Group I, Group II, Group II, Group IV, Group V oil or mixtures thereof. In one embodiment the oil of lubricating viscosity may be an API Group I, Group II, Group III, Group IV oil or mixtures thereof. In one embodiment the oil of lubricating viscosity may be an API Group II, Group III or Group IV oil or mixtures thereof. In one embodiment the oil of lubricating viscosity may be an API Group II or Mixtures thereof.

**[0082]** In one embodiment the oil of lubricating viscosity is other than a white oil base stock.

**[0083]** The amount of the oil of lubricating viscosity present is typically the balance remaining after subtracting from 100 wt % the sum of the static friction reducing friction modifier, the corrosion inhibitor and the other performance additives (described below).

**[0084]** The lubricating composition may be in the form of a concentrate and/or a fully formulated lubricant. If the composition is in the form of a concentrate (which may be combined with additional oil to form, in whole or in part, a finished lubricant), the ratio of the of components (a), (b) and (d) to the oil of lubricating viscosity (c) and/or to diluent oil include the ranges of 1:99 to 99:1 by weight, or 80:20 to 10:90 by weight.

#### Other Performance Additive

**[0085]** The composition of the invention optionally further includes at least one other performance additive. The other performance additives include detergents, viscosity index improvers (also referred to as viscosity modifiers), antiwear agents, friction modifiers other than the static reducing friction modifier of the invention, friction stabilising agents, antioxidants, dispersants, foam inhibitors, demulsifiers, pour point depressants, seal swelling agents and mixtures thereof.

#### Antiwear Agent

**[0086]** The antiwear agent may be a phosphorus-containing acid, salt, or ester, or mixtures thereof.

**[0087]** The antiwear agent may be metal-containing or metal free (prior to being mixed with other components).

**[0088]** The antiwear agent may be derived from a phosphoric acid, phosphorous acid, thiophosphoric acid, thiophosphorous acid, or mixtures thereof.

**[0089]** The antiwear agent may include (i) a non-ionic phosphorus compound; (ii) an amine salt of a phosphorus compound; (iii) an ammonium salt of a phosphorus compound; (iv) a monovalent metal salt of a phosphorus compound, such as a metal dialkyldithiophosphate or a metal dialkylphosphate; or (v) mixtures of (i), (ii), (iii) or (iv).

**[0090]** In one embodiment the antiwear agent comprises a metal dialkyldithiophosphate or a metal dialkylphosphate. The alkyl groups of the dialkyldithiophosphate and/or the dialkylphosphate may be linear or branched containing 2 to 20 carbon atoms, provided that the total number of carbons is sufficient to make the metal dialkyldithiophosphate or -phosphate oil soluble. The metal of the metal dialkyldithiophosphate and/or dialkylphosphate typically includes monovalent or divalent metals. Examples of suitable metals include sodium, potassium, copper, calcium, magnesium, barium or zinc. In one embodiment the antiwear agent is a zinc dialkylphosphate.

**[0091]** Examples of a suitable zinc dialkyldithiophosphate (often referred to as ZDDP, ZDP or ZDTP) include zinc

di-(amyl)dithiophosphate, zinc di-(1,3-dimethylbutyl)dithiophosphate, zinc di-(heptyl)dithiophosphate, zinc di-(octyl) dithiophosphate di-(2-ethylhexyl)dithiophosphate, zinc di-(nonyl)dithiophosphate, zinc di-(decyl)dithiophosphate, zinc di-(dodecyl)dithiophosphate, zinc di-(dodecylphenyl)dithiophosphate, zinc di-(heptylphenyl)dithiophosphate, or mixtures thereof. In one embodiment the zinc dialkyldithiophosphate comprises a mixed alkyl ZDDP compound, wherein the alkyl groups include 2-methylpropyl and amyl. In one embodiment the zinc dialkyldithiophosphate comprises a mixed alkyl ZDDP compound, wherein one alkyl group comprises isopropyl and at least one of 1,3-dimethylbutyl, 2-ethylhexyl and iso-octyl.

**[0092]** In one embodiment the antiwear agent is other than metal dialkyldithiophosphate.

**[0093]** In one embodiment the antiwear agent comprises an ammonium or amine salt of a phosphorus-containing acid or ester.

**[0094]** The amine salt of a phosphorus acid or ester includes phosphoric acid esters and amine salts thereof; dialkyldithiophosphoric acid esters and amine salts thereof; amine salts of phosphites; and amine salts of phosphoruscontaining carboxylic esters, ethers, and amides; and mixtures thereof.

**[0095]** The amine salt of a phosphorus acid or ester may be used alone or in combination. In one embodiment the amine salt of a phosphorus compound is derived from an amine salt of a phosphorus compound, or mixtures thereof.

**[0096]** In one embodiment the amine salt of a phosphorus acid or ester includes a partial amine salt-partial metal salt compounds or mixtures thereof. In one embodiment the amine salt of a phosphorus acid or ester further comprises a sulphur atom in the molecule.

**[0097]** The amines which may be suitable for use as the amine salt include primary amines, secondary amines, tertiary amines, and mixtures thereof. The amines include those with at least one hydrocarbyl group, or, in certain embodiments, two or three hydrocarbyl groups. The hydrocarbyl groups may contain 2 to 30 carbon atoms, or in other embodiments 8 to 26, or 10 to 20, or 13 to 19 carbon atoms.

**[0098]** Primary amines include ethylamine, propylamine, butylamine, 2-ethylhexylamine, octylamine, and dodecylamine, as well as such fatty amines as n-octylamine, n-decylamine, n-dodecylamine, n-tetradecylamine, n-hexadecylamine, n-octadecylamine and oleyamine. Other useful fatty amines include commercially available fatty amines such as "Armeen®" amines (products available from Akzo Chemicals, Chicago, Ill.), such as Armeen C, Armeen O, Armeen O L, Armeen T, Armeen H T, Armeen S and Armeen S D, wherein the letter designation relates to the fatty group, such as coco, oleyl, tallow, or stearyl groups.

**[0099]** Examples of suitable secondary amines include dimethylamine, diethylamine, dipropylamine, dibutylamine, diamylamine, dihexylamine, diheptylamine, methylethylamine, ethylbutylamine, di-cocoalkyl amine (or di-cocoamine) and ethylamylamine. The secondary amines may be cyclic amines such as piperidine, piperazine and morpholine.

**[0100]** The amine may also be a tertiary-aliphatic primary amine. The aliphatic group in this case may be an alkyl group containing 2 to 30, or 6 to 26, or 8 to 24 carbon atoms. Tertiary alkyl amines include monoamines such as tert-butylamine, tert-hexylamine, 1-methyl-1-amino-cyclohexane, tert-octylamine, tert-decylamine, tertdodecylamine, tert-tetradecylamine, tert-hexadecylamine, tert-octadecylamine, tert-tetracosanylamine, and tert-octacosanylamine.

**[0101]** In one embodiment the amine salt of a phosphorus acid or ester includes an amine with C11 to C14 tertiary alkyl primary groups or mixtures thereof. In one embodiment the amine salt of a phosphorus compound includes an amine with C14 to C18 tertiary alkyl primary amines or mixtures thereof. In one embodiment the amine salt of a phosphorus compound includes an amine with C18 to C22 tertiary alkyl primary amines or mixtures thereof.

**[0102]** Mixtures of amines may also be used in the invention. In one embodiment a useful mixture of amines is "Primene® 81R" and "Primene® JMT." Primene® 81R and Primene® JMT (both produced and sold by Rohm & Haas) are mixtures of C11 to C14 tertiary alkyl primary amines and C18 to C22 tertiary alkyl primary amines respectively.

**[0103]** In one embodiment the amine salt of a phosphorus acid or ester is the reaction product of a  $C_{14}$  to  $C_{18}$  alkylated phosphoric acid with Primene  $81R^{TM}$  (produced and sold by Rohm & Haas) which is a mixture of C11 to C14 tertiary alkyl primary amines.

**[0104]** Examples of the amine salt of a phosphorus acid or ester include the reaction product(s) of isopropyl, methylamyl (1,3-dimethylbutyl or mixtures thereof), 2-ethylhexyl, heptyl, octyl or nonyl dithiophosphoric acids with ethylene diamine, morpholine, or Primene  $81R^{TM}$ , and mixtures thereof.

[0105] In one embodiment a dithiophosphoric acid may be reacted with an epoxide or a glycol. This reaction product is further reacted with a phosphorus acid, anhydride, or lower ester (where "lower" signifies 1 to 8, or 1 to 6, or 1 to 4, or 1 to 2 carbon atoms in the alcohol-derived portion of the ester). The epoxide includes an aliphatic epoxide or a styrene oxide. Examples of useful epoxides include ethylene oxide, propylene oxide, butene oxide, octene oxide, dodecene oxide, styrene oxide and the like. In one embodiment the epoxide is propylene oxide. The glycols may be aliphatic glycols having 1 to 12, or 2 to 6, or 2 to 3 carbon atoms. The dithiophosphoric acids, glycols, epoxides, inorganic phosphorus reagents and methods of reacting the same are described in U.S. Pat. Nos. 3,197,405 and 3,544,465. The resulting acids may then be salted with amines. An example of suitable dithiophosphoric acid is prepared by adding phosphorus pentoxide (about 64 grams) at 58° C. over a period of 45 minutes to 514 grams of hydroxypropyl O,O-di(1,3-dimethylbutyl)phosphorodithioate (prepared by reacting di(1,3-dimethylbutyl)-phosphorodithioic acid with 1.3 moles of propylene oxide at 25° C.). The mixture is heated at 75° C. for 2.5 hours, mixed with a diatomaceous earth and filtered at 70° C. The filtrate contains 11.8% by weight phosphorus, 15.2% by weight sulphur, and an acid number of 87 (bromophenol blue).

**[0106]** In one embodiment the antiwear agent comprises a non-ionic phosphorus compound. Typically the non-ionic phosphorus compound may have an oxidation of +3 or +5. The different embodiments comprise phosphite ester, phosphate esters, or mixtures thereof.

**[0107]** In one embodiment the antiwear agent comprises a non-ionic phosphorus compound that is a hydrocarbyl phosphite. The hydrocarbyl phosphite of the invention includes those represented by the formula:



wherein each R<sup>III</sup> may be independently hydrogen or a hydrocarbyl group, with the proviso that at least one of the R<sup>III</sup> groups is hydrocarbyl.

**[0108]** Each hydrocarbyl group of R''' may contain at least 2 or 4 carbon atoms. Typically, the combined total sum of carbon atoms present on both R''' groups may be less than 45, less than 35 or less than 25. Examples of suitable ranges for the number of carbon atoms present on both R'' groups includes 2 to 40, 3 to 24 or 4 to 20. Examples of suitable hydrocarbyl groups include propyl, butyl, pentyl, hexyl dodecyl, butadecyl, hexadecyl, or octadecyl groups. Generally the hydrocarbyl phosphite is soluble or at least dispersible in oil. In one embodiment the hydrocarbyl phosphite may be dibutyl hydrogen phosphite or a  $C_{16-18}$  alkyl hydrogen phosphorus compound is found in column 9, line 48 to column 11, line 8 of U.S. Pat. No. 6,103,673.

#### Other Friction Modifiers

**[0109]** Another friction modifier (i.e. other than component (a) of the invention) may be present, including fatty amines, borated glycerol esters, fatty acid amides, non-borated fatty epoxides, borated fatty epoxides, alkoxylated fatty amines, borated alkoxylated fatty amines, metal salts of fatty acids, fatty imidazolines, metal salts of alkyl salicylates, condensation products of carboxylic acids or polyalkylene-polyamines, or amides of hydroxyalkyl compounds.

**[0110]** In one embodiment the other friction modifier may include a fatty acid ester of glycerol. The final product may be in the form of a metal salt, an amide, an imidazoline, or mixtures thereof. The fatty acids may contain 6 to 24 or 8 to 18 carbon atoms. The fatty acids may branched or straightchain, saturated or unsaturated. Suitable acids include 2-eth-ylhexanoic, decanoic, oleic, stearic, isostearic, palmitic, myristic, palmitoleic, linoleic, lauric, and linolenic acids, and the acids from the natural products tallow, palm oil, olive oil, peanut oil, corn oil, and Neat's foot oil. In one embodiment the fatty acid is oleic acid.

[0111] When in the form of a metal salt, typically the metal may be zinc or calcium and the products may be overbased. Examples are overbased calcium salts and basic oleic acidzinc salt complexes. The zinc salt may be acidic, neutral or basic (overbased). These salts may be prepared from the reaction of a zinc containing reagent with a carboxylic acid or salt thereof. A useful method of preparation of these salts is to react zinc oxide with a carboxylic acid. Useful carboxylic acids include carboxylic acids of the formula R<sup>1</sup>COOH where R<sup>1</sup> is an aliphatic or alicyclic hydrocarbon radical. In one embodiment  $\hat{R}^1$  is a fatty group such as stearyl, oleyl, linoleyl, palmityl, or mixtures thereof. In one embodiment the zinc salt contains stoichiometric excess of zinc over the amount needed to prepare a neutral salt. The stoichiometry includes zinc present from 1.1 to 1.8 times the stoichiometric, or 1.3 to 1.6 times the stoichiometric amount of zinc. The zinc carboxylates are known in the art and are described in U.S. Pat. No. 3,367,869. Metal salts may also include calcium salts. Examples may include overbased calcium salts.

**[0112]** When in the form of an amide, the condensation product may be prepared with ammonia, or with primary or secondary amines such as diethylamine and diethanolamine. **[0113]** When in the form of an imidazoline, the cyclic condensation product of a fatty acid ester of glycerol with a diamine or polyamine such as a polyethylenepolyamine. In one embodiment the other friction modifier is the condensation product of a C8 to C24 fatty acid with a polyalkylene polyamine, and in particular, the product of isostearic acid with tetraethylenepentamine.

**[0114]** In one embodiment the other friction modifier may be an alkoxylated alcohol. A detailed description of suitable alkyoxylated alcohols is described in paragraphs 19 and 20 of US Patent Application 2005/0101497. The alkoxylated amines are also described in U.S. Pat. No. 5,641,732 in column 7, line 15 to column 9, line 25.

[0115] In one embodiment the other friction modifier may be an alkoxylated amine e.g., an ethoxylated amine derived from 1.79% Ethomeen T-12 and 0.90% Tomah PA-1 as described in Example E of U.S. Pat. No. 5,703,023, column 28, lines 30 to 46. Other suitable alkoxylated amine compounds include commercial alkoxylated fatty amines known by the trademark "ETHOMEEN" and available from Akzo Nobel. Representative examples of these ETHOMEENTM materials is ETHOMEEN™ C/12 (bis[2-hydroxyethyl]-cocoamine); ETHOMEEN™ C/20 (polyoxyethylene[10]cocoamine); ETHOMEEN<sup>™</sup> S/12 (bis[2-hydroxyethyl] soyamine); ETHOMEEN<sup>TM</sup> T/12 (bis[2-hydroxyethyl] allow-amine); ETHOMEEN™ T/15 (polyoxyethylene-[5] tallowamine); ETHOMEEN<sup>TM</sup> 0/12 (bis[2-hydroxyethyl] oleyl-amine); ETHOMEEN<sup>TM</sup> 18/12 (bis[2-hydroxyethyl] octadecylamine); and **ETHOMEEN™** 18/25 (polyoxyethylene[15]octadecylamine). Fatty amines and ethoxylated fatty amines are also described in U.S. Pat. No. 4,741,848.

**[0116]** In one embodiment the other friction modifier may be a reaction product of an isomerised alkenyl substituted succinic anhydride and a polyamine as described in U.S. Pat. No. 5,840,663 in column 2, lines 18 to 43. Specific embodiments of the friction modifier described in U.S. Pat. No. 5,840,663 are further disclosed in column 3, line 23 to column 4, line 35. Preparative examples are further disclosed in column 4, line 45 to column 5, line 37 of U.S. Pat. No. 5,840,663. **[0117]** In one embodiment the other friction modifier may be an alkylphosphonate mono- or di-ester sold commercially by Rhodia under the trademark Duraphos® DMODP.

**[0118]** In one embodiment the other friction modifier other may be a borated fatty epoxide, known from Canadian Patent No. 1,188,704. These oil-soluble boron-containing compositions are prepared by reacting, at a temperature from  $80^{\circ}$  C. to  $250^{\circ}$  C., boric acid or boron trioxide with at least one fatty epoxide. The fatty epoxide typically contains at least 8 carbon atoms in the fatty groups of the epoxide.

**[0119]** The borated fatty epoxides may be characterised by the method for their preparation which involves the reaction of two materials. Reagent A may be boron trioxide or any of the various forms of boric acid including metaboric acid (HBO<sub>2</sub>), orthoboric acid (H<sub>3</sub>BO<sub>3</sub>) and tetraboric acid (H<sub>2</sub>B<sub>4</sub>O<sub>7</sub>). Boric acid, and especially orthoboric acid. Reagent B may be at least one fatty epoxide. The molar ratio of reagent A to reagent B is generally 1:0.25 to 1:4, or 1:1 to 1:3, or about 1:2. The borated fatty epoxides may be prepared

by merely blending the two reagents and heating them at temperature of  $80^{\circ}$  to  $250^{\circ}$  C., or  $100^{\circ}$  to  $200^{\circ}$  C., for a period of time sufficient for reaction to take place. If desired, the reaction may be effected in the presence of a substantially inert, normally liquid organic diluent. During the reaction, water is evolved and may be removed by distillation.

#### Friction Stabilising Agents

**[0120]** In one embodiment the lubricating composition further comprises a friction stabilising agent. Friction stabilising agents include  $H_3PO_4$ ,  $H_3PO_3$  or mixtures thereof. Typically  $H_3PO_4$  is commercially available in 85% solution.

#### Dispersant

**[0121]** In one embodiment the additive concentrate further comprises a dispersant. The dispersant may be a succinimide dispersant (for example N-substituted long chain alkenyl succinimides), a Mannich dispersant, an ester-containing dispersant, a condensation product of a fatty hydrocarbyl mono-carboxylic acylating agent with an amine or ammonia, an alkyl amino phenol dispersant, a hydrocarbyl-amine dispersant, a polyether dispersant or a polyetheramine dispersant.

**[0122]** In different embodiments the dispersant may be a succinimide, succinic acid ester, or Mannich dispersant.

[0123] In several embodiments the N-substituted long chain alkenyl succinimides contain an average of at least 8, or 30, or 35 up to 350, or to 200, or to 100 carbon atoms. In one embodiment, the long chain alkenyl group is derived from a polyalkene characterised by an M<sub>2</sub> (number average molecular weight) of at least 500. Generally, the polyalkene is characterised by an M<sub>2</sub> of 500, or 700, or 800, or even 900 up to 5000, or to 2500, or to 2000, or even to 1500 or 1200. In one embodiment the long chain alkenyl group is derived form polyolefins. The polyolefins may be derived from monomers including monoolefins having 2 to 10 carbon atoms such as ethylene, propylene, 1-butene, isobutylene, and 1-decene. An especially useful monoolefin source is a C<sub>4</sub> refinery stream having a 35 to 75 weight percent butene content and a 30 to weight percent isobutene content. Useful polyolefins include polyisobutylenes having a number average molecular weight of 400 to 5000, in another instance of 400 to 2500, and in a further instance of 400 or 500 to 1500. The polyisobutylene may have a vinylidene double bond content of 5 to 69%, in a second instance of 50 to 69%, and in a third instance of 50 to 95%.

**[0124]** In one embodiment the succinimide dispersant comprises a polyisobutylene-substituted succinimide, wherein the polyisobutylene-substituent has a number average molecular weight of 400 to 5000.

**[0125]** Succinimide dispersants and their methods of preparation are more fully described in U.S. Pat. Nos. 4,234, 435 and 3,172,892. The succinimide dispersant may also be prepared by either direct alkylation or chlorine-route.

**[0126]** Suitable ester-containing dispersants are typically high molecular weight esters. These materials are described in more detail in U.S. Pat. No. 3,381,022.

**[0127]** Mannich dispersants are the reaction product of a hydrocarbyl-substituted phenol, an aldehyde, and an amine or ammonia. The hydrocarbyl substituent of the hydrocarbyl-substituted phenol may have 10 to 400 carbon atoms, in another instance 30 to 180 carbon atoms, and in a further instance 10 or 40 to 110 carbon atoms. This hydrocarbyl

substituent may be derived from an olefin or a polyolefin. Useful olefins include alpha-olefins, such as 1-decene, which are commercially available.

**[0128]** Hydrocarbyl-amine dispersants are hydrocarbylsubstituted amines. The hydrocarbyl-substituted amine may be formed by heating a mixture of a chlorinated olefin or polyolefin such as a chlorinated polyisobutylene with an amine such as ethylenediamine in the presence of a base such as sodium carbonate as described in U.S. Pat. No. 5,407,453. **[0129]** Polyether dispersants include polyetheramines, polyether amides, polyether carbamates, and polyether alcohols. Polyetheramines and their methods of preparation are described in greater detail in U.S. Pat. No. 6,458,172, columns 4 and 5.

**[0130]** In one embodiment the invention further comprises at least one dispersant derived from polyisobutylene, an amine and zinc oxide to form a polyisobutylene succinimide complex with zinc. The polyisobutylene succinimide complex with zinc may be used alone or in combination.

**[0131]** The dispersants may also be post-treated by conventional methods by a reaction with any of a variety of agents. Among these are boron, urea, thiourea, dimercaptothiadiazoles, carbon disulphide, aldehydes, ketones, carboxylic acids, hydrocarbon-substituted succinic anhydrides, maleic anhydride, nitriles, epoxides, phosphorus compounds and/or metal compounds. In one embodiment the dispersant is a borated dispersant. Typically the borated dispersant comprises a succinimide dispersant which in turn comprises a polyisobutylene succinimide, wherein the polyisobutylene has a number average molecular weight of 400 to 5000.

**[0132]** In one embodiment the dispersant is phosphorylated dispersant, or a borated phosphorylated dispersant.

**[0133]** In one embodiment the dispersant may be prepared by heating (i) a dispersant material described above (for example N-substituted long chain alkenyl succinimides), (ii) 2,5-dimercapto-1,3,4-thiadiazole or a hydrocarbyl-substituted 2,5-dimercapto-1,3,4-thiadiazole, or oligomers thereof, (iii) a borating agent, and (iv) optionally a dicarboxylic acid of an aromatic compound selected from the group consisting of 1,3 diacids and 1,4 diacids; or (v) optionally a phosphorus acid compound, said heating being sufficient to provide a product of (i), (ii), (iii) and optionally (iv) or (v), which is soluble in an oil of lubricating viscosity. The dispersant prepared by heating is described in more detail in US Patent Applications US04/027094 and 60/654,164 (now WO 2006/091387).

#### Antioxidants

**[0134]** In one embodiment the lubricating composition further comprises an antioxidant. The antioxidant may be present in ranges of 0 wt % to 10 wt %, 0.01 wt % to 5 wt %, or 0.05 wt % to 3 wt % of the lubricating composition.

**[0135]** Suitable antioxidants include molybdenum compounds such as molybdenum dithiocarbamates, sulphurised olefins, sulphides (including hydroxylalkyl sulphides such as 1-(tert-dodecylthio)-2-propanol or tert-nonyl mercaptan reacted with propylene oxide (mole ratio 1:1)), hindered phenols including ester-substituted hindered phenols, aminic compounds such as phenylalphanaphthylamines or alkylated diphenylamines (typically nonyl diphenylamine, di-nonyl diphenylamine, octyl diphenylamine, di-octyl diphenylamine), or mixtures thereof.

#### Viscosity Modifiers

**[0136]** In one embodiment the lubricating composition further comprises a viscosity modifier or dispersant viscosity

modifier (also referred to as DVMs). The viscosity modifier may be present at 0 wt % to 12 wt %, 0.1 wt % to 10 wt % or 1 wt % to 8 wt % of the lubricating composition.

**[0137]** Viscosity modifiers include hydrogenated copolymers of styrene-butadiene, ethylene-propylene copolymers, polyisobutenes, hydrogenated styrene-isoprene polymers, hydrogenated isoprene polymers, polymath-acrylates, poly-acrylates, polyalkyl styrenes, alkenyl aryl conjugated diene copolymers, polyolefins, and esters of maleic anhydride-styrene copolymers.

**[0138]** Dispersant viscosity modifiers include functionalised polyolefins, for example, ethylene-propylene copolymers that have been functionalized with the reaction product of maleic anhydride and an amine, a polymethacrylate functionalised with an amine, or styrene-maleic anhydride copolymers reacted with an amine; may also be used in the composition of the invention.

#### Detergents

**[0139]** In one embodiment the lubricating composition further comprises a detergent Detergents include neutral or overbased detergents, Newtonian or non-Newtonian, basic salts of alkali, alkaline earth or transition metals with one or more of a phenate, a sulphurised phenate, a sulphonate, a carboxylic acid, a phosphorus acid, a saligenin, an alkylsalicylate, and a salixarate. The alkali metal includes sodium. The alkaline earth metal may be calcium, magnesium or barium. In different embodiments the detergent may be a magnesium sulphonate or a calcium sulphonate.

**[0140]** Other optional components include foam inhibitors, including polydimethyl siloxane, fluorosilicone, copolymers of ethyl acrylate and 2-ethylhexylacrylate and optionally vinyl acetate; demulsifiers including trialkyl phosphates, polyethylene glycols, polyethylene oxides, polypropylene oxides and (ethylene oxide-propylene oxide) polymers; pour point depressants including esters of maleic anhydride-styrene, polymethacrylates, polyacrylates or polyacrylamides. **[0141]** Seal swell agents including 3-(decyloxy)tetrahydro-1,1-dioxide thiophene, decyloxysulpholane, phthalate esters, Exxon Necton37<sup>™</sup> (FN 1380) and Exxon Mineral

## esters, Exxon Necton37<sup>TM</sup> (FN 1380) and Exxon Mineral Seal Oil (FN 3200) may also be present in the lubricating composition.

#### INDUSTRIAL APPLICATION

**[0142]** The method of the invention is useful for lubricating a variety of transmissions including an automatic transmission or a manual transmission. In one embodiment the mechanical device is an automatic transmission.

**[0143]** The automatic transmission includes continuously variable transmissions (CVT), infinitely variable transmissions (IVT), Toroidal transmissions, continuously slipping torque converted clutches (CSTCC), stepped automatic transmissions or dual clutch transmissions (DCT).

**[0144]** The following examples provide illustrations of the invention. These examples are non-exhaustive and are not intended to limit the scope of the invention.

#### EXAMPLES

**[0145]** A conventional automatic transmission fluid is evaluated for friction and squawk performance using a test methodology as defined below. The conventional automatic transmission fluid is evaluated as (i) a new fluid, (ii) a new fluid that is aged at 150° C. for 100 hours, and (iii) the aged

fluid of (ii) is then aged for 100 hours at  $150^{\circ}$  C. Fluid (iii) has therefore been aged for a total of 200 hours at  $150^{\circ}$  C.

**[0146]** The automatic transmission fluid is evaluated in a ZF®-GK rig first utilising standard methods to evaluate frictional performance, and then further evaluated for squawk performance using the procedure set forth in ZF test procedure "PVL44\_Quietschversuche\_6HP26E\_V2.0.pv". The procedure "PVL44\_Quietschversuche\_6HP26E\_V2.0.pv" is available from ZF Passau GmbH, Test Systems, Tittlinger Strasse 28, D-94034 Passau, Germany. In order to obtain squawk performance data the GK-rig employed is configured by addition of ZF Squawk Modifications, including the addition of a data acquisition recorder capable of recording squawk. The results obtained are:

Test Procedure	Test Fluid	Test Parameter(limit)	Test Result
GWK Friction	ATF with 12 wt % of the top treat	Slope (increase with speed)	Increasing
1050	additive concentrate of the invention added	μ5 (0.110 to 0.113)	0.109
GK E Clutch	invention added	torque	
Squawk Test		oscillation $<50$ Nm at p $\leq$ 1.625 N/mm <sup>2</sup>	
100 Hrs before	ATF fluid with no		Fail, >50 Nm
run in	top treat additive concentrate		at 0.9 N/mm <sup>2</sup>
100 Hrs after	ATF fluid with no		Fail, >50 Nm
run in	top treat additive concentrate		at 0.75 N/mm <sup>2</sup>
200 Hrs	ATF fluid with no		Fail, >50 Nm
additional aging at 150° C.	top treat additive concentrate		at 0.5 N/mm <sup>2</sup>
Before running	ATF with 12 wt		Pass, no
in	% of the top treat additive		oscillation >50 Nm
	concentrate of the invention added		
After running in	ATF with 12 wt		Pass, no
The funning in	% of the top treat		oscillation >50
	additive concentrate of the		Nm
	invention added		
100 Hrs	ATF with 12 wt		Pass, no
additional aging	% of the top treat		oscillation >50
at 150° C.	additive		Nm
	concentrate of the		
	invention added		

**[0147]** Overall the data indicates that the automatic transmission fluid treated with the additive concentrate as a top treat package is capable of minimising noise, shudder, chattering, or squawking, whilst also not adversely affecting the frictional balance and clutch holding capacity, nor increasing lubricant waste. This allows for the transmission to operate with appropriate frictional performance and minimised noise, shudder, chatter or squawk.

**[0148]** As used herein, the term "hydrocarbyl substituent" or "hydrocarbyl group" is used in its ordinary sense, which is well-known to those skilled in the art. Specifically, it refers to a group having a carbon atom directly attached to the remainder of the molecule and having predominantly hydrocarbon character. Examples of hydrocarbyl groups include:

**[0149]** (i) hydrocarbon substituents, that is, aliphatic (e.g., alkyl or alkenyl), alicyclic (e.g., cycloalkyl, cycloalkenyl) substituents, and aromatic-, aliphatic-,

and alicyclic-substituted aromatic substituents, as well as cyclic substituents wherein the ring is completed through another portion of the molecule (e.g., two substituents together form a ring);

- **[0150]** (ii) substituted hydrocarbon substituents, that is, substituents containing non-hydrocarbon groups which, in the context of this invention, do not alter the predominantly hydrocarbon nature of the substituent (e.g., halo (especially chloro and fluoro), hydroxy, alkoxy, mercapto, alkylmercapto, nitro, nitroso, and sulphoxy);
- **[0151]** (iii) hetero substituents, that is, substituents which, while having a predominantly hydrocarbon character, in the context of this invention, contain other than carbon in a ring or chain otherwise composed of carbon atoms, and encompass substituents as pyridyl, furyl, thienyl and imidazolyl. Heteroatoms include sulphur, oxygen, nitrogen. In general, no more than two, preferably no more than one, non-hydrocarbon substituent will be present for every ten carbon atoms in the hydrocarbyl group; typically, there will be no non-hydrocarbon substituents in the hydrocarbyl group.

**[0152]** It is known that some of the materials described above may interact in the final formulation, so that the components of the final formulation may be different from those that are initially added. The products formed thereby, including the products formed upon employing lubricant composition of the present invention in its intended use, may not be susceptible of easy description. Nevertheless, all such modifications and reaction products are included within the scope of the present invention; the present invention encompasses lubricant composition prepared by admixing the components described above.

[0153] Each of the documents referred to above is incorporated herein by reference. Except in the Examples, or where otherwise explicitly indicated, all numerical quantities in this description specifying amounts of materials, reaction conditions, molecular weights, number of carbon atoms, and the like, are to be understood as modified by the word "about." Unless otherwise indicated, each chemical or composition referred to herein should be interpreted as being a commercial grade material which may contain the isomers, by-products, derivatives, and other such materials which are normally understood to be present in the commercial grade. However, the amount of each chemical component is presented exclusive of any solvent or diluent oil, which may be customarily present in the commercial material, unless otherwise indicated. It is to be understood that the upper and lower amount, range, and ratio limits set forth herein may be independently combined. Similarly, the ranges and amounts for each element of the invention may be used together with ranges or amounts for any of the other elements.

**[0154]** While the invention has been explained in relation to its preferred embodiments, it is to be understood that various modifications thereof will become apparent to those skilled in the art upon reading the specification. Therefore, it is to be understood that the invention disclosed herein is intended to cover such modifications as fall within the scope of the appended claims.

#### What is claimed is:

**1**. A method of lubricating a transmission by supplying to the transmission a lubricating composition further treated with an additive concentrate comprising:

(a) 4 wt % to 67 wt % of a static friction reducing friction modifier; (b) 0.1 wt % to 5 wt % of a corrosion inhibitor;

(c) 3 wt % to 96 wt % of an oil of lubricating viscosity; and (d) 0 wt % to 20 wt % of other performance additives,

wherein the static friction reducing friction modifier is an amide represented by the formula  $R^1R^2N$ —C(O) $R^3$ , wherein  $R^1$  and  $R^2$  are each independently hydrocarbyl groups of at least 6 carbon atoms and  $R^3$  is a hydroxyalkyl group of 1 to 6 carbon atoms or a group formed by the condensation of said hydroxyalkyl group, through a hydroxyl group thereof, with an acylating agent.

2. The method of claim 1, wherein the additive concentrate contains the static friction reducing friction modifier at 10 wt % to 50 wt %.

3. The method of claim 1, wherein the additive concentrate contains the static friction reducing friction modifier at 20 wt % to 40 wt %.

4. The method of claim 1, wherein the additive concentrate contains the corrosion inhibitor at 0.2 wt % to 3 wt %.

**5**. The method of claim **1**, wherein the additive concentrate is supplied to the transmission as a service-fill top treat.

6. The method of claim 1, wherein the additive concentrate is supplied to the transmission as a service-fill top treat at 0.1 wt % to 30 wt %.

7. The method of claim 1, wherein the additive concentrate is supplied to the transmission as a service-fill top treat at 1 wt % to 15 wt %.

**8**. A method of lubricating a transmission containing a lubricant comprising supplying to the lubricant within the transmission an additive concentrate comprising:

(a) 4 wt % to 67 wt % of a static friction reducing friction modifier;

(b) 0.1 wt % to 5 wt % of a corrosion inhibitor;

(c) 3 wt % to 96 wt % of an oil of lubricating viscosity; and (d) 0 wt % to 20 wt % of other performance additives,

wherein the static friction reducing friction modifier is an amide represented by the formula  $R^1R^2N$ —C(O) $R^3$ , wherein  $R^1$  and  $R^2$  are each independently hydrocarbyl groups of at least 6 carbon atoms and  $R^3$  is a hydroxyalkyl group of 1 to 6 carbon atoms or a group formed by the condensation of said hydroxyalkyl group, through a hydroxyl group thereof, with an acylating agent.

9. An additive concentrate comprising:

(a) 4 wt % to 67 wt % of a static friction reducing friction modifier;

(b) 0.1 wt % to 5 wt % of a corrosion inhibitor;

(c) 3 wt % to 96 wt % of an oil of lubricating viscosity; and (d) 0 wt % to 20 wt % of other performance additives,

wherein the static friction reducing friction modifier is an amide represented by the formula  $R^1R^2N$ —C(O) $R^3$ , wherein  $R^1$  and  $R^2$  are each independently hydrocarbyl groups of at least 6 carbon atoms and  $R^3$  is a hydroxyalkyl group of 1 to 6 carbon atoms or a group formed by the condensation of said hydroxyalkyl group, through a hydroxyl group thereof, with an acylating agent.

**10**. A method of lubricating a transmission by supplying to the transmission a lubricating composition further treated with an additive concentrate comprising:

(a) 4 wt % to 67 wt % of a static friction reducing friction modifier;

(b) 0.1 wt % to 5 wt % of a corrosion inhibitor;

(c) 3 wt % to 96 wt % of an oil of lubricating viscosity; and (d) 0 wt % to 20 wt % of other performance additives,

wherein the static friction reducing modifier comprises a tertiary amine, wherein the amine contains three substituent hydrocarbyl groups, two of which are alkyl groups, and the amine is represented by the formula:

 $R^4 R^5 N R^6$ 

wherein  $R^4$  and  $R^5$  are each independently an alkyl group of at least 6 carbon atoms and  $R^6$  is a polyhydroxyl-containing alkyl group or a polyhydroxyl-containing alkoxyalkyl group.

**11**. A method of lubricating a transmission by supplying to the transmission a lubricating composition further treated with an additive concentrate comprising:

(a) 4 wt % to 67 wt % of a static friction reducing friction modifier;

(b) 0.1 wt % to 5 wt % of a corrosion inhibitor;

(c) 3 wt % to 96 wt % of an oil of lubricating viscosity; and (d) 0 wt % to 20 wt % of other performance additives,

wherein the static friction reducing modifier comprises an amine, wherein the amine is secondary or tertiary amine being represented by the formula:

 $R^7 R^8 N R^9$ 

wherein  $\mathbb{R}^7$  and  $\mathbb{R}^8$  are each independently an alkyl group of at least 6 carbon atoms and  $\mathbb{R}^9$  is a hydroxyl-containing alkyl group, a hydroxyl-containing alkoxyalkyl group, an aminecontaining alkyl group, a hydrocarbyl group, or hydrogen, provided that when  $\mathbb{R}^9$  is H, then at least one of  $\mathbb{R}^7$  and  $\mathbb{R}^8$  is an alkyl group of 8 to 16 carbon atoms.

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