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(54) METHOD OF LUBRICATING A MANUAL TRANSMISSION

METHODE ZUM SCHMIEREN EINES SCHALTGETRIEBES

METHODE DE LUBRIFIER UN TRANSMISSION MANUEL

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(56) References cited:
WO-A1-92/07047 **WO-A1-2009/064827**
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EP 2 510 078 B1

Description

FIELD OF INVENTION

[0001] The present invention relates to a method of lubricating a manual transmission comprising supplying to the manual transmission a lubricating composition comprising an oil of lubricating viscosity and an aromatic hydroxy-substituted compound. The invention further provides for the use of the lubricating composition comprising the aromatic hydroxy-substituted compound as at least one of a filterability aid and a wax crystal growth modifier/gelation inhibitor in the driveline device.

BACKGROUND OF THE INVENTION

[0002] In driveline devices (in particular, a manual transmission) lubricants, whilst circulating in the device, frequently pass through filters. In some instances additives, waxes, or other contaminants may become trapped in the filter, thereby causing a decrease in lubricant flow and/or filter blockage. Decreasing lubricant flow or filter blockage may be more apparent at low temperature or with lubricants containing brightstock. It is known that waxes, in general, can be suppressed by pour-point depressants or agents which improve flow or change the size and shape of the deposits (such as wax crystals) and, in this way, counteract increases in the viscosity of the oils.

[0003] U.S. 6,495,495 discloses a filterability improver for mineral oil distillates and heavy base oils containing wax material consisting essentially of a blend containing from about 30% to about 70% of an alkyl ester polymer and from about 70% to about 30% naphthenic oil, based upon the total weight of said blend, wherein such filterability improver prevents filter blockage due to wax formation at ambient temperatures. The filterability improver may be used in manual transmission oils, axle factory fill oils, and extended drain oils.

[0004] German Patent No. DE 11 47 799 B1 discloses oil-soluble copolymers of ethylene and vinyl acetate, having molecular masses between 1,000 and 3,000 g/mol, that are added to petroleum distillate propellants or fuels. The copolymers that contain 60% to 99% by weight of ethylene and 1% to 40% by weight of vinyl acetate.

[0005] Other polymers employed as agents which improve flow contain, for example, 1-hexene, as described in EP 184,083, or diisobutylene, as set forth in EP 203,554, in addition to ethylene and vinyl acetate. Copolymers of ethylene, alkenecarboxylic acid esters, vinyl esters and/or vinyl ketones are also used as pour-point depressants and for improving the flow properties of crude oils and middle distillates.

[0006] EP 111,883 discloses that ethylene and vinyl acetate, copolymers of ethylene, alkenecarboxylic acid esters, vinyl esters and/or vinyl ketones are also used as pour-point depressants and for improving the flow properties of crude oils and middle distillates.

[0007] U.S. Patents 5,039,437 and 5,082,470 disclose alkyl phenolformaldehyde condensates additives for improving the low temperature flow properties of hydrocarbon oils. The polymer composition has a number average molecular weight of at least about 3,000 and a molecular weight distribution of at least about 1.5; in the alkylated phenol reactant the alkyl groups are essentially linear, have between 6 and 50 carbon atoms, and have an average number of carbon atoms between about 12 and 26; and not more than about 10 mole % of the alkyl groups on the alkylated phenol have less than 12 carbon atoms and not more than about 10 mole % of the alkyl groups on the alkylated phenol have more than 26 carbon atoms.

[0008] U.S. Patents 4,565,460; 4,559,155; 4,575,526 and 4,613,342 all disclose additive combinations for improving the cold flow properties of hydrocarbon fuel compositions. The composition includes a pour point depressant which can be a hydrocarbyl-substituted phenol of the formula $(R^*)_a$-Ar-$(OH)_b$ wherein R* is a hydrocarbyl group selected from the group consisting of hydrocarbyl groups of from about 8 to about 39 carbon atoms and polymers of at least 30 carbon atoms. Ar is an aromatic moiety which can include linked polynuclear aromatic moieties represented by the general formula ar-(-Lng-ar-)-$_w$$(Q)_{mw}$ wherein w is an integer of 1 to about 20. Each Lng is a bridging linkage of the type including alkylene linkages (e.g., -CH_2-among others).

[0009] U.S. Patent 5,171,330 discloses methacyclophanes with substituents, obtained from the condensation reaction of a resorcin with a product containing an aldehyde group, followed by a reaction with halides of organic acids or alkyl halides.

[0010] U.S. Patent 5,707,946 discloses a pour point depressant which is the reaction product of a hydrocarbyl-substituted phenol having a number average of greater than 30 carbon atoms in the hydrocarbyl-substituent, and an aldehyde of 1 to about 12 carbon atoms, or a source therefore.

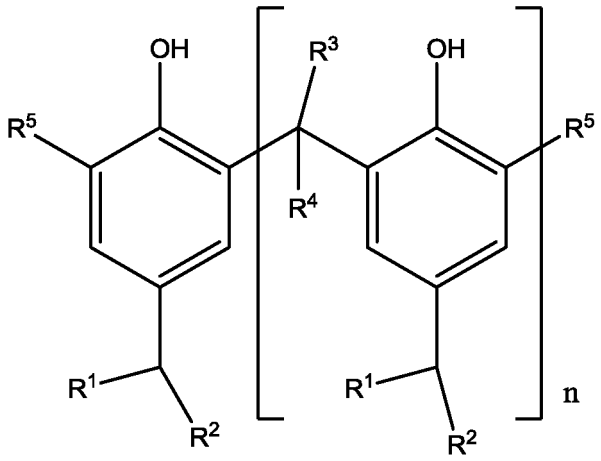
[0011] International publication WO 2009/064827 discloses pour point depressant composition comprising the reaction product of: (a) a hydrocarbyl-substituted phenol which is the reaction product of (i) phenol and (ii) an olefin; and (b) an aldehyde; wherein the olefin has a vinylidene end group content of at least about 10 mole % and less than about 85 mole %, wherein the reaction between (a) and (b) is catalyzed, and wherein the catalyst comprises an acid catalyst or a base catalyst.

SUMMARY OF THE INVENTION

[0012] The inventors of this invention have discovered that the lubricating composition disclosed herein may be capable of at least one of increased filterability, wax crystal growth reduction or modification, and reduced or minimised gelation in a driveline device.

[0013] As used herein the term "linear hydrocarbyl" means that the hydrocarbyl group has a carbon chain that is substantially free of branching, apart from the carbon atom that is the point of attachment to the phenol ring shown in formula (1) below.

[0014] In one embodiment the invention provides a method of lubricating a driveline device comprising supplying to the driveline device a lubricating composition comprising an oil of lubricating viscosity and a compound represented by formula (1):



formula (1)

wherein

each R^1 is independently hydrogen or a linear hydrocarbyl group containing at least 1, or at least 3, or at least 4, or at least 6, or at least 8, or at least 10 carbon atoms (typically R^1 is a linear hydrocarbyl group containing at least 1 carbon atom);
each R^2 is independently a linear hydrocarbyl group containing at least 10, or at least 12, or at least 14, or at least 16 carbon atoms (R^2 may contain carbon atoms in the range of 10 to 60, or 16 to 50, or 16 to 40, or 16 to 30, or 18 to 24, or 20 to 28, or 22 to 26);
each R^3 and R^4 is independently hydrogen or a linear hydrocarbyl group containing 1 to 5, or 1 to 2 carbon atoms (typically R^3 and R^4 are both hydrogen);
each R^5 is independently hydrogen or a linear hydrocarbyl group containing 1 to 10, or 1 to 5, or 1 to 2 carbon atoms (typically R^5 may be methyl or hydrogen, or R^5 may be hydrogen); and
n is 1 to 20, or 3 to 15, or 4 to 8, or 6 to 8.

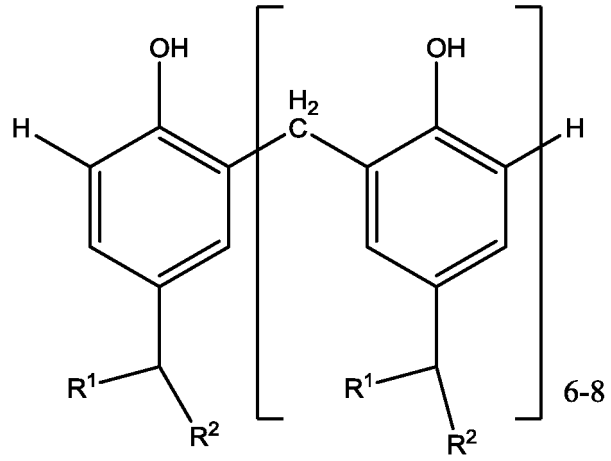
[0015] A person skilled in the art will appreciate that the compound of formula (1) may have repeat units shown in random or block architecture.

[0016] A person skilled in the art will appreciate that the compound of formula (1), during preparation, may have a portion of compounds that alkylate phenol at the ortho- position relative to a hydroxy group of phenol. Compounds having alkylated ortho- substitution may then couple at the para-position relative to the hydroxyl group. Consequently, a person skilled in the art will realize that compounds of this type are also encompassed by the present invention. In addition, a portion of phenol groups may not be alkyated; and, if incorporated into the structure of formula (1), would contain a portion of unsubstituted phenol in formula (1). The person skilled in the art will also appreciate that in some instances the phenol may contain lower alkylated impurities (e.g., methyl, or ethyl, or propyl groups) that may also become incorporated into the structure of formula (1). Consequently, a person skilled in the art will realize that compounds of this type are also encompassed by the present invention.

[0017] Typically, the compound of formula (1) as defined above is the dominant product (typically present at least 50 mole % or at least 70 mole %) in the reaction to prepare compounds of the present invention.

[0018] In one embodiment the invention provides a method of lubricating a driveline device comprising supplying to

the driveline device a lubricating composition comprising an oil of lubricating viscosity and a compound represented by formula (1a):



formula (1a)

wherein

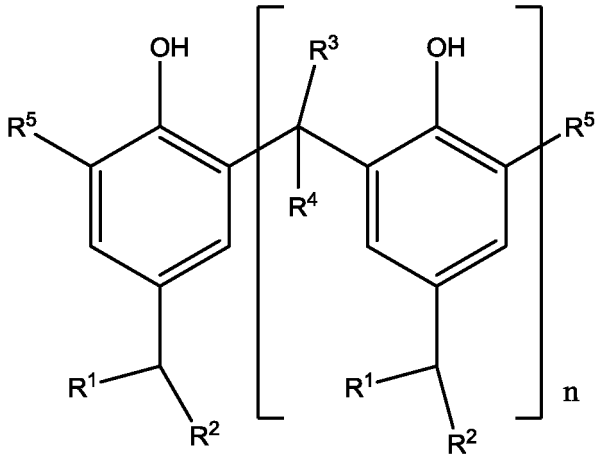
each R^1 is independently hydrogen or a linear hydrocarbyl group containing at least 1, or at least 3, or at least 4, or at least 6, or at least 8, or at least 10 carbon atoms (typically R^1 is a linear hydrocarbyl group containing at least 1 carbon atoms); and
each R^2 is independently a linear hydrocarbyl group containing at least 10, or at least 12, or at least 14, or at least 16 carbon atoms (R^2 may contain carbon atoms in the range of 16 to 50, or 16 to 40, or 16 to 30, or 18 to 24).

[0019] In one embodiment the mechanical device may be an axle gear or a manual transmission (typically a manual transmission).

[0020] In one embodiment the lubricating composition may have a SAE viscosity grade from XW-Y, wherein X is 70, 75, 80 or 85 and Y is 80, 85, 90, or 140. Typically, Y is greater than X.

[0021] The oil of lubricating viscosity is a bright stock. The bright stock is present from 80 wt % to 95 wt % of the lubricating composition.

[0022] In one embodiment the invention provides for the use of a lubricating composition comprising an oil of lubricating viscosity and a compound of formula (1) as at least one of a filterability aid and a wax crystal growth/gelation inhibitor (may also be referred to as a wax crystal growth modifier/gelation modifier) in a driveline device (typically a manual transmission), wherein the compound of formula (1) is represented by:



formula (1)

wherein R^1 to R^5 and n are described above.

DETAILED DESCRIPTION OF THE INVENTION

[0023] The present invention provides a method and use as disclosed herein.

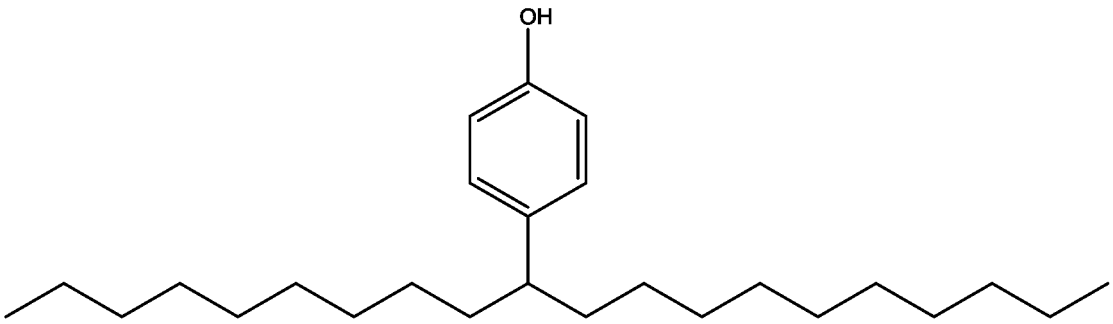
[0024] In one embodiment the compound of the invention may be represented by the formula (1) herein, wherein R^1 may be hydrogen; R^2 may be a linear hydrocarbyl group containing at least 10 carbon atoms (typically 10 to 100, or 16 to 70, or 16 to 40 carbon atoms); R^3 and R^4 may be hydrogen; R^5 may be hydrogen; and n may be 4 to 8, or 6 to 8.

[0025] In one embodiment the compound of the invention may be represented by the formula (1) described herein, wherein R^1 may independently be a linear hydrocarbyl group containing at least 1 (or at least 3, or at least 6) carbon atoms; R^2 may be a linear hydrocarbyl group containing at least 10 carbon atoms (typically R^2 may contain carbon atoms in the range of 10 to 60, or 16 to 50, or 16 to 40, or 16 to 30, or 18 to 24, or 20 to 28, or 22 to 26); R^3 and R^4 may be hydrogen; R^5 may be hydrogen; and n may be 1 to 20, or 3 to 15, or 4 to 8, or 6 to 8.

[0026] R^1 and R^2 may be derived from an olefin or polyolefin (typically an olefin), where the polyolefin or olefin is reacted with a phenol to form the hydrocarbyl-substituted phenol of the present invention.

[0027] Examples of a suitable olefin include dodecene, tetradecene, hexadecene, octadecene, eicosene, doeicosene, tetraeicosene, hexaeicosene or octaeicosene, or mixtures thereof. The olefin may be a mixture of 15 to 18, or 16 to 18, or 16 to 22, or 24 to 28, or 26 to 30 carbon atoms. In one embodiment the olefin may be a mixture of 24 to 28 carbon atoms.

[0028] The carbon atom bonded to both R^1 and R^2 may be a carbon atom of the olefin. As a consequence the structural unit R^1R^2HC-, the point of attachment to the phenol ring in the formula above may vary the number of carbon atoms present in R^1 and R^2 depending on where the double bond of the olefin is positioned at the time of alkylation. For example if the olefin is eicosene, a carbocation intermediate formed during alkylation of phenol may be nearly equally likely to be located along the carbon chain on any carbon atoms due to migration of the double bond or cation along the carbon chain. For illustrative purposes the resultant product may have R^1 having 9 carbon atoms, R^2 having 10 carbon atoms and it may be represented by formula (2):



[0029] Alternatively, the resultant product may have R^1 having 8 carbon atoms, R^2 having 11 carbon atoms, or R^1 having 5 carbon atoms, R^2 having 14 carbon atoms, or R^1 having 3 carbon atoms, R^2 having 16 carbon atoms, R^1 having 1 carbon atoms, R^2 having 18 carbon atoms and so on. However, since it is believed that the carbocation is more stable if it is a secondary carbocation (rather than a primary carbocation) there is likely to be a tendency for the carbocation to react when in a more central (or internal) position. In view of the above, the compound of the invention may be considered as a mixture of varying lengths of hydrocarbyl groups represented by the structural unit R^1R^2HC-. Often the carbocation may favour one end of the hydrocarbyl group such as at the second, third, fourth or fifth carbon.

[0030] Since the compound of formula (1) may be considered a mixture, the mole percent of compounds defined by formula (1) that is, in which R^1 is H or C_1 or higher, may be at least 30 mole percent, or at least 40 mole percent, or at least 45 mole percent, or at least 50 mole percent of molecules formed during preparation of compounds of the invention. For example, the mole percent of compounds of formula (1) may be 30 to 99.9 mole percent, or 40 to 90 mole percent, or 55 to 75 mole percent of molecules formed during preparation of compounds of the invention.

[0031] The attachment of a hydrocarbyl group to the phenol group of the compound of the invention may be accomplished by a number of techniques well known to those skilled in the art, for example, a Friedel-Crafts reaction. The Friedel-Crafts reaction may react an olefin (e.g., a polymer containing an olefinic bond), or halogenated or hydrohalogenated derivative thereof, with a phenol in the presence of a Lewis acid catalyst. Methods and conditions for carrying out such reactions are well known to those skilled in the art. See, for example, the discussion in the article entitled, "Alkylation of Phenols" in "Kirk-Othmer Encyclopedia of Chemical Technology", Third Edition, Vol. 2, pages 65-66, Interscience Publishers.

[0032] The compound of the invention may be prepared by known processes to prepare novolak chemistry. For

example the compound may be prepared by reacting an aromatic hydroxy-substituted compound and an aldehyde. International publication WO 2009/064827, and U.S. Patents 5,707,946; 5,171,330; 4,565,460; 4,559,155; 4,575,526; 4,613,342; 5,039,437 and 5,082,470 all disclose methods of preparing aromatic hydroxy-substituted compounds.

[0033] The aldehyde may have 1 to 5 or 1 to 2 carbon atoms. Examples of a suitable aldehyde include formaldehyde, acetaldehyde, propionaldehyde, butyraldehyde, isobutyraldehyde, and pentanal. Formaldehyde may be supplied as a solution, but is more commonly used in the polymeric form, as paraformaldehyde. Polymeric aldehydes, such as paraformaldehyde, may be considered a reactive equivalent of, or a source for, an aldehyde. Aqueous solutions of aldehydes such as formalin may also be considered a reactive equivalent of, or a source for, an aldehyde.

[0034] The compound is present in the lubricating composition in a range including 100 ppm to 2000 ppm, or 200 ppm to 1000 ppm, or 300 ppm to 750 ppm or 400 ppm to 600 ppm.

Oils of Lubricating Viscosity

[0035] The lubricating composition comprises an oil of lubricating viscosity. The oil of lubricating viscosity in one embodiment includes bright stock. In other embodiments, the oil of lubricating viscosity may include oils such as natural and synthetic oils, oil derived from hydrocracking, hydrogenation, and hydrofinishing, unrefined, refined, re-refined oils or mixtures thereof. A more detailed description of unrefined, refined and re-refined oils is provided in International Publication WO2008/147704, paragraphs [0054] to [0056]. A more detailed description of natural and synthetic lubricating oils is described in paragraphs [0058] to [0059] respectively of WO2008/147704. Synthetic oils may also 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.

[0036] Oils of lubricating viscosity may also be defined as specified in April 2008 version of "Appendix E - API Base Oil Interchangeability Guidelines for Passenger Car Motor Oils and Diesel Engine Oils", section 1.3 Sub-heading 1.3. "Base Stock Categories". In one embodiment the oil of lubricating viscosity may be an API Group II or Group III oil. In one embodiment the oil of lubricating viscosity may be an API Group III base oil (typically including hydrocracked/hydroisomerized base oil).

[0037] The amount of the oil of lubricating viscosity present is typically the balance remaining after subtracting from 100 wt % the sum of the amount of the compound of the invention and the other performance additives.

[0038] The lubricating composition may be in the form of a concentrate and/or a fully formulated lubricant. If the lubricating composition of the invention (comprising the additives disclosed herein) 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 these additives to the oil of lubricating viscosity 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 Additives

[0039] The lubricating composition described herein optionally further includes at least one other performance additives. The other performance additives include at least one of metal deactivators, detergents, dispersants, viscosity modifiers, friction modifiers, corrosion inhibitors, dispersant viscosity modifiers, antiwear agents, extreme pressure agents, antiscuffing agents, antioxidants, foam inhibitors, demulsifiers, pour point depressants (other than the compound of formula (1)), seal swelling agents and mixtures thereof. Typically, fully-formulated lubricating oil will contain one or more of these performance additives.

Dispersants

[0040] Dispersants are known and include for example an N-substituted long chain alkenyl succinimide, a Mannich base, or mixtures thereof. Examples of N-substituted long chain alkenyl succinimides include polyisobutylene succinimides, wherein the polyisobutylene from which it is derived has a number average molecular weight in the range 350 to 5000, or 500 to 3000, or 750 to 1150.

[0041] The dispersants may also be post-treated by conventional methods by a reaction with any of a variety of agents. Among these are boron compounds (such as boric acid), urea, thiourea, dimercaptothiadiazoles, carbon disulphide, aldehydes, ketones, carboxylic acids such as terephthalic acid, hydrocarbonsubstituted succinic anhydrides, maleic anhydride, nitriles, epoxides, and phosphorus compounds. In one embodiment the post-treated dispersant is borated. In one embodiment the post-treated dispersant is reacted with dimercaptothiadiazoles.

Detergents

[0042] Detergents are known and include neutral or overbased detergents, i.e., ones prepared by conventional proc-

esses known in the art. Suitable detergent substrates include, phenates, sulphur containing phenates, sulphonates, salixarates, salicylates, carboxylic acids, phosphorus acids, alkyl phenols, sulphur coupled alkyl phenol compounds, or saligenins. In one embodiment the detergent includes a magnesium or calcium sulphonate, or mixtures thereof.

Antioxidant

[0043] Antioxidant compounds are known and include sulphurised olefins, diphenylamines (such as dinonyl diphenylamine), hindered phenols, molybdenum dithiocarbamates, and mixtures thereof. Antioxidant compounds may be used alone or in combination.

[0044] The hindered phenol antioxidant often contains a secondary butyl and/or a tertiary butyl group as a sterically hindering group. The phenol group is often further substituted with a hydrocarbyl group and/or a bridging group linking to a second aromatic group. Examples of suitable hindered phenol antioxidants include 2,6-di-tert-butylphenol, 4-methyl-2,6-di-tert-butylphenol, 4-ethyl-2,6-di-tert-butylphenol, 4-propyl-2,6-di-tert-butylphenol or 4-butyl-2,6-di-tert-butylphenol, or 4-dodecyl-2,6-di-tert-butylphenol. In one embodiment the hindered phenol antioxidant may be an ester and may include, e.g., Irganox™ L-135 from Ciba. Suitable examples of molybdenum dithiocarbamates which may be used as an antioxidant include commercial materials sold under the trade names such as Vanlube 822™ and Molyvan™ A from R. T. Vanderbilt Co., Ltd., and Adeka Sakura-Lube™ S-100, S-165 and S-600 from Asahi Denka Kogyo K. K and mixtures thereof.

Viscosity Modifiers

[0045] In addition to the polymers described herein as part of the invention, the lubricating composition may optionally further contain other known viscosity modifiers. The viscosity modifiers may be hydrogenated styrene-butadiene rubbers, ethylene-propylene copolymers, hydrogenated styrene-isoprene polymers, hydrogenated diene polymers, polyalkyl styrenes, polyolefins, esters of maleic anhydride-styrene copolymers, or mixtures thereof.

Antiwear Agent

[0046] The lubricating composition optionally further includes at least one antiwear agent. Examples of suitable antiwear agents include oil soluble amine salts of phosphorus compounds, sulphurised olefins, metal dihydrocarbyldithiophosphates (such as zinc dialkyldithiophosphates), thiocarbamate-containing compounds such as thiocarbamate esters, thiocarbamate amides, thiocarbamic ethers, alkylene-coupled thiocarbamates, and bis(S-alkyldithiocarbamyl) disulphides.

[0047] In one embodiment the oil soluble phosphorus amine salt antiwear agent includes an amine salt of a phosphorus acid ester or mixtures thereof. The amine salt of a phosphorus acid ester may include phosphoric acid esters and amine salts thereof; dialkyldithiophosphoric acid esters and amine salts thereof; amine salts of phosphites; and amine salts of phosphorus-containing carboxylic esters, ethers, and amides; and mixtures thereof. The amine salt of a phosphorus acid ester may be used alone or in combination.

[0048] In one embodiment the oil soluble phosphorus amine salt includes partial amine salt-partial metal salt compounds or mixtures thereof. In one embodiment the phosphorus compound further includes a sulphur atom in the molecule. In one embodiment the amine salt of the phosphorus compound may be ashless, i.e., metal-free (prior to being mixed with other components).

[0049] 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.

[0050] 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, Illinois), such as Armeen C, Armeen O, Armeen OL, Armeen T, Armeen HT, Armeen S and Armeen SD, wherein the letter designation relates to the fatty group, such as coco, oleyl, tallow, or stearyl groups.

[0051] Examples of suitable secondary amines include dimethylamine, diethylamine, dipropylamine, dibutylamine, diamylamine, dihexylamine, diheptyl-amine, methylethylamine, ethylbutylamine and ethylamylamine. The secondary amines may be cyclic amines such as piperidine, piperazine and morpholine.

[0052] 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.

[0053] In one embodiment the phosphorus acid amine salt includes an amine with C11 to C 14 tertiary alkyl primary amino groups or mixtures thereof. In one embodiment the phosphorus acid amine salt includes an amine with C14 to C18 tertiary alkyl primary amines or mixtures thereof. In one embodiment the phosphorus acid amine salt includes an amine with C18 to C22 tertiary alkyl primary amines or mixtures thereof.

[0054] Mixtures of amines may also be used in the invention. In one embodiment a useful mixture of amines is "Primene® 81R" or "Primene® JMT." Primene® 81R and Primene® JMT (both produced and sold by Rohm & Haas) are mixtures of C11 to C 14 tertiary alkyl primary amines and C18 to C22 tertiary alkyl primary amines respectively.

[0055] In one embodiment oil soluble amine salts of phosphorus compounds include a sulphur-free amine salt of a phosphorus-containing compound which may be obtained/obtainable by a process comprising: reacting an amine with either (i) a hydroxy-substituted di-ester of phosphoric acid, or (ii) a phosphorylated hydroxy-substituted di- or tri- ester of phosphoric acid. A more detailed description of compounds of this type is provided in International Application PCT/US08/051126 (or equivalent to US Application 11/627405 and US-2008-0182770).

[0056] In one embodiment the hydrocarbyl amine salt of an alkylphosphoric acid ester is the reaction product of a C 14 to C18 alkylated phosphoric acid with Primene 81R™.

[0057] Examples of hydrocarbyl amine salts of dialkyldithiophosphoric acid esters include the reaction product(s) of isopropyl, methyl-amyl (4-methyl-2-pentyl or mixtures thereof), 2-ethylhexyl, heptyl, octyl or nonyl dithiophosphoric acids with ethylene diamine, morpholine, or Primene 81R™, and mixtures thereof.

[0058] In one embodiment the 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. 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, and styrene oxide. In one embodiment the epoxide may be propylene oxide. The glycols may be aliphatic glycols having from 1 to 12, or from 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. Patent numbers 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(4-methyl-2-pentyl)phosphorodithioate (prepared by reacting di(4-methyl-2-pentyl)-phosphorodithioic acid with 1.3 moles of propylene oxide at 25 °C). The mixture may be 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).

[0059] The dithiocarbamate-containing compounds may be prepared by reacting a dithiocarbamate acid or salt with an unsaturated compound. The dithiocarbamate containing compounds may also be prepared by simultaneously reacting an amine, carbon disulphide and an unsaturated compound. Generally, the reaction occurs at a temperature from 25 °C to 125 °C.

[0060] Examples of suitable olefins that may be sulphurised to form an the sulphurised olefin include propylene, butylene, isobutylene, pentene, hexane, heptene, octane, nonene, decene, undecene, dodecene, undecyl, tridecene, tetradecene, pentadecene, hexadecene, heptadecene, octadecene, nonadecene, eicosene or mixtures thereof. In one embodiment, hexadecene, heptadecene, octadecene, eicosene or mixtures thereof and their dimers, trimers and tetramers are especially useful olefins. Alternatively, the olefin may be a Diels-Alder adduct of a diene such as 1,3-butadiene and an unsaturated ester, such as butyl acrylate.

[0061] Another class of sulphurised olefin includes fatty acids and their esters. The fatty acids are often obtained from vegetable oil or animal oil; and typically contain 4 to 22 carbon atoms. Examples of suitable fatty acids and their esters include triglycerides, oleic acid, linoleic acid, palmitoleic acid or mixtures thereof. Often, the fatty acids are obtained from lard oil, tall oil, peanut oil, soybean oil, cottonseed oil, sunflower seed oil or mixtures thereof. In one embodiment fatty acids and/or ester are mixed with olefins.

[0062] In an alternative embodiment, the ashless antiwear agent may be a monoester of a polyol and an aliphatic carboxylic acid, often an acid containing 12 to 24 carbon atoms. Often the monoester of a polyol and an aliphatic carboxylic acid is in the form of a mixture with a sunflower oil or the like, which may be present in the friction modifier mixture from 5 to 95, in several embodiments from 10 to 90, or from 20 to 85, or 20 to 80 weight percent of said mixture. The aliphatic carboxylic acids (especially a monocarboxylic acid) which form the esters are those acids typically containing 12 to 24, or from 14 to 20 carbon atoms. Examples of carboxylic acids include dodecanoic acid, stearic acid, lauric acid, behenic acid, and oleic acid.

[0063] Polyols include diols, triols, and alcohols with higher numbers of alcoholic OH groups. Polyhydric alcohols include ethylene glycols, including di-, tri- and tetraethylene glycols; propylene glycols, including di-, tri- and tetrapropylene glycols; glycerol; butane diol; hexane diol; sorbitol; arabitol; mannitol; sucrose; fructose; glucose; cyclohexane diol; erythritol; and pentaerythritols, including di- and tripentaerythritol. Often the polyol is diethylene glycol, triethylene glycol, glycerol, sorbitol, pentaerythritol or dipentaerythritol.

[0064] The commercially available monoester known as "glycerol monooleate" is believed to include 60 ± 5 percent

by weight of the chemical species glycerol monooleate, along with 35 ± 5 percent glycerol dioleate, and less than 5 percent trioleate and oleic acid. The amounts of the monoesters, described above, are calculated based on the actual, corrected, amount of polyol monoester present in any such mixture.

Extreme Pressure Agents

[0065] Extreme Pressure (EP) agents that are soluble in the oil include sulphur- and chlorosulphur-containing EP agents, chlorinated hydrocarbon EP agents and phosphorus EP agents. Examples of such EP agents include chlorinated wax; sulphurised olefins (such as sulphurised isobutylene), organic sulphides and polysulphides such as dibenzyldisulphide, bis-(chlorobenzyl) disulphide, dibutyl tetrasulphide, sulphurised methyl ester of oleic acid, sulphurised alkylphenol, sulphurised dipentene, sulphurised terpene, and sulphurised Diels-Alder adducts; phosphosulphurised hydrocarbons such as the reaction product of phosphorus sulphide with turpentine or methyl oleate; phosphorus esters such as the dihydrocarbon and trihydrocarbon phosphites, e.g., dibutyl phosphite, diheptyl phosphite, dicyclohexyl phosphite, pentylphenyl phosphite; dipentylphenyl phosphite, tridecyl phosphite, distearyl phosphite and polypropylene substituted phenol phosphite; metal thiocarbamates such as zinc dioctyldithiocarbamate and barium heptylphenol diacid; amine salts of alkyl and dialkylphosphoric acids or derivatives including, for example, the amine salt of a reaction product of a dialkyldithiophosphoric acid with propylene oxide and subsequently followed by a further reaction with P_2O_5; and mixtures thereof (as described in US 3,197,405).

[0066] Corrosion inhibitors that may be useful in the compositions of the invention include fatty amines, octyl octanamide, condensation products of dodecenyl succinic acid or anhydride and/or a fatty acid such as oleic acid with a polyamine.

[0067] Foam inhibitors that may be useful in the compositions of the invention include 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.

[0068] Pour point depressants (other than the compound of the invention) may include polyalphaolefins, esters of maleic anhydride-styrene copolymers, poly(meth)acrylates, polyacrylates or polyacrylamides. In one embodiment the pour point depressant may be (i) esters of maleic anhydride-styrene copolymers, or (ii) polymethacrylates. The pour point depressant may be present at 0 wt % to 0.5 wt %, or 0.1 wt % to 0.4 wt % of the lubricating composition.

[0069] Friction modifiers that may be useful in the compositions of the invention include fatty acid or fatty alkyl derivatives such as amines, esters, epoxides, fatty imidazolines, condensation products of carboxylic acids and polyalkylene-polyamines and amine salts of alkylphosphoric acids.

[0070] In several embodiments a suitable lubricating composition includes the compound of the formula disclosed herein that may be present (on an actives basis) in ranges as shown in the following table.

Table

	Embodiments (of lubricant composition)			
	A	B	C	D
Compound of Invention (ppm)	100-2000	200-1000	300-750	400-600
Other Performance Additives (wt %)	0-15	0.01-15	0.5-10	0.5-10
Oil of Lubricating Viscosity (wt %)		Balance to 100 %		

[0071] The following examples provide an illustration of the invention. These examples are non exhaustive and are not intended to limit the scope of the invention.

EXAMPLES

[0072] Preparative Example 1: A 3L flask under nitrogen is charged with a C_{24-28} alkylphenol (1000g, 2.222 mol) and heated to 85 °C. Sulphuric acid (3.46 g) is added dropwise over approximately 10 minutes. The flask is then heated to 105 °C and paraformaldehyde (81.6 g, 2.717 mol) is charged over four hours. The flask is then heated to 135 °C over two hours and then held at this temperature for a further two hours to collect any aqueous distillate. The reaction is then cooled to 121 °C and solid calcium hydroxide (3.16 g) charged to the flask, followed by a 50% aqueous solution of sodium hydroxide 30 minutes later. The flask is then held at 121°C for two hours, then heated to 152°C and held there for a further two hours with a high nitrogen sparge to quench the reaction and collect any additional/remaining aqueous distillate. The flask is then cooled to about 70°C and an aromatic solvent (Solvesso™ 150, 1022 g) charged to the flask. After 30 minutes the mixture is discharged and filtered to give a dark oil.

[0073] Comparative lubricant 1 (COMPL1) is a manual transmission lubricant comprising 7 wt % of a commercially available additive package, 400 ppm of a vinyl acetate copolymer (commercially available from The Lubrizol Corporation

under the trade name LZ®8056F, 0.3 wt % of a pour point depressant (derived from a polymethacrylate) and the balance to 100 wt % with bright stock lubricant.

[0074] Comparative lubricant 2 (COMPL2) is a manual transmission lubricant comprising 7 wt % of a commercially available additive package, 400 ppm of the material of Example 1 of International publication WO 2009/064827, and the balance to 100 wt % with bright stock lubricant.

[0075] Inventive lubricant 1 (INVL1) is a manual transmission lubricant comprising 7 wt % of a commercially available additive package, 400 ppm of the product of preparative example 1, 0.3 wt % of a pour point depressant (derived from an esterified maleic anhydride styrene), and the balance to 100 wt % with bright stock lubricant.

[0076] Lubricants COMPL1, COMPL2 and INVL1 are evaluated to determine filterability by the methodology described in ISO Standard ISO13357-2:1998(E), except the filter pressure is 2 bar (or 200 kPa) and a 5 μm membrane is used. Stage 2 filterability (as noted in section 4.3 of the procedure) is a ratio expressed as a percentage between the flow rate at the start of the filtration and the flow rate between 200 ml and 300 ml of filtered volume. The ratio is calculated by the formula given in section 10.2 of the procedure. The results obtained are:

Example	COMPL1	COMPL2	INVL1
Filterability Percent Ratio	unfilterable	unfilterable	91
Footnote: Unfilterable indicates that the filtration time exceeds 7200 seconds.			

[0077] The results indicate that the example of the invention has improved filterability compared with comparative examples COMPL1 and COMPL2 in a manual transmission containing bright stock. In particular both COMPL1 and COMPL2 fail the filterability test because both lubricants cause the filter to block. In contrast, the inventive lubricant allows the lubricant to pass stage 2 with a 91 % flow rate.

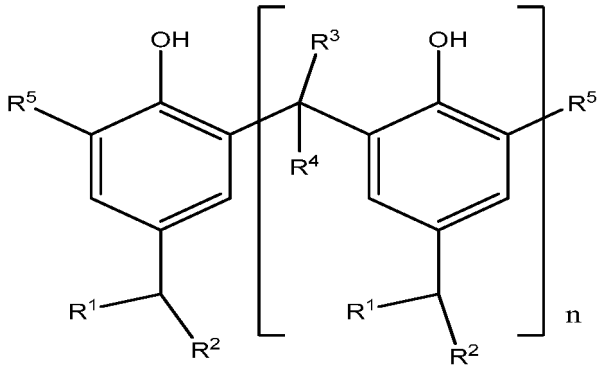
[0078] It is known that some of the materials described above may interact in the final formulation, so that the components of the final formulation may be different from those that are initially added. The products formed thereby, including the products formed upon employing lubricant composition of the present invention in its intended use, may not be susceptible of easy description.

[0079] 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.

[0080] 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: hydrocarbon substituents, including aliphatic, alicyclic, and aromatic substituents; 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; and hetero substituents, that is, substituents which similarly have a predominantly hydrocarbon character but contain other than carbon in a ring or chain. A more detailed definition of the term "hydrocarbyl substituent" or "hydrocarbyl group" is described in paragraphs [0118] to [0119] of International Publication WO2008147704 (corresponding to US-2010-0197536).

Claims

1. A method of lubricating a manual transmission comprising supplying to the manual transmission a lubricating composition comprising
   an oil of lubricating viscosity, wherein the oil of lubricating viscosity comprises bright stock present from 80 wt % to 95 wt % of the lubricating composition; and
   a compound represented by formula (1):



formula (1)

wherein

each R^1 is independently hydrogen or a linear hydrocarbyl group containing at least 1, or at least 3, or at least 4, or at least 6 carbon atoms;
each R^2 is independently a linear hydrocarbyl group containing at least 10 carbon atoms;
each R^3 and R^4 are independently hydrogen or a linear hydrocarbyl group containing 1 to 5, or 1 to 2 carbon atoms;
each R^5 is independently hydrogen or a linear hydrocarbyl group containing 1 to 10, or 1 to 5, or 1 to 2 carbon atoms; and
n is 1 to 20, or 3 to 15, or 4 to 8, or 6 to 8;

wherein the compound of formula (1) is present in the lubricating composition in a range including 100 ppm to 2000 ppm.

2. The method of claim 1, wherein each R^1 is independently a linear hydrocarbyl group containing at least 1 (or at least 3 or at least 6) carbon atoms; each R^2 is independently a linear hydrocarbyl group containing at least 10 carbon atoms; R^3 and R^4 are hydrogen; R^5 is hydrogen; and n is 4 to 8, or 6 to 8.

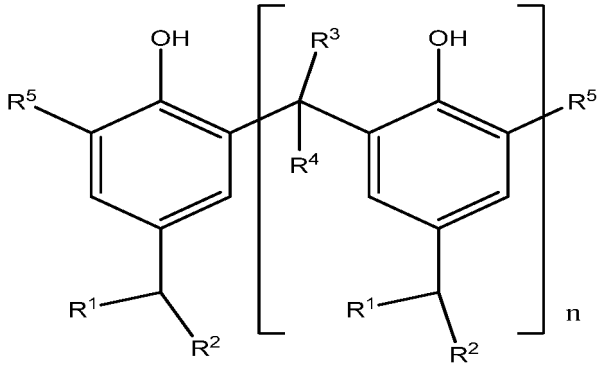
3. The method of claim 1, wherein each R^1 is hydrogen; each R^2 is a linear hydrocarbyl group containing at least 10 carbon atoms; R^3 and R^4 are hydrogen; R^5 is hydrogen; and n is 4 to 8, or 6 to 8.

4. The method of any preceding claim, wherein the lubricating composition has a SAE viscosity grade of XW-Y, wherein X is 70, 75, 80 or 85 and Y is 80, 85, 90, or 140.

5. The method of any preceding claim, wherein the lubricating composition further comprises compounds represented by formula (1) but being alkylated at the ortho-position and coupled at the para-position, relative to the hydroxyl.

6. The method of any preceding claim, wherein the compound of formula (1) is present in the lubricating composition in a range including 200 ppm to 1000 ppm, or 300 ppm to 750 ppm, or 400 ppm to 600 ppm.

7. The use of a lubricating composition comprising an oil of lubricating viscosity wherein the oil of lubricating viscosity comprises bright stock present from 80 wt % to 95 wt % of the lubricating composition and a compound of formula (1) as a filterability aid in a manual transmission, wherein the compound of formula (1) is represented by:



formula (1)

wherein

each R^1 is independently hydrogen or a linear hydrocarbyl group containing at least 1, or at least 3, carbon atoms;
each R^2 is independently a linear hydrocarbyl group containing at least 10 carbon atoms;
each R^3 and R^4 are independently hydrogen or a linear hydrocarbyl group containing 1 to 5, or 1 to 2 carbon atoms;
each R^5 is independently hydrogen or a linear hydrocarbyl group containing 1 to 10, or 1 to 5, or 1 to 2 carbon atoms; and
n is 1 to 20, or 3 to 15, or 4 to 8, or 6 to 8;

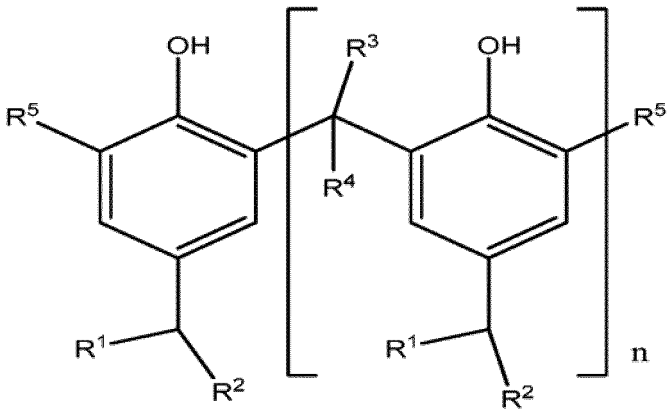
wherein the compound of formula (1) is present in the lubricating composition in a range including 100 ppm to 2000 ppm.

8. The use of claim 7, wherein the lubricating composition further comprises compounds represented by formula (1) but being alkylated at the ortho-position and coupled at the para-position, relative to the hydroxyl.

Patentansprüche

1. Verfahren zum Schmieren eines Handschaltgetriebes, bei dem man das Handschaltgetriebe mit einer Schmiermittelzusammensetzung versorgt, die Folgendes umfasst:

ein Öl mit Schmierviskosität, wobei das Öl mit Schmierviskosität Brightstock in einer Menge von 80 Gew.-% bis 95 Gew.-%, bezogen auf die Schmiermittelzusammensetzung, umfasst; und
eine Verbindung, die durch Formel (1) wiedergegeben wird:



Formel (1)

wobei

R^1 jeweils unabhängig für Wasserstoff oder eine lineare Hydrocarbylgruppe mit mindestens 1 oder mindestens 3 oder mindestens 4 oder mindestens 6 Kohlenstoffatomen steht;
R^2 jeweils unabhängig für eine lineare Hydrocarbylgruppe mit mindestens 10 Kohlenstoffatomen steht;
R^3 und R^4 jeweils unabhängig für Wasserstoff oder eine lineare Hydrocarbylgruppe mit 1 bis 5 oder 1 bis 2 Kohlenstoffatomen stehen;
R^5 jeweils unabhängig für Wasserstoff oder eine lineare Hydrocarbylgruppe mit 1 bis 10 oder 1 bis 5 oder 1 bis 2 Kohlenstoffatomen steht; und
n für 1 bis 20 oder 3 bis 15 oder 4 bis 8 oder 6 bis 8 steht;

wobei die Verbindung der Formel (1) in der Schmiermittelzusammensetzung in einem Bereich einschließlich 100 ppm bis 2000 ppm vorliegt.

2. Verfahren nach Anspruch 1, wobei R^1 jeweils unabhängig für eine lineare Hydrocarbylgruppe mit mindestens 1 (oder mindestens 3 oder mindestens 6) Kohlenstoffatomen steht; R^2 jeweils für eine lineare Hydrocarbylgruppe mit mindestens 10 Kohlenstoffatomen steht; R^3 und R^4 für Wasserstoff stehen; R^5 für Wasserstoff steht; und n für 4 bis 8 oder 6 bis 8 steht.

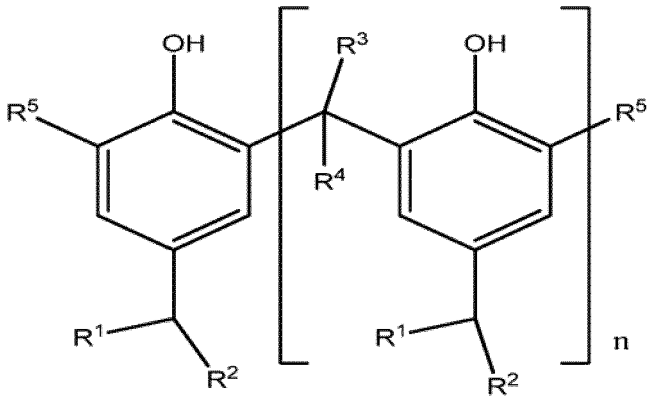
3. Verfahren nach Anspruch 1, wobei R^1 jeweils für Wasserstoff steht; R^2 jeweils unabhängig für eine lineare Hydrocarbylgruppe mit mindestens 10 Kohlenstoffatomen steht; R^3 und R^4 für Wasserstoff stehen; R^5 für Wasserstoff steht; und n für 4 bis 8 oder 6 bis 8 steht.

4. Verfahren nach einem der vorhergehenden Ansprüche, wobei die Schmiermittelzusammensetzung eine SAE-Viskositätsklasse XW-Y aufweist, wobei X für 70, 75, 80 oder 85 steht und Y für 80, 85, 90 oder 140 steht.

5. Verfahren nach einem der vorhergehenden Ansprüche, wobei die Schmiermittelzusammensetzung ferner Verbindungen umfasst, die durch Formel (1) wiedergegeben werden, aber in der ortho-Position zur Hydroxylgruppe alkyliert und in der para-Position zur Hydroxylgruppe gekuppelt sind.

6. Verfahren nach einem der vorhergehenden Ansprüche, wobei die Verbindung der Formel (1) in der Schmiermittelzusammensetzung in einem Bereich einschließlich 200 ppm bis 1000 ppm oder 300 ppm bis 750 ppm oder 400 ppm bis 600 ppm vorliegt.

7. Verwendung einer Schmiermittelzusammensetzung, umfassend ein Öl mit Schmierviskosität, wobei das Öl mit Schmierviskosität Brightstock in einer Menge von 80 Gew.-% bis 95 Gew.-%, bezogen auf die Schmiermittelzusammensetzung, umfasst, und eine Verbindung der Formel (1) als Filtrierbarkeitshilfe in einem Handschaltgetriebe, wobei die Verbindung der Formel (1) wiedergegeben wird durch:



Formel (1)

wobei

R^1 jeweils unabhängig für Wasserstoff oder eine lineare Hydrocarbylgruppe mit mindestens 1 oder mindestens 3 Kohlenstoffatomen steht;
R^2 jeweils unabhängig für eine lineare Hydrocarbylgruppe mit mindestens 10 Kohlenstoffatomen steht;

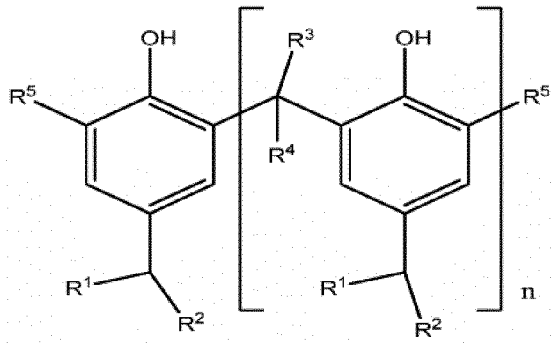
R^3 und R^4 jeweils unabhängig für Wasserstoff oder eine lineare Hydrocarbylgruppe mit 1 bis 5 oder 1 bis 2 Kohlenstoffatomen stehen;
R^5 jeweils unabhängig für Wasserstoff oder eine lineare Hydrocarbylgruppe mit 1 bis 10 oder 1 bis 5 oder 1 bis 2 Kohlenstoffatomen steht; und
n für 1 bis 20 oder 3 bis 15 oder 4 bis 8 oder 6 bis 8 steht;

wobei die Verbindung der Formel (1) in der Schmiermittelzusammensetzung in einem Bereich einschließlich 100 ppm bis 2000 ppm vorliegt.

8. Verwendung nach Anspruch 7, wobei die Schmiermittelzusammensetzung ferner Verbindungen umfasst, die durch Formel (1) wiedergegeben werden, aber in der ortho-Position zur Hydroxylgruppe alkyliert und in der para-Position zur Hydroxylgruppe gekuppelt sind.

Revendications

1. Procédé de lubrification d'une transmission manuelle comprenant la fourniture à la transmission manuelle d'une composition lubrifiante comprenant
une huile de viscosité lubrifiante, l'huile de viscosité lubrifiante comprenant une base lubrifiante de haute viscosité présente à 80 % en poids à 95 % en poids de la composition lubrifiante ; et
un composé représenté par la formule (1) :



formule (1)

dans laquelle

chaque R_1 représente indépendamment un hydrogène ou un groupe hydrocarbyle linéaire contenant au moins 1, ou au moins 3, ou au moins 4, ou au moins 6 atomes de carbone ;
chaque R_2 représente indépendamment un groupe hydrocarbyle linéaire contenant au moins 10 atomes de carbone ;
chaque R_3 et chaque R_4 représentent indépendamment un hydrogène ou un groupe hydrocarbyle linéaire contenant 1 à 5, ou 1 à 2 atomes de carbone ;
chaque R_5 représente indépendamment un hydrogène ou un groupe hydrocarbyle linéaire contenant 1 à 10, ou 1 à 5, ou 1 à 2 atomes de carbone ; et
n vaut 1 à 20, ou 3 à 15, ou 4 à 8, ou 6 à 8 ;

le composé de formule (1) étant présent dans la composition lubrifiante dans une gamme couvrant 100 ppm à 2000 ppm.

2. Procédé de la revendication 1, dans lequel chaque R_1 représente indépendamment un groupe hydrocarbyle linéaire contenant au moins 1 atome de carbone (ou au moins 3 ou au moins 6) ; chaque R_2 représente indépendamment un groupe hydrocarbyle linéaire contenant au moins 10 atomes de carbone ; R_3 et R_4 sont des hydrogènes ; R_5 est un hydrogène ; et n vaut 4 à 8, ou 6 à 8.

3. Procédé de la revendication 1, dans lequel chaque R_1 représente un hydrogène ; chaque R_2 représente un groupe hydrocarbyle linéaire contenant au moins 10 atomes de carbone ; R_3 et R_4 sont des hydrogènes ; R_5 est un

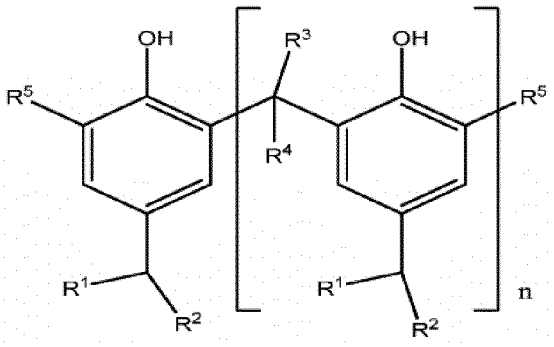
hydrogène ; et n vaut 4 à 8, ou 6 à 8.

4. Procédé d'une quelconque revendication précédente, dans lequel la composition lubrifiante a un grade de viscosité SAE de XW-Y, dans lequel X est égal à 70, 75, 80 ou 85 et Y est égal à 80, 85, 90 ou 140.

5. Procédé d'une quelconque revendication précédente, dans lequel la composition lubrifiante comprend en outre des composés représentés par la formule (1), mais étant alkylés à la position ortho et couplés à la position para, par rapport à l'hydroxyle.

6. Procédé d'une quelconque revendication précédente, dans lequel le composé de formule (1) est présent dans la composition lubrifiante dans une gamme couvrant 200 ppm à 1000 ppm, ou 300 ppm à 750 ppm, ou 400 ppm à 600 ppm.

7. Utilisation d'une composition lubrifiante comprenant une huile de viscosité lubrifiante, l'huile de viscosité lubrifiante comprenant une base lubrifiante de haute viscosité présente à 80 % en poids à 95 % en poids de la composition lubrifiante et un composé de formule (1) comme adjuvant de filtrabilité dans une transmission manuelle, le composé de formule (1) étant représenté par :



formule (1)

dans laquelle

chaque R_1 représente indépendamment un hydrogène ou un groupe hydrocarbyle linéaire contenant au moins 1 ou au moins 3 atomes de carbone ;
chaque R_2 représente indépendamment un groupe hydrocarbyle linéaire contenant au moins 10 atomes de carbone ;
chaque R_3 et chaque R_4 représentent indépendamment un hydrogène ou un groupe hydrocarbyle linéaire contenant 1 à 5, ou 1 à 2 atomes de carbone ;
chaque R_5 représente indépendamment un hydrogène ou un groupe hydrocarbyle linéaire contenant 1 à 10, ou 1 à 5, ou 1 à 2 atomes de carbone ; et
n vaut 1 à 20, ou 3 à 15, ou 4 à 8, ou 6 à 8 ;

le composé de formule (1) étant présent dans la composition lubrifiante dans une gamme couvrant 100 ppm à 2000 ppm.

8. Utilisation de la revendication 7, dans laquelle la composition lubrifiante comprend en outre des composés représentés par la formule (1), mais étant alkylés à la position ortho et couplés à la position para, par rapport à l'hydroxyle.

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- US 6495495 B **[0003]**
- DE 1147799 B1 **[0004]**
- EP 184083 A **[0005]**
- EP 203554 A **[0005]**
- EP 111883 A **[0006]**
- US 5039437 A **[0007] [0032]**
- US 5082470 A **[0007] [0032]**
- US 4565460 A **[0008] [0032]**
- US 4559155 A **[0008] [0032]**
- US 4575526 A **[0008] [0032]**
- US 4613342 A **[0008] [0032]**
- US 5171330 A **[0009] [0032]**
- US 5707946 A **[0010] [0032]**
- WO 2009064827 A **[0011] [0032] [0074]**
- WO 2008147704 A **[0035] [0080]**
- US 08051126 W **[0055]**
- US 11627405 B **[0055]**
- US 20080182770 A **[0055]**
- US 3197405 A **[0058] [0065]**
- US 3544465 A **[0058]**
- US 20100197536 A **[0080]**

Non-patent literature cited in the description

- Alkylation of Phenols. Kirk-Othmer Encyclopedia of Chemical Technology. Interscience Publishers, vol. 2, 65-66 **[0031]**