

Description

This invention relates to a method of preparing certain boron-containing compositions as well as additive concentrates and lubricant compositions containing such boron-containing compositions.

5 The use of various boron-containing compounds as extreme pressure and anti-wear additives for lubricating oils has been suggested in the prior art.

There continues to be a need for lubricants of sufficient lubricity to provide lubricating between the bearing surfaces of moving metal components where the bearing surfaces are subjected to large forces at the point of contact. These forces and the accompanying friction result in the generation of heat which elevates the metal
10 temperatures. If the pressure is sufficiently high and the relative motion between surfaces sufficient, very high metal temperatures can result. Thus, in extreme cases, the contacting metal surfaces may actually weld one to the other. With less extremes of pressure, inadequate lubrication between surfaces is manifested in accelerated wear, scuffing, etc.

In addition to anti wear and extreme pressure properties, lubricants for use in limited slip differentials require
15 unusual frictional properties. Limited slip differentials are unlike conventional differentials in that they have the ability to move a vehicle when only one wheel has traction. This advantage is accomplished through an internal clutch that restricts conventional differential action and tends to make the left and right rear axle shafts turn as one. There are a variety of clutch arrangements employed to accomplish this result but most rely on the force of friction to restrict the free turning of the axle shaft. Since the clutches are continuously contacted by the differential lubricant, the restricting force depends on the coefficient of friction imparted to the engaging parts by
20 said lubricant. The coefficient of friction must be compromised. On the other hand, it must be high enough to allow the clutch plates to seize, thus allowing the vehicle to move in instances when one rear wheel has low traction and, on the other hand, it must be sufficiently low to allow the clutch plates to slip and accommodate conventional differential action such as occurs when the vehicle turns corners. The ability of a lubricant to impart
25 a coefficient of friction within this fairly wide effective range is referred to as effectiveness.

Another important aspect of frictional properties relates to the chatter or noise caused by the differential action of clutches. Chatter is generated by a stick-slip action of the engaging parts of the clutches. Differential manufacturers have found that stick-slip is primarily the result of the static coefficient of friction being greater than the dynamic coefficient of friction. That is, when the ratio of the static coefficient of friction to the dynamic
30 coefficient of friction is greater than one.

Many diverse additives for enhancing anti-wear, extreme pressure and friction modifying properties are well known in the prior art. Examples of such additives are chlorinated waxes, alkyl polysulfides, alkyl phosphites, alkyl phosphates, metal dithiophosphates, sulfurized sperm oil, sulfurized olefins, alkali metal borates, and the like. However, a need still exists for materials which can provide these properties and combinations of these
35 properties.

U.S. Patent 4,512,903 describes the preparation of amides from mono- or polyhydroxy-substituted aliphatic monocarboxylic acids and primary or secondary amines. The amides are reported to be useful as friction reducing agents when incorporated into fuels or lubricants. U.S. Patent 4,406,802 describes various borated additive compositions which are useful in lubricating compositions as multi-functional additive compounds having friction-reducing and oxidation and corrosion-inhibiting characteristics. In general, the boron-containing compounds are borates of mixed alcohols, amides, amines, and hydroxy esters, ethoxylated amines and ethoxylated amides and mixtures thereof.
40

U.S. Patent No. 4 472 289 describes lubricant and fuel compositions obtained by adding to a lubricant or fuel a product made by reacting a boron compound with a mixture of vicinal diols and (1) long chain alcohols or (2) hydroxyl-containing alkyl carboxylates, to reduce friction and fuel consumption in internal combustion engines.
45

WO 84/04323 describes inhibitors for preventing the corrosion of metal surfaces that contact aqueous systems containing them. The inhibitors comprises mixtures of monoamine salts of polycarboxylic acids and boric compounds.

50 This invention is directed to the preparation of boron-containing compositions and to the use of such compositions in lubricants and fuels.

Thus, in one aspect, the invention provides a method of preparing a boron-containing composition which comprises reacting

(A) a hydroxy-substituted amide of the formula
55 $(\text{HO})_n\text{-R-(C(O)NR'R'')}_m$ (I)

wherein R is a polyvalent hydrocarbyl group, R' is a hydrocarbyl group and R'' is hydrogen or a hydrocarbyl group, m is 1 or 2, and n is an integer from 1 to 10 provided that only one free hydroxyl group is attached per carbon atom of the hydrocarbyl group and that when m is 1, n is 1; with

(B) a boron compound selected from boric acid, boron trioxide, boron halides, boron amides and boron esters and wherein (A) is reacted with (B) at an elevated temperature up to but not including the decomposition temperature of any reactants or the product of the reaction and wherein the molar ratio of the hydroxyl group to the boron compound is from about 0.5:1 to about 4:1.

In a preferred aspect, the invention provides a method of preparing boron-containing compositions which comprises reacting

(A) an amide of a hydroxy-substituted carboxylic having the formula



wherein R is a polyvalent aliphatic group containing from 1 to 6 carbon atoms, R' is an aliphatic group containing from about 6 to about 30 carbon atoms, R'' is hydrogen or is as defined for R', m is 1 or 2, and n is an integer from 1 to about 5 provided that when m is 1, n is 1; with

(B) a boron compound selected from boric acid, boron oxide, boron halides, boron amides and boron esters in a ratio of hydroxyl group to boron of from about 0.5:1 to about 3:1 wherein (A) is reacted with (B) at an elevated temperature up to but not including the decomposition temperature of any reactants or product thereof.

In addition to the method of preparing boron-containing compositions, the present invention also relates to the use of such compositions in lubricant and fuel compositions. The boron-containing compositions of the invention are incorporated into lubricating compositions to improve fuel economy, and reduce friction and wear.

Preferred features and embodiments of the invention are described below.

The boron-containing compositions of the present invention may be prepared by reacting at least one hydroxy-substituted amide (A), with a boron compound (B).

Reactant (A):

Reactant (A) is a hydroxy-substituted amine of formula (I) as defined above.

In the above Formula I, R is a polyvalent hydrocarbyl group such as, for example, alkylene, alkenylene, alkynylene, arylene or alkarylene groups containing generally from about 1 to 10 carbon atoms. The hydrocarbyl group R may be aliphatic, alicyclic, aromatic, aliphatic- and alicyclic-substituted aromatic, aromatic-substituted aliphatic, etc. Preferably, the hydrocarbyl group R is an alkylene group containing from 1 to about 5 carbon atoms, and n is from 1 to about 5. Even more generally, R will contain from 1 to about 3 carbon atoms, and n will be from 1 to 3. The hydrocarbyl groups R' and R'' may contain up to about 150 carbon atoms, and preferably will contain from about 4 to about 30 carbon atoms.

The amides useful as reactant (A) can be prepared by the reaction of (A-1) a hydroxy-substituted carboxylic acid or an anhydride thereof with (A-2) at least one primary or secondary hydrocarbyl amine.

The hydroxy-substituted carboxylic acids useful in the preparation of reactant (A) may be represented by the formula



wherein m, n and R are as defined above.

Suitable hydroxy-substituted monocarboxylic acids include hydroxyacetic acid, glycolic acid, mandelic acid, 2-hydroxypropionic acid, 3-hydroxypropionic acid, 2-hydroxybutyric acid, 3-hydroxybutyric acid and, 4-hydroxybutyric acid.

The hydroxy-substituted monocarboxylic acids also may be aromatic hydroxy-carboxylic acids such as salicylic acid, 3-chloro-2-hydroxy-benzoic acid, 3-methyl-2-hydroxy-benzoic acid, 2-hydroxy-1-naphthoic acid, 1-hydroxy-2-naphthoic acid, etc.

Examples of hydroxy-substituted dicarboxylic acids useful in the method of the present invention include tartronic acid, tartaric acid, malic acid, dihydroxy-maleic acid, etc. The corresponding anhydrides also may be used.

Hydroxy-substituted amides useful as reactant (A) in the present invention also may be prepared by the reaction of a lactone of the formula



wherein R* is a divalent hydrocarbyl group, with at least one primary or secondary hydrocarbyl amine. Generally, R* will be a divalent hydrocarbyl group containing up to about 5 to 6 carbon atoms. Examples of suitable lactones include gamma-butyrolactone, valerolactone and caprolactone.

The amines which are reacted with the above described hydroxy-substituted carboxylic acids or anhydrides

thereof, or the lactones of formula (III), are primary or secondary hydrocarbyl amines having the general formula

$$R'R''NH \quad (IV)$$

wherein R' is a hydrocarbyl group and R'' is hydrogen or a hydrocarbyl group. Generally, the hydrocarbyl group R' and R'' will contain up to about 150 carbon atoms and will more often be aliphatic hydrocarbyl groups containing from about 4 to about 30 carbon atoms.

In one preferred embodiment, the hydrocarbyl amines which are useful in preparing the amides of the present invention are primary hydrocarbyl amines containing from about 4 to about 30 carbon atoms in the hydrocarbyl group, and more preferably from about 8 to about 20 carbon atoms in the hydrocarbyl group. The hydrocarbyl group may be saturated or unsaturated. Representative examples of primary saturated amines are those known as aliphatic primary fatty amines and commercially known as "Armeen" primary amines (products available from Armak Chemicals, Chicago, Illinois). Typical fatty amines include alkyl amines such as N-hexylamine, N-octylamine, N-decylamine, N-dodecylamine, N-tetradecylamine, N-pentadecylamine, N-hexadecylamine, N-octadecylamine, (stearyl amine), etc. These Armeen primary amines are available in both distilled and technical grades. While the distilled grade will provide a purer reaction product, the desirable amides will form in reactions with the amines of technical grade.

Primary amines in which the hydrocarbon chain comprises olefinic unsaturation also are quite useful. Thus, the R' and R'' groups may contain one or more olefinic unsaturation depending on the length of the chain, usually no more than one double band per 10 carbon atoms. Representative amines are dodecenyamine, myristoleylamine, palmitoleylamine, oleylamine and linoleylamine. Such unsaturated amines also are available under the Armeen tradename.

Also suitable are mixed fatty amines such as Armak's Armeen-C, Armeen-O, Armeen-OL, Armeen-T, Armeen-HT, Armeen S and Armeen SD. Secondary amines include dialkylamines having two of the above alkyl groups including such commercial fatty secondary amines as Armeen 2C and Armeen HT, and also mixed dialkylamines where R' is a fatty amine and R'' may be a lower alkyl group (1-9 carbon atoms) such as methyl, ethyl, n-propyl, i-propyl, butyl, etc., or R'' may be an alkyl group bearing other non-reactive or polar substituents (CN, alkyl, carbalkoxy, amide, ether, thioether, halo, sulfoxide, sulfone) such that the essentially hydrocarbon character of the radical is not destroyed. The fatty polyamine diamines include mono- or dialkyl, symmetrical or asymmetrical ethylene diamines, propane diamines (1,2, or 1,3), and polyamine analogs of the above. Suitable commercial fatty polyamines are "Duomeen C" (N-coco-1,3-diaminopropane), "Duomeen S" (N-soya-1,3-diaminopropane), "Duomeen T" (N-tallow-1,3-diaminopropane), or "Duomeen O" (N-oleyl-1,3-diaminopropane). "Duomeens" are commercially available diamines described in Product Data Bulletin No. 7-10R1 of Armak Chemical Co., Chicago, Illinois.

Other primary amines useful in the preparation of the amides are the primary ether amines R''OR'NH₂ wherein R' is a divalent alkylene group having 2 to 6 carbon atoms and R'' is a hydrocarbyl group of about 5 to about 150 carbon atoms. These primary ether amines are generally prepared by the reaction of an alcohol R''OH with an unsaturated nitrile. The R'' group of the alcohol can be a hydrocarbon-based group having up to about 150 carbon atoms. Typically, and for efficiency and economy, the alcohol is a linear or branched aliphatic alcohol with R'' having up to about 50 carbon atoms, preferably up to 26 carbon atoms and most preferably R'' has from 6 to 20 carbon atoms. The nitrile reactant can have from 2 to 6 carbon atom with acrylonitrile being most preferred. Ether amines are known commercial products which are available under the name SURFAM™ produced and marketed by Mars Chemical Company, Atlanta, Georgia. Typical of such amines are those having from about 150 to about 400 molecular weight. Preferred etheramines are exemplified by those identified as SURFAM P14AB (branched C₁₄), SURFAM P16A (linear C₁₆), SURFAM P17AB (branched C₁₇). The carbon chain lengths (i.e., C₁₄, etc.) of the SURFRAMS described above and used hereinafter are approximate and include the oxygen ether linkage. For example, a C₁₄ SURFRAM would have the following general formula



The preparation of the hydroxy-substituted amides utilized in the preparation of the boron-containing compositions of the present invention is effected by techniques well known in the art. The amides generally will be prepared by reacting approximately equal amounts of the acid or anhydride thereof with one or more of the primary or secondary amines described above. When the hydroxy-substituted carboxylic acid (or derivative) is a dicarboxylic acid (or derivative), and the amine is a primary amine, the reaction product may be the corresponding amide, imide or a mixture of amide and imide depending on the reaction conditions.

In general, the reaction of the hydroxy-substituted carboxylic acid (or derivative) with the primary or secondary amine is conducted at atmospheric, super-atmospheric or sub-atmospheric pressure at temperatures ranging from about room temperature to about decomposition temperature of the mixture or of any products formed from the reaction mixture. Preferably, the reaction is carried out at atmospheric pressure and at temperatures below about 200°C. A preferred range of temperatures at atmospheric pressure is from about 60 to about 180°C. Generally, the reaction is conducted until water evolution ceases.

The preparation of the hydroxy-substituted carboxylic acid amides can be carried out conveniently in solvents, particularly those which can withstand relatively high reaction temperatures and which are inert toward the reactants and the desired product. Various hydrocarbon solvents are particularly useful, and these include, for example, toluene, xylene, ethyl benzene, benzene, and mineral oils. More volatile solvents such as toluene and xylene are preferred where it is desired to remove the solvent by conventional techniques at the end of the reaction. Mineral oils are desirable as solvents for the reaction when the oil does not have to be removed from the reaction product such as when the product is to be used as additives for lubricants. Alternatively, in some instances, no solvent at all is used.

The following examples illustrate the preparation of reactant (A) useful in the preparation of the boron-containing compositions of the present invention. Unless otherwise indicated in the following examples, and elsewhere in the specification and claims, all parts and percentages are by weight and all temperatures are in degrees centigrade.

Example 1-A

A mixture of 600 parts of toluene and 1897 parts of oleylamine (Armeen O) is prepared and heated to the reflux temperature whereupon 760.2 parts of hydroxyacetic acid (70% in water) is added dropwise. Water is removed as an azeotrope, and the residue is stripped of volatile materials under partial vacuum and filtered. The filtrate is the desired amide.

Example 2-A

A mixture of 588 parts of Armeen C (mixed primary fatty amines primarily cocoamine) and 150 parts of toluene is prepared and heated to a temperature of about 90°C whereupon 317.5 parts of hydroxyacetic acid (67% in water) are added dropwise under a nitrogen sweep. Water is removed as an azeotrope and the residue is stripped under mild vacuum. The stripped residue is the desired product containing 5.41% nitrogen (theory, 5.22%).

Example 3-A

A mixture of 1540 parts of di-cocoamine (Armeen 2C), 300 parts of tartaric acid and 2 parts of paratoluenesulfonic acid is prepared and heated to about 90°C and water is removed by distillation. After all of the water is removed, the temperature of the reaction mixture is raised to about 200°C and filtered through a filter aid. The filtrate is the desired product containing 2.99% nitrogen.

Example 4-A

A mixture of 150 parts (1.0 mole) of tartaric acid, 261 parts (1.0 mole) of oleylamine (Armeen O), and 350 parts of toluene is stirred and heated to 90-100°C. The viscous mixture then is heated to 140-170°C while removing toluene and water. The residue is filtered through a filter aid, and the filtrate is the desired product containing 3.4% nitrogen (theory, 3.7%).

Example 5-A

A mixture of 540 parts of oleylamine and 200 parts of toluene is prepared, and 180 parts of lactic acid is added dropwise at a temperature of about 80-100°C while maintaining a nitrogen atmosphere. The reaction mixture is heated to reflux, and water is removed. The residue is stripped under a vacuum and filtered through filter aid. The filtrate is the desired product containing 4.13% nitrogen (theory, 4.09%).

Example 6-A

A mixture of 548 parts (2 moles) of Armeen 18 (octadecylamine) and 200 parts of xylene is heated until the mixture is fluid whereupon 180 parts (2 moles) of lactic acid are added dropwise. When the addition of the lactic acid is completed, the mixture is heated at reflux (at about 140°C) while blowing with nitrogen, and the water is removed. The residue is the desired product.

Example 7-A

A mixture of 680 parts (2 moles) of Duomeen T (N-tallow-1,3-diaminopropane) and 200 parts of xylene is heated until fluid whereupon 360 parts (4 moles) of lactic acid are added in small increments. The reaction mixture is heated at reflux (about 140°C) while stirring and blowing with nitrogen. Water is removed, and after all the water is removed, the mixture is stripped to a temperature of 195°C at 900 Pa (7 mm Hg). The residue is filtered, and the filtrate is the desired product.

Example 8-A

A mixture of 416 parts (1.46 moles) of bis(2-hydroxyethyl) cocoamine (Ethomeen C₁₂), 219 parts (1.46 moles) of tartaric acid and 800 parts of toluene is prepared containing a few drops of titanium tetrabutoxide. The mixture is heated at the reflux temperature while removing water for a period of about 4 to 5 hours. Paratoluenesulfonic acid (1 part) is added to the mixture which is heated at reflux temperature while additional water is removed. The residue remaining in the reaction vessel is the desired product.

Example 9-A

A mixture of 450 parts (3 moles) of tartaric acid, 432 parts of amino propyl morpholine and 500 parts of xylene is heated with stirring while removing water as an azeotrope. The remaining xylene is then removed from the reaction mixture. The residue is the desired product.

Example 10-A

A mixture of 228 parts of caprolactone and 540 parts of oleylamine (Armeen 0) is prepared and heated to a temperature of about 110°C thereafter to a temperature of about 140°C over a period of two days. On cooling, the desired amide is recovered containing 3.65% nitrogen (theory, 3.65%).

The boron-containing compositions of the present invention are prepared by reacting (A) at least one of the above-identified hydroxy-substituted amides with (B) a boron compound selected from boric acid, boron trioxide, boron halides, boron amides and boron esters. Boron trioxide will react first with water which is present in the reaction mixture to form boric acid, which then reacts with reactant (A). Boric acid is the preferred boron compound, and any of the various forms of boric acid may be used, including metaboric acid (HBO₂) orthoboric acid (H₃BO₃) and tetraboric acid (H₂B₄O₇). The esters of these acids include, for example, the methyl, ethyl and propyl esters, with the methyl esters being most readily available and therefore the most often used.

The reaction between the hydroxy-substituted amide (reactant (A)) and boron compound (B) can be conducted at an elevated temperature up to but not including the decomposition temperature of any of the reactants or the product of the reaction. The decomposition point is that temperature (a+) which there is sufficient decomposition of any reactant or product such as to interfere with the production of the desired products. Generally the reaction between reactants (A) and (B) is conducted at a temperature within the range of from about 80° to about 200°C.

The amount of reactant (A) reacted with reactant (B) may be varied over a wide range and generally, the amounts are selected to provide a hydroxyl group to boron mole ratio in the mixture of from about 0.5:1 to about 4:1, and more generally, from about 1:1 to about 3:1. The reaction frequently is effective in the presence of a substantially inert, normally liquid organic diluent, typically an aromatic hydrocarbon such as toluene or xylene, a chlorinated aromatic hydrocarbon such as chlorobenzene, or an ether such as ethylene glycol dimethylether. Alternatively, reactants (A) and (B) can be reacted in the absence of added solvent.

The reaction of the hydroxy-substituted amide (reactant (A)) with the boron compound results in the formation of water which can be removed during the reaction, and the formation and removal of water provides a convenient method for determining when the reaction is completed. In some instances, all of the boron compound added to the reaction mixture does not react, and the unexpected boron compound remains as solid material which can be separated by filtration at the end of the reaction.

The following examples illustrate the preparation of the boron-containing compositions of the present invention.

Example I

A mixture of 1645 parts (5 moles) of the product of Example 1-A, 154.5 parts (2.5 moles) of boric acid and 400 parts of toluene is prepared and heated to the reflux temperature while removing water. The residue is

stripped to 160°C/3.3 kPa (25 mm Hg), filtered while hot through a filter aid, and the filtrate is the desired product containing 1.74% boron (theory, 1.58%).

Example II

A mixture of 268 parts (1 mole) of the product of Example 2-A, 30.9 parts (0.5 mole) of boric acid and 100 parts of toluene is prepared and heated to the reflux temperature while removing water. The reaction mixture then is stripped to 120°C at 3.3 kPa (25 mm Hg). The residue is the desired product containing 1.77% boron (theory, 1.92%).

Example III

A mixture of 132 parts (0.35 mole) of the product of Example 4-A, 10.8 parts (0.17 mole) of boric acid and about 200 parts of toluene is heated to the reflux temperature, and water is removed. The reaction mixture is vacuum stripped to 140°C/2.7 - 5.3 kPa (20-40 mm Hg). The residue in the reaction vessel is the desired product containing 1.3% boron (theory, 1.3%).

Example IV

A mixture of 342 parts (1 mole) of the product of Example 5-A, 30.9 parts (0.5 mole) of boric acid and 100 parts of toluene is heated to the reflux temperature with removal of water. The reaction mixture is stripped to 120°C/3.3 kPa (25 mm Hg), and the residue is the desired product containing 1.63% boron (theory, 1.52%).

Example V

A mixture of 213 parts (0.3 mole) of the boron-containing product of Example IV, 102.6 parts (0.3 mole) of the product of Example 5-A and 100 parts of toluene is heated to reflux while removing water. The reaction mixture is stripped to 150°C/4 kPa (30 mm Hg), and the residue is the desired product containing 1.10% boron (theory, 1.04%).

Example VI

A mixture of 384 parts (1 mole) of the product of Example 10-A, 30.9 parts (0.5 mole) of boric acid and 100 parts of toluene is heated to reflux while removing water under a nitrogen sweep. The reaction mixture then is stripped to 130°C/2.7 kPa (20 mm Hg), and the residue is the desired product containing 1.51% boron (theory, 1.30%).

The boron-containing compositions of the invention can be effectively employed in a variety of lubricating compositions formulated for a variety of uses. The boron-containing compositions are useful particularly as friction-reducing and anti-wear agents. The use of the boron-containing compositions in lubricants results in improved fuel economy. These lubricating compositions are based on diverse oils of lubricating viscosity, including natural and synthetic lubricating oils and mixtures thereof. These lubricating compositions containing the subject additive are effective as crankcase lubricating oils for sparkignited and compression-ignited internal combustion engines, including automobile and truck engines, two-cycle engines, aviation piston engines, marine and low-load diesel engines, and the like. Also, automatic transmission fluids, transaxle lubricants, gear lubricants, metal-working lubricants, hydraulic fluids, and other lubricating oil and grease compositions can benefit from the incorporation of the subject additive.

Natural oils include animal oils and vegetable oils (e.g., castor oil, lard oil) as well as 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. Oils of lubricating viscosity derived from coal or shale are also useful. Synthetic lubricating oils include hydrocarbon oils and halosubstituted hydrocarbon oils such as polymerized and interpolymers of olefins (e.g., polybutylenes, polypropylenes, propylene-isobutylene copolymers, chlorinated polybutylenes, etc.); poly(1-hexenes), poly(1-octenes), poly(1-decenes), etc. and mixtures thereof; alkylbenzenes (e.g., dodecylbenzenes, tetradecylbenzenes, dinonylbenzenes, di-(2-ethylhexyl)-benzenes, etc.); polyphenyls (e.g., biphenyls, terphenyls, alkylated polyphenyls, etc.); alkylated diphenyl ethers and alkylated diphenyl sulfides and the derivatives, analogs and homologs thereof and the like.

Alkylene oxide polymers and interpolymers and derivatives thereof where the terminal hydroxyl groups have been modified by esterification, etherification, etc., constitute another class of known synthetic lubricating oils that can be used. These are exemplified by the oils prepared through polymerization of ethylene oxide or

propylene oxide, the alkyl and aryl ethers of these polyoxyalkylene polymers (e.g., methylpolyisopropylene glycol ether having an average molecular weight of about 1000, diphenyl ether of polyethylene glycol having a molecular weight of about 500-1000, diethyl ether of polypropylene glycol having a molecular weight of about 1000-1500, etc.) or mono- and polycarboxylic esters thereof, for example, the acetic acid esters, mixed C₃-C₈ fatty acid esters, or the C₁₃Oxo acid diester of tetraethylene glycol.

Another suitable class of synthetic lubricating oils that can be used comprises the esters of dicarboxylic acids (e.g., phthalic acid, succinic acid, alkylsuccinic acids, alkenylsuccinic acids, maleic acid, azelaic acid, suberic acid, sebacic acid, fumaric acid, adipic acid, linoleic acid dimer, malonic acid, alkyl malonic acids, alkenyl malonic acids, etc.) with a variety of alcohols (e.g., butyl alcohol, hexyl alcohol, dodecyl alcohol, 2-ethylhexyl alcohol, ethylene glycol, diethylene glycol monoether, propylene glycol, etc.) Specific examples of these esters include dibutyl adipate, di(2-ethylhexyl) sebacate, di-n-hexyl fumarate, dioctyl sebacate, diisooctyl azelate, diisodecyl azelate, dioctyl phthalate, didecyl phthalate, dieicosyl sebacate, the 2-ethylhexyl diester of linoleic acid dimer, the complex ester formed by reacting one mole of sebacic acid with two moles of tetraethylene glycol and two moles of 2-ethylhexanoic acid and the like.

Esters useful as synthetic oils also include those made from C₅ to C₁₂ monocarboxylic acids and polyols and polyol ethers such as neopentyl glycol, trimethylol propane, pentaerythritol, dipentaerythritol, tripentaerythritol, etc.

Silicon-based oils such as the polyalkyl-, polyaryl-, polyalkoxy-, or polyaryloxy-siloxane oils and silicate oils comprise another useful class of synthetic lubricants (e.g., tetraethyl silicate, tetraisopropyl silicate, tetra-(2-ethylhexyl)silicate, tetra-(4-methyl-hexyl)silicate, tetra-(p-tert-butylphenyl) silicate, hexyl-(4-methyl-2-pentoxo)disiloxane, poly(methyl) siloxanes, poly(methylphenyl)siloxanes, etc.). Other synthetic lubricating oils include liquid esters of phosphorus-containing acids (e.g., tricresyl phosphate, trioctyl phosphate, diethyl ester of decane phosphonic acid, etc.), polymeric tetrahydrofurans and the like.

Unrefined, refined and rerefined oils, either natural or synthetic (as well as mixtures of two or more of any of these) of the type disclosed above can be used in the concentrates of the present invention. Unrefined oils are those obtained directly from a natural or synthetic source without further purification treatment. For example, a shale oil obtained directly from retorting operations, a petroleum oil obtained directly from primary distillation or ester oil obtained directly from an esterification process and used without further treatment would be an unrefined oil. 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. Many such purification techniques are known to those skilled in the art such as solvent extraction, secondary distillation, acid or base extraction, filtration, percolation, etc. Rerefined oils are obtained by processes similar to those used to obtain refined oils applied to refined oils which have been already used in service. Such rerefined oils are also known as reclaimed or reprocessed oils and often are additionally processed by techniques directed to removal of spent additives and oil breakdown products.

Generally the lubricants of the present invention contain an amount of one or more of the compositions of this invention sufficient to provide them with improved friction-reducing, anti-wear and/or extreme pressure properties. Normally the amount employed will be about 0.01% to about 20%, preferably about 0.1% to about 10% of the total weight of the lubricating composition. This amount is exclusive of solvent/diluent medium. In lubricating compositions operated under extremely adverse conditions, such as lubricating compositions for marine diesel engines, the compositions of this invention may be present in amounts of up to about 30% by weight, or more, of the total weight of the lubricating composition.

The invention also contemplates the use of other additives in combination with the compositions of this invention. Such additives include, for example, detergents and dispersants of the ash-producing or ashless type, corrosion- and oxidation-inhibiting agents, pour point depressing agents, extreme pressure agents, anti-wear agents, color stabilizers and anti-foam agents.

The ash-producing detergents are exemplified by oil-soluble neutral and basic salts of alkali or alkaline earth metals with sulfonic acids, carboxylic acids, or organic phosphorus acids characterized by at least one direct carbon-to-phosphorus linkage such as those prepared by the treatment of an olefin polymer (e.g., polyisobutene having a molecular weight of 1000) with a phosphorizing agent such as phosphorus trichloride, phosphorus heptasulfide, phosphorus pentasulfide, phosphorus trichloride and sulfur, white phosphorus and a sulfur halide, or phosphorothioic chloride. The most commonly used salts of such acids are those of sodium, potassium, lithium, calcium, magnesium, strontium and barium.

The term "basic salt" is used to designate metal salts wherein the metal is present in stoichiometrically larger amounts than the organic acid radical. The commonly employed methods for preparing the basic salts involve heating a mineral oil solution of an acid with a stoichiometric excess of a metal neutralizing agent such as the metal oxide, hydroxide, carbonate, bicarbonate, or sulfide at a temperature of about 50°C and filtering the resulting mass. The use of a "promoter" in the neutralization step to aid the incorporation of a large excess

of metal likewise is known. Examples of compounds useful as the promoter include phenolic substances such as phenol, naphthol, alkylphenol, thiophenol, sulfurized alkylphenol, and condensation products of formaldehyde with a phenolic substance; alcohols such as methanol, 2-propanol, octyl alcohol, cellosolve, carbitol, ethylene glycol, stearyl alcohol, and cyclohexyl alcohol; and amines such as aniline, phenylenediamine, phenothiazine, phenyl-beta-naphthylamine, and dodecylamine. A particularly effective method for preparing the basic salts comprises mixing an acid with an excess of a basic alkaline earth metal neutralizing agent and at least one alcohol promoter, and carbonating the mixture at an elevated temperature such as 60-200°C.

Ashless detergents and dispersants are so called despite the fact that, depending on its constitution, the dispersant may upon combustion yield a non-volatile material such as boric oxide or phosphorus pentoxide; however, it does not ordinarily contain metal and therefore does not yield a metal-containing ash on combustion. Many types are known in the art, and any of them are suitable for use in the lubricant compositions of this invention. The following are illustrative:

(1) Reaction products of carboxylic acids (or derivatives thereof) containing at least about 34 and preferably at least about 54 carbon atoms with nitrogen containing compounds such as amine, organic hydroxy compounds such as phenols and alcohols, and/or basic inorganic materials. Examples of these "carboxylic dispersants" are described in British Patent 1,306,529 and in many U.S. Patents including the following:

3,163,603	3,351,552	3,541,012
3,184,474	3,381,022	3,543,678
3,215,707	3,399,141	3,542,680
3,219,666	3,415,750	3,567,637
3,271,310	3,433,744	3,574,101
3,272,746	3,444,170	3,576,743
3,281,357	3,448,048	3,630,904
3,306,908	3,448,049	3,632,510
3,311,558	3,451,933	3,632,511
3,316,177	3,454,607	3,697,428
3,340,281	3,467,668	3,725,441
3,341,542	3,501,405	4,234,435
3,346,493	3,522,179	Re 26,433

(2) Reaction products of relatively high molecular weight aliphatic or alicyclic halides with amines, preferably polyalkylene polyamines. These may be characterized as "amine dispersants" and examples thereof are described for example, in the following U.S. Patents:

3,275,554	3,454,555
3,438,757	3,565,804

(3) Reaction products of alkyl phenols in which the alkyl group contains at least about 30 carbon atoms with aldehydes (especially formaldehyde) and amines (especially polyalkylene polyamines), which may be characterized as "Mannich dispersants". The materials described in the following U.S. Patents are illustrative:

	2,459,112	3,442,808	3,591,598
	2,962,442	3,448,047	3,600,372
5	2,984,550	3,454,497	3,634,515
	3,036,003	3,459,661	3,649,229
	3,166,516	3,461,172	3,697,574
	3,236,770	3,493,520	3,725,277
10	3,355,270	3,539,633	3,725,480
	3,368,972	3,558,743	3,726,882
	3,413,347	3,586,629	3,980,569

(4) Products obtained by post-treating the carboxylic, amine or Mannich dispersants with such reagents as urea, thiourea, carbon disulfide, aldehydes, ketones, carboxylic acids, hydrocarbon-substituted succinic anhydrides, nitriles, epoxides, boron compounds, phosphorus compounds or the like. Exemplary materials of this kind are described in the following U.S. Patents:

20	3,036,003	3,282,955	3,493,520	3,639,242
	3,087,936	3,312,619	3,502,677	3,649,229
25	3,200,107	3,366,569	3,513,093	3,649,659
	3,216,936	3,367,943	3,533,945	3,658,836
	3,254,025	3,373,111	3,539,633	3,697,574
	3,256,185	3,403,102	3,573,010	3,702,757
30	3,278,550	3,442,808	3,579,450	3,703,536
	3,280,234	3,455,831	3,591,598	3,704,308
	3,281,428	3,455,832	3,600,372	3,708,422

Preferably, the boron-containing compositions of this invention are diluted with a substantially inert, normally liquid organic diluent such as mineral oil, naphtha, benzene, toluene or xylene, to form an additive concentrate. These concentrates usually contain from about 10% to about 90% by weight of the boron-containing compositions of this invention and may contain, in addition, one or more other additives known in the art or described hereinabove. The remainder of the concentrate is the substantially inert normally liquid diluent.

The boron-containing compositions of the invention are useful particularly for improving the anti-wear properties of lubricants containing little or no phosphorus, especially lubricants containing less than 0.1% phosphorus especially as a phosphorodithioate. In such low phosphorus lubricants, it is preferred to use a boron-containing composition derived from a hydroxy-containing ester.

The boron-containing compositions of the invention also are useful as fuel additives. The fuel compositions of the present invention contain a major proportion of a normally liquid fuel, usually a hydrocarbonaceous petroleum distillate fuel such as motor gasoline as defined by ASTM Specification D439 and diesel fuel or fuel oil as defined by ASTM Specification D396. Normally liquid fuel compositions comprising non-hydrocarbonaceous materials such as alcohols, ethers, organo-nitro compounds and the like (e.g., methanol, ethanol, diethyl ether, methyl ethyl ether, nitromethane) are also within the scope of this invention as are liquid fuels derived from vegetable or mineral sources such as corn, alfalfa, shale and coal. Normally liquid fuels which are mixtures of one or more hydrocarbonaceous fuels and one or more non-hydrocarbonaceous materials are also contemplated. Examples of such mixtures are combinations of gasoline and ethanol and of diesel fuel and ether. Particularly preferred is gasoline, that is, a mixture of hydrocarbons having an ASTM distillation range from about 60°C at the 10% distillation point to about 205°C at the 90% distillation point.

Generally, these fuel compositions contain a property improving amount of the boron-containing compositions of this invention; usually this amount is about 1 to about 50,000 parts by weight, preferably about 4 to about 5000 parts, of the composition of this invention per million parts of fuel.

The fuel compositions can contain, in addition to the boron-containing composition of this invention, other additives which are well known to those of skill in the art. These include antiknock agents such as tetraalkyl lead compounds, lead scavengers such as haloalkanes (e.g., ethylene dichloride and ethylene dibromide), deposit preventers or modifiers such as triaryl phosphates, dyes, cetane improvers, antioxidants such as 2,6-ditertiary-butyl-4-methyl-phenol, rust inhibitors such as alkylated succinic acids and anhydrides, bacteriostatic agents, gum inhibitors, metal deactivators, demulsifiers, upper cylinder lubricants and anti-icing agents.

In certain preferred fuel compositions the boron compositions of this invention are combined with an ashless dispersant in gasoline. Suitable ashless dispersants include esters of mono- or polyols and high molecular weight mono- or polycarboxylic acid acylating agents containing at least 30 carbon atoms in the acyl moiety. Such esters are well known to those skilled in the art. See, for example, French Patent 1,396,645; British Patents 981,850; 1,055,337 and 1,306,529; and U.S. Patents 3,255,108; 3,311,558; 3,331,776; 3,346,354; 3,522,179; 3,579,750; 3,542,680; 3,381,022; 3,639,242; 3,697,428; and 3,708,522.

Generally, the weight ratio of the composition of this invention to the aforesaid ashless dispersant is between about 0.1:1 and about 10:1, preferably between about 1:1 and about 10:1.

The boron-containing compositions of this invention can be added directly to the fuel, or they can be diluted with a substantially inert, normally liquid organic diluent such as naphtha, benzene, toluene, xylene or a normally liquid fuel as described above, to form an additive concentrate. These concentrates generally contain from about 10% to about 90% by weight of the composition of this invention and may contain, in addition one or more other conventional additives known in the art or described hereinabove.

Claims

1. A method of preparing a boron-containing composition which comprises reacting
(A) a hydroxy-substituted amide of the formula



wherein R is a polyvalent hydrocarbyl group, R' is a hydrocarbyl group and R'' is hydrogen or a hydrocarbyl group, m is 1 or 2, and n is an integer from 1 to 10 provided that only one free hydroxyl group is attached per carbon atom of the hydrocarbyl group and that when m is 1, n is 1; with

- (B) a boron compound selected from boric acid, boron trioxide, boron halides, boron amides and boron esters and wherein (A) is reacted with (B) at an elevated temperature up to but not including the decomposition temperature of any reactants or the product of the reaction and wherein the molar ratio of the hydroxyl group to the boron compound is from about 0.5:1 to about 4:1.

2. A method of claim 1 wherein R' and R'' are each independently aliphatic hydrocarbyl groups containing from 4 to about 30 carbon atoms.

3. A method of claim 1 or claim 2 wherein the hydrocarbyl group R is an alkylene, alkenylene, alkynylene, arylene or alkarylene group containing from 1 to about 10 carbon atoms.

4. A method of any preceding claim wherein (B) is boric acid.

5. A method of any preceding claim wherein the reaction is conducted at a temperature within the range of from about 80 to about 200°C.

6. A method of any preceding claim wherein the hydroxy-substituted amide (A) is prepared by the reaction of

- (A-1) a hydroxy-substituted carboxylic acid of the formula



- or an anhydride thereof; with

- (A-2) at least one primary or secondary hydrocarbyl amine of the general formula



wherein m, n, R, R' and R'' are as defined in claim 1.

7. A method of claim 6 wherein R contains from 1 to about 6 carbon atoms, and n is an integer from 1 to about 5.

8. A method of claim 6 or claim 7 where (A-1) and (A-2) are heated at an elevated temperature up to about the decomposition point of the reactants or the product mixture and wherein about equivalent amounts of (A-1) and (A-2) are reacted.

9. A method of preparing boron-containing compositions which comprises reacting

- (A) an amide of a hydroxy-substituted carboxylic having the formula



wherein R is a polyvalent aliphatic group containing from 1 to 6 carbon atoms, R' is an aliphatic group containing from about 6 to about 30 carbon atoms, R'' is hydrogen or is as defined for R', m is 1 or 2, and n is

The fuel compositions can contain, in addition to the boron-containing composition of this invention, other additives which are well known to those of skill in the art. These include antiknock agents such as tetraalkyl lead compounds, lead scavengers such as haloalkanes (e.g., ethylene dichloride and ethylene dibromide), deposit preventers or modifiers such as triaryl phosphates, dyes, cetane improvers, antioxidants such as 2,6-ditertiary-butyl-4-methyl-phenol, rust inhibitors such as alkylated succinic acids and anhydrides, bacteriostatic agents, gum inhibitors, metal deactivators, demulsifiers, upper cylinder lubricants and anti-icing agents.

In certain preferred fuel compositions the boron compositions of this invention are combined with an ashless dispersant in gasoline. Suitable ashless dispersants include esters of mono- or polyols and high molecular weight mono- or polycarboxylic acid acylating agents containing at least 30 carbon atoms in the acyl moiety. Such esters are well known to those skilled in the art. See, for example, French Patent 1,396,645; British Patents 981,850; 1,055,337 and 1,306,529; and U.S. Patents 3,255,108; 3,311,558; 3,331,776; 3,346,354; 3,522,179; 3,579,750; 3,542,680; 3,381,022; 3,639,242; 3,697,428; and 3,708,522.

Generally, the weight ratio of the composition of this invention to the aforesaid ashless dispersant is between about 0.1:1 and about 10:1, preferably between about 1:1 and about 10:1.

The boron-containing compositions of this invention can be added directly to the fuel, or they can be diluted with a substantially inert, normally liquid organic diluent such as naphtha, benzene, toluene, xylene or a normally liquid fuel as described above, to form an additive concentrate. These concentrates generally contain from about 10% to about 90% by weight of the composition of this invention and may contain, in addition one or more other conventional additives known in the art or described hereinabove.

Claims

1. A method of preparing a boron-containing composition which comprises reacting
(A) a hydroxy-substituted amide of the formula



wherein R is a polyvalent hydrocarbyl group, R' is a hydrocarbyl group and R'' is hydrogen or a hydrocarbyl group, m is 1 or 2, and n is an integer from 1 to 10 provided that only one free hydroxyl group is attached per carbon atom of the hydrocarbyl group and that when m is 1, n is 1; with

(B) a boron compound selected from boric acid, boron trioxide, boron halides, boron amides and boron esters and wherein (A) is reacted with (B) at an elevated temperature up to but not including the decomposition temperature of any reactants or the product of the reaction and wherein the molar ratio of the hydroxyl group to the boron compound is from about 0.5:1 to about 4:1.

2. A method of claim 1 wherein R' and R'' are each independently aliphatic hydrocarbyl groups containing from 4 to about 30 carbon atoms.

3. A method of claim 1 or claim 2 wherein the hydrocarbyl group R is an alkylene, alkenylene, alkynylene, arylene or alkarylene group containing from 1 to about 10 carbon atoms.

4. A method of any preceding claim wherein (B) is boric acid.

5. A method of any preceding claim wherein the reaction is conducted at a temperature within the range of from about 80 to about 200°C.

6. A method of any preceding claim wherein the hydroxy-substituted amide (A) is prepared by the reaction of

(A-1) a hydroxy-substituted carboxylic acid of the formula



or an anhydride thereof; with

(A-2) at least one primary or secondary hydrocarbyl amine of the general formula



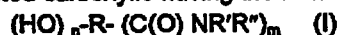
wherein m, n, R, R' and R'' are as defined in claim 1.

7. A method of claim 6 wherein R contains from 1 to about 6 carbon atoms, and n is an integer from 1 to about 5.

8. A method of claim 6 or claim 7 where (A-1) and (A-2) are heated at an elevated temperature up to about the decomposition point of the reactants or the product mixture and wherein about equivalent amounts of (A-1) and (A-2) are reacted.

9. A method of preparing boron-containing compositions which comprises reacting

(A) an amide of a hydroxy-substituted carboxylic having the formula



wherein R is a polyvalent aliphatic group containing from 1 to 6 carbon atoms, R' is an aliphatic group containing from about 6 to about 30 carbon atoms, R'' is hydrogen or is as defined for R', m is 1 or 2, and n is



an integer from 1 to about 5 provided that when m is 1, n is 1; with
(B) a boron compound selected from boric acid, boron oxide, boron halides, boron amides and boron esters
in a ratio of hydroxyl group to boron of from about 0.5:1 to about 3:1 wherein (A) is reacted with (B) at an
elevated temperature up to but not including the decomposition temperature of any reactants or product
thereof.

10. A method of claim 9 wherein (A) is derived from the reaction of
(A-1) a hydroxy-substituted carboxylic acid



or an anhydride thereof; with

(A-2) at least one primary or secondary hydrocarbyl amine of the general formula



wherein m, n, R, R' and R'' are as defined in claim 9.

11. A method of claim 9 or claim 10 wherein (A) is an amide of hydroxyacetic acid or lactic acid and (B) is boric acid.

12. An additive concentrate comprising a substantially inert, normally liquid organic solvent/diluent and about 10-90% by weight of a boron-containing composition prepared by a method of any one of claims 1 to 11.

13. A lubricating composition comprising a major amount of a lubricating oil and a minor amount of at least one boron-containing composition prepared by a method of any one of claims 1 to 11.

Patentansprüche

1. Verfahren zur Herstellung einer Bor enthaltenden Zusammensetzung, umfassend die Umsetzung
(A) eines hydroxysubstituierten Amides der Formel



in der R einen mehrwertigen Kohlenwasserstoffrest bedeutet, R' einen Kohlenwasserstoffrest darstellt und R'' ein Wasserstoffatom oder einen Kohlenwasserstoffrest bedeutet, m 1 oder 2 ist und n eine ganze Zahl von 1 bis 10 ist, mit der Maßgabe, daß nur eine freie Hydroxylgruppe pro Kohlenstoffatom des Kohlenwasserstoffrestes gebunden ist und daß n 1 ist, wenn m 1 ist; mit

(B) einer Borverbindung, ausgewählt aus Borsäure, Bortrioxid, Borhalogeniden, Boramiden und Borestern, und wobei (A) mit (B) bei einer erhöhten Temperatur bis zu, jedoch nicht einschließlich der Zersetzungstemperatur eines der Umsetzungsteilnehmer oder des Produktes der Umsetzung umgesetzt wird, und wobei das Molverhältnis der Hydroxylgruppe zur Borverbindung etwa 0,5:1 bis etwa 4:1 beträgt.

2. Verfahren nach Anspruch 1, wobei R' und R'' jeweils unabhängig aliphatische Kohlenwasserstoffreste mit 4 bis etwa 30 Kohlenstoffatomen bedeuten.

3. Verfahren nach Anspruch 1 oder Anspruch 2, wobei der Kohlenwasserstoffrest R ein Alkyl-, Alkenyl-, Alkynyl-, Aryl- oder Alkarylrest mit 1 bis etwa 10 Kohlenstoffatomen ist.

4. Verfahren nach einem der vorangehenden Ansprüche, wobei (B) Borsäure ist.

5. Verfahren nach einem der vorangehenden Ansprüche, wobei die Umsetzung bei einer Temperatur im Bereich von etwa 80 bis etwa 200°C durchgeführt wird.

6. Verfahren nach einem der vorangehenden Ansprüche, wobei das hydroxysubstituierte Amid (A) hergestellt wird durch die Umsetzung von

(A-1) einer hydroxysubstituierten Carbonsäure der Formel



oder einem Anhydrid davon, mit

(A-2) mindestens einem primären oder sekundären Kohlenwasserstoffamin der allgemeinen Formel



wobei m, n, R, R' und R'' wie in Anspruch 1 definiert sind.

7. Verfahren nach Anspruch 6, wobei R 1 bis etwa 6 Kohlenstoffatome enthält und n eine ganze Zahl von 1 bis etwa 5 ist.

8. Verfahren nach Anspruch 6 oder Anspruch 7, wobei (A-1) und (A-2) auf eine erhöhte Temperatur bis etwa dem Zersetzungspunkt der Umsetzungsteilnehmer oder des Produktgemisches erhitzt wird und wobei etwa äquivalente Mengen von (A-1) und (A-2) umgesetzt werden.

9. Verfahren zur Herstellung von borhaltigen Zusammensetzungen, umfassend die Umsetzung

(A) eines Amids einer hydroxysubstituierten Carbonsäure der Formel



in der R einen mehrwertigen aliphatischen Rest mit 1 bis 6 Kohlenstoffatomen bedeutet, R' einen aliphatischen Rest mit etwa 6 bis etwa 30 Kohlenstoffatomen darstellt, R'' ein Wasserstoffatom bedeutet oder



wie R' défini est, m 1 ou 2 est et n un nombre entier de 1 à 5, avec la condition, que si n est 1, si m est 1, avec

(B) d'un composé de bore, choisi parmi l'acide borique, le trioxyde de bore, les halogénures de bore, les amides de bore et les esters de bore et où (A) est mis à réagir avec (B) à une température élevée pouvant aller, limite exclue, jusqu'à la température de décomposition de l'un quelconque des corps en réaction ou du produit de la réaction, et où le rapport molaire du groupe hydroxyle au composé du bore est compris entre environ 0,5:1 et environ 4:1.

10. Procédé selon la revendication 9, dans lequel (A) est dérivé de la réaction de



ou d'un anhydride d'un tel acide avec

(A-2) au moins un hydrocarbyle amine primaire ou secondaire de la formule générale



où m, n, R, R' et R'' sont tels que définis dans la revendication 1.

11. Procédé selon la revendication 9 ou la revendication 10, dans lequel (A) est un amide d'acide hydroxyacétique ou d'acide lactique et (B) l'acide borique.

12. Concentrat additionnel, comprenant un inerte essentiellement, normalement liquide organique, solvant/diluant et environ 10 à 90 p.-% d'une composition borée, préparé selon une des revendications 1 à 11.

13. Lubrifiant, comprenant une plus grande quantité d'huile lubrifiante et une plus petite quantité au moins d'une composition borée, préparé selon une des revendications 1 à 11.

Revendications

1. Un procédé de préparation d'une composition contenant du bore, qui comprend la réaction de

(a) un amide hydroxylé de la formule



où R est un groupe hydrocarbyle polyvalent, R' est un groupe hydrocarbyle et R'' est de l'hydrogène ou un groupe hydrocarbyle, m est 1 ou 2 et n est un nombre entier de 1 à 10, avec les conditions qu'il y a un seul groupe hydroxyle libre attaché par atome de carbone du groupe hydrocarbyle et que quand m est 1, alors n est 1 ; avec

(B) un composé du bore choisi parmi l'acide borique, le trioxyde de bore, les halogénures de bore, les amides de bore et les esters de bore et où (A) est mis à réagir avec (B) à une température élevée pouvant aller, limite exclue, jusqu'à la température de décomposition de l'un quelconque des corps en réaction ou du produit de la réaction, et où le rapport molaire du groupe hydroxyle au composé du bore est compris entre environ 0,5:1 et environ 4:1.

2. Un procédé selon la revendication 1, dans lequel R' et R'' sont chacun indépendamment des groupes hydrocarbyle aliphatiques contenant de 4 à environ 30 atomes de carbone.

3. Un procédé selon la revendication 1 ou la revendication 2, dans lequel le groupe hydrocarbyle R est un groupe alcoyle, alcényle, alcynyle, aryle ou alcaryle contenant de 1 à environ 10 atomes de carbone.

4. Un procédé selon l'une quelconque des revendications précédentes, dans lequel (B) est de l'acide borique.

5. Un procédé selon l'une quelconque des revendications précédentes, dans lequel la réaction est conduite à une température comprise entre environ 80 et environ 200°C.

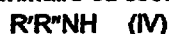
6. Un procédé selon l'une quelconque des revendications précédentes, dans lequel l'amide hydroxylé (A) est préparé par la réaction de

(A-1) un acide carboxylique hydroxylé de la formule



ou un anhydride d'un tel acide avec

(A-2) au moins un hydrocarbyle amine primaire ou secondaire de la formule générale



où m, n, R, R' et R'' sont tels que définis dans la revendication 1.

7. Un procédé selon la revendication 6, dans lequel R contient de 1 à environ 6 atomes de carbone et n est un nombre entier de 1 à environ 5.

8. Un procédé selon la revendication 6 ou la revendication 7, dans lequel (A-1) et (A-2) sont chauffés à une température élevée pouvant aller jusqu'à environ le point de décomposition des corps en réaction ou du mélange de produits et où on fait réagir des quantités à peu près équivalentes de (A-1) et de (A-2).



9. Un procédé de préparation de compositions contenant du bore, qui comprend la réaction de (A) un amide d'un acide carboxylique hydroxylé ayant la formule



où R est un groupe aliphatique polyvalent contenant de 1 à 6 atomes de carbone, R' est un groupe aliphatique contenant d'environ 6 à environ 30 atomes de carbone, R'' est de l'hydrogène ou est tel que défini pour R', m est 1 ou 2 et n est un nombre entier de 1 à environ 5, avec la condition que quand m est 1, alors n est 1 ; avec

(B) un composé du bore choisi parmi l'acide borique, l'oxyde de bore, les halogénures de bore, les amidures de bore et les esters de bore, avec un rapport du groupe hydroxyle au bore compris entre environ 0,5:1 et environ 3;1, où (A) est mis à réagir avec (B) à une température élevée, pouvant aller, limite exclue, jusqu'à la température de décomposition de l'un quelconque des corps en réaction ou du produit.

10. Un procédé selon la revendication 9, dans lequel (A) est dérivé de la réaction de (A-1) un acide carboxylique hydroxylé



ou un anhydride d'un tel acide avec

(A-2) au moins une hydrocarbyle amine primaire ou secondaire de la formule générale $R'R''NH$ (VIII)

où m, n, R, R' et R'' sont tels que définis dans la revendication 9.

11. Un procédé selon la revendication 9 ou la revendication 10, dans lequel (A) est un amide de l'acide hydroxyacétique ou de l'acide lactique et (B) est l'acide borique.

12. Un concentré d'additif comprenant un solvant/diluant organique normalement liquide substantiellement inerte et environ 10-90 % en poids d'une composition contenant du bore préparée par un procédé selon l'une quelconque des revendications 1 à 11.

13. Une composition lubrifiante comprenant une quantité majeure d'une huile lubrifiante et une quantité mineure d'au moins une composition contenant du bore préparée par un procédé selon l'une quelconque des revendications 1 à 11.

