

[54] LUBRICANT CONTAINING NEUTRALIZED
ALKALI METAL BORATES

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[58] Field of Search 252/18, 25, 32.5, 33.4,
252/49.6, 49.7, 49.8, 49.9

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[57]

ABSTRACT

An extreme pressure lubricating composition having excellent water tolerance properties and excellent compatibility with other lubricating oil additives comprises (A) an oil of lubricating viscosity, (B) an alkali metal borate at least partially neutralized with the acidic anion of phosphoric acid or sulfuric acid, and (C) a mixture of lipophilic dispersants.

4 Claims, No Drawings

LUBRICANT CONTAINING NEUTRALIZED ALKALI METAL BORATES

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention concerns extreme pressure (EP) lubricants.

High load conditions often occur in gear sets such as those used in automotive transmissions and differentials, pneumatic tools, gas compressors, centrifuges, high pressure hydraulic systems, metal working and similar devices as well as in many types of bearings. Lubricants containing extreme pressure agents are used in these types of services in order to minimize wear. For the most part, EP agents have been organic or metallo-organic compounds which are oil soluble or easily incorporated as a stable dispersion in oil. Most of the prior art EP agents are chemically reactive: they contain chlorine, sulfur, or phosphorus. They provide a protective coating by reacting with the metal surfaces of the gears or bearings at the high temperatures produced under extreme pressure loading.

Recently, Peeler, U.S. Pat. No. 3,313,727, disclosed an EP lubricant produced by dispersing an hydrated alkali metal borate in a nonpolar lubricating oil. The borate, water, and an emulsifier were introduced into the nonpolar medium. The mixture was then agitated to disperse the aqueous solution in the oil and heated to dehydrate the alkali metal borate. Peeler also disclosed that conventional additives such as rust inhibitors, detergents, foam inhibitors, etc., could be present in the finished lubricating composition containing the borate.

The borate-containing oils described by Peeler have, however, a very serious deficiency in service. If the lubricant comes into contact with water, the borate crystallizes out of oil and forms hard granules. These granules cause severe noise in the lubricated system and can severely damage the gears or bearings themselves. Further, borate lost by crystallization decreases the EP function of the lubricant.

2. Description of the Prior Art

The Peeler patent is described above. U.S. Pat. No. 2,987,476 describes dispersing an "inorganic boric acid compound" in a substantially nonpolar organic liquid by mixing an organic liquid, a lyophilic surface active agent, a water-miscible organic liquid, and an organic ester of boric acid. A metal base is then added to the mixture to hydrolyze the organic ester. The water-miscible liquid (which may be a monohydric alcohol) is removed after formation of the dispersed inorganic boric acid compound. U.S. Pat. Nos. 2,753,305 and 3,338,835 describe aqueous solutions containing polyhydric alcohols and metal borates. U.S. Pat. Nos. 2,780,597 and 3,000,819 describe lubricants containing minor amounts of inorganic phosphates. U.S. Pat. No. 3,313,729 discloses a soap base lubricant containing an alkali metal pyrophosphate and tetraborate. Gear lubrication is discussed in Guthrie, *Petroleum Products Handbook*, 1st Ed., McGraw-Hill Book Co. (1960), pp. 9-47 through 9-49, and in Boner, *Gear and Transmission Lubricants*, Reinhold Publ. Corp. (1964).

SUMMARY OF THE INVENTION

I have now invented a novel lubricant composition having superior EP, water tolerance, and compatibility properties comprising (A) an oil of lubricating viscos-

ity, (B) an alkali metal borate at least partially neutralized with the acidic anion of phosphoric acid or sulfuric acid, and (C) a mixture of lipophilic dispersants.

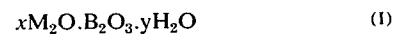
These compositions are highly stable EP lubricants. They perform well in EP tests, such as the four-ball test. They are useful in a number of gear and bearing lubrication applications, particularly in automotive differentials. In contrast to most other EP lubricants, they are, in most cases, essentially noncorrosive to the metal surfaces of the gears. Many of the lubricants are liquids, while others have a soft and pliable consistency. Further, many are also transparent, a property which is highly advantageous where visual appearance is important.

DETAILED DESCRIPTION OF THE INVENTION

The compositions of this invention are lubricants having improved extreme pressure, water tolerance, and compatibility properties comprising (A) amorphous particles of less than 1 micron in size of a hydrated alkali metal borate at least partially neutralized with the acidic anion of phosphoric acid or sulfuric acid, (B) a mixture of lipophilic dispersants and (C) a nonpolar oil of lubricating viscosity.

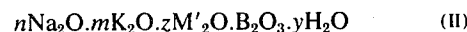
25 The Hydrated Borate

The hydrated borates of this lubricant composition are hydrated alkali metal borates of the formula:



wherein x represents a number from 0.75-3 and y represents a number up to 5, usually from 0.5 to 4 and preferably 1.5 to 3, and M represents one or a mixture of alkali metals, usually potassium or sodium. Preferably x will represent a number of from 0.8 to 1.2 and y will represent a number of 2 times x . Where only a single alkali metal is present, the compounds of Formula I will include sodium metaborate, sodium diborate, potassium metaborate, potassium diborate, and similar materials.

Where M represents two or more alkali metals, the formula above in fact signifies a mixture of alkali metal borates. These mixtures can be represented by the following formula:



wherein n represents a number of from 0.3 to 2.7, m represents a number from 0.3 to 2.7, and z represents a number of from 0 to 1.0 such that the sum of n , m and z represents a number of from 0.75 to 3, M' represents an alkali metal other than sodium or potassium, such as lithium, rubidium, cesium, and the like, and y is as defined above. Preferably z represents 0; i.e., that the mixture of alkali metal borates contain only sodium and potassium as the alkali metal components.

Formulae I and II are meant to be empirical and not structural. The exact structure in which the borates exist in the composition is unknown and varies with different amounts of water of hydration, the presence of different alkali metals and the ratio of the alkali metals to the boron-containing materials. Throughout the specification and the claims, all numerical values for quantities related to the borates are based on these empirical formulae. Where a mixture of borates is in-

volved, numerical values for the quantities of other materials refer to the entire borate mixture.

The borate is dispersed as particles throughout the lubricating oil by means of an emulsifying agent or dispersant as described below. The borate particles are glass-like and are essentially entirely all less than 1 micron in diameter and for the most part less than one-half micron in diameter.

The hydrated alkali metal borate described above is at least partially neutralized to make it compatible with other lubricating oil additives such as dithiophosphates, sulfurized fats and the like. Neutralization causes some loss of extreme pressure activity of the borate. However, this loss of EP activity is rather small and is acceptable if the borate is neutralized with the acid anion of sulfuric or phosphoric acid.

The acid anion can be added in any form in which the anion is not completely neutralized. For example, the anion can be added in the form of the acid or monohydric (or dihydric in the case of phosphoric acid) salts of alkali or alkaline earth metals. Preferably the acid or alkaline earth metal monobasic salts are used. Most preferably, the acid is used. Less of it is required than of the mono- and di-basic salts.

The completely neutralized salts of strong bases offer almost no advantage since they are already substantially neutral and incapable of neutralizing the borate. However, the completely neutralized salts of weak bases such as ammonia can be used.

The quantity of acid anion used can vary widely depending upon its form. Preferably, however, sufficient anion is used to bring the pH of an aqueous solution of the neutralized borate into the range of 6 to 8, preferably 6.5 to 7.5.

The proper pH is easily achieved because of the method of preparing the lubricating compositions of this invention. The desired amount of borate salt is dissolved in water and the acid anion is added until the desired pH is achieved. Alternatively, a solution of one or a mixture of alkali metal hydroxides can be neutralized with boric acid until the proper alkali metal to boron ratio is obtained. This mixture is then neutralized with the acid anion to the desired pH.

The neutralized borate will be present in from 1 to 15 weightpercent, usually 2 to 10 weight percent, and preferably 3 to 7 weight percent of the total composition.

The Dispersant Mixture

The borate and acid anion salt are dispersed in the lubricating oil medium by a mixture of lipophilic dispersants. In general, any lipophilic dispersants which are compatible with the borate and acid anion salt may be used. However, certain dispersant mixtures are preferred because they provide a high degree of water tolerance to the lubricating oil composition, i.e., these mixtures permit the present compositions to function in the presence of water without crystallization of the borate and/or acid anion salt.

These preferred dispersant mixtures contain two components: a lipophilic anionic surface-active agent (dispersant) and a lipophilic nonionic surface-active agent (dispersant). (The expression "lipophilic" as employed herein is synonymous with "hydrophobic," which means a compound having a greater affinity for fats, oil and the like than water and which is readily soluble in organic liquids having electric dipole moments of 0.5 Debye unit or less.) The concentrations of the

anionic dispersant and the nonionic dispersant in the mixture will be in the range of 10-99 weight percent anionic dispersant and 90-1 weight percent nonionic dispersant. Preferably, there will be 25-75 weight percent anionic dispersant and 75-25 weight percent nonionic dispersant and more preferably there will be 59-95 weight percent anionic dispersant and 50-5 weight percent nonionic dispersant.

The Anionic Dispersant

The lipophilic anionic surface-active agents are Group I and Group II metal-containing dispersants. These materials are well known in the art. The dispersants are salts of Group I and Group II metals in which the anionic portion of the salt contains an oil-solubilizing group. They can be used alone or as mixtures.

The oil-solubilizing group generally has at least 9 and usually 12-18 or more carbon atoms, preferably from about 12 to 200 carbon atoms. The oil-solubilizing groups are usually, but not necessarily, hydrocarbyl groups. Hydrocarbyl groups are organic radicals composed of carbon and hydrogen except for minor, sometimes adventitious, amounts of other elements, such as oxygen, chlorine, etc. The term denotes an aliphatic or aromatic radical, or a radical which is a combination thereof, e.g., aralkyl. Preferably, the hydrocarbyl group is relatively free of aliphatic unsaturation, i.e., ethyleneic and acetylenic, particularly acetylenic. When the hydrocarbyl groups are of low molecular weight, the average number of hydrocarbyl substituents per dispersant molecule may be greater than 1. The hydrocarbyl groups are preferably aliphatic, having preferably from 0-2 sites of ethyleneic saturation and most preferably 0-1 site. Hydrocarbyl groups derived from a polyolefin, itself derived from olefins (normally 1-olefins) having from 2-6 carbon atoms (ethylene being copolymerized with an olefin of at least 3 carbon atoms), or from a high molecular weight petroleum-derived hydrocarbon, are preferred, and of these, polyisobutylene is most preferred.

Illustrative sources for the high molecular weight hydrocarbyl substituents are petroleum mineral oil such as naphthenic bright stocks, polypropylene, polyisobutylene, poly-1-butene, copolymers of ethylene and isobutylene, polypropylene and isobutylene, poly-1-pentene, poly-4-methyl-1-pentene, poly-1-hexene, and poly-3-methylbutylene-1, etc.

The acid reacting functional group can be a variety of well-known groups which include the sulfonic acid group, the phenolic group and the carboxylic acid group. These groups, when in the form of the metal salts, are known as sulfonates, phenates and carboxylates, respectively.

The term "sulfonates" is intended to encompass the salts of sulfonic acids derived from petroleum products. Such acids are well known in the art. They can be obtained by treating petroleum products with sulfuric acid or sulfur trioxide. The acids thus obtained are known as petroleum sulfonic acids and the salts as petroleum sulfonates. Most of the compounds in the petroleum product which become sulfonated contain an oil-solubilizing group as discussed above. Also included within the meaning of sulfonates are the salts of sulfonic acids of synthetic alkyl aryl compounds. These acids also are prepared by treating an alkyl aryl compound with sulfuric acid or sulfur trioxide. At least one alkyl substituent of the aryl ring is an oil-solubilizing

group as discussed above. The acids thus obtained are known as alkyl aryl sulfonic acids and the salts as alkyl aryl sulfonates. The sulfonates wherein the alkyl is straight-chain are the well-known linear alkyl sulfonates (LAS).

The acids obtained by sulfonation are converted to the metal salts by neutralizing with a basic reacting alkali or alkaline earth metal compound to yield the Group I or Group II metal sulfonates. Generally, the acids are neutralized with an alkali metal base. Alkaline earth metal salts are obtained from the alkali metal salt by metathesis. Alternatively, the sulfonic acid can be neutralized directly with an alkaline earth metal base.

The sulfonates can then be overbased although for purposes of this invention, overbasing is not necessary. Overbased materials and methods of preparing such materials are well known to those skilled in the art. See, for example, LeSeur, U.S. Pat. No. 3,496,105, issued Feb. 17, 1970, particularly at cols. 3 and 4.

The term "carboxylates" encompasses the salts of carboxylic acids. Acids which are useful in the instant invention generally contain at least 12 and generally 15 or more carbon atoms, part of which are contained in the oil-solubilizing group discussed above for the sulfonates. Examples include palmitic, stearic, myristic, oleic, linoleic, etc., acids. Other carboxylic acids include the cyclic acids. Among these are acids containing an aryl group, i.e., benzene, naphthalene, etc., substituted with an oil-solubilizing radical or radicals having a total of at least 15-18 carbon atoms or more, e.g., alkyl benzoic acid, alkyl naphthoic acid, alkyl salicylic acid, etc. Preferred are the cyclic acids which contain a cycloaliphatic group substituted with an oil-solubilizing group. Examples of such acids are dilauryl decahydronaphthalene carboxylic acid, the petroleum naphthenic acids, e.g., alkyl cyclohexane carboxylic acid, etc. The petroleum naphthenic acids are preferred. The salts of this preferred class of cycloaliphatic acids are commonly known as naphthenates.

The term "phenates" encompasses the salts of oil-soluble phenols. The phenols contain at least 12 and generally 18 or more carbon atoms, part of which is contained in the oil-solubilizing group discussed above for the sulfonates. These phenols can be obtained by alkylating phenol, e.g., reacting phenol with an olefin such as tetrapropylene. The alkylphenols can be further reacted such as by the "Mannich" reaction with formaldehyde and an amine, preferably a monoamine to yield higher molecular weight complex compounds. A wide variety of such phenols are available and well known.

The sulfonates, phenates and carboxylates are present in the lubricating oil composition in the form of their Group I and Group II metal salts. Group I metals include lithium, sodium and potassium. Group II metals include strontium, magnesium, calcium and barium, of which the latter three are preferred.

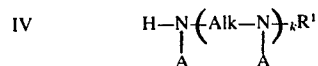
The Nonionic Dispersant

The lipophilic nonionic surface-active agents include those generally referred to as "ashless detergents." Preferably, the nonionic surfactants have a HLB factor (hydrophilic-lipophilic balance) below about 7 and preferably below about 5. These ashless detergents are well known and include hydrocarbyl-substituted amines, amides and cyclo-imides. The hydrocarbyl group or groups act as the oil-solubilizing group as discussed above.

The hydrocarbyl-substituted amines are derived from ammonia, monoamines, and polyamines. An example of amines include ethylamine, butylamine, piperazine, diethylene triamine, trimethylene diamine, di(trimethylene) triamine, dipropylene triamine, tripropylene tetramine, triethylene tetramine, tetraethylene pentamine, pentaethylene hexamine, etc. These amines encompass alkyl-substituted amines, e.g., N-methyl ethylene diamine, N,N'-dimethyl ethylene diamine, N,N-dimethyl propylene diamine, N-hydroxy-ethyl ethylene diamine, etc. Amines having up to about 12 amino nitrogens are especially preferred. The hydrocarbyl-substituted amines are prepared, in general, by reaction of a halogen-substituted hydrocarbon with the amine. Details of such preparations and further descriptions of some of these hydrocarbyl-substituted amines can be found in Honnen and Anderson, U.S. Pat. Nos. 3,565,804 and 3,438,757.

The hydrocarbyl-substituted amides and cyclic imides are derived from the reaction of hydrocarbyl-substituted carboxylic acids, anhydrides, acid chlorides, etc., with certain of the amines described above. A preferred dispersant is the reaction product of hydrocarbyl-substituted succinic acid or anhydride with amines containing at least one primary or secondary amino nitrogen. The polyalkylene polyamines fulfill this requirement as do the substituted polyalkylene polyamines and, for that matter, ammonia.

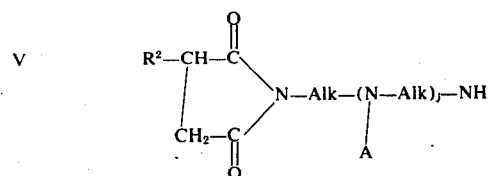
The alkylene polyamines have the formula:



wherein k is an integer of from 1 to 10, preferably 1 to 6, A and R^1 each represent hydrogen or a substantially hydrocarbon radical and Alk represents an alkylene radical having less than 8 carbon atoms.

Of the compounds represented by Formula IV, the ethylene amines are especially useful. Particularly useful are those ethylene amines of Formula IV wherein A and R^1 represent hydrogen, Alk represents ethylene and k represents an integer of from 3 to 5. The ethylene amines are described in some detail under the heading "Ethylene Amines" in *Encyclopedia of Chemical Technology*, Kirk-Othmer, Interscience Publishers, New York, Vol. 5 (1950), pp. 898-905.

An important class of the hydrocarbyl-substituted cyclic imides are the N-substituted alkyl succinimides derived from alkyl succinic acid or anhydride and the alkylene polyamines described above. These compounds are generally considered to have the formula:



wherein R^2 is a hydrocarbon radical having a molecular weight from about 400 to about 3,000 (that is, it contains about 30 to about 200 carbon atoms), Alk is alkylene radical of from 2 to 10, preferably 2 to 6 carbon atoms, and most preferably 2 to 3 carbon atoms, A is

as described above and j is a number of from 0 to 9, preferably 0 to 5, and more preferably 2 to 3. The reaction product of the alkyl succinic acid or anhydride and the alkylene polyamine will be a mixture of compounds, including succinamic acids and succinimides. However, it is customary to designate this reaction product as a "succinimide" corresponding to Formula V, since that is the principal component of the reaction mixture. The preparation and a more detailed discussion of succinimides is found in U.S. Pat. Nos. 3,202,678; 3,024,237; and 3,172,892.

These N-substituted alkyl succinimides can be prepared by reacting maleic anhydride with an olefinic hydrocarbon. The resulting alkyl succinic anhydride is reacted with the alkylene polyamine.

The radical R^2 of the above formula is derived from an olefin containing from 2 to 5 carbon atoms by polymerization to form a hydrocarbon having a molecular weight ranging from about 400 to about 3,000. Suitable olefins include ethylene, propylene, 1-butene, 2-butene, isobutene and mixtures thereof. The methods of polymerizing the olefins are well known to those skilled in the art.

The bis-succinimides also find use in this invention. The bis-succinimides are prepared by reacting hydrocarbyl substituted succinic acids or anhydrides with an amine containing at least two primary and/or secondary nitrogens. About 2 moles of the acid or anhydride are used per mole of amine. Exemplary bis-succinimides include the polyisobutenyl bis-succinimides of ethylene diamine, diethylene triamine, triethylene tetramine, tetraethylene pentamine, or N-methyl dipropylene triamine, etc. See for example Benoit, U.S. Pat. No. 3,438,899.

Another group of nonionic dispersants are the pentaerythritol derivatives. Particular derivatives which find use in this invention include those in which pentaerythritol is combined with a polyolefin and maleic anhydride or with a polyolefin and a phosphorus sulfide. The polyolefins are the polymers of monomeric olefins having 2-6 carbon atoms, such as polyethylene, polypropylene, polybutene, polyisobutylene, and the like. Such olefins generally contain a total of 20-250 carbon atoms and preferably 30-150 carbon atoms. The phosphorus sulfides include P_2S_3 , P_2S_5 , P_4S_7 , P_4S_9 , and related materials. Of these, P_2S_5 (phosphorus pentasulfide) is preferred principally because of its availability.

Other nonionic emulsifiers which may be used include polymethacrylates and copolymers of polymethacrylate or polyacrylate with vinyl pyrrolidone, acrylamide or methacrylamide.

The dispersants or mixtures thereof of the lipophilic surface-active agents will generally be present in from 0.25-5 weight percent, more usually from about 0.5-3 weight percent of the total composition. The amount of dispersant required will vary with the particular mixture used and the total amount of borate in the oil. Generally about 0.05 to about 0.5, more usually about 0.1 to 0.3 part by weight of the dispersant or mixtures thereof will be used per part by weight of the borate. In concentrates, the concentration of the dispersant or mixtures thereof will be based on the quantity of borate contained in the concentrate rather than as a fixed percentage of the concentrate. Generally, the upper ranges of the dispersant concentration will be used with the upper ranges of the borate concentration.

The Lubricating Oil Medium

The nonpolar lubricating oil can be any fluid of lubricating viscosity which is inert under the conditions necessary to disperse the borate and the acid anion salt in the fluid. Particularly, the oil should be nonsaponifiable. Fluids of lubricating viscosity generally have viscosities from 35 to 50,000 Saybolt Universal Seconds (SUS) at 100°F. The fluid can be derived from either natural or synthetic sources. Included among the natural hydrocarbonaceous oils are paraffin-base, naphthenic-base, or mixed base oils. Synthetic oils include polymers of various olefins, generally of from 2 to 6 carbon atoms, alkylated aromatic hydrocarbons, etc. Nonhydrocarbon oils include polyalkylene oxides, e.g., polyethylene oxide, aromatic ethers, silicones, etc. The preferred media are the hydrocarbonaceous media, both natural and synthetic. Preferred are those hydrocarbonaceous oils having SAE viscosity numbers of 5 to 250W (see Guthries, pp. 9-13) and particularly preferred are those having SAE viscosity numbers in the range of 75-250.

Preparation of the Lubricating Composition

The novel compositions of this invention are prepared by dehydrating a water-in-oil dispersion of an aqueous solution of the alkali metal borate-acid anion salt mixture to provide a dispersion of the hydrated alkali metal borate and acid anion salt in the oil medium.

One method of preparation is to add the mixture of dispersants and an aqueous solution of the alkali metal borate and acid anion salt to the inert nonpolar oil medium. The mixture is vigorously agitated to provide the water-in-oil dispersion followed by heating at a temperature and for a time which provides the desired degree of dehydration of the alkali metal borate.

Alternatively, an aqueous solution of one or more alkali metal hydroxides, a second solution of boric acid, and a third solution of phosphoric acid, or sulfuric acid or a mixture thereof are added together with agitation and then added, with agitation, to the oil medium containing the dispersants. After dispersion of the water in the oil, the mixture is heated to the desired degree of dehydration.

The temperature to which the dispersion is heated will generally be at least 250°F, more usually at least between 275° and 325°F. Temperatures of up to 450°F may be used, although it is preferred that the temperature not exceed 350°F. Lower temperatures may be used at reduced pressures. Generally, the process is carried out at atmospheric pressure and at temperatures in the preferred range described above.

The period of heating will depend upon the degree of dehydration desired, the amount of water present and the temperature at which dehydration is carried out. Time is not a critical factor and will be determined for the most part by the variables mentioned.

The water initially present will be sufficient to dissolve the inorganic salts and excess should be avoided as it will prolong dehydration. Generally, from about 0.25 to about 4 and usually about 0.5 to about 1.5 parts by weight of water will be used per part by weight of the combined total of the borate and acid anion salt compounds.

Other Additives

Other materials may also be present in the lubricating compositions of this invention. Such materials may be added to enhance some of the properties which are imparted to the lubricating medium by the alkali metal borate/acid anion salt combination or to provide other

desirable properties to the lubricating medium. These include additives such as rust inhibitors, antioxidants, oiliness agents, film inhibitors, viscosity index improvers, pour point depressants, etc. Usually, these will be in the range of from about 0–0.1 to 5 percent weight and preferably in the range of about 0.1–2 percent weight of the total composition. A particular advantage is often gained by using a foam inhibitor which generally is present from about 0.5 to about 50 parts per million based on the total composition.

Another group of additives which can be used to great advantage are those which contain zinc cations. The presence of zinc cations in a lubricating composition containing dispersed alkali metal borates and acid anion salts provides a synergistic reduction in wear as determined by the 4-Ball Scar Test method. Compositions containing sodium borate and monosodium phosphate yield a four-ball scar of approximately the same dimensions as a composition containing a zinc di-n-octyl dithiophosphate. However, a lubricating composition containing all three of these materials in the same proportions as they were used individually yields a wear scar a little more than half as wide as those obtained when the additives were used alone.

The particular compound by means of which the zinc cation is present in the lubricating compositions is not critical. For example, as mentioned above, the zinc cation can be present in the form of a zinc dialkyl dithiophosphate. Alternatively, the zinc can be present in the form of a mixed sulfonate such as a sodium/zinc petroleum sulfonate. The only important criteria is that the zinc form part of a molecule which dissolves in the lubricating oil medium or is readily dispersed therein. This means that the compounds of which the zinc cation forms a part will have oil-solubilizing groups such as those present in the lipophilic dispersants described above.

The following examples are included to demonstrate the efficacy of the lubricating oil composition of this invention. All parts and percentages are by weight unless otherwise indicated.

EXAMPLE 1

This example demonstrates the extreme pressure (EP) properties of lubricating compositions containing at least partially neutralized borates. Test compositions were prepared as follows:

The inorganic salts are dissolved in sufficient water to effect a complete solution. The solution is then added to a neutral solvent-refined lubricating oil having a viscosity of 126 SUS at 100°F containing the dispersants indicated in Table I below. The oil-water mixture is vigorously agitated, for example in a blender, until the aqueous solution is uniformly dispersed in the oil. The dispersion is then heated, while maintaining agita-

tion, to the indicated temperature to cause dehydration and form a concentrate. A portion of the concentrate is added to a SAE 90 grade lubricating oil in sufficient quantity to yield the concentrations of additives shown in Table I.

The compositions obtained are tested for EP properties in the well known "four-ball" test. This test is described in Bonner, supra, pp. 222–224. In this test three steel balls (one-half-inch diameter) of the type commonly used in ball bearings are placed in a steel cup and clamped into position. A fourth ball of the same type is held rigidly on the end of a shaft which rotates about a vertical axis. The balls are immersed in the test lubricant and the fourth ball is forced against the other three under a measured load. The fourth ball is then rotated at a designated speed for a fixed period. At the end of this period the wear scar diameters on the three fixed balls are measured and averaged and the average scar size reported as the result of the test. The smaller the wear scar, the better the EP characteristics of the lubricant. A satisfactory EP lubricant has a four-ball scar preferably not greater than 0.5 mm. and more preferably not greater than 0.45 mm.

Unless otherwise indicated the conditions for the four-ball wear test are as follows: the load on the test device is 50 kg., the fourth ball is rotated at 1,730 rpm for 30 minutes, and the test lubricant is at room temperature at the beginning of the test.

The test results reported in Table I below show the compatibility, crystallization, and EP properties of several borate-containing oils. In these tests dispersant A is a commercially available neutral calcium sulfonate made from a neutral, solvent-refined paraffinic base oil having a viscosity of 480 SUS at 100°F. It contains 1.67% calcium. Dispersant B is an overbased version of Dispersant A. It has a base ratio of 9.3 and contains 11.4% calcium. Dispersant C is a commercially available overbased magnesium sulfonate having an alkalinity value of 300. Dispersant D is a polyisobutenyl succinimide produced by the reaction of a succinic anhydride substituted with a isobutene radical having a number average molecular weight of 950 with tetraethylene pentamine. The mol ratio of the amine to the anhydride is 0.75. Dispersant E is similar to Dispersant D except that the mol ratio of amine to anhydride is 0.87.

Compatibility of the borate-containing oil compositions with sulfurized fats, zinc dialkyl dithiophosphates and the like is determined by placing a sample in a 4-dram vial and storing in an oven at 300°F for 24 hours. The amount of precipitate is reported in millimeters. Crystallization is determined by stirring 10 grams of water with 100 grams of oil composition, boiling 8 grams of water off at 230°F, allowing the sample to cool and observing the quantity and hardness of crystals, which form.

TABLE I

No.	Compatibility and Water Tolerance			Compatibility		Crystallization	mm Scar 4-Ball
	% W NaBO ₂	% W NaH ₂ PO ₄	Additives	Dispersants, % W	mm Precipitate		
1	5.0	0		2.0 A	0	yes	0.39
2	5.0	0		0.5 D, 0.5 A	0	no	0.45
3	5.0	0		0.67 D, 0.33 B	trace	no	0.52
4	5.0	0	1% Zn DTP*	0.75 E, 0.25 A	completely gelled	—	—
5	1.5	3.4		0.67 D, 0.33 C	0	no	0.68
6	1.5	3.4	1% Zn DTP*	0.67 D, 0.33 C	0	no	0.38

*Zinc Di-n-octyl dithiophosphate

The above results show that sodium metaborate exhibits excellent EP properties, but when used with a simple dispersant, also crystallizes when in contact with water. This problem is solved by using a mixed dispersant system; however, some EP performance is lost. Composition 4 shows that the borate is incompatible with a conventional extreme pressure agent such as zinc dithiophosphate. Neutralization of the borate causes some loss of EP performance, but Composition 6 shows that the combination of the borate with the conventional EP agent yields excellent EP performance without compatibility or crystallization problems.

EXAMPLE 2

This example shows compatibility with zinc dithiophosphate is obtained by at least partially neutralizing the sodium metaborate with monosodium phosphate. It also shows improved extreme pressure performance is obtained by using a combination of the neutralized borate and the zinc dithiophosphate. Preparation is the same as in Example 1. Dispersants C and D are the same as those described above in Example 1. Compatibility and crystallization are also determined as in Example 1.

TABLE II

DITHIOPHOSPHATE COMPATIBILITY BY NEUTRALIZATION									
No.	% W NaBO ₂	% W NaH ₂ PO ₄	% W ZnDTP	Dispersants % W	pH Aqueous Solution	Compatibility mm precipitate	Crystallization	mm Scar 4-Ball	
1	1.3	3.6	0	0.67 D; 0.33 C	6.6	0	OK - slight	0.61	
2	1.3	3.6	1	" "	6.6	0	OK - soft ppt.	0.44	
3	1.7	3.4	0	" "	7.2	0	OK - trace	0.75	
4	1.7	3.4	1	" "	7.2	0	OK	0.38	
5	1.7	5	0	" "	6.5	0	OK	0.58	
6	1.7	5	1	" "	6.5	0	OK	0.44	

EXAMPLE 3

This example shows neutralizing with phosphoric and sulfuric acid rather than the salts of the acid anions. Compositions 1 and 2 were dehydrated at 350°F. The remaining compositions were dehydrated at 300°F. Preparation is otherwise as in Example 1.

TABLE III

NEUTRALIZATION WITH ACIDS									
No.	% W NaBO ₂	% W/Acid	% W ZnDTP	Dispersants % W	Compatibility mm Precipitate	Crystallization	mm Scar 4-Ball		
1	3.6	1.4 H ₃ PO ₄	0	0.76 D; 0.38 C	trace	OK	0.59		
2	3.6	1.4 "	1	" "	trace	OK-some - soft	0.38		
3	3.3	1.7 "	0	" "	—	OK	0.61		
4	3.3	1.7 "	1	" "	—	OK-some - soft	0.39		
5	3.5	1.5 H ₂ SO ₄	0	" "	trace	OK-slight	0.67		
6	3.5	1.5 "	1	" "	trace	OK-slight	0.43		
7	3.5	1.5 "	0	0.76 D; 0.38 C	trace	OK	0.67		
8	3.5	1.5 "	1	1% glycerin D; 0.38 C	trace	OK	0.48		
				1% glycerin					

The above data demonstrates that excellent compatibility with other extreme pressure agents such as the zinc dithiophosphate is obtained when the borate is neutralized with phosphoric or sulfuric acid.

EXAMPLE 4

This example demonstrates the synergistic effect of combining an alkali metal borate-phosphate EP agent with a zinc cation containing compound.

An aqueous solution of sodium metaborate is titrated to neutrality with monosodium phosphate. The neutral solution is then combined with a neutral, solvent-refined lubricating oil having a viscosity of 126 SUS at

100°F containing Dispersants C and D. The oil-water mixture is vigorously agitated and dehydrated as described in Example 1. A portion of this concentrate is then added to a SAE 90 grade lubricating oil in an amount to give 0.37 percent Dispersant C, 0.67 percent Dispersant D, and the quantities of borate and phosphate shown in Table IV. A second oil is prepared by adding 1 percent of a zinc di(n-octyl)dithiophosphate to a SAE 90 oil containing the same amounts of the dispersants. A third oil is prepared by adding 1 percent of the dithiophosphate to a portion of the first oil which contains the neutralized borate. These oils were tested in the four-ball test. The results are shown below in Table IV.

TABLE IV

THE EFFECT OF ADDING ZINC TO BORATE-PHOSPHATE CONTAINING LUBRICANTS				
Percent of Additive			4-Ball Scar.	
No.	NaBO ₂	NaH ₂ PO ₄	Dithiophosphate	Diameter, mm
1	2.2	3.1	0	0.615, 0.619
2	0	0	1	0.567, 0.562
3	2.2	3.1	1	0.370, 0.375

The synergistic extreme pressure performance ob-

tained by the combined use of the neutralized borate additive with the zinc cation containing additive is demonstrated by Oil No. 3.

EXAMPLE 5

This example demonstrates that it is the presence of

the zinc cation rather than the presence of a specific additive such as zinc dialkyldithiophosphate which provides the synergistic extreme pressure properties.

In this example four lubricating oils are prepared using the same solvent-refined oil of Example 4. To this oil is added Dispersants C and D. An aqueous solution of sodium metaborate substantially neutralized with monosodium phosphate is dispersed in the oil. The dispersion is then dehydrated. This concentrate is added to a SAE grade oil such that the oil contains 1.67 percent sodium metaborate, 3.35 percent monosodium phosphate, 0.38 percent Dispersant C and 0.77 percent Dispersant D. To this oil are added different combina-

tions of additives which contain the various functional groups of the zinc dialkyldithiophosphate. The various additives are used at treatment levels which yield approximately the same molar content of that particular functional group as was present in Oil No. 3 of Example 3. The additives used are a mixed sodium-zinc petroleum sulfonate, tricresyl phosphate, and a sulfurized isobutene (Anglamol 32). The results of testing various combinations of these three additives are presented in Table V.

TABLE V

THE EFFECT OF THE FUNCTIONAL GROUPS OF ZINC DIALKYL DITHIOPHOSPHATE				4-Ball Scar Diameter mm
No.	Percent of Additive Na-Zn Sulfonate	Tricresyl Phosphate	Sulfurized Polybutene	
1	1.3	0.4	0.2	.38
2	1.3	0.4	—	.41
3	1.3	—	0.2	.40
4	—	0.4	0.2	.56

The above results show that excellent extreme pressure performance is obtained with all oils except Oil No. 4 which did not contain the zinc cation. Thus, the zinc cation is the important factor in obtaining synergistic extreme pressure performance rather than the entire zinc dialkyl-dithiophosphate molecule used in Example 4.

I claim:

1. A lubricating composition comprising (1) an oil of

lubricating viscosity and dispersed therein (2) an amount effective to impart extreme pressure properties to said oil of a hydrated borate of the formula:



wherein M represents an alkali metal, x represents a positive number of 0.75 to 3, and y represents a positive number of 0.5 to 4.5, said borate having been neutralized with sufficient acidic anions of phosphoric acid, sulfuric acid, or mixtures thereof such that an aqueous solution of said borate has a pH of 6-8.

2. The composition of claim 1 wherein said neutralized borate is dispersed in said lubricating oil by a mixture of dispersants, said mixture comprising 10-99 weight percent of a lipophilic anionic surface-active agent and 90-1 weight percent of a lipophilic nonionic surface-active agent and wherein said mixture of dispersants comprises 0.25 to 5 weight percent of said composition and said neutralized borate comprises 1 to 15 weight percent of said composition.

3. The composition of claim 2 wherein said lipophilic anionic surface-active agent is an alkaline earth metal sulfonate, phenate, naphthenate or mixture thereof and said lipophilic nonionic surface-active agent is an alkyl succinimide of an alkylene polyamine.

4. The composition of claim 3 wherein the alkali metal of said alkali metal borate is sodium or potassium and an aqueous solution of said neutralized borate has a pH of 6.5 to 7.5.

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