

[54]	<b>EXTREME PRESSURE AGENT AND ITS PREPARATION</b>	3,243,372	3/1966	Dreher et al.....	252/51.5 A
		3,254,025	5/1966	LeSuer.....	252/33
		3,313,727	4/1967	Peeler.....	252/33.4
[75]	Inventors: <b>John M. King, San Rafael; Nicolaas Bakker, Pinole, both of Calif.</b>	3,396,108	8/1968	Caruso.....	252/18
		3,480,548	11/1969	Hellmuth et al.....	252/33.4
[73]	Assignee: <b>Chevron Research Company, San Francisco, Calif.</b>	3,565,802	2/1971	West et al.....	252/25
		3,819,521	6/1974	Sims.....	252/33
		3,829,381	8/1974	LeSuer.....	252/33.4

[22] Filed: **Mar. 22, 1974**

[21] Appl. No.: **453,898**

*Primary Examiner*—Delbert E. Gantz  
*Assistant Examiner*—I. Vaughn  
*Attorney, Agent, or Firm*—G. F. Magdeburger; C. J. Tonkin

[52] U.S. Cl..... **252/33.4; 252/33**

[51] Int. Cl.<sup>2</sup>. **C10M 1/40; C10M 3/34; C10M 5/22; C10M 7/38**

[58] Field of Search ..... **252/33, 33.4, 18, 25**

[56] **References Cited**

**UNITED STATES PATENTS**

2,987,476	6/1961	Hartley et al.....	252/18
3,242,080	3/1966	Wiley et al.....	252/33.4

[57] **ABSTRACT**

A particulate dispersion of an alkali metal borate is prepared by contacting boric acid with an alkali metal carbonate overbased metal sulfonate within an oleophilic liquid reaction medium.

**23 Claims, No Drawings**

## EXTREME PRESSURE AGENT AND ITS PREPARATION

### DESCRIPTION OF THE INVENTION

Numerous additives are incorporated into lubricating oils and greases to enhance their lubricating properties. A wide variety of materials have been employed to increase the load-carrying capacity of lubricants employed under boundary or extreme pressure (EP) conditions. When moving surfaces are separated by oil or a grease, as the load is increased and the clearance is reduced between the surfaces, the condition of boundary, or thin-film, lubrication is reached. Metal-to-metal contact occurs and wear or seizure results. Under these conditions, the effectiveness of lubricants in reducing wear or friction varies widely. At still higher loads, the condition commonly known as extreme pressure lubrication is reached. Scuffing, galling, and rapid wear or seizure may occur. Welding of two contacting surfaces occurs followed by metal transfer (galling) or cleavage and production of metal fragments.

In order to avoid the undesirable effects which result when using an uncompounded lubricant under high load conditions, extreme pressure agents are added. For the most part, the extreme pressure agents have been oil-soluble agents containing a chemically reactive element, e.g., chlorine, sulfur, or phosphorus, which reacts with the metal surface at the high temperatures produced under load conditions. This chemical bond to the EP agent then provides relatively good boundary protection.

Recently, a new type of additive has been developed which, unlike the chemically reactive chlorine, sulfur or phosphorus containing EP agent, does not react with the metal surfaces to become chemically bonded thereto. Instead, this extreme pressure additive is a dispersion of microparticulate alkali metal borates which is believed to deposit on the metal surface a viscous lubricating film. These borates and their preparation are disclosed in U.S. Pat. No. 3,313,727.

The microparticulate metal borates are prepared by dissolving an alkali metal borate, or its precursors, in water and emulsifying the aqueous solution in oil to form a micro-emulsion. The emulsion is then dehydrated, leaving amorphous or glassy particles of the hydrated alkali metal borate dispersed within the lubricating oil.

Although the borate dispersions prepared in this manner have excellent extreme pressure properties, there is a need to prepare borate dispersions by a method which does not involve the use of a water-in-oil emulsion and its subsequent dehydration. In addition, certain borate dispersions exhibit a compatibility problem with other lubricating oil additives such as phenates, sulfurized fats, and zinc dithiophosphates.

It is therefore an object of this invention to provide an improved method for preparing a dispersion of microparticles of an alkali metal borate in a lubricating oil or grease.

It is an additional object of this invention to provide a method for preparing borate dispersions having EP properties which does not involve the employment of a water-in-oil emulsion.

It is an additional object of this invention to provide a borate dispersion having improved EP properties. An additional object of this invention is to provide a borate

dispersion which has improved compatibility with other lubricating oil additives.

The aforementioned objects and their attendant advantages may be realized by a particulate dispersion of an alkali metal borate prepared by contacting boric acid with an alkali metal carbonate overbased alkali or alkaline earth metal sulfonate within a stable inert oleophilic liquid reaction medium. Overbased materials are characterized by a metal content in excess of that stoichiometrically required by the reaction of the metal with the particular sulfonic acid. The base ratio is the ratio of the chemical equivalents of excess metal in the product to the chemical equivalents of the metal required to neutralize the sulfonic acid.

By preparing the alkali metal borates by the reaction of boric acid with the alkali metal carbonate overbased alkali or alkaline earth metal sulfonate, the necessity for using a water-in-oil emulsion is avoided. In addition, it was discovered that the borate dispersions prepared by the method of this invention have improved compatibility with other additives which are normally incorporated into lubricating oils and include phenates, sulfurized fats, and zinc dithiophosphates.

### DETAILED DESCRIPTION OF THE INVENTION

The borate dispersions of this invention are prepared by contacting within an oleophilic reaction medium (1) boric acid and (2) an alkali metal carbonate overbased alkali or alkaline earth metal sulfonate. The resulting alkali metal borate particles prepared by this method are believed to have the empirical formula:



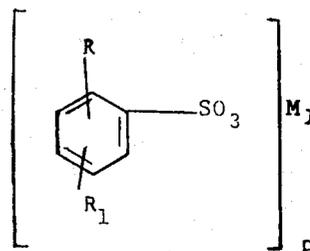
wherein: M is an alkali metal selected from the group consisting of lithium, sodium, and potassium and preferably sodium or potassium; x is a number from 0.8 to 3.5 and preferably from 1.0 to 3.0, and y is a number from 0 to 8; and preferably from 0 to 1.

The borate particles prepared by the method of this invention form a stable dispersion in the oleophilic liquid reaction medium. These particles are almost entirely less than 0.1 micron and more usually less than 0.05 micron. The product may be filtered to remove the larger microparticles.

The alkali metal carbonate overbased alkaline earth or alkali metal sulfonates are prepared by overbasing a neutral alkali or alkaline earth metal sulfonate with an alkali metal carbonate.

### NEUTRAL METAL SULFONATE

The neutral alkali or alkaline earth metal sulfonates which may be overbased in the practice of this invention can comprise any oil-soluble alkali or alkaline earth metal sulfonate. Preferably, these sulfonates are aromatic and have the following generalized chemical formula:



wherein: R is hydrogen or an alkyl having from 10 to 22 carbons (preferably from 15 to 21) and preferably attached to the benzene ring through a secondary carbon atom; R<sub>1</sub> is an alkyl having from 3 to 10 carbons when R is an alkyl or an alkyl having from 8 to 22 carbons when R is hydrogen. M<sub>1</sub> is an alkali or alkaline earth metal; and *p* is an integer from 1 to 2 and sufficient to make M<sub>1</sub> electro-neutral.

In a particular embodiment the neutral metal sulfonate is a dialkylbenzene sulfonate of the above formula wherein R is a straight chain aliphatic hydrocarbon radical of 17 to 21 carbon atoms, usually having at least 2 homologs present, and having secondary carbon attachment to the benzene ring and R<sub>1</sub> is a branched chain alkyl group of 3 to 10 carbon atoms, more usually from 4 to 9 carbon atoms, having at least 1 homolog present, and preferably having at least two homologs present, and there being at least 1 branch of 1 to 2 carbon atoms, more usually of 1 carbon atom, i.e., methyl, per 2 carbon atoms, along the longest chain. The attachment of the shorter alkyl group will generally be secondary or tertiary. Particular compositions have R<sub>1</sub> with an average of 5 to 8 carbon atoms.

Usually, the difference in average number of carbon atoms between the short and long chain alkyl groups will be at least 10 and more usually at least 12, and not more than 16.

The preferred dialkylbenzene sulfonates which find use in the practice of this invention will generally have small amounts of monoalkylbenzene sulfonate, wherein the alkyl group is of from 17 to 21 carbon atoms, present within the admixture. Preferably, the amount of the monoalkylbenzene sulfonate will not exceed 30% and more preferably the monoalkylbenzene sulfonate will not exceed 20% by weight of the total sulfonate. Generally, it will be in the range of about 5 to 20 weight percent.

The positions of the alkyl group and the sulfonate on the benzene ring in relation to each other are not critical to this invention. Generally, most of the isomeric possibilities will be encountered — with the particular isomers having the least steric hindrance being predominant. Also, there will be a broad spectrum of isomers based on the carbon of the alkyl group bonded to the benzene ring, depending on the method of preparation and the reactants used in the preparation.

Illustrative short chain alkyl groups are isopropyl, tert.-butyl, neopentyl, diisobutyl, dipropenyl, tripropenyl, etc.

Illustrative of the long chain alkyl groups are heptadecyl, octadecyl, nonadecyl, eicosyl and heneicosyl.

The total number of carbon atoms in the alkyl groups will generally be in the range of at least about 20 and less than about 28. While small amounts of the dialkylbenzenes may be outside this range, the average number of carbon atoms of the alkyl groups over the total composition will be within the range.

In a particular embodiment, a monoalkyl polypropyl benzene fraction having a boiling range of about 318°–478°F (ASTM D 447) containing from 4 to 9 carbon atoms (an average of 6 carbon atoms) in the side chain and an average molecular weight of about 167 is alkylated with a substantially straight chain C<sub>17</sub>–C<sub>21</sub> cracked wax alpha-olefin. The molecular weight of the resulting dialkylbenzene mixture has an average value in the range of 350 to 450 and preferably 400 to 420.

The average molecular weights of the alkylated benzenes used to prepare the sulfonate will generally be in the range of about 350 to 460, more usually in the range of about 375 to 425.

The monoalkyl benzenes can be prepared by simply reacting benzene with a mono-olefin in a simple alkylation process. Typical alkylation catalysts include Friedel-Crafts catalysts such as hydrogen fluoride, aluminum chloride, phosphoric acid, etc. The alkylation temperatures will ordinarily be in the range of about 4°C (40°F) to 38°C (100°F).

The particular dialkyl benzenes can be prepared in substantially the same manner. A description of its preparation is disclosed in U.S. Pat. No. 3,470,097.

The mono- or dialkylbenzenes may then be readily sulfonated, using conventional sulfonation procedures and agents, including oleum, chlorosulfonic acid, sulfur trioxide (complexed or thin film dilution techniques) and the like.

Various methods may be used to neutralize the sulfonic acid obtained, these methods being extensively described in the art. See for example U.S. Pat. Nos. 2,485,861, 2,402,325 and 2,732,344.

The neutralization step is conveniently conducted by contacting the sulfonated alkyl or dialkyl benzenes with an aqueous alkali metal hydroxide solution. The product is a neutral alkali metal sulfonate. The neutral alkaline earth metal sulfonate is prepared by a simple metal exchange process. The alkali metal sulfonate is contacted with an alkaline earth metal salt, typically the halide salt, and the mixture heated. The exchange process may be accomplished at temperatures of 50° to 150°C and contact times of 0.5 to 10 hours, usually from 1 to 3 hours.

Ordinarily, the neutralized product will be mildly overbased, having from about 0.02 to 0.7 mol percent excess of basic metal over that required for neutralizing the acid. Alkalinity values of these neutral compositions will generally be in the range of about 1 to 30, more usually from about 1 to 10 mg.KOH/g.

Specific examples of exemplary metal sulfonates which may be overbased for use in this invention are disclosed in U.S. Pat. Nos. 3,691,075, 3,629,209, 3,595,790, and 3,537,996. These patents are herein incorporated by reference.

Illustrative individual compositions are sodium isopropyl eicosylbenzene sulfonate, potassium or barium tert.-butyl nonadecylbenzene sulfonate, calcium dipropenyl octadecylbenzene sulfonate, calcium diisobutyl octadecylbenzene sulfonate, sodium (propylene trimer) nonadecylbenzene sulfonate, barium isopropyl eicosylbenzene sulfonate, etc.

#### OVERBASING OF THE NEUTRAL METAL SULFONATE

Various methods of overbasing neutral metal sulfonates have been reported in the literature. See for example U.S. Pat. Nos. 2,695,910, 3,282,835 and 3,155,616, as well as Canadian Patent 570,814. The preferred method employs a method similar to that described in U.S. Pat. No. 3,155,616.

The overbasing process can be conveniently conducted by charging to a suitable reaction zone the neutral metal sulfonate, and an inert hydrocarbon solvent. An alkali metal base (usually an alkali metal hydroxide) dissolved in a C<sub>1</sub> to C<sub>4</sub> alkanol is added while the mixture is agitated and maintained at a temperature and pressure sufficient to remove from the liquid mix-

ture by distillation most of the alkanol charged. Carbon dioxide or another suitable acid gas (hydrogen sulfide, sulfur dioxide, etc.) is then contacted with the reaction medium, preferably sparged or bubbled through the liquid mixture. The introduction of the acid gas is continued until its absorption rate into the mixture ceases or substantially subsides. Generally, from 0.2 to 1.6 equiv. and more usually from 0.9 to 1.3 equiv. of acid gas will be absorbed by the mixture for every equivalent of alkali metal base present.

The crude reaction product is then heated to strip out the residual alkanol and water of reaction. The stripping will generally be conducted at temperatures below 150°C and usually below 125°C.

After stripping the alkanol and water, the product may be filtered.

The stripping of the hydrocarbon diluent will generally be carried out at temperatures below 200°C and will usually not exceed 175°C, depending on the hydrocarbon diluents used. Preferably, when xylene is used, the temperature will not exceed 150°C.

Occasionally, the final product will be filtered again to remove any adventitious particulate matter which may still be present.

In a different embodiment, the hydrocarbon diluent is first stripped and then the product is filtered. Also, further addition of oil may be made to obtain a product having a somewhat lower viscosity. The choice of the particular route will depend on the equipment, the materials used, their physical properties, and the product desired.

The alkanol used, preferably methanol, will generally have from about 0.01 to 1 weight percent water, more usually from about 0.1 to 0.7% water. The alkanol will generally be present from about 0.1 to 20, more usually from about 1 to 10 weight parts per part of alkali metal base.

The hydrocarbon diluent will be one having a boiling point higher than alkanol to permit its retention when the alcohol is removed during processing. The boiling point should generally be less than about 280°C and preferably less than about 250°C. Usually, the hydrocarbon diluent will form an azeotrope with water. The usual diluents contain aromatic hydrocarbons of 7 to 10 carbon atoms, having boiling points in the range of about 100° to 180°C. These include toluene, xylene, cumene and cymene. The hydrocarbonaceous diluent can be present in an amount to form about a 5 to 20 weight percent dispersion of alkali metal base in the initial composition, usually an 8 to 15 weight percent dispersion.

The amount of overbasing varies greatly depending upon the amount of borate dispersion ultimately wanted. Typically from 1 to 20 equivalents of alkali metal base will be used per equivalent of neutral metal sulfonate, more usually from about 5 to 15 equivalents of alkali metal base per equivalent of neutral metal sulfonate. Thus, alkalinity values range from 50 to 460 mg.KOH/g, and preferably from about 150 to 300 mg.KOH/g.

It should be recognized that mixtures of alkali metal carbonates may be employed as well as mixtures of alkali and alkaline earth metal sulfonates. Thus, a sodium and potassium carbonate overbased sodium and calcium sulfonate may be present in the same mixture which may be further reacted with the boric acid to form the borate particles of the instant invention.

## OLEOPHILIC REACTION MEDIUM

The overbased metal sulfonate is contacted with boric acid within a suitable oleophilic reaction medium. As referred to herein oleophilic is defined as a property of a substance having a strong affinity to oils. The liquid oleophilic medium is generally present in the preparation of the overbased sulfonate and hence extraneous addition of the medium is normally not necessary. The oleophilic reaction medium can comprise any stable, inert, organic oil having a viscosity ranging from 50 to 1000 SUS at 38°C (100°F) and preferably from 50 to 350 SUS at 38°C (100°F).

Examples of stable organic oils which may be employed include a wide variety of hydrocarbon lubricating oils such as naphthenic base, paraffinbase, and mixed base lubricating oils. Other oleophilic oils include oils derived from coal products and synthetic oils, e.g., alkylene polymers (such as polymers of propylene, butylene, etc., and mixtures thereof), alkylene oxide-type polymers (e.g., alkylene oxide polymers prepared by polymerizing alkylene oxide, e.g., propylene oxide polymers, etc., in the presence of water or alcohols, e.g., ethyl alcohol), liquid esters of acids of phosphorus, alkyl benzenes, polyphenols (e.g., biphenols and terphenols), alkyl biphenol ethers, polymers of silicon, e.g., hexyl(4-methyl-2-pentoxy)disilicone, poly(methyl)siloxane, and poly(methylphenyl)siloxane, etc. The oleophilic lubricating oils may be used individually or in combinations, whenever miscible or whenever made so by use of mutual solvents.

When concentrates are desired, the viscosity of the overbased sulfonate in the oleophilic reaction medium is generally too high for normal processing. In these instances, it is preferred that a light hydrocarbon diluent be employed to reduce the viscosity of the reaction medium. The diluent may be aliphatic or aromatic and boiling below 200°C and preferably below 150°C. Exemplary aromatic diluents include benzene, toluene, xylene, etc., exemplary aliphatic diluents include cyclohexane, the heptanes, octanes, etc. The diluent should not boil below 70°C and preferably not below 100°C.

At the end of the processing steps, the diluent may be stripped from the system. Any of the conventional stripping techniques may be employed.

## PREPARATION OF BORATE DISPERSION

The alkali metal borate dispersion may be prepared, in a preferred embodiment, by the following steps: a suitable reaction vessel is charged with the alkali metal carbonate overbased metal sulfonate within the oleophilic reaction medium (typically the hydrocarbon medium employed to prepare the overbased metal sulfonate). The boric acid is then charged to the reaction vessel and the contents vigorously agitated.

The reaction is conducted for a period of 0.5 to 7 hours, usually from 1 to 3 hours at a reaction temperature of 20° to 200°C, preferably from 20° to 150°C and more preferably from 40° to 125°C. At the end of the reaction period, the temperature is raised to 100° to 250°C, preferably from 100° to 150°C to strip the medium of any residual alcohol and water. The stripping may be done at atmospheric pressure or under reduced pressure of 700 mm. to 10 mm.Hg.

The amount of boric acid charged to the reaction medium depends upon what type of alkali metal borate is desired. If a tetraborate is desired 2 molar parts of boric acid is charged per molar equivalent part of over-



amine, pentaethylene hexamine, 1,2-diaminopropane, N,N-di(1-methyl-2-aminomethyl)amine, etc.

A second group of important nonionic dispersants comprises certain pentaerythritol derivatives. Particular derivatives which find use in this invention are 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 to 6 carbon atoms, such as polyethylene, polypropylene, polybutene, polyisobutylene, and the like. Such olefins generally contain a total of 20 to 250 carbon atoms and preferably 30 to 150 carbon atoms. The phosphorus sulfides include P2S3, P2S5, P4S7, P4S3 and related materials. Of these, P2S5 (phosphorus pentasulfide) is preferred principally because of its ready availability.

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

If the lipophilic nonionic surface-active agent is employed, it will generally be present in about 0.01 to 5 weight percent, more usually from about 0.1 to 3 weight percent, of the final composition. The actual amount of dispersant required will vary with the particular dispersant used and the total amount of borate in the oil. Generally, about 0.001 to 1, more usually about 0.01 to 0.5, part by weight of nonionic surface-active agent will be used per part by weight of the borate. In the concentrates the mixture concentration will be based on the relationship to borate rather than on the fixed percentage limits of the lubricant, noted above. Generally, the upper ranges of the nonionic surface active agent concentration will be used with the upper ranges of the alkali metal borate concentration.

Other materials may also be present as additives in the composition of this invention. Such materials may be added for enhancing some of the properties which are imparted to the lubricating medium by the alkali metal borate or providing other desirable properties to the lubricating medium. These include additives such as rust inhibitors, antioxidants, oiliness agents, viscosity index improvers, etc. Usually, these will be in the range from about 0.1 to 5 weight percent, preferably in the range from about 0.1 to 2 weight percent, of the total composition. An antifoaming agent may also be added with advantage. The amount required will generally be about 0.5 to 50 ppm, based on the total composition.

The borate dispersions are preferable employed in lubricating oils, such as, gear and bearing oils, cutting oils, pneumatic oils, greases, etc. The concentration of the alkali metal borate present within the lubricating oil (oleophilic reaction medium) may vary from 0.1 to 20 weight percent depending upon the particular application.

The borate dispersion may also be employed in a grease to impart extreme pressure properties. The grease composition may be prepared by adding a thickening agent to the borate dispersion in the oleophilic lubricating oil. The thickening agent may be added directly to the borate dispersion or produced "in-situ" within the oleophilic oil. Typical thickening agents which may be employed include organic or metal organic thickeners such as polyurea, alkali metal terephthalamate, lithium hydroxystearate, calcium complex soap, aluminum complex soap, polymeric thickener or combinations thereof.

Exemplary polyurea greases which may be employed are disclosed in U.S. Pat. No. 3,243,372. These greases are prepared by reacting, within the lubricating oil to be thickened, a polyamine having from 2 to 20 carbons, a diisocyanate having from 6 to 16 carbons and a monoamine or monoisocyanate each having from 10 to 30 carbons. Typically, these greases contain from 5 to 15 weight percent of the polyurea thickener although lesser amounts may be used if other thickening agents are present. A particularly preferred polyurea is a tetraurea prepared by reacting one molar part of ethylene diamine with two molar parts of tolylen diisocyanate and two molar parts of a monoamine having from 16 to 20 carbons.

Exemplary sodium terephthalamate greases are disclosed in U.S. Pat. Nos. 2,820,012 and 2,892,778. These greases may be prepared by reacting a monoester of terephthalic acid with an alkali metal base in the presence of a solvent. A particularly preferred grease contains from 8-15 weight percent of a sodium N-(hydrocarbyl) terephthalamate having from 5 to 24 carbons in the hydrocarbyl group such as sodium N-octadecyl terephthalamate.

The lithium hydroxy stearate greases are the most widely employed multi-purpose grease. These greases have the properties which render them particularly suitable for use in the practice of this invention. The lithium thickening agent is typically prepared by reacting lithium hydroxide with hydrogenated castor oil and is present within a lubricating oil at a concentration of 10 to 20 percent.

Another class of high temperature greases which may be employed is the calcium complex grease. These greases are composed of 5-20 percent of a calcium soap, e.g., calcium hydroxystearate, 4-20 percent of calcium acetate and 1-10 percent of calcium carbonate. A small amount of calcium hydroxide may also be employed. Exemplary greases of this type are described in U.S. Pat. Nos. 3,186,944 and 3,159,575.

Exemplary aluminum complex greases are described in U.S. Pat. Nos. 3,476,684 and 3,514,400. These greases are prepared by incorporating into a lubricating oil from 5-20 percent of the reaction product of a long chain fatty acid, an aromatic acid and aluminum isopropoxide.

The amount of thickener employed in making the greases of this invention varies depending upon the type thickener, type of lubricating oil, hardness of the grease desired and the presence of other additives. When greases having the preferred hardness of No. 2-4 NLGI (ASTM work penetration varying from 340 to 175) are employed, the amount of thickener generally varies from 5 to 25 weight percent and more usually from 8 to 15 weight percent of the grease composition.

#### EXAMPLE I

This example is presented to illustrate the preparation of a dialkyl benzene sulfonate which may be used to prepare the overbased metal sulfonates of this invention.

Benzene is alkylated using a tetramer polypropylene fraction and HF alkylation catalyst, a reaction temperature of about 18°C. (65°F), and efficient mixing. The hydrocarbon phase is separated, washed and fractionated. The lower alkyl benzene fraction (boiling range 159°C (318°F) to 248°C (478°F), ASTM D 447 distillation) is collected as feed for the second stage alkylation with a mixture of straight chain 1-olefins. The average

molecular weight of the above branched chain alkylbenzene is 164. This corresponds to an average of 6 carbon atoms per alkyl group in the mixture. The overall alkyl carbon atom content corresponding to the above boiling range is the C4-C9 range.

Using the above branched chain monoalkylbenzene and a substantially straight chain C17-C21 1-alkene fraction obtained from cracked wax, and hydrogen fluoride catalyst, the desired dialkylbenzene is produced in a stirred, continuous reactor.

The 1-alkene feed has the following characteristics.

Average mol weight	268	
Average number of carbon atoms per molecule	19	
Olefin distribution, weight percent:		
C17	2	
C18	22	
C19	39	
C20	32	
C21	5	
Reaction conditions:		
LHSV	2	
Temperature	38°C	(100°F)
Monoalkylbenzene to alpha-olefin, mol ratio	2-1	
Hydrocarbon to HF ratio, volume	2.3-1	

After reaction the settled product is separated into an organic phase and a lower HF acid phase. The crude dialkylbenzene organic phase is washed and then fractionated by distillation. A minor amount of forecut, mainly monoalkylbenzene, is collected up to an overhead temperature of about 232°C (450°F) at 10 mm.Hg. The balance of the distillate is the desired product, and has an average molecular weight of about 405. The difference between the average carbon atom content of the alkyl-chain types is about 13.

The dialkylbenzene is charged to a stirred reaction vessel fitted for temperature control along with 130 neutral oil which is substantially free of sulfonatable material. The volume ratio of the two materials is 3½ to 4, respectively, and to this mixture is added, over a period of several hours, 2 volumes of 25% oleum. The reaction temperature is maintained at about 38°C (100°F). Two phases developed in the settled mixture, the lower being a spent mineral acid phase and the upper being the desired sulfonic acid phase.

The separated sulfonic acid-oil mixture is then neutralized with one volume of 50% aqueous caustic diluted with 15 volumes of aqueous 2-butanol. During the neutralization the temperature is maintained below about 43°C (110°F), and after completion thereof the neutral solution is heated and maintained at 60°C (140°F) during a second phase separation. Two phases developed, a lower brine-alcohol solution and an upper neutral alcohol-sodium sulfonate solution.

#### EXAMPLE 2

The preparation of a neutral calcium sulfonate is illustrated in this example. A 3-liter glass flask is charged with 80 grams of calcium chloride and 800 milliliters of water. Thereafter, 1,500 grams of the sodium sulfonate solution as prepared by the method of Example 1 is charged to the flask. The contents are heated to 30°C (85°F) under agitation and maintained at these conditions for one hour. The contents are allowed to phase separate and the water layer drawn off. 800 milliliters of distilled water is admixed with the

sulfonate and heated for one hour. The phases are allowed to separate and the aqueous phase drawn off. The sulfonate is washed three additional times with water and one time with an aqueous isobutyl alcohol solution. The mixture is heated to 112°C to remove any residual water and isobutyl alcohol. 500 milliliters of toluene is added to the sulfonate and the admixture filtered through Celite 512. The product is stripped at 185°C under 3 mm.Hg pressure to yield 740 grams of neutral calcium sulfonate. Analysis of the product reveals

Wt % sulfated ash	6.09
Wt % metal	1.92 calcium

#### EXAMPLE 3

This example is presented to illustrate the preparation of a sodium carbonate overbased calcium sulfonate which may be employed to prepare the borate dispersions of this invention. A neutral sodium sulfonate is prepared by sulfonating a 480 neutral oil (RPM 480) in a manner as described in Example 1 and neutralizing the resulting organic sulfonic acid with sodium hydroxide. The resulting sodium sulfonate is then subjected to calcium metathesis as described in Example 2 to yield a neutral calcium sulfonate (calcium content of about 1.67 weight percent).

A 2-liter flask is charged with 200 grams of the neutral calcium sulfonate and 750 ml of an aliphatic hydrocarbon diluent having a boiling range of 158°C to 202°C and containing 17 percent aromatics. The solution is heated to 110°C and a solution of 60 grams of sodium hydroxide in 300 ml of methanol are added over a 138-minute period at 110°C.

Upon completion of the sodium hydroxide addition the temperature is raised to 130°C and carbon dioxide is passed into the solution at a rate equal to its maximum carbon dioxide absorption. The carbonation continued for 60 minutes at 130°C and then the contents of the flask are heated to 150°C. A total of 38 grams of CO<sub>2</sub> are absorbed. The contents are cooled and filtered. The filtered product is then stripped to 165°C at 5 mm of Hg pressure. A total of 229 grams of product is recovered. The product had an alkalinity value of 253 mg KOH/gm.

#### EXAMPLE 4

A two-liter flask is charged with 200 grams of a neutral calcium sulfonate of the type described in Example 3 along with 750 milliliters of an aliphatic hydrocarbon diluent having a boiling range from 158°C to 202°C and containing 17 percent aromatics. A solution of 60 grams of sodium hydroxide in 300 milliliters of methanol is added to the flask through a dropping funnel. The temperature is maintained between 23°C and 40°C during the addition which took 69 minutes. Simultaneously with the NaOH addition, carbon dioxide is passed through the flask contents at a rate equal to its maximum carbon dioxide absorption. Carbonation continued for fifteen minutes after the addition of the sodium hydroxide/methanol solution had stopped. A total of 52 grams of carbon dioxide is absorbed. The contents of the flask is stripped by heating to a temperature of 125°C. the contents are then cooled and filtered. The filtered product is then stripped to 165°C at 5 mm of Hg pressure. A total of 249 grams of the prod-

uct is recovered. The product has an alkalinity value of 259 mg KOH/g.

#### EXAMPLE 5

This is another example demonstrating the preparation of a sodium carbonate overbased calcium sulfonate which may be used to prepare the borate dispersions of this invention. A neutral sodium sulfonate is prepared by sulfonating a 480 neutral oil (RPM 480) in a manner as described in Example 1 and neutralizing the resulting organic sulfonic acid with sodium hydroxide. The resulting sodium sulfonate is then subjected to calcium metathesis as described in Example 2 to yield a neutral calcium sulfonate calcium content of about 1.67 weight percent.

A 2-liter flask is charged with 200 grams of the neutral calcium sulfonate and 750 ml of an aliphatic hydrocarbon diluent of the type described in Example 3. Contents of the flask are heated to 110°C and a solution of 60 grams of sodium hydroxide in 300 ml of methanol is introduced into the flask over an 82-minute period at 110°C. One minute after the start of the sodium hydroxide addition, carbon dioxide is bubbled into the flask contents at a rate equivalent to that of the sodium hydroxide addition, until a total of 41 grams of carbon dioxide are absorbed. At the end of the sodium hydroxide addition, the temperature is increased to 140°C and 507 ml are taken off overhead.

The product is cooled, filtered and stripped to a temperature of 175°C under 5 mm.Hg pressure. A total of 271 grams of product is recovered. The alkalinity value is 302 mg.KOH/g, and the base ratio is 10.6:1.

#### EXAMPLE 6

This example is presented to illustrate the preparation of a sodium carbonate overbased sodium sulfonate, 140 grams of a 57 weight percent sodium sulfonate solution is butanol and prepared by the method of Example 1 are charged to a 2-liter flask along with 325 grams of a 37.5 wt % sodium sulfonate solution in butanol and prepared by neutralizing a sulfonated 480 neutral hydrocarbon oil with sodium hydroxide. The contents are heated to 165°C under 600 mm vacuum and 263 grams are taken off overhead.

Thereafter 750 milliliters of an aliphatic hydrocarbon diluent described in Example 3 are charged to the flask. The solution is heated to 110°C and 60 grams of sodium hydroxide dissolved in 300 milliliters of methanol are slowly added to the flask over a 100-minute period at 110°C. Simultaneously, carbon dioxide is bubbled into the solution at a rate equivalent to that of the sodium hydroxide addition. After completion of the sodium hydroxide addition, the contents are heated to 150°C with the carbon dioxide flow continued for 10 additional minutes. A total of 515 milliliters are taken off overhead. A total of 45 grams of carbon dioxide is absorbed. The resulting sodium carbonate overbased sodium sulfonate solution is filtered and the filtrate stripped to a temperature of 175°C at 5 mm mercury pressure. A total of 282 grams of product are recovered with an alkalinity value of 299 mg KOH/gm.

#### EXAMPLE 7

A 2-liter flask is charged with 280 grams of a sodium carbonate overbased calcium sulfonate prepared by the method of Example 5 (alkalinity value of 308), 600 ml of an aliphatic hydrocarbon solvent described in Example 3 and 95 grams of boric acid. The contents of the

flask are heated to 125°C and thereafter allowed to cool to 20°C under a pressure of 160 mm of mercury. A total of 90 ml are removed overhead and thereafter an additional 95 grams of boric acid are charged to the flask and heated to 125°C. The contents are allowed to cool to 100°C under a pressure of 160 mm of mercury. The product is filtered and stripped to 165°C at 5 mm of mercury pressure and 368 grams of product are recovered. Analysis of the product reveals Boron content — 7.6 weight percent. Carbon dioxide content — 0.23 weight percent; alkalinity value — 215 mg.KOH/g.

#### EXAMPLE 8

A 2-liter flask is charged with 140 grams of a sec-butanol solution containing 57.4 weight percent of a neutral sodium sulfonate prepared by the method of Example 1 and 325 grams of a sec-butanol solution containing 37.5 weight percent of a neutral sodium sulfonate prepared by sulfonating a 480 neutral oil (RPM 480) and neutralizing with sodium hydroxide. The contents are stripped of 262 grams of alcohol. Thereafter, 750 ml of a petroleum aliphatic thinner of the type disclosed in Example 3 are mixed with the contents of the flask. Flask contents are heated to 110°C and a solution of 60 grams of sodium hydroxide in 300 ml of methanol are added to the flask over a 93-minute period at 110°C. Simultaneously, carbon dioxide is bubbled into the flask contents at a rate equivalent to that of the sodium hydroxide addition until a total of 38 grams of CO<sub>2</sub> have been absorbed. The temperature is raised to 150°C and 515 ml are taken off overhead. The reaction mixture is allowed to cool to 60°C and, 93 grams of boric acid are added, and the temperature raised to 120°C over a one-hour period. The contents are allowed to cool under a pressure of 160 mm of mercury. Thereafter, an additional 93 grams of boric acid are charged to the flask contents and the temperature raised to 135°C over a 70-minute period. A vacuum of 600 mm of mercury is applied to the flask to cool the product. Product is filtered and stripped to 165°C at 5 mm of mercury pressure. 367 grams of product are recovered. Analysis of the product reveals an alkalinity value of 233 mg.KOH/g; a boron content of 7.7 weight percent and a carbon dioxide content of 0.12 weight percent.

#### EXAMPLE 9

The example is presented to demonstrate the preparation of an exemplary potassium borate dispersion. A 2-liter flask is charged with (1) 140 grams of a sec-butanol solution containing 57.4 weight percent of a neutral sodium sulfonate prepared by the method of Example 1 and 325 grams of a sec-butanol solution containing 37.5 weight percent of a neutral sodium sulfonate prepared by sulfonating a 480 neutral oil (RPM 480) and neutralizing with sodium hydroxide. The contents are stripped of 262 grams of alcohol. 750 ml of a petroleum aliphatic thinner of the type employed in Example 3 are mixed with the contents of the flask. Flask contents are then heated to 110°C and a solution of 107 grams of potassium hydroxide in 300 ml of methanol are added to the flask over a 94-minute period at 110°C. Simultaneously carbon dioxide is bubbled into the flask contents at a rate equivalent to that of the potassium hydroxide addition until a total of 43 grams of CO<sub>2</sub> are absorbed. The temperature is raised to 150°C and 525 ml are taken off overhead. The reac-

15

tion mixture is allowed to cool to 60°C and 102 grams of boric acid are charged to the flask, the temperature raised to 135°C in approximately 45 minutes. The temperature is maintained at 135° for an additional 30 minutes, then the contents are allowed to cool under a pressure of 160 mm of mercury. 63 ml are taken off overhead. An additional 102 grams of boric acid is added to the flask and the temperature slowly raised to 145°C in one hour. The reaction mixture is cooled, filtered and stripped to 165°C under a pressure of 5 mm of mercury. 396 grams of product is recovered. Analysis of the product reveals that it has an alkalinity value of 223, a boron content of 7.1 weight percent and a carbon dioxide content of 0.62 weight percent.

## EXAMPLE 10

A 2-liter flask is charged with 280 grams of sodium carbonate overbased calcium sulfonate having an alkalinity value of 308 and prepared by the method of Example 5 along with 600 ml of an aliphatic hydrocarbon thinner of the type described in Example 3 and 95 grams of boric acid. The contents of the flask are slowly heated to 120°C over a one-hour period. The contents are slowly cooled to 60°C by applying a vacuum of 600 mm of mercury to the flask contents. A total of 59 ml are recovered overhead. Thereafter, 95 grams of boric acid are charged to the flask and heated to 120°C over a one-hour period. The contents are then cooled to 65° under a pressure of 160 mm and 87 ml are taken off overhead. Thereafter, a third batch of 95 grams of boric acid are charged to the flask and the contents heated to 140°C in 1½ hours. A vacuum of 600 mm of mercury is slowly applied to the flask and the contents allowed to cool. 200 ml are taken off overhead. The product was filtered and stripped to 165°C at 5 mm of mercury pressure. 417 grams of product was recovered. The product is analyzed and found to have an alkalinity value of 187 mg KOH/g; a boron content of 11.7 weight percent and carbon dioxide content of 0.01 weight percent.

## EXAMPLE 11

A 2-liter flask is charged with 380 grams of sodium carbonate overbased calcium sulfonate as described in Example 5, along with 400 milliliters of an aliphatic thinner of the type described in Example 3. The contents of the flask are heated to 40°C and thereafter 122 grams of boric acid are added. The temperature of the solution is slowly raised to 150°C in approximately 105 minutes. The product is then cooled, filtered and stripped to recover 435 grams of product. The filtration and stripping step is described in Example 8. Analysis of the product reveals an alkalinity value of 262 mg KOH/g, a boron content of 5.36 weight percent and a carbon dioxide content of 4.27 percent. The high carbon dioxide content reveals that the product is a mixture of sodium tetraborate and sodium carbonate.

293 grams of this product along with 500 milliliters of an aliphatic thinner of the type described in Example 3 are charged to a two-liter fournecked flask. Thereafter 82 grams of additional boric acid are added to the flask. The contents are slowly heated to a temperature of 135°C over an hour period. The contents are cooled and then heated to 110°C and a solution of 51 grams of sodium hydroxide in 260 milliliters of methanol are added over a 70-minute period at 110°C. An additional 140 milliliters of aliphatic thinner of the type described in Example 3 are then added to the flask and the tem-

16

perature of the contents raised to 150°C. The product is then filtered and stripped in the manner described in Example 8. A total of 364 grams of sodium metaborate product is recovered. Analysis of the product reveals an alkalinity value of 386 mg KOH/g and a boron content of 8.06 weight percent.

## EXAMPLE 12

This example is presented to illustrate the preparation of a metaborate dispersion. A 2-liter flask is charged with 132 grams of a sodium sulfonate solution prepared by neutralizing a sulfonated 480 neutral oil in a manner described in Example 3. The sodium sulfonate solution contains 38 weight percent sodium sulfonate and 62 weight percent butanol. 133 grams of RPM Hydrocarbon Neutral Oil having a viscosity of 126 SUS at 100°F are added to the flask. The contents of the flask are stripped to 165°C and 600 mm mercury vacuum and 81 grams are taken off overhead. Thereafter, 17 grams of a polyisobutenyl succinimide dispersant (formed by the reaction of polyisobutenyl succinic anhydride with tetraethylene pentamine with the succinimide product containing 2.2 percent nitrogen) and 750 milliliters of an aliphatic diluent of the type described in Example 3 are added to the flask. The contents are heated to 110°C under vigorous agitation and a solution of 50 grams of sodium hydroxide in 250 milliliters of methanol are slowly added over an 80-minute period at 110°C. Simultaneously with the addition of the sodium hydroxide solution, carbon dioxide is bubbled into the flask contents at a rate equivalent to that of the sodium hydroxide addition. After 28 grams of carbon dioxide are absorbed, the flask contents are heated to 150°C and 455 milliliters are taken off overhead.

The contents are cooled to 60°C and two aliquots of 78 grams of boric acid are added (a total of 156 grams are added). The temperature is raised to 125°C then cooled to 60°C after the first aliquot and the temperature raised to 135°C after the addition of the second aliquot. A slight vacuum is applied and a total of 161 milliliters are taken off overhead. The resulting product is a tetraborate.

To convert the tetraborate to a metaborate, the flask contents are diluted with 250 milliliters of the aliphatic diluent described above and heated to 110°C. Thereafter, a solution of 50 grams of sodium hydroxide in 250 milliliters of methanol are slowly added over an eighty-minute period at 110°C.

The temperature is raised to 150°C and thereafter the reaction mixture is cooled to 75°C. The product is filtered and then stripped to 160°C at 5 mm Hg. pressure.

382 grams of product are recovered which upon analysis revealed

Nitrogen Content	(wt %)	0.089
CO <sub>2</sub>	(wt %)	0.04
Boron	(wt %)	7.68
Alkalinity value		355 mg KOH/gm

## EXAMPLE 13

The procedure of Example 12 is repeated except that the 132 grams of sodium sulfonate solution is replaced by 50 grams of a calcium sulfonate prepared by the method of Example 3. The calcium sulfonate is overbased by the procedure of Example 3 and reacted with

boric acid to form the tetraborate and thereafter converted to the metaborate. A total of 365 grams of product is recovered and analysis reveals the following:

Nitrogen Content	(wt %)	0.081
CO <sub>2</sub> "	(wt %)	0.17
Calcium "	(wt %)	0.05
Boron "	(wt %)	7.15
Alkalinity value		339 mg KOH/gm

#### EXAMPLE 14

This example is presented to illustrate the effectiveness of the instant compositions in lubricating a system under high load conditions. In this example several representative lubricating oils containing the additives of this invention are tested in two extreme pressure tests. In one test, the extreme pressure characteristics are determined by using the well-known "Four-Ball Test" machine. This test is described in ASTM 2266-67 under the following test conditions:

Temperature	130°F
Speed	1200 rpm
Load	20 kg
Duration of Test	1 Hour

In each test, three one-half-inch diameter steel balls

posed of the aforesaid base oil containing a sodium carbonate overbased sulfonate as prepared by Example 6. Test Lubricant C is the aforesaid base oil containing 10 wt. % of a sodium metaborate prepared by the method of U.S. Pat. No. 3,313,727 (Example 1 of patent) and containing a neutral calcium petroleum sulfonate. Test Lubricant D is the aforesaid base oil containing sodium borate prepared by the method of Example 7. Test Lubricant E is the aforesaid base oil containing a sodium borate produced by the method of Example 8. Test Lubricant F is the aforesaid base oil containing a potassium borate produced by the method of Example 9. Test Lubricant G is composed of the aforesaid base oil containing a sodium borate produced by the method of Example 10. Test Lubricant H is the aforesaid base oil containing a sodium borate prepared by the method of Example 12. Test Lubricant I is the aforesaid base oil containing a sodium borate prepared by the method of Example 13. The table following depicts the test results of the above test lubricants in the Four Ball and Timken Tests. Also shown in the table is the compatibility of the metal borate with various additives. In order to pass this test the borate containing oil must not gel in the presence of an equal volume of Chevron Multiservice Gear Lube 90 after 65 hours at 180°F, and must be clear in the presence of 2 wt. % of a conventional sulfurized calcium phenate after 65 hours at 180°F.

TABLE I

Test No.	Test Sample	Borate Type	Extreme Pressure Properties			Timken <sup>(1)</sup> Lbs.	Compatibility Pass/Fail
			Additive Conc. (Wt.%)	Wear	4-Ball Test Weld (Pass Lbs.)		
1.	A	None	—	0.68	130	5	—
2.	B	Na <sub>2</sub> CO <sub>3</sub>	13.5	0.45	—	20	—
3.	C	NaBO <sub>2</sub>	10	0.25	225	100	Fail
4.	D	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub>	8.2	0.51	195	65	Pass
5.	E	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub>	10	0.45	195	100	Pass
6.	F	K <sub>2</sub> B <sub>4</sub> O <sub>7</sub>	8.3	0.40	185	100	—
7.	G	Na <sub>2</sub> O <sub>3</sub> (B <sub>2</sub> O <sub>3</sub> )	10	0.52	190	55	Pass
8.	H	NaBO <sub>2</sub>	10	0.26	255	100	—
9.	I	NaBO <sub>2</sub>	10	0.29	250	100	Pass

are clamped together and immersed in the test lubricant. A fourth ball is then rotated at the selected rpm in contact with the other three balls. The 20-kg load is applied to the rotating ball, forcing the same against the three stationary balls. Test is run for one hour and at the end of this run, the three stationary balls are observed for wear scars. The average scar size in millimeters is reported. The size of the scar is indicative of the extreme pressure character of the lubricant with a smaller scar representing a superior extreme pressure lubricant.

In a second Four-Ball Test, increasing load is applied to the fourth ball until the four balls seize or weld together. The load at the weld point is noted and the load immediately preceding the weld is noted. The higher the load applied to the ball without welding, the better the extreme pressure properties of the lubricant.

Another test which determines the extreme pressure capabilities of the lubricant is the so-called Timken Test. The procedure and mechanics of this test are set forth in ASTM 2782-69T. The Timken Test reveals the antiwear characteristics of the lubricant with a higher load and higher contact pressure indicating the better antiwear properties.

The test lubricants employed in this example include the following:

Test Lubricant A is a base oil comprising SAE 90 Gear Oil without additives. Test Lubricant B is com-

We claim:

1. A process for preparing a particulate alkali metal borate dispersion which comprises contacting an alkali metal carbonate overbased alkali or alkaline earth metal sulfonate with boric acid within a stable inert oleophilic liquid reaction medium, wherein said contacting is conducted at a temperature of 20° to 200°C for a period of 0.5 to 7 hours and wherein the molar ratio of boric acid to alkali metal carbonate is from 1 to 3.

2. The process defined in claim 1 wherein said contacting is conducted at a temperature of 40° to 125°C for a period of 1 to 3 hours.

3. The process defined in claim 2 wherein from one to two molar parts of boric acid are contacted for each molar equivalent part of alkali metal carbonate overbased alkali or alkaline earth metal sulfonate.

4. The process defined in claim 3 wherein said alkali metal carbonate is sodium or potassium carbonate and said alkali or alkaline earth metal sulfonate is sodium, calcium or barium sulfonate.

5. The process defined in claim 4 wherein said particulate alkali metal borate has from 0 to 8 waters of hydration.

6. The process defined in claim 5 wherein said alkali metal borate is a sodium metaborate, wherein said alkali metal carbonate is sodium carbonate, and said

alkali or alkaline earth metal sulfonate is calcium sulfonate.

7. A process for preparing a particulate alkali metal tetraborate dispersion which comprises contacting at a temperature of 20° to 200°C for a period of 0.5 to 7 hours two molar parts of boric acid with each molar equivalent part of an alkali metal carbonate overbased alkali or alkaline earth sulfonate within a stable inert oleophilic liquid reaction medium to form an intermediate alkali metal tetraborate dispersion and thereafter contacting said alkali metal tetraborate with two molar parts of an alkali metal hydroxide per molar part of said alkali metal tetraborate to prepare an alkali metal metaborate dispersion.

8. The process defined in claim 7 wherein said alkali metal carbonate is sodium or potassium carbonate and said alkali or alkaline earth metal sulfonate is sodium, calcium or barium sulfonate.

9. The process defined in claim 8 wherein a lipophilic nonionic surface active agent is also present during the contacting of said alkali metal tetraborate and said alkali metal hydroxide.

10. The process defined in claim 9 wherein said lipophilic nonionic surface active agent is an N-substituted alkyl succinimide derived from reacting alkenyl succinic acid or anhydride with an alkylene polyamine.

11. A particulate dispersion of an alkali metal borate having a particle size below about 0.1 micron wherein said particulate alkali metal borate is prepared by contacting at a temperature of 20° to 200°C and for a period of 0.5 to 7 hours boric acid with an alkali metal carbonate overbased metal sulfonate within a stable inert oleophilic liquid reaction medium wherein the molar ratio of said boric acid to said alkali metal carbonate is from 1 to 3.

12. The composition defined in claim 11 wherein said alkali metal carbonate is sodium or potassium carbonate and wherein said alkali or alkaline earth metal sulfonate is sodium, calcium or barium sulfonate.

13. The composition defined in claim 12 wherein said particulate alkali metal borate has from 0 to 8 waters of hydration.

14. A particulate dispersion of an alkali metal borate prepared by contacting at a temperature of 20° to 200°C and for a period of 0.5 to 7 hours two molar parts of boric acid with each molar equivalent part of an alkali metal carbonate overbased alkali or alkaline earth metal sulfonate within a stable inert oleophilic liquid reaction medium to form an alkali metal tetraborate dispersion which is then reacted with two molar parts of an alkali metal hydroxide per molar part of said

alkali metal tetraborate to form an alkali metal metaborate dispersion.

15. The composition defined in claim 14 wherein said alkali metal carbonate is sodium or potassium carbonate and wherein said alkali or alkaline earth metal sulfonate is sodium, calcium or barium sulfonate.

16. The composition defined in claim 14 wherein said alkali metal carbonate is sodium carbonate and wherein said overbased alkali metal or alkaline earth metal sulfonate is calcium sulfonate.

17. Composition defined in claim 15 wherein an N-substituted alkenyl succinimide derived from the reaction of alkenyl succinic acid or anhydride with an alkylene polyamine is present in said stable inert oleophilic liquid reaction medium.

18. A lubricating composition comprising a major portion of a hydrocarbon oil of lubricating viscosity and from 0.1 to 20 weight percent of particulate alkali metal borate dispersed in said oil, said alkali metal borate being prepared by reacting at a temperature of 20° to 200°C and for a period of 0.5 to 7 hours an alkali metal carbonate overbased alkali or alkaline earth metal sulfonate with boric acid, wherein the molar ratio of said boric acid to said alkaline metal carbonate is from 1 to 3.

19. A lubricating composition defined in claim 18 wherein said alkali metal carbonate is sodium or potassium carbonate and said alkali or alkaline earth metal sulfonate is sodium, calcium or barium sulfonate.

20. The composition defined in claim 18 wherein said particulate alkali metal borate has a particle size of less than 0.1 microns.

21. A grease composition comprising a major portion of a lubricating oil, from 5 to 25 weight percent of an organic or metal organic thickener selected from the group consisting of polyurea, alkali metal terephthalate, lithium hydroxy stearate, calcium complex soap and aluminum complex soap, and from 0.1 to 20 weight percent of an alkali metal borate prepared by reacting at a temperature of 20° to 200°C and for a period of 0.5 to 7 hours an alkali metal carbonate overbased alkali or alkaline earth metal sulfonate with boric acid, wherein the molar ratio of said boric acid to said alkali metal carbonate is from 1 to 3.

22. The grease composition defined in claim 21 wherein said alkali metal carbonate is sodium carbonate and said alkali or alkaline earth metal sulfonate is sodium, calcium or barium sulfonate.

23. The grease composition defined in claim 22 wherein said organic or metal organic thickener is a polyurea thickener.

\* \* \* \* \*

55

60

65