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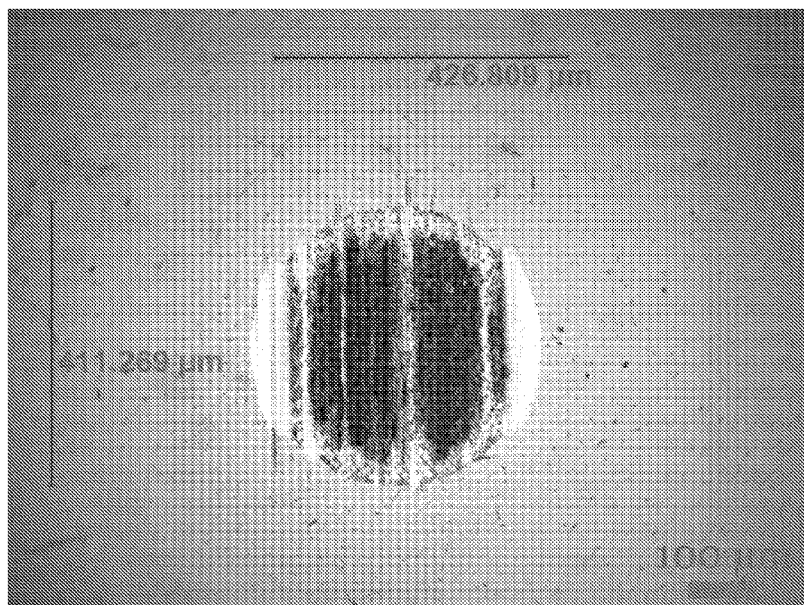


FIG. 8

(57) Abstract: Boron soap compounds are provided. Lubricating grease compositions are provided. The lubricating grease compositions include a lubricating oil and at least one boron soap compound. The lubricating grease compositions can optionally include one or more additive.

BORON SOAP COMPOUNDS FOR LUBRICATING GREASE COMPOSITIONS

CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] This application claims priority to U.S. Provisional Application No. 62/221,745 filed on September 22, 2015, which is incorporated fully herein by reference.

TECHNICAL FIELD

[0002] This disclosure relates to boron soap compounds. The compounds can be used to prepare lubricating grease compositions.

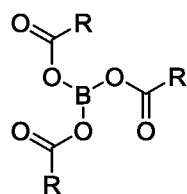
BACKGROUND

[0003] Lubricating grease compositions typically include a thickening agent, additives, and a base oil, or lubricating oil. The thickening agent may be any material that, in combination with the lubricating oil, will produce a solid to semi-fluid structure. Common thickening agents include simple and complex soaps such as lithium, calcium, sodium, and aluminum soaps. These thickening agents generally do not reduce friction and wear of tribological contacts when employed in lubricating grease compositions. Additives may be added to a lubricant to modify its properties. Typical additives are solid lubricants, antioxidants, corrosion inhibitors, anti-wear, and extreme pressure additives. Solid lubricant additives such as graphite, molybdenum disulfide, tungsten disulfide, polytetrafluoroethylene, boron nitride, and boric acid may be added to grease compositions to impart friction reducing and wear reducing properties.

SUMMARY

[0004] The invention provides a soap thickening agent that reduces wear and friction at the interface without the aid of an additive. Since fewer components in a lubricating grease results in less competition for the limited surface sites it may be more efficient. The improved thickening agent for lubricating grease compositions may provide friction reducing and anti-wear properties to the lubricating grease without using additional additives.

[0005] In one aspect, disclosed is a compound of formula (I)



(I),

wherein each R is independently alkyl, alkenyl or aryl, and each R is substituted or unsubstituted; and wherein two R groups together may form a ring.

[0006] Compositions comprising compounds of formula (I), and methods and processes for making and using the compounds and compositions are further described herein.

BRIEF DESCRIPTION OF THE DRAWINGS

[0007] FIG. 1 shows a temperature vs. time plot of a low temperature process for producing disclosed boron soap greases.

[0008] FIG. 2 shows a temperature vs. time plot of a high temperature process for producing disclosed boron soap greases.

[0009] FIG. 3 shows cone penetration analysis for disclosed boron soap greases.

[0010] FIG. 4 shows infrared analysis for a disclosed boron soap grease.

[0011] FIG. 5 shows cone and plate rheological analysis for disclosed simple and complex boron soap greases.

[0012] FIG. 6 shows wear test results of a disclosed boron soap grease comparatively to other lubricants.

[0013] FIG. 7 shows an image of a ball wear test conducted with a base oil sans the disclosed boron soap compounds.

[0014] FIG. 8 shows an image of a ball wear test conducted with a disclosed boron soap grease.

DETAILED DESCRIPTION

[0015] The present disclosure relates to boron soap compounds, lubricating grease compositions comprising the boron soap compounds, methods for preparing the compounds and compositions, and methods for using the compositions. The boron soap compounds can have formula (I). The lubricating grease compositions include at least one compound of

formula (I) and a lubricating oil. The lubricating grease compositions may also include one or more additives.

[0016] Compounds of formula (I) may be useful components in the lubricating grease composition due to their advantageous lubricating properties. For example, compounds of formula (I) may impart anti-wear and anti-fretting properties to the lubricating grease compositions that are advantageous over existing thickeners and lubricants. Aluminum-based soaps known within the art fail to have this dual function of thickener and anti-wear agent. This is because the decomposition products of aluminum grease are abrasive aluminum oxides. In contrast to the aluminum greases, the disclosed boron compounds of formula (I) may decompose into boric acid or other boron compounds that may be lubricious. Accordingly, the compounds of formula (I) may act as a thickening agent and a lubricant in the compositions, thereby obviating the need for both a thickening agent and a lubricant additive common in existing lubricating compositions, or simply decreasing the need for high percentages of additives within the lubricating compositions. Alternatively, compounds of formula (I) may be combined with additional thickening agents to create compositions that possesses advantageous properties over existing lubricating compositions.

[0017] The combination of the components of the lubricating grease composition of the present disclosure can result in lubricating grease compositions in which the individual components provide compositions with sufficient friction reduction and viscosity that results in a better than expected improvement in lubricating capability.

1. Definitions

[0018] Unless otherwise defined, all technical and scientific terms used herein have the same meaning as commonly understood by one of ordinary skill in the art. In case of conflict, the present document, including definitions, will control. Preferred methods and materials are described below, although methods and materials similar or equivalent to those described herein can be used in practice or testing of the present invention. All publications, patent applications, patents and other references mentioned herein are incorporated by reference in their entirety. The materials, methods, and examples disclosed herein are illustrative only and not intended to be limiting.

[0019] The terms “comprise(s),” “include(s),” “having,” “has,” “can,” “contain(s),” and variants thereof, as used herein, are intended to be open-ended transitional phrases, terms, or words that do not preclude the possibility of additional acts or structures. The singular forms “a,” “an” and “the” include plural references unless the context clearly dictates otherwise.

The present disclosure also contemplates other embodiments “comprising,” “consisting of” and “consisting essentially of,” the embodiments or elements presented herein, whether explicitly set forth or not.

[0020] The conjunctive term “or” includes any and all combinations of one or more listed elements associated by the conjunctive term. For example, the phrase “an apparatus comprising A or B” may refer to an apparatus including A where B is not present, an apparatus including B where A is not present, or an apparatus where both A and B are present. The phrases “at least one of A, B, . . . and N” or “at least one of A, B, . . . N, or combinations thereof” are defined in the broadest sense to mean one or more elements selected from the group comprising A, B, . . . and N, that is to say, any combination of one or more of the elements A, B, . . . or N including any one element alone or in combination with one or more of the other elements which may also include, in combination, additional elements not listed.

[0021] The modifier “about” used in connection with a quantity is inclusive of the stated value and has the meaning dictated by the context (for example, it includes at least the degree of error associated with the measurement of the particular quantity). The modifier “about” should also be considered as disclosing the range defined by the absolute values of the two endpoints. For example, the expression “from about 2 to about 4” also discloses the range “from 2 to 4.” The term “about” may refer to plus or minus 10% of the indicated number. For example, “about 10%” may indicate a range of 9% to 11%, and “about 1” may mean from 0.9-1.1. Other meanings of “about” may be apparent from the context, such as rounding off, so, for example “about 1” may also mean from 0.5 to 1.4.

[0022] The term “additive” as used herein, means any substance added to a lubricating grease composition to modify its properties. For example, the additives may be solid lubricants, thickening agents, antioxidants, corrosion inhibitors, anti-wear, and extreme pressure additives.

[0023] The term “alkyl” as used herein, means a straight or branched, saturated hydrocarbon chain containing from 1 to 30 carbon atoms. The term “lower alkyl” or “C₁-C₆ alkyl” means a straight or branched chain hydrocarbon containing from 1 to 6 carbon atoms. The term “C₃-C₇ branched alkyl” means a branched chain hydrocarbon containing from 3 to 7 carbon atoms. The term “C₁-C₄ alkyl” means a straight or branched chain hydrocarbon containing from 1 to 4 carbon atoms. The term “C₆-C₃₀ alkyl” means a straight or branched chain hydrocarbon containing from 6 to 30 carbon atoms. The term “C₁₂-C₁₈ alkyl” means a straight or branched chain hydrocarbon containing from 12 to 18 carbon atoms.

Representative examples of alkyl include, but are not limited to, methyl, ethyl, *n*-propyl, *iso*-propyl, *n*-butyl, *sec*-butyl, *iso*-butyl, *tert*-butyl, *n*-pentyl, isopentyl, neopentyl, *n*-hexyl, 3-methylhexyl, 2,2-dimethylpentyl, 2,3-dimethylpentyl, *n*-heptyl, *n*-octyl, *n*-nonyl, *n*-decyl and *n*-dodecyl.

[0024] The term "alkenyl" as used herein, means a straight or branched, unsaturated hydrocarbon chain containing at least one carbon-carbon double bond and from 2 to 30 carbon atoms. The term "lower alkenyl" or "C₂-C₆ alkenyl" means a straight or branched chain hydrocarbon containing at least one carbon-carbon double bond and from 1 to 6 carbon atoms. The term "C₆-C₃₀ alkenyl" means a straight or branched chain hydrocarbon containing at least one carbon-carbon double bond and from 6 to 30 carbon atoms. The term "C₁₂-C₁₈ alkyl" means a straight or branched chain hydrocarbon containing at least one carbon-carbon double bond and from 12 to 18 carbon atoms. The alkenyl groups, as used herein, may have 1, 2, 3, 4, or 5 carbon-carbon double bonds. The carbon-carbon double bonds may be *cis* or *trans* isomers.

[0025] The term "aryl" as used herein, refers to a phenyl group, or bicyclic aryl or tricyclic aryl fused ring systems. Bicyclic fused ring systems are exemplified by a phenyl group appended to the parent molecular moiety and fused to a phenyl group. Tricyclic fused ring systems are exemplified by a phenyl group appended to the parent molecular moiety and fused to two other phenyl groups. Representative examples of bicyclic aryls include, but are not limited to, naphthyl. Representative examples of tricyclic aryls include, but are not limited to, anthracenyl. The monocyclic, bicyclic, and tricyclic aryls are connected to the parent molecular moiety through any carbon atom contained within the rings, and can be unsubstituted or substituted.

[0026] The term "hydroxyl" as used herein, means -OH.

[0027] The term "amino" as used herein, means -NH₂.

[0028] The term "carboxyl" as used herein, means a carboxylic acid group, or -COOH.

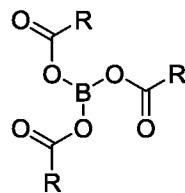
[0029] The term "alkylcarbonyl" as used herein, means an alkyl group, as defined herein, appended to the parent molecular moiety through a carbonyl.

[0030] The term "alkenylcarbonyl" as used herein, means an alkenyl group, as defined herein, appended to the parent molecular moiety through a carbonyl.

[0031] For the recitation of numeric ranges herein, each intervening number there between with the same degree of precision is explicitly contemplated. For example, for the range of 6-9, the numbers 7 and 8 are contemplated in addition to 6 and 9, and for the range 6.0-7.0, the number 6.0, 6.1, 6.2, 6.3, 6.4, 6.5, 6.6, 6.7, 6.8, 6.9, and 7.0 are explicitly contemplated.

2. The Compound of Formula (I)

[0032] In one aspect, disclosed are compounds of formula (I):



(I),

wherein each R is independently alkyl, alkenyl or aryl, and each R is substituted or unsubstituted; and wherein two R groups together may form a ring.

[0033] In certain embodiments, each R is independently alkyl, alkenyl or aryl, and each R is independently substituted with 0-3 substituents selected from the group consisting of hydroxyl, amino, carboxyl and phenyl.

[0034] In certain embodiments, R is alkyl or alkenyl, and R is substituted with 0-3 substituents selected from the group consisting of hydroxyl, amino, carboxyl and phenyl.

[0035] In certain embodiments, R is alkyl or alkenyl, and R is substituted with 0-3 hydroxyl groups.

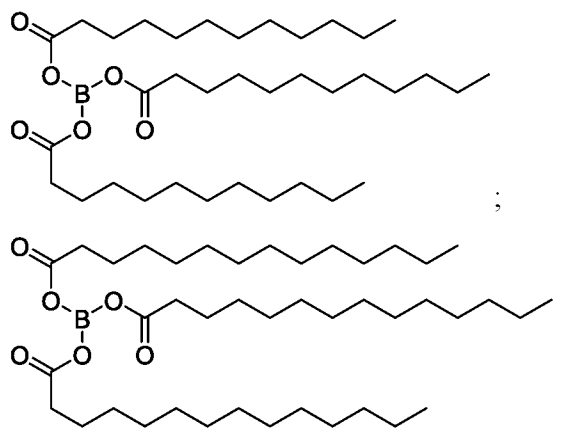
[0036] In certain embodiments, R is C₆-C₃₀ alkyl or C₆-C₃₀ alkenyl, and R is substituted with 0-3 hydroxyl groups.

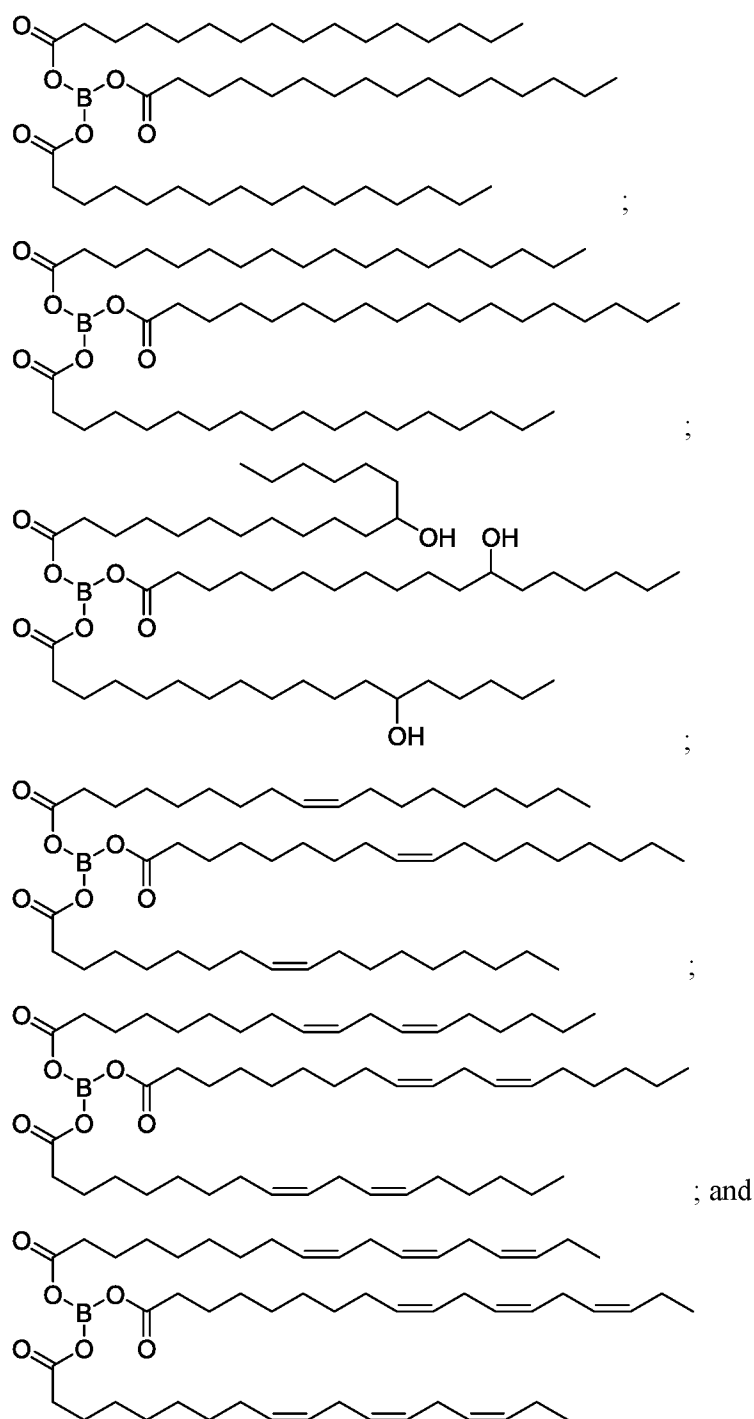
[0037] In certain embodiments, R is C₁₂-C₁₈ alkyl or C₁₂-C₁₈ alkenyl, and R is substituted with 0-3 hydroxyl groups.

[0038] In certain embodiments, R is C₆-C₃₀ alkyl substituted with 0-3 hydroxyl groups.

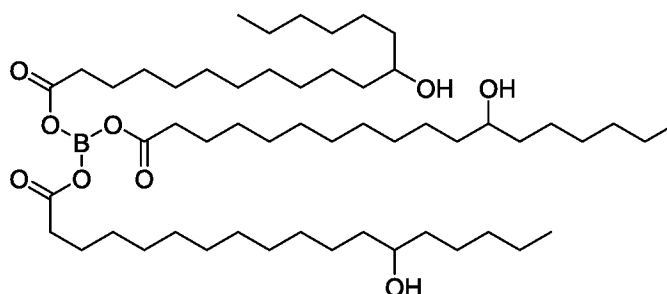
[0039] In certain embodiments, R is C₁₂-C₁₈ alkyl substituted with 0-3 hydroxyl groups.

[0040] In certain embodiments, the compound of formula (I) is selected from the group consisting of:



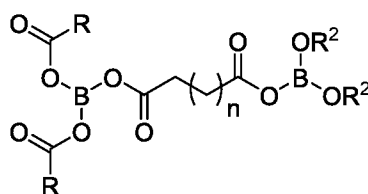


[0041] In an exemplary embodiment, the compound of formula (I) is:



[0042] In certain embodiments, R is substituted with a carboxyl group.

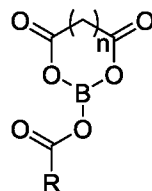
[0043] In certain embodiments, R is substituted with a carboxyl group which is attached to a boron atom. For example, the compound of formula (I) may be the compound of formula (I-a),



(I-a),

wherein R is as defined in any embodiment herein, n is an integer greater than 0, and R² is hydrogen, alkyl, alkylcarbonyl, or alkenylcarbonyl.

[0044] In certain embodiments, two R groups together may form a ring. For example, the compound of formula (I) may be the compound of formula (I-b),



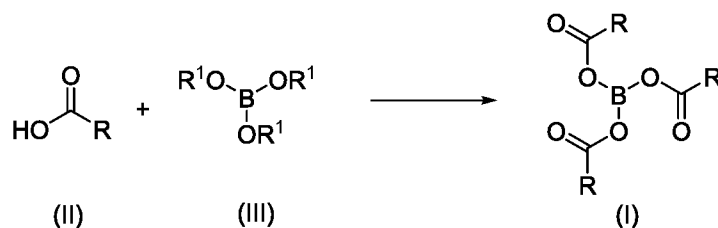
(I-b),

wherein R is as defined in any embodiment herein, and n is an integer greater than 0.

A. Synthesis of the Compound of Formula (I)

[0045] Compounds of formula (I) may be prepared by synthetic processes. Compounds of formula (I), wherein R has the meanings as set forth in the Summary of the Invention unless otherwise noted, can be synthesized as shown in Scheme 1.

Scheme 1. Synthesis of the compound of Formula (I)



[0046] Scheme 1 illustrates that compounds of formula (I) can be prepared by reacting the compound of formula (II) with the compound of formula (III).

[0047] In certain embodiments, reacting the compound of formula (II) with the compound of formula (III) may include employing a solvent as a component of the reaction mixture. The solvent may be any solvent suitable to dissolve the starting materials and promote the reaction to proceed to the desired product (e.g. a compound of formula (I)).

[0048] In certain embodiments, the solvent is a lubricating oil. The lubricating oil may be a mineral oil, poly-alpha-olefin, ester, perfluoropolyether, perfluorinated hydrocarbons, multiply alkylated cyclopentanes, or silicone oil.

[0049] In certain embodiments, the method may further comprise adding an additional solvent. In certain embodiments, the additional solvent may be an organic solvent. In certain embodiments, the additional solvent may be water.

[0050] In certain embodiments, reacting the compound of formula (II) with the compound of formula (III) may include heating the reaction. Heating the reaction may be useful for the reaction to proceed to completion and generate the compound of formula (I). The reaction may be heated at a temperature of about 30°C to about 200°C, about 30°C to about 190°C, about 30°C to about 180°C, about 30°C to about 170°C, about 30°C to about 160°C, about 30°C to about 150°C, about 30°C to about 140°C, about 30°C to about 130°C, about 30°C to about 120°C, about 30°C to about 110°C, about 30°C to about 100°C, about 30°C to about 90°C, about 30°C to about 80°C, about 30°C to about 70°C, about 30°C to about 60°C, about 30°C to about 50°C, about 30°C to about 40°C, about 100°C to about 200°C, about 100°C to about 150°C, about 120°C to about 180°C, or about 140°C to about 160°C.

[0051] In certain embodiments, the reaction may be heated at a temperature of greater than about 100°C, greater than about 110°C, greater than about 120°C, greater than about 130°C, greater than about 140°C, greater than about 150°C, greater than about 160°C, greater than about 170°C, greater than about 180°C, greater than about 190°C, or greater than about 200°C.

[0052] In certain embodiments, the synthesis of the compound of formula (I) comprises the synthesis of the compound of formula (I-a). In certain embodiments, the synthesis of the compound of formula (I) comprises the synthesis of the compound of formula (I-b).

[0053] In certain embodiments, the reaction conditions may be modified to form the compound of formula (I-a) or the compound of formula (I-b).

[0054] The compounds and intermediates may be isolated and purified by methods well-known to those skilled in the art of organic synthesis. Examples of conventional methods for isolating and purifying compounds can include, but are not limited to, chromatography on solid supports such as silica gel, alumina, or silica derivatized with alkylsilane groups, by recrystallization at high or low temperature with an optional pretreatment with activated carbon, thin-layer chromatography, distillation at various pressures, sublimation under vacuum, and trituration, as described for instance in "Vogel's Textbook of Practical Organic Chemistry", 5th edition (1989), by Furniss, Hannaford, Smith, and Tatchell, pub. Longman Scientific & Technical, Essex CM20 2JE, England.

[0055] Reaction conditions and reaction times for each individual step can vary depending on the particular reactants employed and substituents present in the reactants used. Specific procedures are provided in the Examples section. Reactions can be worked up in the conventional manner, e.g. by eliminating the solvent from the residue and further purified according to methodologies generally known in the art such as, but not limited to, crystallization, distillation, extraction, trituration and chromatography. Unless otherwise described, the starting materials and reagents are either commercially available or can be prepared by one skilled in the art from commercially available materials using methods described in the chemical literature. Starting materials, if not commercially available, can be prepared by procedures selected from standard organic chemical techniques, techniques that are analogous to the synthesis of known, structurally similar compounds, or techniques that are analogous to the above described schemes or the procedures described in the synthetic examples section.

[0056] Routine experimentations, including appropriate manipulation of the reaction conditions, reagents and sequence of the synthetic route, protection of any chemical functionality that cannot be compatible with the reaction conditions, and deprotection at a suitable point in the reaction sequence of the method are included in the scope of the invention. Suitable protecting groups and the methods for protecting and deprotecting different substituents using such suitable protecting groups are well known to those skilled in the art; examples of which can be found in PGM Wuts and TW Greene, in Greene's book

titled Protective Groups in Organic Synthesis (4th ed.), John Wiley & Sons, NY (2006), which is incorporated herein by reference in its entirety. Synthesis of the compounds of the invention can be accomplished by methods analogous to those described in the synthetic schemes described hereinabove and in specific examples.

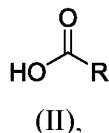
[0057] When an optically active form of a disclosed compound is required, it can be obtained by carrying out one of the procedures described herein using an optically active starting material (prepared, for example, by asymmetric induction of a suitable reaction step), or by resolution of a mixture of the stereoisomers of the compound or intermediates using a standard procedure (such as chromatographic separation, recrystallization or enzymatic resolution).

[0058] Similarly, when a pure geometric isomer of a compound is required, it can be obtained by carrying out one of the above procedures using a pure geometric isomer as a starting material, or by resolution of a mixture of the geometric isomers of the compound or intermediates using a standard procedure such as chromatographic separation.

[0059] It can be appreciated that the synthetic schemes and specific examples as described are illustrative and are not to be read as limiting the scope of the invention as it is defined in the appended claims. All alternatives, modifications, and equivalents of the synthetic methods and specific examples are included within the scope of the claims.

1) Compound of Formula (II)

[0060] The synthesis of compounds of formula (I) may employ compounds of formula (II):



wherein each R is independently alkyl, alkenyl or aryl, and each R is independently substituted or unsubstituted.

[0061] In certain embodiments, R is alkyl, alkenyl or aryl, and R is substituted with 0-3 substituents selected from the group consisting of hydroxyl, amino, carboxyl and phenyl.

[0062] In certain embodiments, R is alkyl or alkenyl, and R is substituted with 0-3 hydroxyl groups.

[0063] In certain embodiments, R is C₆-C₃₀ alkyl or C₆-C₃₀ alkenyl, and R is substituted with 0-3 hydroxyl groups.

[0064] In certain embodiments, R is C₆-C₃₀ alkyl or C₆-C₃₀ alkenyl, and R is substituted with 0-3 hydroxyl groups.

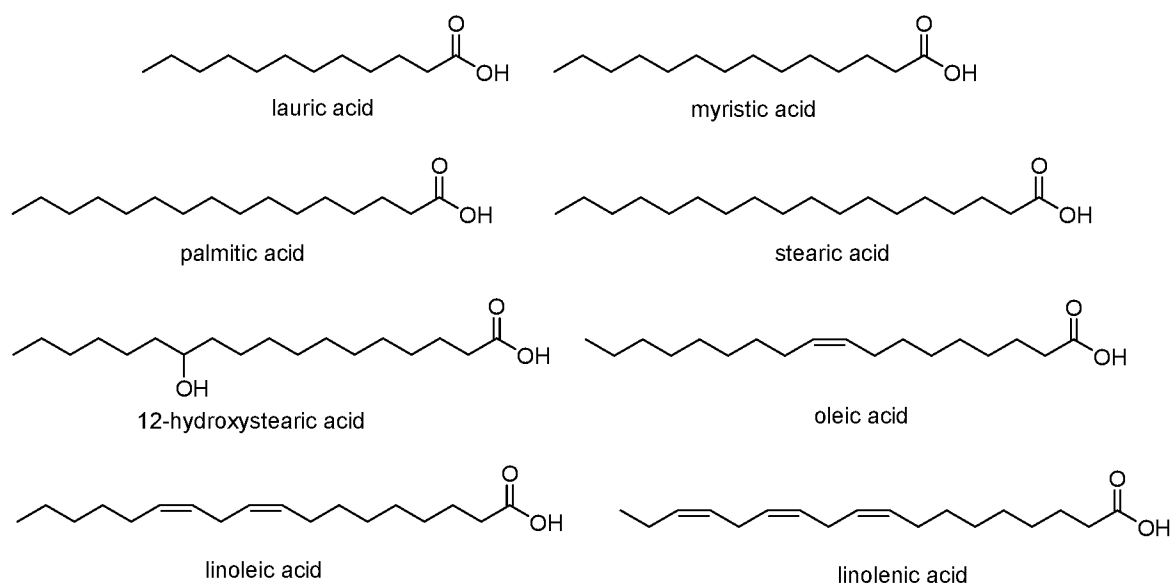
[0065] In certain embodiments, R is C₁₂-C₁₈ alkyl or C₁₂-C₁₈ alkenyl, and R is substituted with 0-3 hydroxyl groups.

[0066] In certain embodiments, R is C₆-C₃₀ alkyl substituted with 0-3 hydroxyl groups.

[0067] In certain embodiments, R is C₁₂-C₁₈ alkyl substituted with 0-3 hydroxyl groups.

[0068] In certain embodiments, the compound of formula (II) is benzoic acid or substituted benzoic acid.

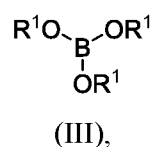
[0069] In certain embodiments, the compound of formula (II) is a fatty acid. Fatty acids are typically major components of soaps. Examples of naturally occurring fatty acids that may be used herein as the compound of formula (II) include lauric acid, myristic acid, palmitic acid, stearic acid, 12-hydroxystearic acid, oleic acid, linoleic acid and linolenic acid:



[0070] In an exemplary embodiment, the compound of formula (II) is 12-hydroxystearic acid.

2) Compound of Formula (III)

[0071] The synthesis of compounds of formula (I) may employ compounds of formula (III):



wherein R¹ is alkyl, aryl or hydrogen.

[0072] In certain embodiments, R¹ is alkyl.

[0073] In certain embodiments, R¹ is C₁-C₆ alkyl.

[0074] In certain embodiments, R¹ is C₁-C₄ alkyl.

[0075] In certain embodiments, R¹ is aryl.

[0076] In an exemplary embodiment, the compound of formula (III) is triisopropyl borate.

3. Lubricating Grease Compositions

[0077] Disclosed are lubricating grease compositions. In certain embodiments, the lubricating grease compositions include a lubricating oil and at least one compound of formula (I). The lubricating grease may have improved anti-wear and anti-fretting properties over existing lubricating greases.

[0078] In certain embodiments, the lubricating grease compositions may comprise, by weight, about 50% to about 99% of a lubricating oil and about 1% to about 50% of at least one compound of formula (I).

[0079] In certain embodiments, the lubricating grease compositions include one compound of formula (I). In certain embodiments, the lubricating grease composition includes more than one compound of formula (I). In certain embodiments, the lubricating grease composition includes 1, 2, 3, 4 or 5 compounds of formula (I).

[0080] The lubricating grease compositions may comprise, by weight, about 1% to about 50%, about 1% to about 45%, about 1% to about 40%, about 1% to about 35%, about 1% to about 30%, about 1% to about 25%, about 1% to about 20%, about 1% to about 15%, about 1% to about 10%, about 1% to about 5%, or about 1% to about 3% of at least one compound of formula (I). The lubricating compositions may comprise, by weight, about 1%, about 2%, about 3%, about 4%, about 5%, about 6%, about 7%, about 8%, about 9%, about 10%, about 15%, about 20%, about 25%, about 30%, about 35%, about 40%, about 45%, or about 50% of at least one compound of formula (I).

[0081] In certain embodiments, the lubricating grease compositions may comprise at least one compound of formula (I), wherein all of the R substituents are the same, (e.g., a simple lubricating grease composition). In other embodiments the lubricating grease compositions may comprise at least one compound of formula (I), wherein all of the R substituents are not the same, (e.g., a complex lubricating grease composition).

[0082] In certain embodiments, the lubricating grease compositions may further comprise one or more additives. The additives are different from the compound of formula (I). Additives may be selected from the group consisting of lithium soaps, calcium soaps, sodium soaps, aluminum soaps, polyurea, calcium sulfonate, clay, polytetrafluoroethylene, graphite, boron nitride and boric acid, or a combination thereof.

[0083] The lubricating grease compositions can be provided in the form of a liquid, a solid, a suspension, a coating, or a powder.

A. Lubricating Oil

[0084] The lubricating grease compositions include at least one lubricating oil. The lubricating oil is typically in the form of a viscous liquid. The oil may be miscible with the compound of formula (I) such that the lubricating grease is a homogeneous composition. The lubricating oil may be a mineral oil, poly-alpha-olefins, esters, perfluorinated hydrocarbons, perfluoropolyether, multiply alkylated cyclopentanes, or silicone oil.

[0085] The lubricating grease compositions may comprise, by weight, about 50% to about 99%, about 55% to about 99%, about 60% to about 99%, about 65% to about 99%, about 70% to about 99%, about 75% to about 99%, about 80% to about 99%, about 85% to about 99%, about 90% to about 99%, about 95% to about 99%, or about 97% to about 99% of the lubricating oil. The lubricating composition may comprise, by weight, about 50%, about 55%, about 60%, about 65%, about 70%, about 75%, about 80%, about 85%, about 90%, about 91%, about 92%, about 93%, about 94%, about 95%, about 96%, about 97%, about 98%, or about 99% of the lubricating oil.

B. Preparing the Lubricating Grease Compositions

[0086] The lubricating grease compositions may be prepared by methods that also synthesize compounds of formula (I) by reacting the compound of formula (II) with the compound of formula (III) as shown in Scheme 1. The compounds of formula (II) and formula (III) may be any embodiment described herein.

[0087] The methods of preparing the lubricating grease compositions may include combining a lubricating oil with the compound of formula (II) and the compound of formula (III) to form a first mixture.

[0088] The method may further comprise adding a solvent to the first mixture. In certain embodiments, the solvent may be an organic solvent. In certain embodiments, the solvent may be water.

[0089] The first mixture may be heated to form a second mixture. The first mixture may be heated at a temperature of about 30°C to about 200°C, about 30°C to about 190°C, about 30°C to about 180°C, about 30°C to about 170°C, about 30°C to about 160°C, about 30°C to about 150°C, about 30°C to about 140°C, about 30°C to about 130°C, about 30°C to about 120°C, about 30°C to about 110°C, about 30°C to about 100°C, about 30°C to about 90°C, about 30°C to about 80°C, about 30°C to about 70°C, about 30°C to about 60°C, about 30°C

to about 50°C, about 30°C to about 40°C, about 100°C to about 200°C, about 100°C to about 150°C, about 120°C to about 180°C, or about 140°C to about 160°C.

[0090] In certain embodiments, the mixture may be heated at a temperature of greater than about 100°C, greater than about 110°C, greater than about 120°C, greater than about 130°C, greater than about 140°C, greater than about 150°C, greater than about 160°C, greater than about 170°C, greater than about 180°C, greater than about 190°C, or greater than about 200°C.

[0091] The second mixture may be treated in any manner known in the art to remove byproducts or excess solvents and form the lubricating grease composition. The byproducts and solvents may be volatile and their removal may be achieved by distillation or evaporation. For example, when triisopropyl borate is the compound of formula (III), isopropanol is formed as a byproduct in the formation of the lubricating grease composition. Isopropanol may then be removed by evaporation from the mixture to form the lubricating grease composition.

[0092] In certain embodiments, additional lubricating oil may be added to form a desired concentration of the final components of the lubricating grease composition.

[0093] In certain embodiments, additional lubricating oil may be added to provide the lubricating grease composition with a desired consistency.

[0094] The method may further comprise adding an additive. The additive may be added to the second mixture before removal of any byproducts or the additive may be added to the composition after removal of any byproducts. The additive may be selected from the group consisting of lithium soaps, calcium soaps, sodium soaps, aluminum soaps, polyurea, calcium sulfonate, clay, polytetrafluoroethylene, graphite, boron nitride and boric acid, or a combination thereof.

C. Properties of the Lubricating Grease Composition

[0095] The lubricating grease compositions may have a combination of desired properties. The lubricating grease compositions may reduce friction and wear of tribological contacts when employed in typical lubricating applications. For example, the compositions may have improved consistency, mechanical stability, dropping point, age hardening, volatility, oil bleed, corrosion prevention, oxidation stability, water content, fretting wear, friction and wear, and EHL film thickness properties.

1) Consistency

[0096] Consistency is the degree to which a plastic material resists deformation under the application of force. In the case of lubricating greases, this is a measure of the relative

hardness or softness and has some relation to flow and dispensing properties. Consistency of the lubricating grease compositions may be measured by ASTM D217, Cone Penetration of Lubricating Grease (60 strokes) and may be reported in terms of NLGI grade. The lubricating grease compositions may have an NLGI grade of 000, 00, 0, 1, 2, 3, 4, 5 or 6, according to ASTM D 217.

2) Mechanical Stability

[0097] The mechanical stability of the lubricating grease composition is a measure of how the grease consistency will change in service when it is subjected to mechanical stress (shear) resulting from the churning action caused by moving elements or vibrations generated by, or external to, the application. Grease softening in a bearing may eventually cause grease to leak from the housing, requiring maintenance and frequent grease replenishment to avoid premature failure resulting from lack of lubricant on the rolling elements. Mechanical stability may be measured using the ASTM D217 prolonged worker test (100,000 double strokes), or the ASTM D1831 Roll Stability test. ASTM D1831 subjects the grease to shearing by rotating a cylinder containing a 5kg roller at 165 rpm for 2 hours. The change in penetration at the end of the tests is a measure of the mechanical stability. This test produces low shearing forces approximately equal to those found in the grease worker used for ASTM D217. Accordingly, the mechanical stability of the lubricating grease compositions may be determined according to ASTM D217, ASTM D1831, or a combination thereof.

3) Dropping Point

[0098] The dropping point of grease is the temperature at which the thickener loses its ability to maintain the base oil within the thickener matrix. This may be due to the thickener melting or the oil becoming so thin that the surface tension and capillary action become insufficient to hold the oil within the thickener matrix. ASTM D2265 may be used to determine the dropping point of grease. The dropping point of the lubricating grease compositions may be greater than or equal to 100°C, greater than or equal to 120°C, greater than or equal to 140°C, greater than or equal to 160°C, greater than or equal to 180°C, greater than or equal to 200°C, greater than or equal to 220°C, greater than or equal to 240°C, greater than or equal to 260°C, greater than or equal to 280°C, greater than or equal to 300°C, greater than or equal to 320°C, greater than or equal to 340°C, or greater than or equal to 360°C, according to ASTM D2265.

4) Corrosion Prevention

[0099] The Emcor Grease Corrosion Test (ASTM D6138) can evaluate the rust preventive properties of greases on bearing components, measuring the ability of a grease to protect a

bearing against corrosion in the presence of water. The test results are evaluated by scoring samples on a scale of 0-5 as follows: 0 = no corrosion; 1 = trace corrosion; 2 = light corrosion; 3 = moderate corrosion; 4 = heavy corrosion; 5 = severe corrosion. The corrosion prevention ability of the lubricating grease compositions may be measured by employing the Emcor test. The lubricating grease compositions may have an Emcor test score of 0, less than or equal to 1, less than or equal to 2, less than or equal to 3, less than or equal to 4, or less than or equal to 5, according to ASTM D6138.

5) Oil Bleed

[00100] Oil release from greases occurs in two distinct modes: static bleed is the release of oil from the thickener matrix in a package or non-moving part; and dynamic bleed is the controlled release of oil and additives in application as a result of application stresses such as rotation. Without bleed, greases may not provide lubrication for the application as designed. The balance between static and dynamic bleed may determine a grease's performance. For example, elevated temperatures may soften grease and lead to excessive oil release (leakage, evaporation) and premature grease ageing. However, grease with a low oil bleed at high temperature may under-perform at ambient temperature because of oil starvation. ASTM D1742 pressure bleed and ASTM D6184 cone bleed are static bleed tests that can be used to determine static oil bleed. ASTM D4425 Centrifugal Oil Bleed is a dynamic bleed test that can be used to determine dynamic oil bleed. The lubricating grease compositions may have a static oil bleed of less than 99%, less than 80%, less than 70%, less than 60%, less than 50%, less than 45%, less than 40%, less than 30%, less than 20%, less than 15%, less than 10%, less than 9%, less than 8%, less than 7%, less than 6%, less than 5%, less than 4%, less than 3%, less than 2%, or less than 1% at a temperature of about 20°C, about 30°C, about 40°C, about 50°C, about 60°C, about 70°C, about 80°C, about 90°C, about 100°C, about 110°C, about 120°C, about 130°C, about 140°C, about 150°C, about 160°C, about 170°C, about 180°C, about 190°C, or about 200°C, according to ASTM D6184.

6) Oxidation Stability

[00101] Oxidative induction time (OIT) measures the oxidative stability of lubricants. ASTM D5483 can be employed to determine the OIT by use of a pressure differential scanning calorimeter (PDSC), which is a thermal analytical technique for evaluating oxidation-thermal stability of lubricating greases using the differential heat flow between sample reference thermocouples under various temperatures and pressures. Oxidation induction time, as determined under the conditions of this test method, can be used as an indication of oxidation stability of the lubricating grease composition. The oxidation

induction time of the lubricating grease compositions may be greater than or equal to 10 minutes, greater than or equal to 20 minutes, greater than or equal to 50 minutes, greater than or equal to 100 minutes, greater than or equal to 150 minutes, greater than or equal to 200 minutes, greater than or equal to 250 minutes, greater than or equal to 300 minutes, greater than or equal to 350 minutes, greater than or equal to 400 minutes, greater than or equal to 450 minutes, greater than or equal to 500 minutes, greater than or equal to 550 minutes, greater than or equal to 600 minutes, greater than or equal to 650 minutes, greater than or equal to 700 minutes, greater than or equal to 750 minutes, or greater than or equal to 800 minutes, at a temperature of about 20°C, about 30°C, about 40°C, about 50°C, about 60°C, about 70°C, about 80°C, about 90°C, about 100°C, about 110°C, about 120°C, about 130°C, about 140°C, about 150°C, about 160°C, about 170°C, about 180°C, about 190°C, or about 200°C, according to ASTM D 5483.

7) Water Content

[00102] ASTM D4377 is the standard test method for determining the amount of water in crude oils by potentiometric Karl Fischer titration. Employment of the Karl Fischer titration method can determine the amount of water in a grease lubricant. The lubricating grease compositions may comprise, by weight, less than or equal to 2% water, less than or equal to 1.9% water, less than or equal to 1.8% water, less than or equal to 1.7% water, less than or equal to 1.6% water, less than or equal to 1.5% water, less than or equal to 1.4% water, less than or equal to 1.3% water, less than or equal to 1.2% water, less than or equal to 1.1% water, less than or equal to 1.0% water, less than or equal to 0.9% water, less than or equal to 0.8% water, less than or equal to 0.7% water, less than or equal to 0.6% water, less than or equal to 0.5% water, less than or equal to 0.4% water, less than or equal to 0.3% water, less than or equal to 0.2% water, or less than or equal to 0.1% water, according to ASTM D4377.

8) Fretting Wear

[00103] The Fafnir Fretting test (ASTM D4170) is a standardized test for evaluating the anti-wear protection provided by a grease to a ball bearing due to fretting wear or false brinelling. This test provides a method for quantifying grease wear protection and comparing multiple greases. The Fafnir test measures weight loss of the grease. The larger the weight loss, the larger the fretting wear. The lubricating grease compositions may have a weight loss of less than or equal to 5 mg, less than or equal to 4.5 mg, less than or equal to 4.0 mg, less than or equal to 3.5 mg, less than or equal to 3.0 mg, less than or equal to 2.5 mg, less than or equal to 2.0 mg, less than or equal to 1.5 mg, less than or equal to 1.0 mg, less than or equal to 0.9 mg, less than or equal to 0.8 mg, less than or equal to 0.7 mg, less than or equal to 0.6

mg, less than or equal to 0.5 mg, less than or equal to 0.4 mg, less than or equal to 0.3 mg, less than or equal to 0.2 mg, or less than or equal to 0.1 mg according to ASTM D4170.

9) Friction and Wear

[00104] Friction and wear properties of lubricating greases can be determined by ASTM D5707. This test method can determine wear properties and coefficient of friction of lubricating greases at selected temperatures and loads specified for use in applications where high-speed vibrational or start-stop motions are present for extended periods of time under initial high Hertzian point contact pressures. This test method has found application in qualifying lubricating greases used in constant velocity joints of front-wheel-drive automobiles and for lubricating greases used in roller bearings. Accordingly, the wear properties and coefficient of friction of the lubricating grease compositions may be determined according to ASTM D5707.

[00105] In addition, the four ball wear test method (ASTM 2266) and the four ball weld and load wear index (ASTM 2596) can determine the wear preventive characteristics of the lubricating grease compositions in sliding applications.

10) EHL Film Thickness

[00106] Elastohydrodynamic lubrication (EHL) occurs when lubricant film pressure is high enough to significantly influence the shape of the film thickness profile. EHL conditions are typically obtained in nonconformal contacts such as ball on cylinder (elliptical contact), ball on ball (circular contact), and cylinder on cylinder (line contact). EHL film thickness is the separation between surfaces of an elastohydrodynamically lubricated contact. The thickness is typically described by a minimum value, h_{min} , which is the smallest separation, and a central value, h_{cen} , which is the separation at the center of Hertzian contact zone. The variation of the thickness inside the contact zone is the film thickness profile. The EHL film thickness of the lubricating grease compositions may be about 0.05 to about 5.0 μm , about 0.05 to about 4.0 μm , about 0.05 to about 3.0 μm , about 0.05 to about 2.0 μm , about 0.05 to about 1.0 μm , about 0.05 to about 0.8 μm , about 0.05 to about 0.6 μm , about 0.05 to about 0.5 μm , about 0.05 to about 0.4 μm , about 0.05 to about 0.3 μm , about 0.05 to about 0.2 μm , or about 0.05 to about 0.1 μm .

4. Use of the Lubricating Composition

[00107] The lubricating grease compositions may be used in any setting which requires a lubricant. The lubricating grease compositions may be applied to mechanisms that can only be lubricated infrequently and in which a lubricating oil or liquid would not stay in position.

The lubricating grease compositions may act as sealants to prevent ingress of water and incompressible materials.

[00108] The lubricating grease compositions may be used for lubricating contacting surfaces that are rolling, sliding, or a combination of rolling and sliding. Examples of such contacting surfaces can be found in rolling-element bearings, ball bearings, linear bearings, and linear actuators, pumps, bolts, studs, springs, linear guides, seals, cables, shafts, gears, chains, and joints.

5. Examples

[00109] The present disclosure has multiple aspects, illustrated by the following non-limiting examples.

Example 1. Preparation of boron soap lubricating grease compositions

Preparation 1 – triisopropyl borate

[00110] 510.3 g of ISO VG 68 PAO base oil and 300.5 g 12-hydroxystearic acid (12HSA) was heated to 120°C (250°F). 67.0 g of tri-isopropyl borate (TIPB) was added and the solution allowed to cool to 93°C (200°F). Approximately 40 ml of water was added and the solution stirred for 20 min at this temperature. The solution was then heated to 150°C (300°F). The solution was cooled to room temperature.

[00111] This grease was measured to have a consistency (ASTM D217) of 362 for an NLGI grade of 0. The cone bleed (ASTM D6184) measured 44.6 %.

Preparation 2 – triisopropyl borate/low-temperature

[00112] 750 grams of ISO 150 base oil and 12HSA was heated to reach about 93°C (200°F) to melt the acid. The amount of 12HSA was as follows: 138.5 g 12HSA for 15% thickener content, 172.3 g 12HSA for 18% thickener content, 196.3 g 12HSA for 20% thickener content, and 261.7 g 12HSA for 25% thickener content. At about 93°C (200°F), the TIPB was added slowly to the kettle. The amount of TIPB was as follows: 46.2 g TIPB for 15% thickener content, 57.4 g TIPB for 18% thickener content, 65.4 g TIPB for 20% thickener content, and 87.2 g TIPB for 25% thickener content. Heat was then applied to initiate the temperature ramp up to the about 121°C (250°F) reaction temperature. It is hypothesized that this temperature should dehydrate the mixture and eliminate the isopropyl alcohol reaction product. The kettle foamed during the heating, in part due to TIPB having a boiling point of 137°C (280°F). Once the 121°C temperature stage was reached, the reaction was allowed to

proceed and was considered complete when the foaming stopped. Approximately 10 ml of water was added to complete the reaction and the temperature was held constant until the foaming stopped again. The batch was then cooled to room temperature while being stirred.

[00113] A plot of the temperature vs. time for this preparation is shown in Figure 1.

[00114] Infrared analysis was performed to further characterize the soap making procedure. Infrared analysis measures the absorption of infrared radiation by the sample. Specific bond stretching or bending adsorbs at a characteristic frequency known as an absorption band. The frequency is converted and is reported as a wavenumber in reciprocal cm. Here there is a band around 1332 cm^{-1} that correlates with the bending modes of the BO_3 triangle. The band around 1395 cm^{-1} correlates well with the B-O bond stretching modes in the BO_3 triangle. Infrared analysis is depicted in Figure 4.

Preparation 3 – triisopropyl borate/high-temperature

[00115] 750 grams of ISO 150 base oil and 196.3g 12HSA (20% thickener) was heated to reach about 93°C (200°F) in order to melt the acid. At about 93°C (200°F), the TIPB (65.4 g TIPB for 20% thickener content) was added slowly to the kettle. Heat was then applied to initiate the temperature ramp up to the 150°C (300°F) reaction temperature. It is hypothesized that this temperature should dehydrate the mixture and eliminate the isopropyl alcohol reaction product. The kettle foamed during the heating, in part due to TIPB having a boiling point of 137°C (280°F). Once the 150°C temperature stage was reached the reaction was allowed to proceed and was considered complete when the foaming stopped. Approximately 10 ml of water was added to complete the reaction and the temperature was held constant until the foaming stopped again. The batch was then cooled to room temperature while being stirred.

[00116] During the high temperature process a white powder was produced. It is hypothesized that this was attributed to volatilized TIPB that reacted with atmospheric water to produce boric acid.

[00117] A plot of the temperature vs. time for this preparation is shown in Figure 2.

Example 2. Preparation of complex boron soap lubricating grease compositions

[00118] This example describes a complex soap of multiple fatty acid materials, in this case 12HSA and octanoic acid. This was manufactured to 20% thickener content and a 2:1 molar ratio of 12HSA to octanoic acid.

[00119] 750 grams of ISO 150 base oil, 156.1g 12HSA and 37.5 g octanoic acid was heated to reach about 93°C (200°F) to melt the acid. At 93°C (200°F), TIPB (78.0 g TIPB for 20% thickener content) was slowly added to the kettle. Heat was then applied to initiate the temperature ramp up to the about 121°C (250°F) reaction temperature. It is hypothesized that this temperature should dehydrate the mixture and eliminate the isopropyl alcohol reaction product. The kettle foamed during the heating, in part due to TIPB having a boiling point of 137°C (280°F). Once the 121°C temperature stage was reached the reaction was allowed to proceed and was considered complete when the foaming stopped. Approximately 10 ml of water was added to complete the reaction and the temperature was held constant until the foaming stopped again. The batch was then cooled to room temperature while being stirred.

Example 3. Characterization of boron soap lubricating grease compositions from Examples 1 & 2

Cone Penetration

[00120] The various concentrations of thickener content from Preparation 2 of Example 1 gave the following cone penetration values. Cone penetration was measured according to ASTM D-1403 at ½ scale. The National Lubricating Grease Institute (NLGI) sets the values for the grades for grease, which are also listed here. Grade 2 grease is a widely used grade. The test measures the drop in tenths of a mm for a standardized cone into grease at a specified temperature. The test determines if the grease thickener matrix has formed. The following values were measured:

Table 1. Cone Penetration Analysis

Percent Thickener	Penetration (0.1 mm)	NLGI grade
15	411	00
20	283	2
25	226	3

[00121] This analysis is further depicted in Figure 3. Overall, cone penetration analysis suggests that the disclosed methods for preparing boron soap-based lubricants results in grease formation.

Cone Bleed

Cone bleed was measured on the manufactured greases including the high temperature process. Cone bleed was performed according to ASTM D-6184 at 100°C for 30 hours. Grease was held in a cone made of stainless steel mesh and heated for the specified time. The amount of oil lost through the mesh was recorded and the percentage lost is reported. The testing gave the following results:

Table 2. Cone Bleed Analysis

Sample	Manufacturing Process	Cone Bleed (%)
Simple Grease	Low Temperature	72
Complex Grease	Low Temperature	98
Simple	High Temperature	77

Cone & Plate Rheometry

[00122] Cone on plate Rheometry was also performed on grease samples of Examples 1 and 2 to determine the yield point. No standard test method exists for this technique yet. A 2° cone was oscillated against a flat plate at 1 Hz and 25°C with the sample between the cone and plate. The stress was increased from 5 to 1000 Pa while the storage (G') and loss (G'') moduli were measured. The results are shown in Figure 5. The results show a pattern that corresponds to that of grease, in particular with a flat linear region at the beginning of the trace followed by a reduction of the moduli values indicating the yield point. The yield point is the stress needed to force the grease to move. Below the yield point the grease acts like a solid.

Example 4. Tribological Characterization of boron soap-based lubricating grease compositions

[00123] The ability of a disclosed simple boron soap grease to reduce wear was evaluated using a modified ASTM D5707-05 test. A simple boron soap grease was synthesized from a reaction between 12 hydroxy-stearic acid and tri-isopropyl borate. This reaction was carried out in the presence of a solvent, in this case a mineral lubricating oil, to form a simple boron soap thickened lubricating grease. The characterized grease was manufactured with 20% thickener and 80% base oil. The base oil was an ISO viscosity grade 150 mineral oil without extreme pressure or anti-wear additives. Although the grease was not milled, or finished, this mixture was shown to have an NLGI Grade 2 consistency using half scale penetration testing after working the grease with 60 strokes (ASTM D217).

[00124] ASTM D5707-05 “Standard Test Method for Measuring Friction and Wear Properties of Lubricating Grease Using a High-Frequency, Linear-Oscillation (SRV) Test Machine,” is a common test for evaluating the reciprocating wear protection provided by greases. The standardized test was written based on the use of an SRV test machine. However, the standard specifically describes a reciprocating wear test that could be conducted on any machine capable of the test conditions.

[00125] The ASTM D5707-05 test specifies the materials, sample geometry, load, oscillation magnitude, oscillation frequency, temperature, and test duration. In addition, the standard also describes the method for determining the amount of wear.

[00126] The reciprocating test sample is specified to be a 10 mm diameter 52100 steel ball with 60 ± 2 HRC hardness and 0.025 ± 0.005 mm Ra roughness. The stationary sample is specified to be a 52100 steel disk with 60 ± 2 HRC hardness and a lapped surface finish of 0.0425 ± 0.0075 mm Ra roughness. The lapped surface should be free of lapping materials. The disk geometry is 24 mm diameter, and 7.85 mm thick.

[00127] The ASTM D5707-05 test is conducted with approximately 0.1 – 0.2 g (about the size of a pea) of grease applied to the contact between the ball and plate. The test is typically conducted with 200 N load applied to the ball, 1 mm oscillation amplitude, and 50 Hz oscillation frequency for 2 hours. The standard states that the test can be conducted at any temperature ranging from ambient to 280°C. The precision of the test, in the written standard, was determined at test temperatures of 50°C and 80°C. Upon the completion of the test, the wear scar on the ball is measured at two locations 90° to each other using a calibrated light microscope. The lengths measured on the ball wear scar are used to compare the amount of wear that occurred with different lubricating greases. A larger wear scar is evidence of more wear on the ball surface.

[00128] A disclosed boron soap grease sample and comparable commercial greases were evaluated using a modified ASTM D5707-05 test. The modified tests were conducted using a Phoenix Tribology Ltd, Plint TE-77 reciprocating wear test machine. The tests were all conducted with steel ball and disk samples that conform to the standard. The test conditions were 200 N load, 1 mm oscillation amplitude, and 20 Hz oscillation frequency for 5 hours. The tests were conducted at a lower frequency, but for a longer duration so that a total of 360,000 cycles were accomplished. This is the same number of cycles as a test run with 50 Hz for 2 hours. In addition, all of the tests were conducted at 50°C with 0.1 mL of grease. The measured volume of grease was applied to the contact with a 3 mL syringe.

[00129] Upon the completion of the tests, the wear scars were measured at two locations 90° to each other using a calibrated light microscope (similar to the standard). The measurements were always made in the direction of sliding, and transverse to the direction of sliding. Instead of reporting the wear scar diameters for each test, a normalized wear area was calculated for each wear track. The normalized wear area was determined by calculating the elliptical area described by the two measured diameters, and normalizing the wear area by the calculated Hertzian contact area of a 10 mm steel ball pressed against a steel plate with 200 N of force. When the analysis is made this way, a normalized wear area of 1 or ~1 suggests that the wear scar is only witness to the Hertzian contact area. This means that there is minimal or no wear damage and only some light scratches or staining. When the normalized wear area becomes 2 or more, this suggests that the wear on the ball was enough to create a contact ~ twice the size of the original (or Hertzian calculated) contact area or larger.

[00130] The modified ASTM D5707-05 (described above) was used to evaluate wear using a disclosed boron soap grease, the base oil from the boron soap grease, and six fully formulated commercial grease products manufactured by 6 different companies. The details from the lubricants tested are listed in Table 3.

Table 3. Lubricant Comparison

Lubricant	Base Oil Type	Viscosity at 40°C (cSt)	Viscosity at 100°C (cSt)	Thickener Type	NLGI Grade
1	synthetic hydrocarbon oil and mineral oil	130	14	Lithium Complex Soap	1
2	mineral oil	148	14	Lithium Complex Soap	1-2
3	mineral oil	-	9.3	Urea	1
4	mineral oil	143.2	13.9	Lithium Complex Soap	2
5	mineral oil	150	15.6	Lithium Complex Soap	2
6	mineral oil	220	19	Lithium Complex Soap	2
Boron Soap Grease	mineral oil	143	14.2	Simple Boron Soap	2
Base Oil	mineral oil	143	14.2	none	-

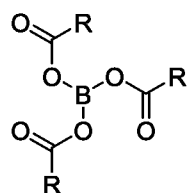
[00131] Six tests were conducted with commercial greases 1 and 2, three tests were conducted with commercial greases 3 – 6, and four tests were conducted with the boron soap grease and its base oil. The normalized wear values are plotted in Figure 6. This data shows that the boron soap grease yielded less wear than four of the commercial greases tested, and similar wear to two of the commercial greases tested. The commercial grease that yielded the least wear had a urea thickener. The urea thickener is known to reduce wear in this type of test. The boron soap grease yielded similar wear to the commercial urea thickened grease, and less wear than the base oil. This suggests that the boron soap thickener can reduce wear without the addition of anti-wear or extreme pressure additives, or with a decreased amount of such additives. Figures 7 and 8 show comparison ball wear from tests with the boron soap grease compared to the base oil.

[00132] It is understood that the foregoing detailed description and accompanying examples are merely illustrative and are not to be taken as limitations upon the scope of the invention, which is defined solely by the appended claims and their equivalents.

CLAIMS

What is claimed is:

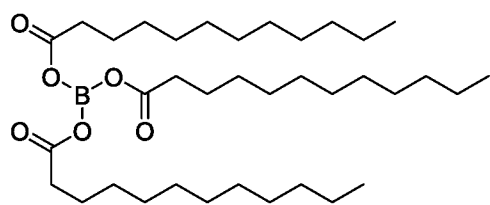
1. A compound of formula (I)



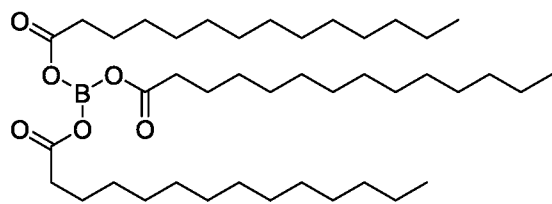
(I),

wherein each R is independently alkyl, alkenyl or aryl, and each R is substituted or unsubstituted; and wherein two R groups together may form a ring.

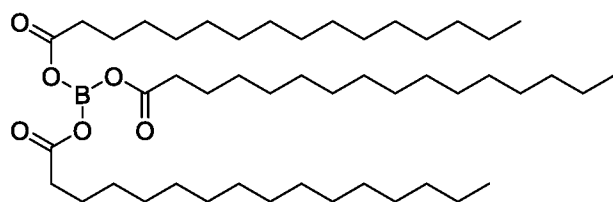
2. The compound of claim 1, wherein R is independently alkyl, alkenyl or aryl, and each R is independently substituted with 0-3 substituents selected from the group consisting of hydroxyl, amino, carboxyl and phenyl.
3. The compound of claim 1 or claim 2, wherein R is alkyl or alkenyl, and R is substituted with 0-3 hydroxyl groups.
4. The compound of any one of claims 1-3, wherein R is C₆-C₃₀ alkyl or C₆-C₃₀ alkenyl, and R is substituted with 0-3 hydroxyl groups.
5. The compound of any one of claims 1-4, wherein R is C₁₂-C₁₈ alkyl or C₁₂-C₁₈ alkenyl, and R is substituted with 0-3 hydroxyl groups.
6. The compound of any one of claims 1-5, wherein the compound is selected from the group consisting of:



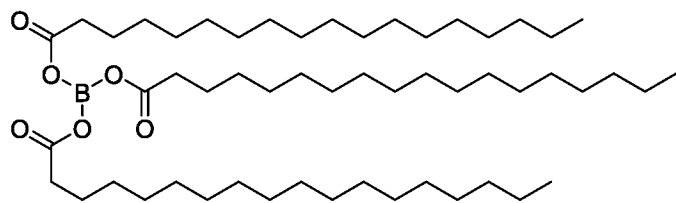
;



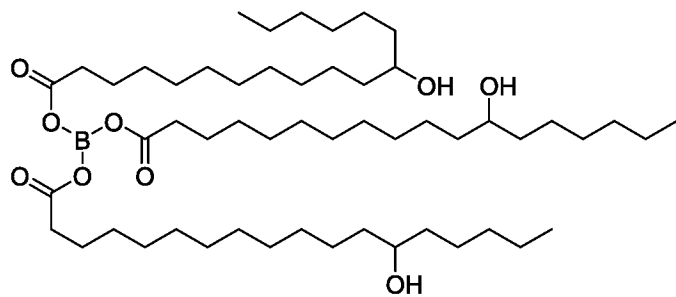
;



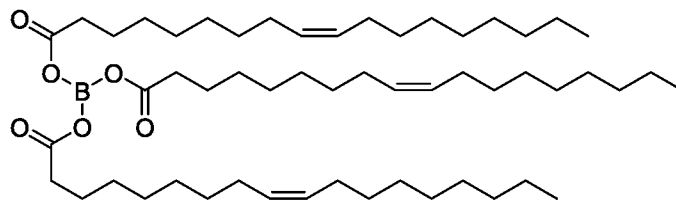
;



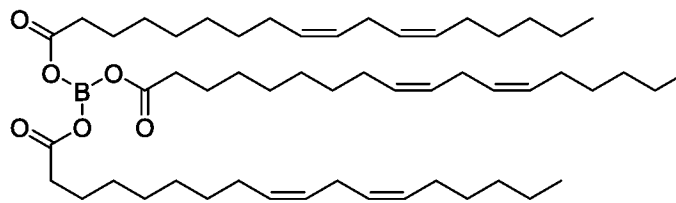
;



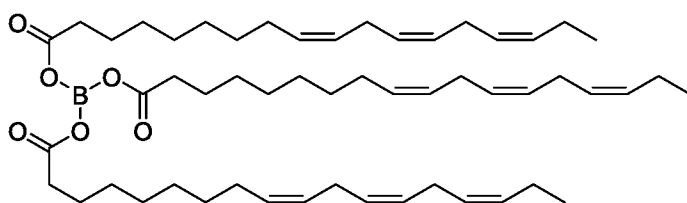
;



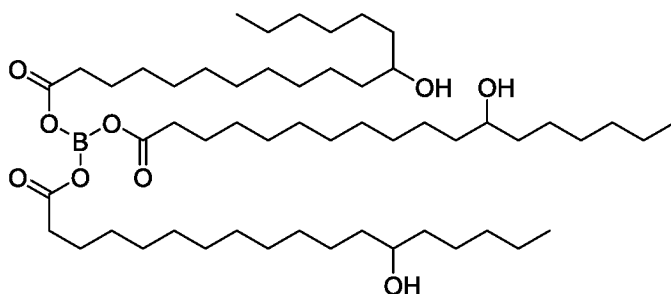
;



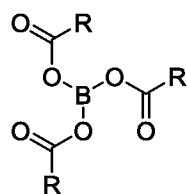
; and



7. The compound of any one of claims 1-6, wherein the compound is



8. A lubricating grease composition comprising a lubricating oil and at least one compound of any one of claims 1-7.
9. A lubricating grease composition comprising, by weight, about 50% to about 99% of a lubricating oil and about 1% to about 50% of at least one compound of any one of claims 1-7.
10. The lubricating grease composition of claim 8 or claim 9 further comprising an additive.
11. The lubricating grease composition of claim 10, wherein the additive is selected from the group consisting of lithium soaps, calcium soaps, sodium soaps, aluminum soaps, polyurea, calcium sulfonate, clay, polytetrafluoroethylene, graphite, boron nitride, boric acid, and a combination thereof.
12. A method of synthesizing a compound of formula (I),

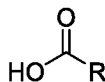


(I),

wherein each R is independently alkyl, alkenyl or aryl, and each R is substituted or unsubstituted; and wherein two R groups together may form a ring;

the method comprising:

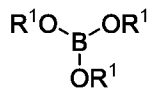
reacting a compound of formula (II),



(II),

wherein each R is independently alkyl, alkenyl or aryl, and each R is independently substituted or unsubstituted;

with a compound of formula (III),



(III),

wherein R¹ is alkyl, aryl or hydrogen;

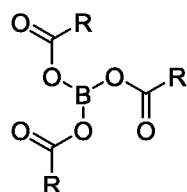
to form the compound of formula (I).

13. The method of claim 12, wherein reacting the compound of formula (II) with the compound of formula (III) further comprises heating at a temperature of about 30°C to about 200°C.

14. The method of claim 12 or claim 13, wherein reacting the compound of formula (II) with the compound of formula (III) is achieved in a solvent.

15. The method of any one of claims 12-14, wherein the compound of formula (I) is a compound of any one of claims 1-7.

16. A method of preparing a lubricating grease composition comprising a lubricating oil and a compound of formula (I),

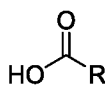


(I),

wherein each R is independently alkyl, alkenyl or aryl, and each R is substituted or unsubstituted; and wherein two R groups together may form a ring;

the method comprising:

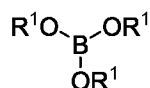
(a) combining a lubricating oil with a compound of formula (II),



(II),

wherein R is alkyl, alkenyl or aryl, and R is substituted or unsubstituted;

and a compound of formula (III),



(III),

wherein R¹ is alkyl, aryl or hydrogen;

to form a first mixture;

(b) heating the first mixture at a temperature of about 30°C to about 200°C to form a second mixture; and

(c) removing byproducts from the second mixture;
to form the lubricating grease composition.

17. The method of claim 16, further comprising adding additional lubricating oil.
18. The method of claim 16 or claim 17, further comprising adding an additive.
19. The method of claim 18, wherein the additive is selected from the group consisting of lithium soaps, calcium soaps, sodium soaps, aluminum soaps, polyurea, calcium sulfonate, clay, polytetrafluoroethylene, graphite, boron nitride, boric acid, and a combination thereof.
20. The method of any one of claims 16-19, wherein the compound of formula (I) is a compound of any one of claims 1-7.

FIGURES

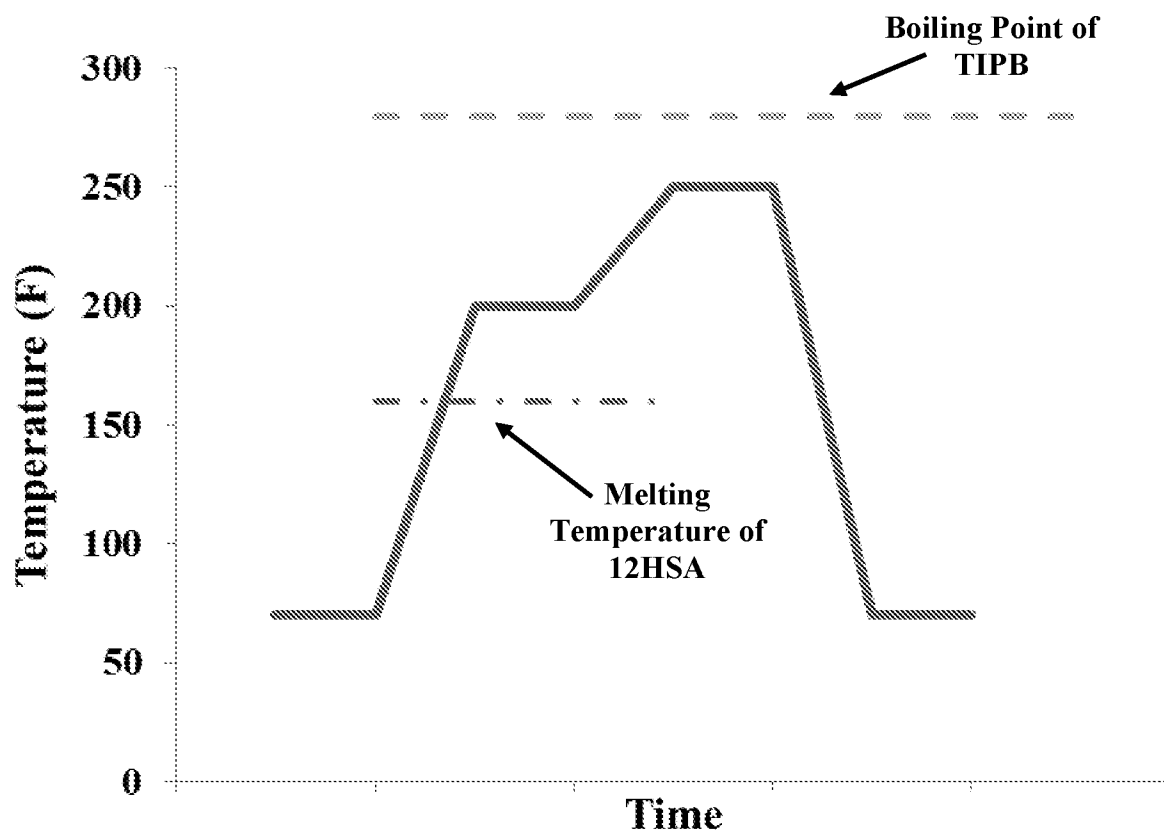
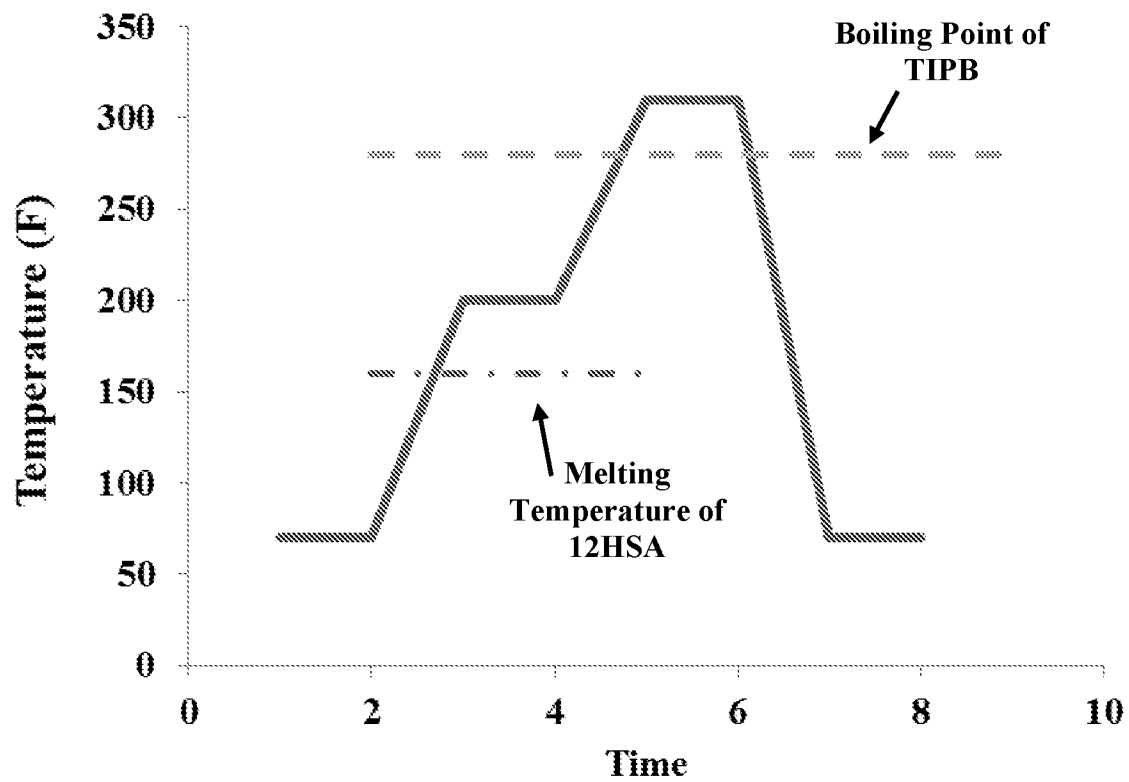
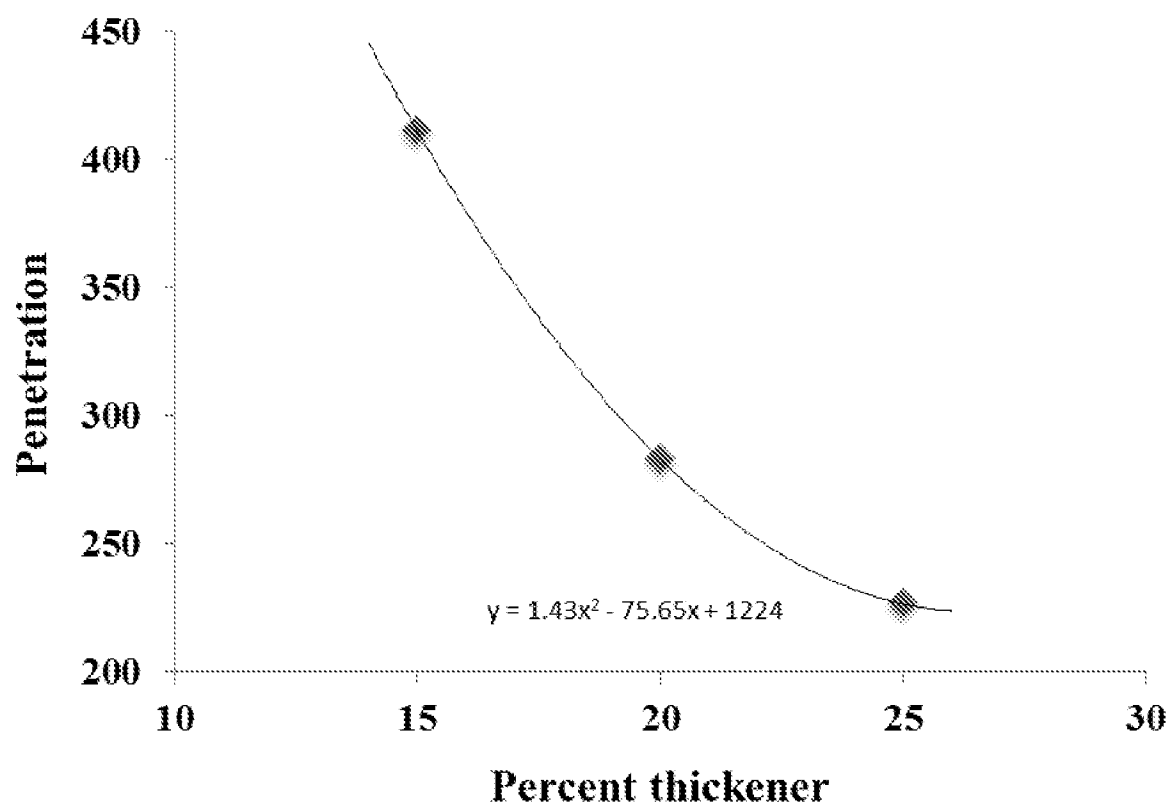


FIG. 1

**FIG. 2**

**FIG. 3**

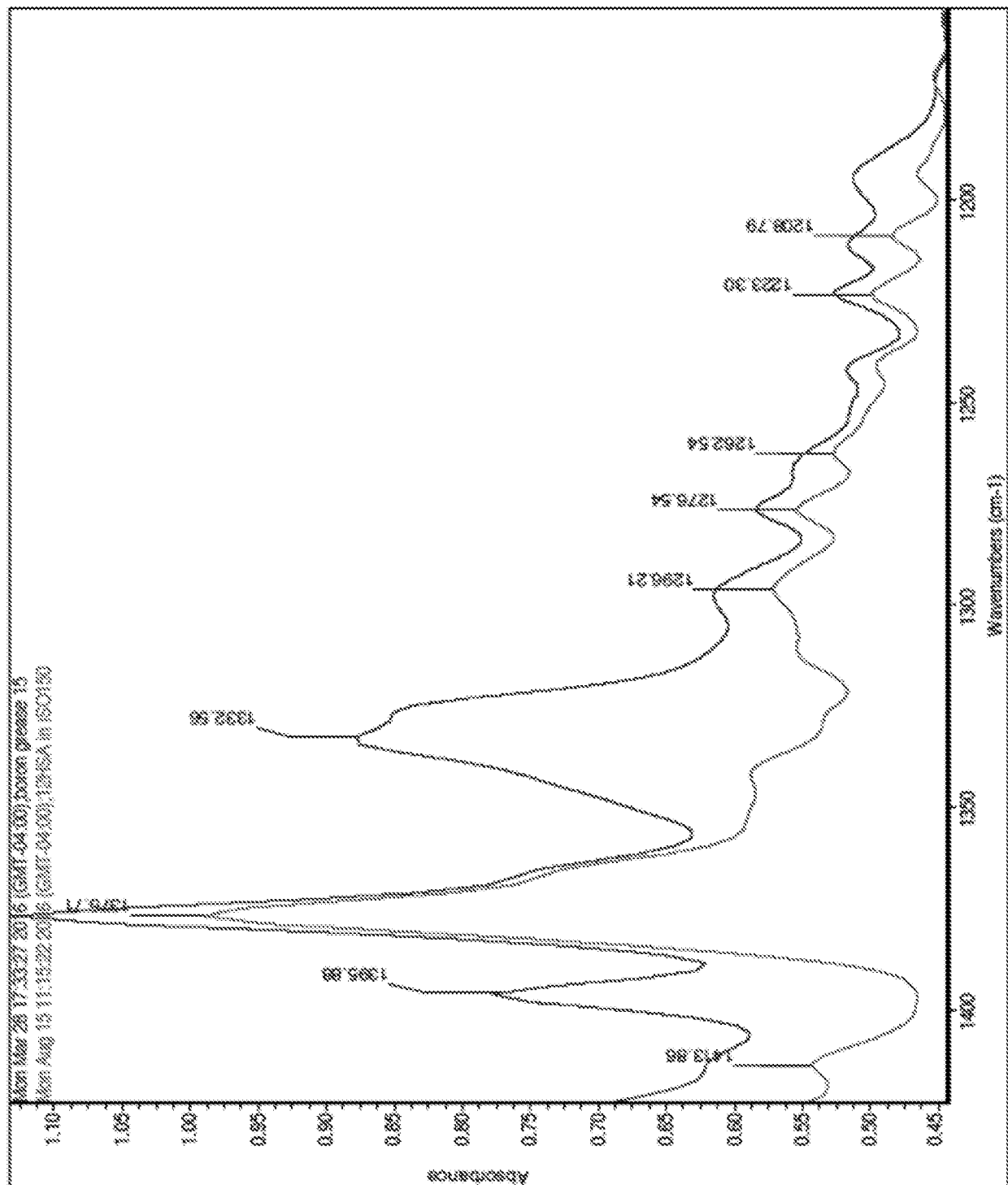


FIG. 4

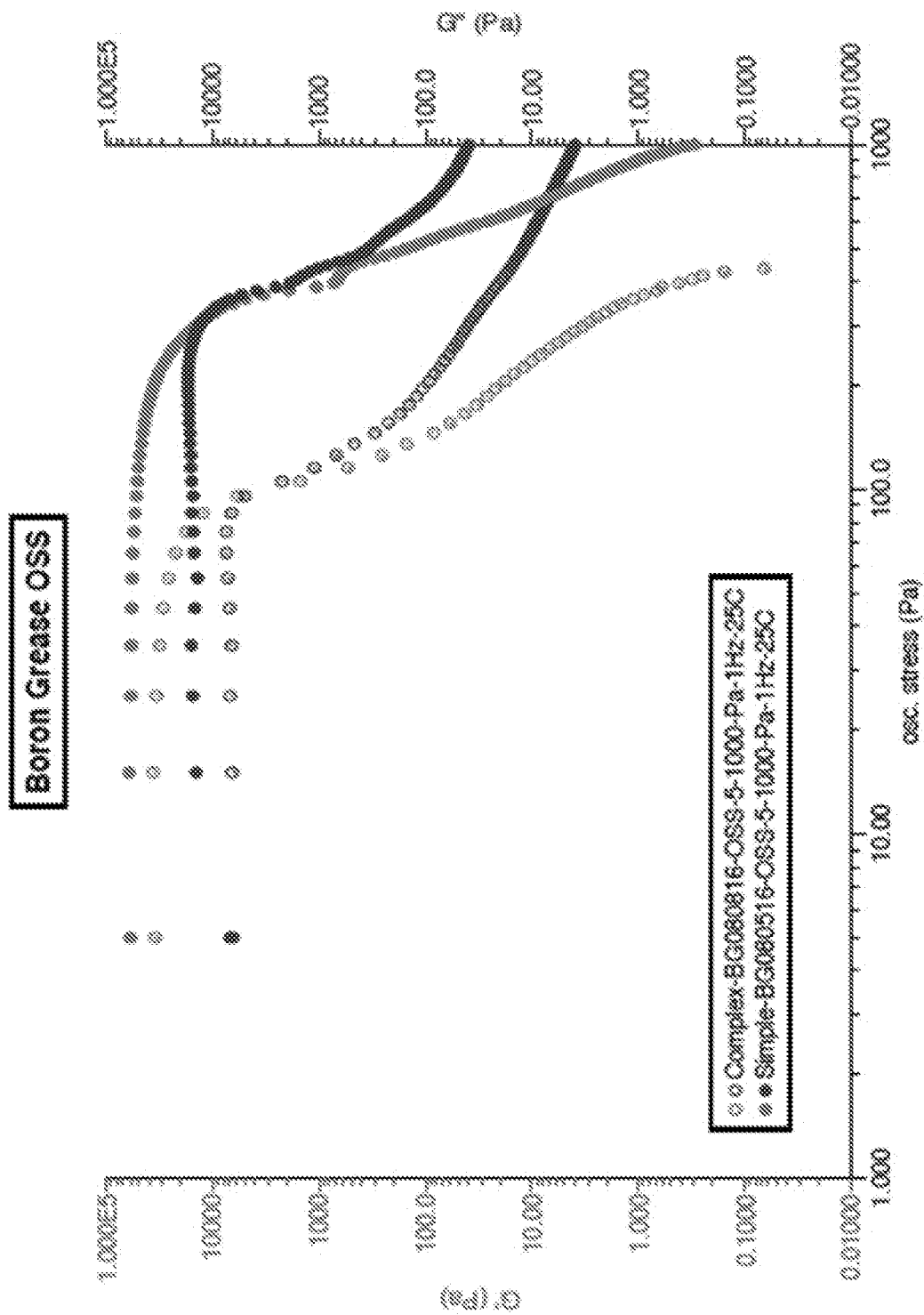


FIG. 5

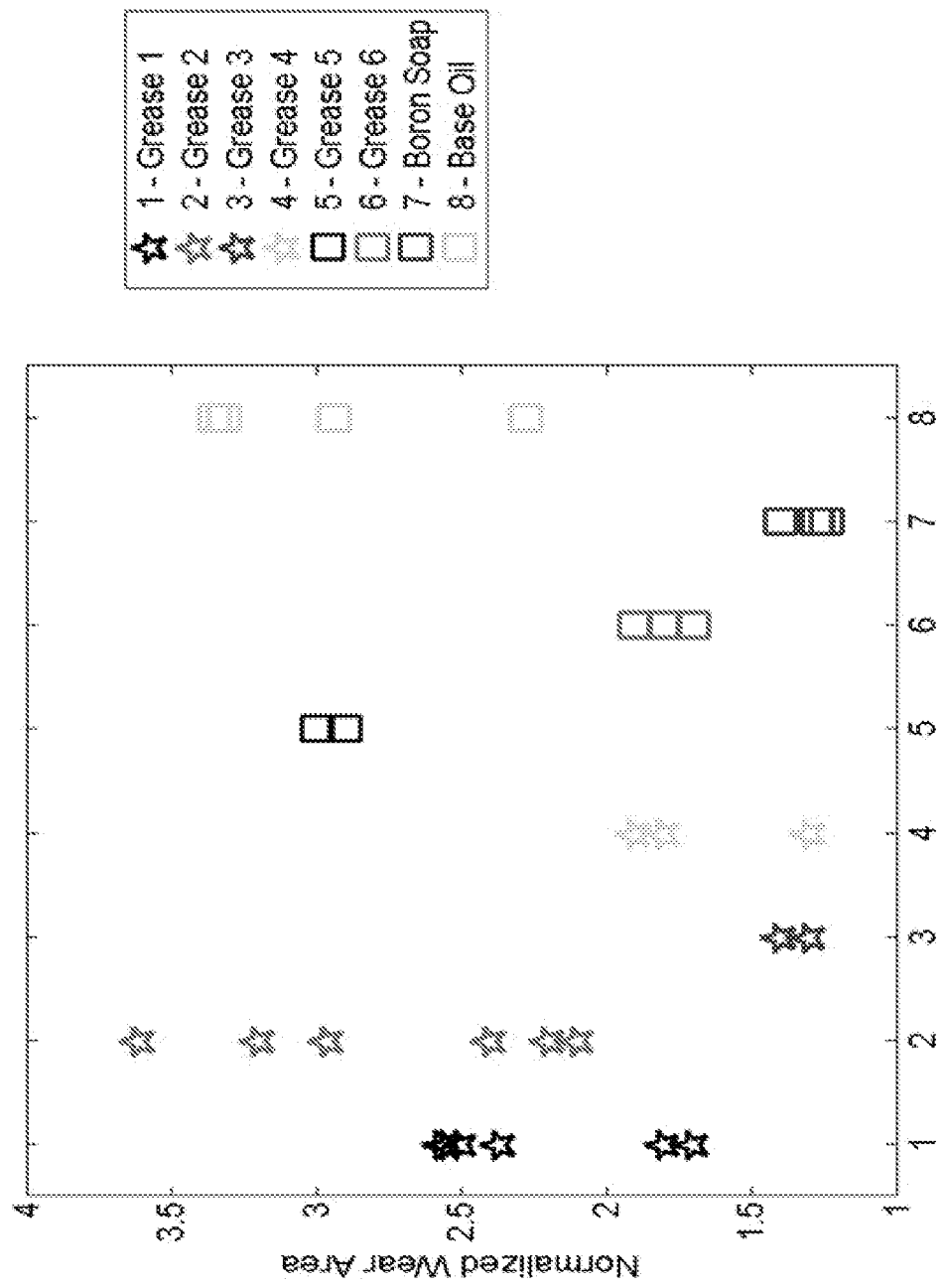
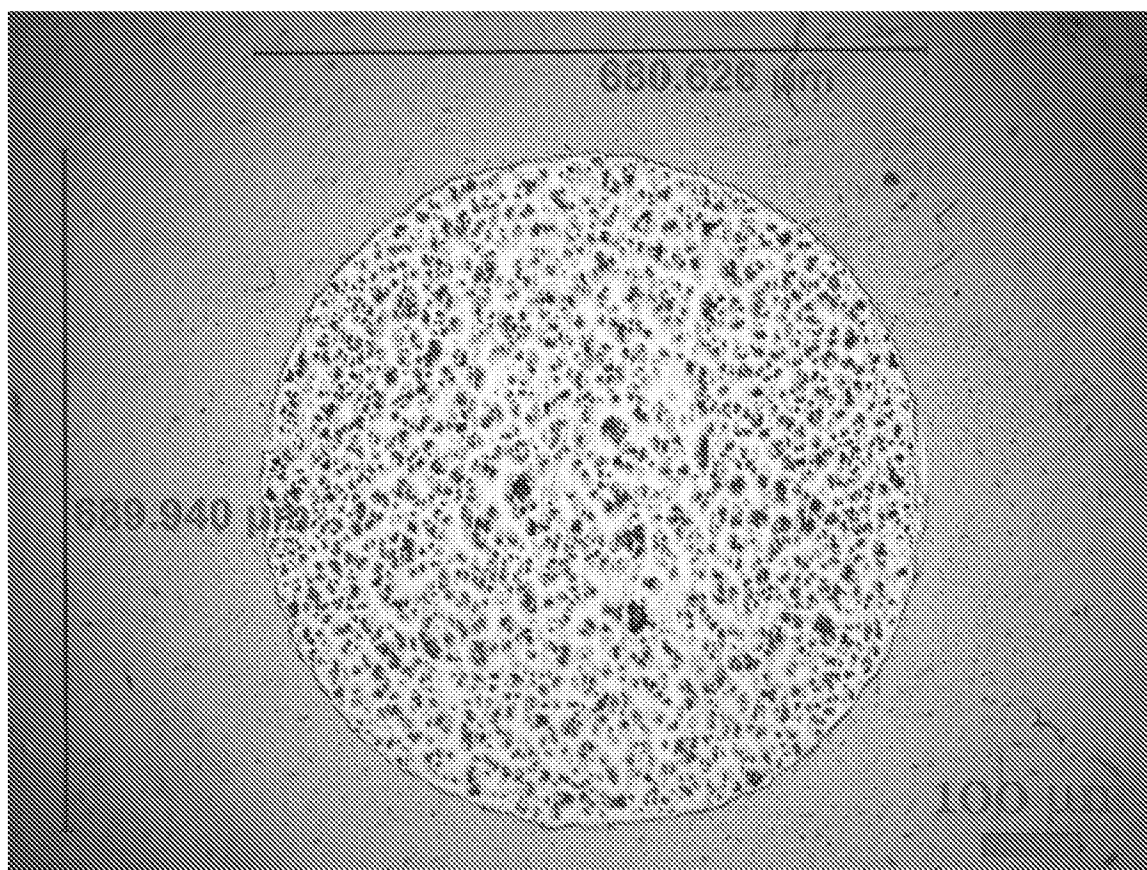


FIG. 6

**FIG. 7**

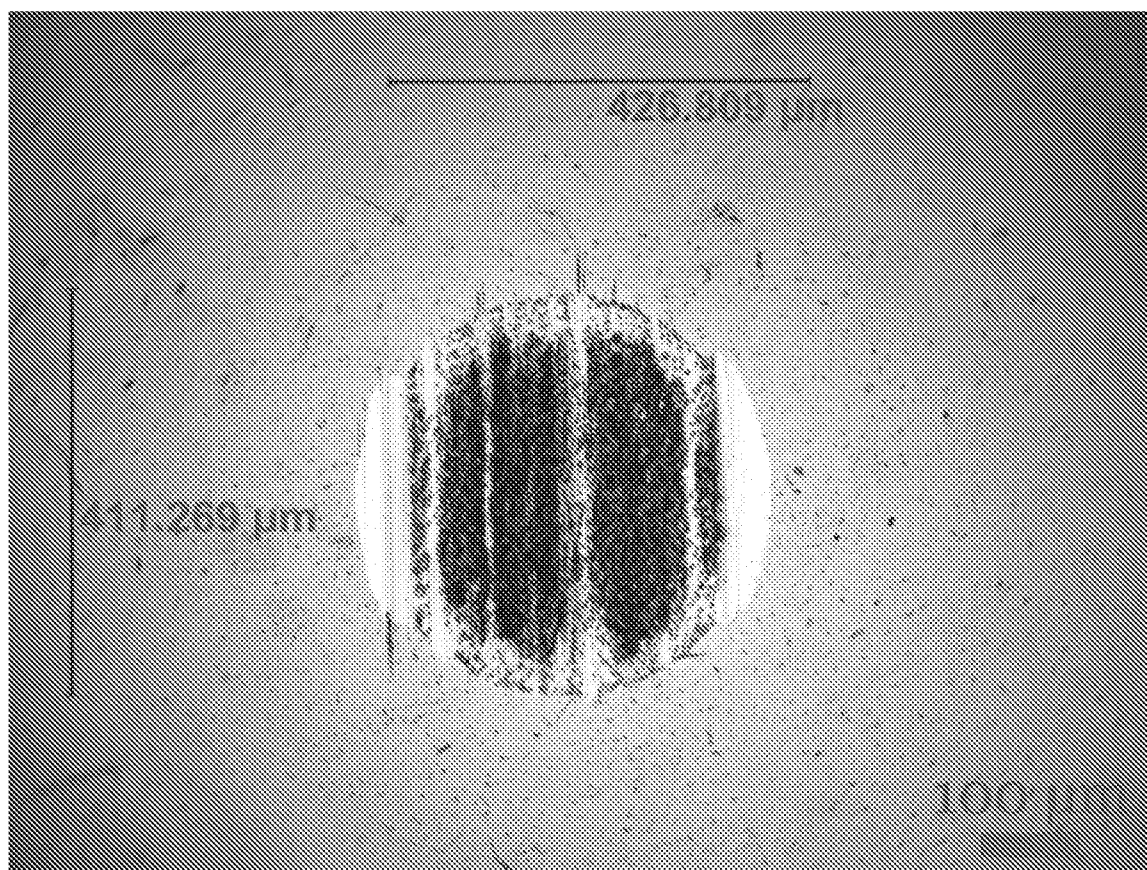


FIG. 8