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(54) **GREASE COMPOSITION**

(57) The grease composition of the present invention comprises at least one base oil selected from a mineral oil and a synthetic oil, and based on the total amount of the grease composition, 2 to 35% by mass of a thickener, 0.05 to 5% by mass of an epoxy compound and 0.1 to 20% by mass of an antiwear additive. The grease of the present invention is good in stability, lubricity and friction characteristics and excellent in long-term reliability even under severe use conditions as compared with conventional grease.

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Description

Technical Field

[0001] The present invention relates to a grease composition.

Background Art

[0002] Conventionally, a grease has been used as a lubricant in a bearing, a constant velocity joint and the like with which an apparatus such as a driving force transmission mechanism is provided.

[0003] In recent years, the apparatus such as a driving force transmission mechanism has tended to be increasingly advanced, higher in performance, higher in speed, smaller in size and longer in lifetime, and a bearing, a constant velocity joint and the like have been used under a high load condition. Therefore, in order to maintain stable lubrication characteristics for a long period of time, levels required for characteristics of a grease, in particular, stability and lubricity as basic performances have been further higher.

[0004] In addition, in an apparatus with a large number of bearing systems rotated at a relatively low speed and bearing positions, the energy saving effect due to a reduction in friction is high. Then, a measure by a reduction in viscosity of a base oil for use in a grease is studied. The reduction in viscosity, however, deteriorates oil film formability in a lubrication part, and thus has limitations with the occurrence of a lubrication failure in mind.

[0005] From the above, a grease that is superior in stability and lubricity and exhibits low friction characteristics is demanded.

[0006] With respect to the enhancement in stability of a grease, Patent Literature 1 discloses a grease in which a thickener, (A) a dialkyldithiocarbamate and (B) an aromatic amine compound are added to a base oil with a polyol ester and an alkyl phenyl ether mixed, Patent Literature 2 discloses a grease composition in which a base oil includes a polyoxyalkylene glycol ether and an additive includes a quinoline type compound and a benzotriazole type compound, Patent Literature 3 discloses a grease in which a base oil is a specific condensed phosphoric acid ester and a specific thickener such as organic bentonite is blended therewith, Patent Literature 4 discloses a lubricant containing in a base oil, fluorinated calcium phosphate also having a thickening effect, Patent Literature 5 discloses a lubricant composition containing a thickener and trimagnesium phosphate in a base oil, Patent Literature 6 discloses a grease containing a thickener and a base oil including a specific poly-α-olefin and a specific ethylene-α-olefin copolymer, and Patent Literature 7 discloses a grease in which a base oil is a specific perfluoropolyether, respectively.

[0007] With respect to the enhancement in lubricity of a grease, Patent Literature 8 discloses a grease containing a powdery oxymolybdenum dithiocarbamate sulfide composition of a specific structure, Patent Literature 9 discloses a conductive grease composition having good lubricity, including an ionic liquid, an alkaline earth metal salt of a higher fatty acid, and a dispersant, Patent Literature 10 discloses a grease mixed with a particulate thickening substance whose rate of increase in viscosity can satisfy the condition expressed by a specific expression, Patent Literature 11 discloses a grease containing a thickener, a specific polytetrafluoroethylene resin powder and a specific zinc dialkyldithiophosphate in a synthetic base oil, Patent Literature 12 discloses a grease in which a base oil is a perfluoropolyether and a thickener is a specific fluororesin, and Patent Literature 13 discloses a grease composition containing a silicone oil, a polyurethane powder and synthetic mica, respectively.

Citation List

Patent Literature

[0008]

[Patent Literature 1] Japanese Patent Application Laid-Open No. 9-3468
[Patent Literature 2] Japanese Patent Application Laid-Open No. 2006-249376
[Patent Literature 3] Japanese Patent Application Laid-Open No. 2010-174209
[Patent Literature 4] Japanese Patent Application Laid-Open No. 2011-21149
[Patent Literature 5] Japanese Patent Application Laid-Open No. 2011-57762
[Patent Literature 6] Japanese Patent Application Laid-Open No. 2011-148908
[Patent Literature 7] Japanese Patent Application Laid-Open No. 2011-256397
[Patent Literature 8] Japanese Patent Application Laid-Open No. 2004-2872
[Patent Literature 9] Japanese Patent Application Laid-Open No. 2007-99826
[Patent Literature 10] Japanese Patent Application Laid-Open No. 2007-231207
[Patent Literature 11] Japanese Patent Application Laid-Open No. 2008-101122

[Patent Literature 12] Japanese Patent Application Laid-Open No. 2010-65171
[Patent Literature 13] Japanese Patent Application Laid-Open No. 2010-138320

Summary of Invention

Technical Problem

[0009] According to studies by the present inventor, however, the greases described in Patent Literatures 1 to 13 above cannot be said to be necessarily sufficient in terms of stability and lubricity under severe use conditions, and have room for improvement in simultaneously satisfying these characteristics and friction characteristics.

[0010] The present invention has been made in view of the problems of the prior art above, and an object thereof is to provide a grease composition that is good in stability, lubricity and friction characteristics and excellent in long-term reliability even under severe use conditions as compared with a conventional grease.

Solution to Problem

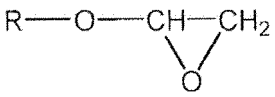
[0011] The present inventors have found that the above problems are solved by blending a thickener, an epoxy compound and an antiwear additive with a specific base oil in respective specific proportions to thereby result in improvements in stability, lubricity and friction characteristics of a grease, leading to completion of the present invention.

[0012] That is, the present invention provides grease compositions according to the following [1] to [10].

[0013] [1] A grease composition comprising at least one base oil selected from a mineral oil and a synthetic oil, and, based on the total amount of the grease composition, 2 to 35% by mass of a thickener, 0.05 to 5% by mass of an epoxy compound and 0.1 to 20% by mass of an antiwear additive.

[0014] [2] The grease composition according to [1], wherein the epoxy compound is at least one selected from a phenyl glycidyl ether type epoxy compound, an alkyl glycidyl ether type epoxy compound, a glycidyl ester type epoxy compound, an alicyclic epoxy compound, an epoxidized aliphatic monoester and an epoxidized vegetable oil.

[0015] [3] The grease composition according to [2], wherein the epoxy compound is an alkyl glycidyl ether represented by the following formula (1):



(1)

wherein R represents a linear or branched alkyl group having 6 to 20 carbon atoms.

[0016] [4] The grease composition according to [3], wherein the epoxy compound is at least one selected from octyl glycidyl ether, 2-ethylhexyl glycidyl ether, nonyl glycidyl ether, decyl glycidyl ether, undecyl glycidyl ether and dodecyl glycidyl ether.

[0017] [5] The grease composition according to any one of [1] to [3], wherein the antiwear additive is at least one selected from an oxygen-containing compound, a nitrogen-containing compound, an organophosphorus compound, an organosulfur compound, an organomolybdenum compound, an alkaline earth metal compound and an organozinc compound.

[0018] [6] The grease composition according to [5], wherein the oxygen-containing compound is at least one selected from an ester, an alcohol, an ether and a carboxylic acid; the nitrogen-containing compound is at least one selected from an aliphatic amine, an aliphatic amide and an aliphatic imide; the organophosphorus compound is at least one selected from a phosphoric acid ester, a phosphorous acid ester and an acidic phosphoric acid ester amine salt; the organosulfur compound is at least one selected from a sulfide compound, and sulfurized fat and oil; the organomolybdenum compound is at least one selected from molybdenum dithiocarbamate and molybdenum dithiophosphate; the alkaline earth metal compound is at least one selected from alkaline earth metal sulfonate salt, phenate salt and salicylate salt; and the organozinc compound is at least one selected from a zinc dialkyl, a zinc dialkyldithiophosphate and a zinc dialkyldithiocarbamate.

[0019] [7] The grease composition according to any one of [1] to [3], wherein the antiwear additive is a solid lubricant.

[0020] [8] The grease composition according to [7], wherein the solid lubricant is at least one selected from molybdenum disulfide, graphite, a tetrafluoroethylene resin powder and boron nitride.

[0021] [9] The grease composition according to any one of [1] to [8], wherein the synthetic oil is at least one selected from a hydrocarbon oil, a diester, a polyol ester and a polyalkylene glycol compound.

[0022] [10] The grease composition according to any one of [1] to [8], wherein the thickener is at least one selected from lithium soaps, calcium soaps, aluminum soaps and a urea compound.

[0023] [11] The grease composition according to [5], containing, as the antiwear additive, an organomolybdenum

compound and an organozinc compound.

[0024] [12] The grease composition according to [5], containing, as the antiwear additive, an organomolybdenum compound, an organozinc compound and an organosulfur compound.

Advantageous Effects of Invention

[0025] According to the present invention, it is possible to provide a grease composition that is good in stability, lubricity and friction characteristics and excellent in long-term reliability even under severe use conditions as compared with a conventional grease.

Description of Embodiments

[0026] Hereinafter, a preferred embodiment of the present invention is described in detail.

[0027] A grease composition according to an embodiment of the present invention comprises at least one base oil selected from a mineral oil and a synthetic oil, and based on the total amount of the grease composition, 2 to 35% by mass of a thickener, 0.05 to 5% by mass of an epoxy compound and 0.1 to 20% by mass of an antiwear additive.

[Base oil]

[0028] As the base oil in the present embodiment, a mineral oil and/or a synthetic oil for use in a lubricating oil may be used.

[0029] The mineral oil includes a paraffinic base oil, a naphthenic base oil and a mixed base oil. These are each a refined lubricating oil fraction obtained by subjecting a crude oil to distillation at ordinary pressure and further distillation at reduced pressure and treating the resulting lubricating oil fraction by an appropriate combination of lubricating oil-refining procedures such as solvent deasphalting, solvent extraction, hydrorefining, hydrocracking, solvent dewaxing, hydrodewaxing and a clay treatment, and may be suitably used. In particular, a step of controlling the composition corresponds to solvent extraction, hydrorefining and hydrocracking, a step of controlling low temperature characteristics such as a pour point corresponds to solvent dewaxing and hydrodewaxing for removal of the wax content, and a clay treatment is a step of removing the nitrogen content mainly to enhance the stability of the base oil.

[0030] In addition, examples include a lubricating base oil such as a wax-cracked/isomerized mineral oil obtained by hydrocracking and/or isomerizing a raw material containing a wax mainly including n-paraffins, such as a slack wax or GTL WAX (gas to liquid wax) produced by a Fischer-Tropsch process or the like. A refined lubricating oil fraction having a different property, obtained from a combination of various raw materials and various refining procedures, may be used singly or in combinations of two or more, and is a suitable base oil.

[0031] In addition, the synthetic oil includes esters such as monoesters, diesters and polyol esters, ethers such as polyoxyalkylene glycols, polyvinyl ethers, dialkyl diphenyl ethers and polyphenylethers, and hydrocarbon oils such as poly-α-olefins (PAO), an ethylene-α-olefin oligomer, alkylbenzenes and alkylnaphthalenes.

[0032] Esters are compounds having various molecular structures, and each of them has particular viscosity characteristics and low temperature characteristics and is a base oil that is characterized by being high in flash point as compared with a hydrocarbon type base oil whose viscosity is the same. Esters may be each obtainable by a dehydration condensation reaction of an alcohol with a carboxylic acid such as a fatty acid, but, in the present invention, a suitable base oil component includes diesters of dibasic acids and monohydric alcohols, polyol esters of polyols (in particular, neopentyl polyol) and monovalent fatty acids, or complex esters of polyols, polyvalent basic acids and monohydric alcohols (or monovalent fatty acids) in terms of chemical stability.

[0033] Dibasic acids include adipic acid, azelaic acid, sebacic acid and dodecanedioic acid, and monohydric alcohols include, as linear monohydric alcohols, butanol, pentanol, hexanol, octanol and decanol, and, as branched monohydric alcohols, 2-ethylhexanol, 3,5,5-trimethylhexanol and isodecanol.

[0034] Among monovalent fatty acids, specifically, linear fatty acids include butanoic acid, pentanoic acid, hexanoic acid, heptanoic acid, octanoic acid, nonanoic acid and oleic acid, and branched fatty acids include branched butanoic acid, branched pentanoic acid, branched hexanoic acid, branched heptanoic acid, branched octanoic acid and branched nonanoic acid. Specifically, α- and/or β-branched fatty acids include isobutanoic acid, 2-methylbutanoic acid, 2-methylpentanoic acid, 2-methylhexanoic acid, 2-ethylpentanoic acid, 2-methylheptanoic acid, 2-ethylhexanoic acid and 3,5,5-trimethylhexanoic acid. As monovalent fatty acids, saturated fatty acids having 4 to 18 carbon atoms, preferably 6 to 12 carbon atoms, can be used.

[0035] As polyhydric alcohols, polyhydric alcohols having 2 to 6 hydroxyl groups, polyhydric alcohols having 4 to 12 carbon atoms, are preferably used. Specifically, polyhydric alcohols include hindered alcohols such as neopentyl glycol, trimethylolethane, trimethylolpropane, trimethylolbutane, di-(trimethylolpropane), pentaerythritol and di-(pentaerythritol).

[0036] Ethers include polyalkylene glycols, polyvinyl ethers and dialkyl diphenyl ethers. Polyalkylene glycols include

polypropylene glycol, polyethylene glycol, and a copolymer of propylene oxide and ethylene oxide. A compound, in which the hydroxyl group at one end is etherified and the hydroxyl group at the remaining end remains as it is, is commonly used, but a compound in which the hydroxyl groups at both ends are etherified is preferable because of being low in hygroscopicity, and the backbone thereof is more preferably an oxypropylene type than an oxyethylene type high in hygroscopicity. In the present embodiment, a polyalkylene glycol compound may be suitably used.

[0037] Among hydrocarbon oils, poly-α-olefins (PAOs) are widely used, and are polymers of α-olefins and thus are characterized by the degree of polymerization. While alkylbenzenes and alkylnaphthalenes are used in the field of a specific lubricating oil, they are classified to a linear type and a branched type depending on the structure of the alkyl group and are different in terms of characteristics, and thus are used depending on the purpose.

[0038] Among these base oils, a mineral oil, diesters, polyol esters, a polyalkylene glycol compound or poly-α-olefins may be preferably used.

[0039] In the present embodiment, one of the mineral oil or one of the synthetic oil may be used singly. In addition, two or more selected from the group consisting of the mineral oil and the synthetic oil may be appropriately combined and blended in appropriate proportions so as to satisfy various performances required depending on the application. Herein, a plurality of the mineral oil type base oils and a plurality of the synthetic oil type base oils may be each used.

[0040] The kinematic viscosity of the base oil at 40°C is preferably 3 to 2000 mm^2/s, more preferably 5 to 1000 mm^2/s and further preferably 8 to 500 mm^2/s in order that the grease composition maintains an appropriate viscous property.

[0041] The content of the base oil is preferably 60% by mass or more and more preferably 70% by mass or more based on the total amount of the grease composition. In addition, the content of the base oil is preferably 95% by mass or less and more preferably 90% by mass or less based on the total amount of the composition. If the content is within this range, lubricity is excellent and it is possible to stably provide a grease.

[Thickener]

[0042] The thickener for use in the grease composition according to the present embodiment is not particularly limited, and all thickeners usually used for a grease composition may be used. Examples of the thickener include metal soaps including lithium, calcium, sodium or aluminum, a urea compound, an imide compound, Bentone, silica gel, a non-soap thickener such as a fluorinated thickener typified by polytetrafluoroethylene, and furthermore an amide compound that is liquid at a temperature equal to or higher than the melting point of the compound and is a gel-like grease at room temperature. Among them, metal soaps and a urea compound are preferable. In addition, among metal soaps, lithium soaps are particularly preferable.

[0043] Lithium soaps include lithium soaps and lithium complex soaps. Lithium soaps include lithium metal soaps such as lithium 12-hydroxystearate and lithium stearate, and lithium complex soaps include lithium soaps with a mixture of a monovalent carboxylic acid and a polyvalent carboxylic acid (preferably, divalent carboxylic acid), specifically, a mixture of a reaction product of 12-hydroxystearic acid and lithium hydroxide and a reaction product of azelaic acid and lithium hydroxide, and the like.

[0044] The urea compound includes a diurea compound, and in particular, examples of the diurea compound include a diurea compound obtained by reacting an aromatic amine, an aliphatic amine, an alicyclic amine or a mixture of two or more of them with an aromatic diisocyanate. Examples of the aromatic diisocyanate include tolylene diisocyanate, diphenylmethane diisocyanate and naphthalene diisocyanate, examples of the aromatic amine include p-toluidine, aniline and naphthylamine, examples of the aliphatic amine include octylamine, nonylamine, decylamine, undecylamine, dodecylamine, tridecylamine and tetradecylamine, and examples of the alicyclic amine include cyclohexylamine and methylcyclohexylamine.

[0045] These thickeners are thickeners that are balanced in terms of characteristics and suitable for practical use in terms of availability and cost. In the present embodiment, the thickener may be used singly or in combinations of two or more. The content of the thickener can be appropriately selected so as to be necessary for achieving required consistency, but is preferably 2 to 35% by mass, preferably 5 to 30% by mass and further preferably 5 to 25% by mass based on the total amount of the grease composition.

[Epoxy compound]

[0046] The grease composition according to the present embodiment comprises an epoxy compound. In the present embodiment, the epoxy compound and an antiwear additive are used in combination while the base oil and the thickener are combined, thereby enabling to enhance stability and wear resistance, and also to simultaneously satisfy these characteristics and friction characteristics. Herein, the epoxy compound itself does not usually exhibit the effect of enhancing wear resistance. In addition, the present inventor has confirmed that the grease composition according to the present embodiment is excellent in wear resistance as compared with the case where no epoxy compound is used and the antiwear additive is used singly. It is thus found that the above effect by the grease composition according to

the present embodiment is a synergetic effect by use of the epoxy compound and the antiwear additive in respective specific contents in combination, and a special effect that is not exerted by a conventional grease composition.

[0047] The type of the epoxy compound includes an alkyl glycidyl ether type epoxy compound, a phenyl glycidyl ether type epoxy compound, a glycidyl ester type epoxy compound, an alicyclic epoxy compound, an epoxidized fatty acid monoester and an epoxidized vegetable oil.

[0048] The alkyl glycidyl ether type epoxy compound specifically includes hexyl glycidyl ether, octyl glycidyl ether, 2-ethylhexyl glycidyl ether, nonyl glycidyl ether, decyl glycidyl ether, undecyl glycidyl ether, dodecyl glycidyl ether, tridecyl glycidyl ether, tetradecyl glycidyl ether, oleyl glycidyl ether, neopentyl glycol diglycidyl ether, trimethylolpropane triglycidyl ether, pentaerythritol tetraglycidyl ether, 1,6-hexanediol diglycidyl ether, sorbitol polyglycidyl ether, polyalkylene glycol monoglycidyl ether and polyalkylene glycol diglycidyl ether.

[0049] The phenyl glycidyl ether type epoxy compound includes phenyl glycidyl ether, methylphenyl glycidyl ether, ethylphenyl glycidyl ether, propylphenyl glycidyl ether and butylphenyl glycidyl ether.

[0050] The glycidyl ester type epoxy compound specifically includes phenyl glycidyl esters, alkyl glycidyl esters and alkenyl glycidyl esters, and examples include glycidyl-2,2-dimethyl octanoate, glycidyl benzoate, glycidyl acrylate and glycidyl methacrylate. Examples include alkyl glycidyl esters in which the number of carbon atoms in the alkyl group is 4 to 18, in particular, 6 to 12.

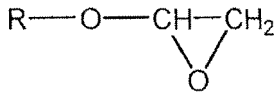
[0051] The alicyclic epoxy compound specifically includes 1,2-epoxycyclohexane, 1,2-epoxycyclopentane, 3,4-epoxycyclohexyl methyl-3',4'-epoxycyclohexane carboxylate, bis(3,4-epoxy-6-methylcyclohexyl methyl)adipate, 2-(7-oxabicyclo[4,1,0]hept-3-yl)-spiro(1,3-dioxane-5,3'-[7]oxabicyclo[4 ,1,0]heptane, 4-(1'-methylepoxyethyl)-1,2-epoxy-2-methylcyclohexane and 4-epoxyethyl-1,2-epoxycyclohexane. Examples include derivatives of epoxycycloalkanes having 5 to 7 carbon atoms.

[0052] Epoxidized fatty acid monoesters include esters of epoxidized fatty acids having 12 to 20 carbon atoms and alcohols having 1 to 8 carbon atoms, or phenols or alkyl phenols, and examples include butyl, hexyl, benzyl, cyclohexyl, methoxyethyl, octyl, phenyl and butyl phenyl esters of epoxy stearic acid.

[0053] The epoxidized vegetable oil includes an epoxy compound of a vegetable oil such as a soybean oil, a flaxseed oil or a cotton seed oil.

[0054] In the present embodiment, the epoxy compound incorporates a carboxylic acid or the like produced along with degradation of the grease into the molecule to stabilize the carboxylic acid or the like, and thus suppresses production of a component that causes corrosion wear and suppresses degradation of the additive to thereby contribute to an enhancement in lubricity.

[0055] Furthermore, the present inventor has found that alkyl glycidyl ethers among the epoxy compounds exhibit a different behavior from other types of epoxy compounds, and result in not only an enhancement in lubricity by stabilization but also a significant increase in the effect of the antiwear additive. In particular, an alkyl glycidyl ether represented by the following formula (1) is preferable because of resulting in not only an enhancement in stability but also a large effect of increasing wear resistance, and furthermore an alkyl glycidyl ether in which R in the formula (1) represents an alkyl group having 8 to 12 carbon atoms is more preferable.



(1)

[wherein R represents a linear or branched alkyl group having 6 to 20 carbon atoms.]

[0056] Preferred alkyl glycidyl ethers include octyl glycidyl ether, 2-ethylhexyl glycidyl ether, nonyl glycidyl ether, decyl glycidyl ether, undecyl glycidyl ether and dodecyl glycidyl ether.

[0057] In the present embodiment, the epoxy compound may be used singly or may be used in combinations of two or more.

[0058] The content of the epoxy compound is 0.05 to 5% by mass, preferably 0.1 to 3% by mass and more preferably 0.1 to 2% by mass based on the total amount of the grease composition. If the content is low, no effect of enhancing stability and wear resistance is exerted, and if the content is too high, characteristics as the grease are not balanced, for example, an organic material in a mechanical system is swollen.

[Antiwear additive]

[0059] The grease composition according to the present-embodiment comprises an antiwear additive. A preferred antiwear additive includes an oxygen-containing compound, a nitrogen-containing compound, an organophosphorus compound, an organosulfur compound, an organomolybdenum compound, a calcium compound and an organozinc compound. In addition, a solid lubricant may also be preferably used. Herein, the antiwear additive may be classified to

an oiliness agent, an antiwear agent and an extreme pressure agent, but such classification is not necessarily critical. In addition, one antiwear additive may correspond to two or three of an oiliness agent, an antiwear agent and an extreme pressure agent. The epoxy compound for use in the present invention is excluded from the antiwear additive in the present invention.

[0060] Among the antiwear additives, the oxygen-containing compound includes higher fatty acids such as oleic acid and stearic acid, higher alcohols such as oleyl alcohol, esters such as glycerol monooleate, and ethers such as glycerol monooleyl ether. These oxygen-containing compounds are typically configured from an oxygen-containing polar group such as a hydroxyl group, a carboxylic acid group, an ether group or an ester group and a saturated or unsaturated hydrocarbon group having 12 to 24 carbon atoms. It is expected in these oxygen-containing compounds that the polar group adsorbs to the surface of a metal sliding portion and the hydrocarbon group forms an oil film to enhance wear resistance. Examples include partial esters in which a hydroxyl group remains, in particular, glycerol partial esters with fatty acids having 12 to 24 carbon atoms.

[0061] In addition, the nitrogen-containing compound includes aliphatic amines such as oleylamine and dodecylamine, aliphatic amides and aliphatic imides. These nitrogen-containing compounds are typically configured from a nitrogen-containing polar group such as an amine group, an amide group or an imide group and a hydrocarbon group having a saturated or unsaturated bond and having 12 to 24 carbon atoms. It is expected in these nitrogen-containing compounds that the polar group adsorbs to the surface of a metal sliding portion and the hydrocarbon group forms an oil film to enhance wear resistance. In particular, examples include aliphatic amines having 12 to 24 carbon atoms.

[0062] In addition, the organophosphorus compound includes phosphoric acid esters, phosphorous acid esters and an acidic phosphoric acid ester amine salt. In the present invention, it is to be noted that a compound containing nitrogen or the like, in addition to phosphorus, as a constituent element, is included in the organophosphorus compound. The organophosphorus compound is typically one having one or more hydrocarbon groups having 6 to 24 carbon atoms. Such a hydrocarbon group includes a linear hydrocarbon group, a branched hydrocarbon group and an aromatic group. The salt compound in the organophosphorus compound includes a compound completely neutralized or a compound partially neutralized. It is expected in such an organophosphorus compound that a lubricant film of iron phosphate or the like is formed on the surface of a metal sliding portion to enhance wear resistance. Examples of phosphoric acid esters include those having an aromatic group, and typically include triaryl phosphates.

[0063] The organosulfur compound includes a monosulfide compound, a disulfide compound, a polysulfide compound, sulfurized fat and oil and sulfurized olefins. It is expected in such an organosulfur compound that a lubricant film of iron sulfide or the like is formed on the surface of a metal sliding portion to enhance wear resistance. Examples include diaryl sulfides, dialkyl sulfides and dialkenyl sulfides. The sulfur element content in the organosulfur compound can be, for example, 2 to 70% by mass, in particular, 5 to 60% by mass.

[0064] The organomolybdenum compound includes molybdenum dithiocarbamate and molybdenum dithiophosphate. In the present invention, it is to be noted that a compound containing sulfur or the like, in addition to molybdenum, as a constituent element, is included in the organomolybdenum compound. It is expected in such an organomolybdenum compound that a lubricant film made of molybdenum disulfide is formed on the surface of a metal sliding portion to enhance wear resistance. Examples include molybdenum dialkyldithiocarbamates and molybdenum dialkyldithiophosphates in which the number of carbon atoms in an alkyl group is 4 to 12.

[0065] The alkaline earth metal compound includes alkaline earth metal sulfonate salt, phenate salt and salicylate salt. The alkaline earth metal may be selected from calcium, magnesium and barium. In the present invention, it is to be noted that a compound containing sulfur or the like, in addition to the alkaline earth metal, as a constituent element is included in the alkaline earth metal compound. In addition, the calcium compound may include a basic component such as calcium carbonate.

[0066] The organozinc compound includes organozinc compounds such as zinc dialkyls, zinc dialkyldithiophosphates and zinc dialkyldithiocarbamates. In the present invention, it is to be noted that a compound containing sulfur, phosphorus or the like, in addition to zinc, as a constituent element is included in the organozinc compound. The organozinc compound typically has one or more linear, branched or aromatic hydrocarbon groups having 6 to 24 carbon atoms. Examples include zinc dialkyldithiophosphates in which the number of carbon atoms in an alkyl group is 4 to 12.

[0067] In addition, a solid lubricant may also be applied to the grease being a semi-solid lubricant. The solid lubricant is an additive that is used in the form of a powder or thin film in order to prevent the surface of a sliding material from being damaged or in order to reduce friction/wear, and includes molybdenum disulfide, tungsten disulfide, graphite, graphite fluoride, melamine cyanurate, a tetrafluoroethylene (PTFE) resin powder, a polyimide resin powder, a high-density polyethylene resin powder, boron nitride, a copper powder, a nickel powder, a tin powder and a silver powder.

[0068] In particular, molybdenum disulfide, graphite, a tetrafluoroethylene resin powder and boron nitride may be suitably used in the present invention.

[0069] In the present embodiment, the antiwear additive may be used singly or may be used in combinations of two or more. The content of the antiwear additive is 0.1 to 20% by mass, preferably 0.2 to 20% by mass, more preferably 0.2 to 7% by mass and further preferably 0.3 to 5% by mass based on the total amount of the grease composition. If

the content is too low, no effect of wear-resistance is exerted, and if the content is too high, the stability of the lubricating oil is deteriorated.

[0070] It is preferable to contain the organomolybdenum compound and the organozinc compound as the antiwear additive. The mass ratio of the content of the organomolybdenum compound to the content of the organozinc compound may be, for example, 0.2 to 10 : 1, in particular, 1 to 5 : 1. In this case, it is possible to use a lithium complex soap as the thickener.

[0071] It is preferable to contain the organomolybdenum compound, the organozinc compound and the organosulfur compound as the antiwear additive. The mass ratio of the content of the organomolybdenum compound to the content of the organozinc compound may be, for example, 1 to 10 : 1, in particular, 1 to 5 : 1, and the mass ratio of the content of the organozinc compound to the content of the organosulfur compound may be, for example, 0.2 to 2 : 1, in particular, 0.5 to 2 : 1. In this case, it is possible to use a lithium soap as the thickener. It is possible to use a dialkyl sulfide having alkyl having 4 to 18, in particular, 6 to 10 carbon atoms as the organosulfur compound.

[Other additives]

[0072] The grease composition according to the present embodiment may contain, in order to improve its performance, additives such as an antioxidant, an antirust agent, a metal deactivator, a corrosion inhibitor, a viscosity index improver, a pour point depressant, a detergent dispersant, an emulsifier and an antifoamer that have been conventionally used for a lubricating oil, as long as the object of the present invention is not impaired.

[0073] The antioxidant includes a phenol type compound like di-tert-butyl-p-cresol, and an amine type compound like alkyl diphenyl amines, the antirust agent includes alkenyl succinic acid esters or partial esters thereof, the metal deactivator includes benzotriazole and gallic acid esters, the corrosion inhibitor includes thiadiazole, the viscosity index improver includes polymethacrylate and polyisobutylene, the pour point depressant includes polyalkylacrylate and polyalkylstyrene, the detergent dispersant includes succinic imide and phosphonates, the emulsifier includes a fatty acid soap and a long-chain alcohol sulfate ester salt, and the antifoamer includes a silicone compound and a polyester compound, respectively.

Examples

[0074] Hereinafter, the present invention is described in more detail based on Examples and Comparative Examples, but the present invention is not limited to such Examples.

[Examples 1 to 28, Comparative Examples 1 to 17]

[0075] In Examples 1 to 28 and Comparative Examples 1 to 17, respective base oils, thickeners and additives shown below were used to prepare respective grease compositions having composition shown in Tables 1 to 6.

(A) Base oil

[0076]

(A-1) Mineral oil: paraffinic refined mineral oil (kinematic viscosity at 40°C: 93.3 mm^2/s; viscosity index: 95; pour point: -15°C; flash point: 260°C)

(A-2) Polyol ester (POE): ester of pentaerythritol and a mixed acid of 2-ethylhexanoic acid and 3,5,5-trimethylhexanoic acid in a mass ratio of 5 : 5 (kinematic viscosity at 40°C: 66.7 mm^2/s; viscosity index: 92; pour point: -40°C; flash point: 248°C)

(A-3) Polyalkylene glycol (PAG): polyoxypropylene in which both ends were blocked by a methyl group (weight average molecular weight: 1000; kinematic viscosity at 40°C: 46.0 mm^2/s; viscosity index: 190; pour point: -45°C; flash point: 218°C)

(A-4) Poly-α-olefin (PAO): polymer of 1-dodecene (kinematic viscosity at 40°C: 100 mm^2/s; viscosity index: 140; pour point: -40°C; flash point: 260°C)

Herein, the kinematic viscosity and the viscosity index of each of the base oils were measured according to JIS K2283, the pour point thereof was measured according to JIS K2269, and the flash point thereof was measured according to JIS K2265.

(B) Thickener

[0077]

(B-1) Lithium soap grease: lithium 12-hydroxystearate (10% by mass) was blended and stirred with any of the base oils, uniformly dissolved therein at about 200°C and then cooled, and additive (C or D) was added thereto at about 80°C to provide a grease. This lithium soap grease was allowed to pass through a roll mill and tested as the grease composition.

(B-2) Urea grease: one in which diphenylmethane-4,4'-diisocyanate was dissolved in any of the base oils (about 80°C) and one in which cyclohexylamine and octadecylamine were dissolved in any of the base oils (about 80°C) were prepared (mass ratio of diphenylmethane-4,4'-diisocyanate/cyclohexylamine/octadecylamine: 9/6/4), both of them were mixed and stirred for reaction (160°C, 30 minutes) and cooled, and thereafter an additive (C or D) was added thereto at about 80°C to provide a urea grease (5% by mass as diurea). This urea grease was allowed to pass through a roll mill and tested as the grease composition.

(B-3) Lithium complex soap grease: 12-hydroxystearic acid and azelaic acid, and lithium hydroxide were blended and stirred with any of the base oils in 10% by mass relative to the total amount of the grease composition, uniformly dissolved therein at about 200°C and then cooled, and additive (C or D) was added thereto at about 80°C to provide a grease. This lithium complex soap grease was allowed to pass through a roll mill and tested as the grease composition.

(C) Epoxy compound

[0078]

(C-1) 2-Ethylhexyl glycidyl ether
(C-2) Dodecyl glycidyl ether
(C-3) Glycidyl-2,2-dimethyl octanoate
(C-4) 3,4-Epoxycyclohexylmethyl-3',4'-epoxycyclohexane carboxylate

(D) Antiwear additive

[0079]

(D-1) Oxygen-containing compound: glycerol monooleate
(D-2) Nitrogen-containing compound: oleylamine
(D-3) Organophosphorus compound: tricresyl phosphate
(D-4) Organosulfur compound A: dibenzyl disulfide
(D-5) Organomolybdenum compound A: molybdenum dialkyldithiophosphate [Mo element content: 8.0% by mass]
(D-6) Alkaline earth metal compound: overbasic Ca sulfonate, [TBN (ASTM D2895): 325 mgKOH/g; Ca: 12.7% by mass; S: 2% by mass]
(D-7) Organozinc compound: zinc dioctyl dithiophosphate
(D-8) Solid lubricant: graphite (average particle size: 10 μm)
(D-9) Organomolybdenum compound B: molybdenum dialkyldithiocarbamate [Mo element content: 10.0% by mass]
(D-10) Organosulfur compound B: dioctyl polysulfide [sulfur element content: 39% by mass]

[0080] With respect to each of the grease compositions in Examples 1 to 28 and Comparative Examples 1 to 17, the following tests were performed.

[SRV test]

[0081] A ball/disc reciprocating kinematic friction/wear test machine, SRV test machine (Optimol Instruments, Instrument SRV), was used to perform seizure load and wear coefficient measurements according to ASTM D5706.

[0082] SUJ2 was used for materials of the ball and disc of the SRV test. The test was started at a temperature of 80°C, an amplitude of 1 mm, a frequency of 50 Hz and a load of 50 N, and the load was increased by 100 N every 2 minutes to perform the operation until seizure occurred (the maximum load set was 2000 N).

[0083] In this test, the seizure load, and the friction coefficient at each load before the seizure can be measured. Tables 1 to 6 show the seizure load, and the friction coefficient at a load lower than the seizure load by 200 N in the test in which each of the grease compositions was used. Herein, "Unstable" in the column "Friction coefficient" in Tables means

that the friction coefficient varied between 0.08 and 0.18 and the value could not be read.

[Oxidation stability test]

[0084] A sample pressurized to 755 KPa by an oxygen tank was heated to 99°C and the pressure drop after a lapse of 100 hours was measured according to the grease oxidation stability test method of JIS K2220. The results obtained are shown in Tables 1 to 3. JIS K2220 defines Class 3 grease for rolling bearing (oxidation stability is good over a wide temperature range) as having a reduction in oxygen pressure of 49 KPa or less, and the oxidation stability of each of the grease compositions can be rated based on the definition.

[Table 1]

	Example 1	Comparative Example 1	Comparative Example 2	Comparative Example 3	Example 2	Comparative Example 4	Example 3	Comparative Example 5
Base oil								
Type	A-1	A-1	A-1	A-1	A-1	A-1	A-1	A-1
Content (% by mass)	Balance	Balance	Balance	Balance	Balance	Balance	Balance	Balance
Thickener (% by mass)								
B-1	10.0	10.0	10.0	10.0	10.0	10.0	-	-
B-2	-	-	-	-	-	-	5.0	5.0
Epoxy compound (% by mass)								
C-1	0.5	-	0.5	-	0.2	-	-	-
C-2	-	-	-	-	-	-	0.3	-
C-3	-	-	-	-	-	-	-	-
C-4	-	-	-	-	-	-	-	-
Antiwear additive (% by mass)								
D-1	1.0	-	-	1.0	-	-	-	-
D-2	-	-	-	-	0.5	0.5	-	-
D-3	-	-	-	-	-	-	1.5	1.5
D-4	-	-	-	-	-	-	-	-
D-5	-	-	-	-	-	-	-	-
D-6	-	-	-	-	-	-	-	-
D-7	-	-	-	-	-	-	-	-
D-8	-	-	-	-	-	-	-	-
SRV test								
Seizure load (N)	700	200	200	400	700	400	800	500
Friction coefficient	0.07	Unstable	Unstable	0.10	0.08	0.11	0.10	0.13
Oxidation stability test								
Amount of reduction in oxygen pressure (KPa)	35	85	38	75	39	70	25	73

[Table 2]

	Example 4	Comparative Example 6	Example 5	Comparative Example 7	Example 6	Comparative Example 8	Example 7	Comparative Example 9
Base oil								
Type	A-1	A-1	A-1	A-1	A-1	A-1	A-1	A-1
Content (% by mass)	Balance	Balance	Balance	Balance	Balance	Balance	Balance	Balance
Thickener (% by mass)								
B-1	10.0	10.0	-	-	10.0	10.0	10.0	10.0
B-2	-	-	5.0	5.0	-	-	-	-
Epoxy compound (% by mass)								
C-1	-	-	-	-	2.0	-	0.2	-
C-2	-	-	-	-	-	-	-	-
C-3	1.0	-	-	-	-	-	-	-
C-4	-	-	0.5	-	-	-	-	-
Antiwear additive (% by mass)								
D-1	-	-	-	-	-	-	-	-
D-2	-	-	-	-	-	-	-	-
D-3	-	-	-	-	-	-	-	-
D-4	1.0	1.0	-	-	-	-	-	-
D-5	-	-	1.0	1.0	-	-	-	-
D-6	-	-	-	-	3.0	3.0	-	-
D-7	-	-	-	-	-	-	0.5	0.5
D-8	-	-	-	-	-	-	-	-
SRV test								
Seizure load (N)	700	500	700	500	1100	700	900	500
Friction coefficient	0.11	0.13	0.07	0.09	0.08	0.11	0.08	0.11
Oxidation stability test								
Amount of reduction in oxygen pressure (KPa)	32	76	35	80	28	92	22	68

[Table 3]

	Example 8	Comparative Example 10	Example 9	Comparative Example 11	Example 10	Comparative Example 12	Example 11	Comparative Example 13
Base oil								
Type	A-1	A-1	A-2	A-2	A-3	A-3	A-1	A-1
Content (% by mass)	Balance	Balance	Balance	Balance	Balance	Balance	Balance	Balance
Thickener (% by mass)								
B-1	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0
B-2	-	-	-	-	-	-	-	-
Epoxy compound (% by mass)								
C-1	-	-	0.15	-	0.1	-	0.3	-
C-2	0.3	-	-	-	-	-	-	-
C-3	-	-	-	-	-	-	-	-
C-4	-	-	-	-	-	-	-	-
Antiwear additive (% by mass)								
D-1	-	-	0.2	0.2	-	-	-	-
D-2	-	-	-	-	-	-		-
D-3	-	-	1.0	1.0	-	-	-	-
D-4	-	-	-	-	0.5	0.5	-	-
D-5	-	-	-	-	0.1	0.1	1.0	1.0
D-6	2.0	2.0	-	-	-	-	2.0	2.0
D-7	1.5	1.5	-	-	-	-	1.5	1.5
D-8	1.0	1.0	-	-	-	-	-	-
SRV test								
Seizure load (N)	1500	1100	1100	700	1000	700	1500	1200
Friction coefficient	0.07	0.10	0.07	0.10	0.07	0.10	0.06	0.09
Oxidation stability test								
Amount of reduction in oxygen pressure (KPa)	20	60	38	75	39	70	21	65

[Table 4]

	Example 12	Example 13	Example 14	Example 15	Example 16	Example 17	Example 18	Example 19
Base oil Type	A-1	A-1	A-1	A-1	A-1	A-4	A-1	A-1
Content (% by mass)	Balance	Balance	Balance	Balance	Balance	Balance	Balance	Balance
Thickener (% by mass)								
B-1	-	10.0	-	-	-	-	-	-
B-3	10.0	-	10.0	10.0	10.0	10.0	10.0	10.0
Epoxy compound (% by mass)								
C-1	0.5	0.5	-	-	-	0.5	1.0	0.5
C-2	-	-	0.5	-	-	-	-	-
C-3	-	-	-	0.5	-	-	-	-
C-4	-	-	-	-	0.5	-	-	-
Antiwear additive (% by mass)								
D-5	1.0	1.0	1.0	1.0	1.0	1.0	5.0	1.0
D-7	1.0	1.0	1.0	1.0	1.0	1.0	5.0	-
D-9	1.0	1.0	1.0	1.0	1.0	1.0	5.0	-
SRV test Seizure load (N)	1500	1400	1400	1400	1400	1500	1700	1200

[Table 5]

	Example 20	Comparative Example 14	Comparative Example 15	Comparative Example 16	Comparative Example 17
Base oil Type	A-1	A-1	A-1	A-1	A-1
Content (% by mass)	Balance	Balance	Balance	Balance	Balance
Thickener (% by mass)					
B-1	-	-	-	-	10.0
B-3	10.0	10.0	10.0	10.0	-
Epoxy compound (% by mass)					
C-1	0.5	-	0.5	-	-
C-2	-	-	-	-	-
C-3	-	-	-	-	-
C-4	-		-	-	-
Antiwear additive (% by mass)					

(continued)

	Example 20	Comparative Example 14	Comparative Example 15	Comparative Example 16	Comparative Example 17
D-5	-	1.0	-	-	0.5
D-7	1.0	1.0	-	-	1.0
D-9	-	1.0	-	-	3.0
D-10	-	--	-	-	1.0
SRV test					
Seizure load (N)	1100	800	400	400	800
Friction coefficient	-	-	-	-	0.07

[Table 6]

	Example 21	Example 22	Example 23	Example 24	Example 25	Example 26	Example 27	Example 28
Base oil								
Type	A-1	A-1	A-1	A-1	A-1	A-2	A-1	A-1
Content (% by mass)	Balance	Balance	Balance	Balance	Balance	Balance	Balance	Balance
Thickener (% by mass)								
B-1	10.0	-	10.0	10.0	10.0	10.0	10.0	10.0
B-2	-	10.0	-	-	-	-	-	-
Epoxy compound (% by mass)								
C-1	0.5	0.5	-	-	-	0.5	1.0	0.5
C-2	-	-	0.5	-	-	-	-	-
C-3	-	-	-	0.5	-	-	-	-
C-4	-	-	-	-	0.5	-	-	-
Antiwear additive (% by mass)								
D-5	0.5	0.5	0.5	0.5	0.5	0.5	2.0	-
D-7	1.0	1.0	1.0	1.0	1.0	1.0	3.0	1.0
D-9	3.0	3.0	3.0	3.0	3.0	3.0	5.0	3.0
D-10	1.0	1.0	1.0	1.0	1.0	1.0	3.0	1.0
SRV test								
Seizure load (N)	1600	1500	1500	1500	1500	1600	1700	1400
Friction coefficient	0.04	0.05	0.05	0.05	0.05	0.04	0.04	0.04

Industrial Applicability

[0085] The grease composition of the present invention is a grease that is significantly improved in terms of stability, lubricity and friction characteristics and that is excellent in reliability so that the initial characteristics may be maintained and the effect of wear-resistance may be kept even under severe lubrication conditions, as compared with a conventional grease. Accordingly, the grease composition of the present invention may be suitably used as a lubricant for various machines, vehicles, driving force transmission mechanisms and the like that are required to have anti-wear performance

and energy saving performance.

Claims

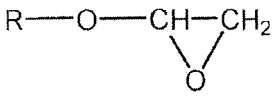
1. A grease composition comprising:

   at least one base oil selected from a mineral oil and a synthetic oil; and
   based on the total amount of the grease composition,

   2 to 35% by mass of a thickener;
   0.05 to 5% by mass of an epoxy compound; and
   0.1 to 20% by mass of an antiwear additive.

2. The grease composition according to claim 1, wherein the epoxy compound is at least one selected from a phenyl glycidyl ether type epoxy compound, an alkyl glycidyl ether type epoxy compound, a glycidyl ester type epoxy compound, an alicyclic epoxy compound, an epoxidized aliphatic monoester and an epoxidized vegetable oil.

3. The grease composition according to claim 2, wherein the epoxy compound is an alkyl glycidyl ether represented by the following formula (1):



(1)

   wherein R represents a linear or branched alkyl group having 6 to 20 carbon atoms.

4. The grease composition according to claim 3, wherein the epoxy compound is at least one selected from octyl glycidyl ether, 2-ethylhexyl glycidyl ether, nonyl glycidyl ether, decyl glycidyl ether, undecyl glycidyl ether and dodecyl glycidyl ether.

5. The grease composition according to any one of claims 1 to 3, wherein the antiwear additive is at least one selected from an oxygen-containing compound, a nitrogen-containing compound, an organophosphorus compound, an organosulfur compound, an organomolybdenum compound, an alkaline earth metal compound and an organozinc compound.

6. The grease composition according to claim 5, wherein
   the oxygen-containing compound is at least one selected from an ester, an alcohol, an ether and a carboxylic acid,
   the nitrogen-containing compound is at least one selected from an aliphatic amine, an aliphatic amide and an aliphatic imide,
   the organophosphorus compound is at least one selected from a phosphoric acid ester, a phosphorous acid ester and an acidic phosphoric acid ester amine salt,
   the organosulfur compound is at least one selected from a sulfide compound, and sulfurized fat and oil,
   the organomolybdenum compound is at least one selected from molybdenum dithiocarbamate and molybdenum dithiophosphate,
   the alkaline earth metal compound is at least one selected from alkaline earth metal sulfonate salt, phenate salt and salicylate salt, and
   the organozinc compound is at least one selected from a zinc dialkyl, a zinc dialkyldithiophosphate and a zinc dialkyldithiocarbamate.

7. The grease composition according to any one of claims 1 to 3, wherein the antiwear additive is a solid lubricant.

8. The grease composition according to claim 7, wherein the solid lubricant is at least one selected from molybdenum disulfide, graphite, a tetrafluoroethylene resin powder and boron nitride.

9. The grease composition according to any one of claims 1 to 8, wherein the synthetic oil type is at least one selected from a hydrocarbon oil, a diester, a polyol ester and a polyalkylene glycol compound.

10. The grease composition according to any one of claims 1 to 8, wherein the thickener is at least one selected from lithium soaps, calcium soaps, aluminum soaps and a urea compound.

11. The grease composition according to claim 5, containing, as the antiwear additive, an organomolybdenum compound and an organozinc compound.

12. The grease composition according to claim 5, containing, as the antiwear additive, an organomolybdenum compound, an organozinc compound and an organosulfur compound.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2013/065600

A. CLASSIFICATION OF SUBJECT MATTER
See extra sheet.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
See extra sheet.

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
Jitsuyo Shinan Koho 1922-1996 Jitsuyo Shinan Toroku Koho 1996-2013
Kokai Jitsuyo Shinan Koho 1971-2013 Toroku Jitsuyo Shinan Koho 1994-2013

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	JP 6-057284 A (Kyodo Yushi Co., Ltd.), 01 March 1994 (01.03.1994), paragraphs [0004] to [0020]; examples (Family: none)	1,2,5,6,9-11 1-12
Y	JP 2008-274076 A (NTN Corp.), 13 November 2008 (13.11.2008), paragraphs [0003] to [0027]; examples & US 2010/0092118 A1 & WO 2008/136386 A1 & DE 112008001121 T	1-12
Y	JP 9-003471 A (The Lubrizol Corp.), 07 January 1997 (07.01.1997), paragraphs [0073] to [0089] & US 5622923 A1 & EP 748862 A2 & DE 69624626 D & SG 38971 A & AU 721561 B & CA 2178692 A1	1-12

☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
05 August, 2013 (05.08.13)	13 August, 2013 (13.08.13)
Name and mailing address of the ISA/ Japanese Patent Office Facsimile No.	Authorized officer Telephone No.

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2013/065600

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP 2010-168593 A (Kyodo Yushi Co., Ltd.), 05 August 2010 (05.08.2010), paragraphs [0005] to [0021] (Family: none)	1-12
X	JP 5-230486 A (Kyodo Yushi Co., Ltd.), 07 September 1993 (07.09.1993),	1,2,5-8,10, 11
Y	paragraphs [0004] to [0021]; examples (Family: none)	1-12
Y	JP 9-157678 A (NTN Corp.), 17 June 1997 (17.06.1997), paragraphs [0010] to [0060]; examples & US 5789354 A1	1-12

Form PCT/ISA/210 (continuation of second sheet) (July 2009)

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2013/065600

Continuation of A. CLASSIFICATION OF SUBJECT MATTER
(International Patent Classification (IPC))

C10M169/02(2006.01)i, *C10M101/02*(2006.01)i, *C10M105/36*(2006.01)i, *C10M105/38*(2006.01)i, *C10M107/34*(2006.01)i, *C10M115/08*(2006.01)i, *C10M117/02*(2006.01)i, *C10M125/02*(2006.01)i, *C10M125/22*(2006.01)i, *C10M125/26*(2006.01)i, *C10M129/06*(2006.01)i, *C10M129/16*(2006.01)i, *C10M129/18*(2006.01)i, *C10M129/40*(2006.01)i, *C10M129/54*(2006.01)i, *C10M129/76*(2006.01)i, *C10M133/06*(2006.01)i, *C10M133/16*(2006.01)i, *C10M135/06*(2006.01)i, *C10M135/10*(2006.01)i, *C10M135/18*(2006.01)i, *C10M135/22*(2006.01)i, *C10M135/30*(2006.01)i, *C10M137/04*(2006.01)i, *C10M137/08*(2006.01)i, *C10M137/10*(2006.01)i, *C10M147/02*(2006.01)i, *C10M159/22*(2006.01)i, *C10M159/24*(2006.01)i, *C10N10/02*(2006.01)n, *C10N10/04*(2006.01)n, *C10N10/06*(2006.01)n, *C10N10/12*(2006.01)n, *C10N30/06*(2006.01)n, *C10N40/04*(2006.01)n, *C10N50/10*(2006.01)n

(According to International Patent Classification (IPC) or to both national classification and IPC)

Continuation of B. FIELDS SEARCHED
Minimum documentation searched (International Patent Classification (IPC))

C10M169/02, C10M101/02, C10M105/36, C10M105/38, C10M107/34, C10M115/08, C10M117/02, C10M125/02, C10M125/22, C10M125/26, C10M129/06, C10M129/16, C10M129/18, C10M129/40, C10M129/54, C10M129/76, C10M133/06, C10M133/16, C10M135/06, C10M135/10, C10M135/18, C10M135/22, C10M135/30, C10M137/04, C10M137/08, C10M137/10, C10M147/02, C10M159/22, C10M159/24, C10N10/02, C10N10/04, C10N10/06, C10N10/12, C10N30/06, C10N40/04, C10N50/10

Minimum documentation searched (classification system followed by classification symbols)

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