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(54) **GREASE COMPOSITION FOR CONSTANT VELOCITY JOINT**

(57) The grease composition for constant velocity joints of the invention is **characterized by** comprising a lubricating base oil, a thickener, carbon black with a mean particle size of not greater than 500 nm, and an organic molybdenum compound. A grease composition for constant velocity joints according to the invention can

achieve a highly satisfactory balance between anti-flaking, anti-seizure and anti-wear without using lead compounds, and can sufficiently maintain these properties with long-term use.

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## Description

### Technical Field

5 [0001] The present invention relates to a grease composition for constant velocity joints.

### Background Art

10 [0002] Constant velocity joints are joints for a shaft transmitting a driving force from a transmission of a car to its tires. Types of constant velocity joints include fixed constant velocity joints such as Barfield joints, Rzeppa joints and under-cutting free joints, and slide type constant velocity joints such as double-offset joints, tripod joints, and cross-groove joints; and the like.

15 [0003] Constant velocity joints must exhibit performance such as anti-flaking, anti-seizure, anti-wear and low-friction properties. Such performance is ensured by using constant velocity joint greases that in most cases are produced by mixing a base grease comprising a lubricating oil base and lithium soap or urea-based thickener, with additives such as molybdenum disulfide and lead compounds.

[Patent document 1] Japanese Unexamined Patent Publication HEI No. 04-304300

[Patent document 2] Japanese Unexamined Patent Publication HEI No. 06-57283

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### Disclosure of the Invention

#### Problems to be Solved by the Invention

25 [0004] The higher performance and smaller and lighter weights of automobiles in recent years has led to a tendency toward increasing loads on constant velocity joints. However, the aforementioned conventional greases are not always satisfactory from the viewpoint of anti-flaking, anti-seizure, anti-wear and low-friction properties, and much room for improvement still remains for achieving high-performance, long-lived constant velocity joints used under heavy loads.

30 [0005] As mentioned above, lead compounds are sometimes used as additives in conventional greases, and the use of lead compounds is preferably minimized due to human safety and environmental considerations.

[0006] It is an object of the present invention to provide a grease composition for constant velocity joints that achieves a highly satisfactory balance between anti-flaking, anti-seizure, anti-wear and low-friction properties without using lead compounds, and that can sufficiently maintain these properties with long-term use.

#### Means for Solving the Problems

[0007] In order to achieve this object, the grease composition for constant velocity joints is characterized by comprising a lubricating base oil, a thickener, carbon black with a mean particle size of not greater than 500 nm, and an organic molybdenum compound.

40 [0008] By combining the aforementioned carbon black with an organic molybdenum compound and a thickener in a lubricating base oil, it is possible to achieve a high-level balance between anti-flaking, anti-seizure and anti-wear, and to sufficiently maintain these properties with long-term use. Thus, a grease composition for constant velocity joints according to the invention can effectively increase performance and lengthen the practical life of constant velocity joints without using lead compounds.

45 [0009] The grease composition for constant velocity joints according to the invention preferably further comprises at least one selected from among sulfur-based extreme pressure agents, phosphorus-based extreme pressure agents and zinc-based extreme pressure agents. This will permit further improvements in anti-flaking, anti-seizure and anti-wear.

50 [0010] Moreover, the grease composition for constant velocity joints according to the invention preferably also further comprises at least one selected from among complexes of fatty acid salts and carbonates, wherein the fatty acid is overbased by the carbonate, and organic acid salts. This will permit further improvements in anti-flaking and anti-seizure.

### Effect of the Invention

55 [0011] According to the invention there is provided a grease composition for constant velocity joints that can achieve a high-level balance between anti-flaking, anti-seizure and anti-wear, and can sufficiently maintain these properties with long-term use, without the use of lead compounds.

## Best Mode for Carrying Out the Invention

[0012] Preferred embodiments of the invention will now be described in detail.

[0013] As lubricating base oils for the grease composition for constant velocity joints of the invention there may be mentioned mineral oils and/or synthetic oils. As mineral oils there may be mentioned mineral oils obtained by lubricating oil production processes ordinarily employed in the petroleum refining industry, and more specifically these include naphthene-based and paraffin-based mineral oils purified by subjecting crude oil to atmospheric distillation or vacuum distillation to yield lubricating oil fractions, and then applying one or more types of treatment such as solvent deasphalting, solvent extraction, hydrocracking, solvent dewaxing, catalytic dewaxing, hydrogenation refining, sulfuric acid cleaning, clay treatment and the like.

[0014] As synthetic oils there may be mentioned, specifically, poly  $\alpha$ -olefins such as polybutene, 1-octene oligomers and 1-decene oligomers, and their hydrogenated forms; diesters such as ditridecyl glutarate, di-2-ethylhexyl adipate, diisodecyl adipate, ditridecyl adipate and di-3-ethylhexyl sebacate, polyol esters such as trimethylolpropane caprylate, trimethylolpropane pelargonate, pentaerythritol 2-ethyl hexanoate and pentaerythritol pelargonate, alkylnaphthalenes; alkylbenzenes; polyoxyalkylene glycols; polyphenyl ether; dialkyldiphenyl ether; silicone oils, and mixtures thereof.

[0015] The kinematic viscosity of the lubricating base oil at 100°C is preferably 2-40 mm<sup>2</sup>/s and more preferably 3-20 mm<sup>2</sup>/s. The viscosity index of the lubricating base oil is preferably 90 or greater and more preferably 100 or greater.

[0016] According to the invention, any one of the aforementioned lubricating base oils may be used alone or two or more thereof may be used in combination, but preferably naphthene-based mineral oils are used from the standpoint of excellent anti-flaking, anti-seizure and anti-wear. The boot used for the constant velocity joint is composed of a rubber material such as chloroprene rubber (CR) or chlorinated polyethylene rubber (CM), or a resin material such as a polyester-based thermoplastic elastomer (TPE) or the like, and naphthene-based mineral oils are preferred from the viewpoint of compatibility with the boot material.

[0017] Here, "compatibility with the boot material" means minimal adverse effect on the boot material and a property of adequately preventing grease leakage due to strength reduction or damage to the boot.

[0018] Also, "naphthene-based mineral oil" means the lubricating oil fraction obtained by refining of naphthene crude oil with a high naphthene content. Specifically, there may be mentioned lubricating oil fractions obtained by atmospheric distillation and vacuum distillation of naphthene crude oil, and then refinement by one type of treatment alone or two or more types of treatment in appropriate combination selected from among solvent deasphalting, solvent extraction, hydrocracking, solvent dewaxing, catalytic dewaxing, hydrogenation refining, sulfuric acid cleaning, clay treatment and the like.

[0019] The properties of the naphthene-based mineral oil used for the invention are not particularly restricted, but the naphthene-based mineral oil preferably has a %Cn of 35-70, more preferably 40-65 and even more preferably 45-60. Also, the naphthene-based mineral oil preferably has a %Cp of 20-55, more preferably 25-50 and even more preferably 30-45. Here the %Cn and %Cp are, respectively, the %Cn and %Cp measured based on "Standard Test Method for Calculation Distribution and Structural Group Analysis of Petroleum Oils by the n-d-M Method" specified by ASTM-D-3238.

[0020] The aniline point of the naphthene-based mineral oil is preferably not higher than 90°C, more preferably not higher than 85°C and even more preferably not higher than 80°C. Here, "aniline point" means the aniline point measured according to "Test Method for Petroleum Product Aniline Point and Mixed Aniline Point" of JIS K 2256.

[0021] The pour point of the naphthene-based mineral oil is preferably not higher than -20°C, more preferably not higher than -30°C and even more preferably not higher than -40°C. Here, "pour point" means the pour point measured according to "Test Method For Crude Oil and Petroleum Product Pour Point and Petroleum Product Clouding Point" of JIS K 2269.

[0022] From the standpoint of reducing adverse effects on thermal stability, the sulfur content of the naphthene-based mineral oil is preferably not greater than 1500 ppm by mass, more preferably not greater than 800 ppm by mass, even more preferably not greater than 500 ppm by mass, yet more preferably not greater than 100 ppm by mass and most preferably not greater than 50 ppm by mass based on the total amount of the naphthene-based mineral oil. Here, "sulfur content" means the sulfur content measured according to "Inductively Coupled Plasma-Atomic Emission Method", an attachment to "Crude Oil and Petroleum Products - Test Method For Sulfur Content" of JIS K 2541, and the sulfur content includes disulfide sulfurs, mercaptanes, alkyl sulfides, alkyl disulfides, thiophane, thiophene, sulfonic acid and the like.

[0023] From the standpoint of reducing adverse effects on thermal stability, the nitrogen content of the naphthene-based mineral oil is preferably not greater than 500 ppm by mass, more preferably not greater than 200 ppm by mass, even more preferably not greater than 150 ppm by mass, yet more preferably not greater than 100 ppm by mass and most preferably not greater than 50 ppm by mass based on the total amount of the naphthene-based mineral oil. Here, "nitrogen content" means the nitrogen content measured according to the trace coulometric titration method specified by "Crude Oil and Petroleum Products - Test Method For Nitrogen Content" of JIS K 2609, and the nitrogen content includes inorganic ammonia compounds such as ammonia, ammonium sulfate, ammonium carbonate and ammonium chloride, and heterocyclic compounds such as pyridine, quinoline and naphthene bases.

**[0024]** From the standpoint of low-temperature pour properties, the viscosity index of the naphthene-based mineral oil is preferably at least -10, more preferably at least 0, even more preferably at least 10, yet more preferably at least 20 and most preferably at least 30. Here, "viscosity index" means the viscosity index calculated according to the "Crude Oil and Petroleum Products - Kinematic viscosity Test Method and Viscosity Index Calculation Method" of JIS K 2283.

**[0025]** When the grease composition of the invention comprises a naphthene-based mineral oil, the content of the naphthene-based mineral oil is preferably 3-40 % by mass and more preferably 5-30 % by mass based on the total amount of the composition. If the naphthene-based mineral oil content is less than 3 % by mass, the addition will tend to be insufficient for improving the anti-flaking, anti-seizure, anti-wear and compatibility with the boot material. Also, if the naphthene-based mineral oil content exceeds 40 % by mass, an improving effect in the anti-flaking, anti-seizure and anti-wear will be obtained, but compatibility with the boot material will tend to be reduced.

**[0026]** As thickeners there may be used various types of thickeners including soap-based thickeners such as metal soaps and complex metal soaps, and non-soap-based thickeners such as bentone, silica gel and urea-based thickeners (urea compounds, urea/urethane compounds, urethane compounds, etc.). From the viewpoint of heat resistance, urea compounds, urea/urethane compounds, urethane compounds and their mixtures are preferred.

**[0027]** As specific examples of soap-based thickeners there may be mentioned sodium soaps, calcium soaps, aluminum soaps and lithium soaps.

**[0028]** As examples of urea-based thickeners there may be mentioned urea compounds such as diurea compounds, triurea compounds, tetraurea compounds and polyurea compounds (other than diurea compounds, triurea compounds and tetraurea compounds), urethane compounds such as urea compounds, urea/urethane compounds and diurethane compounds, and mixtures thereof, among which diurea compounds, urea/urethane compounds, diurethane compounds and mixtures thereof are preferred.

**[0029]** As preferred examples of urea-based thickeners there may be mentioned compounds represented by general formula (1) below. The compounds represented by general formula (1) include diurea compounds, urea/urethane compounds and diurethane compounds.

**[0030]**

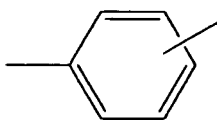


In general formula (1),  $\text{R}^1$  represents a divalent organic group and preferably a divalent hydrocarbon group. As divalent hydrocarbon groups there may be mentioned, specifically, straight-chain or branched alkylene groups, straight-chain or branched alkenylene groups, cycloalkylene groups, arylene groups, alkylarylene groups, arylalkylene groups and the like. The number of carbon atoms of the divalent organic group represented by  $\text{R}^1$  is preferably 6-20 and more preferably 6-15.

**[0031]** As preferred examples of divalent organic groups represented by  $\text{R}^1$  there may be mentioned ethylene, 2,2-dimethyl-4-methylhexylene and groups represented by the following formulas (2)-(11), among which groups represented by formulas (3) and (5) are preferred.

**[0032]**

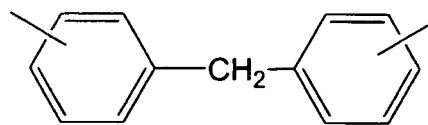
[Chemical Formula 1]



(2)

[Chemical Formula 2]

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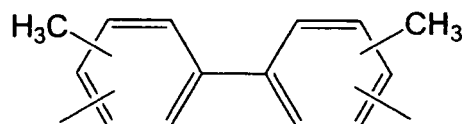


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[Chemical Formula 3]

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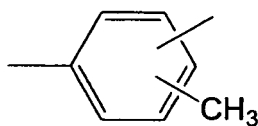


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[Chemical Formula 4]

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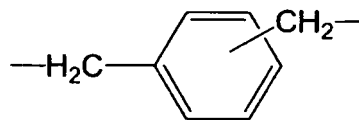


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[Chemical Formula 5]

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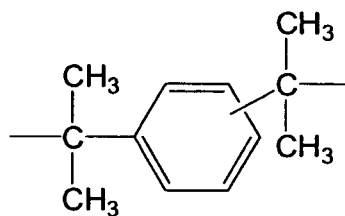


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[Chemical Formula 6]

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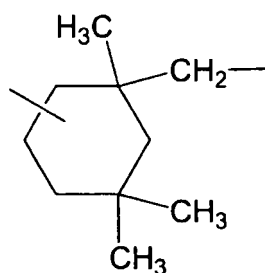


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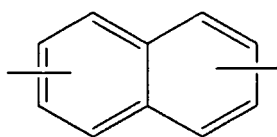
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## [Chemical Formula 7]



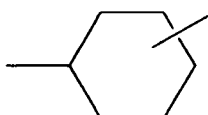
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## [Chemical Formula 8]



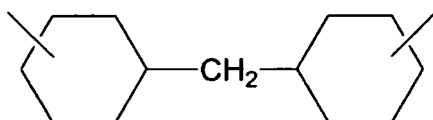
(9)

## [Chemical Formula 9]



(10)

## [Chemical Formula 10]



(11)

In general formula (1), A and B may be the same or different, and each represents an  $\text{NHR}^2$ ,  $\text{-NR}^3\text{R}^4$  or  $\text{-OR}^5$  group. Here,  $\text{R}^2$ ,  $\text{R}^3$ ,  $\text{R}^4$  and  $\text{R}^5$  may be the same or different, and each represents a monovalent organic group and preferably a C6-20 monovalent hydrocarbon group.

**[0033]** As examples of C6-20 monovalent hydrocarbon groups represented by  $\text{R}^2$ ,  $\text{R}^3$ ,  $\text{R}^4$  and  $\text{R}^5$  there may be mentioned straight-chain or branched alkyl, straight-chain or branched alkenyl, cycloalkyl, alkylcycloalkyl, aryl, alkylaryl

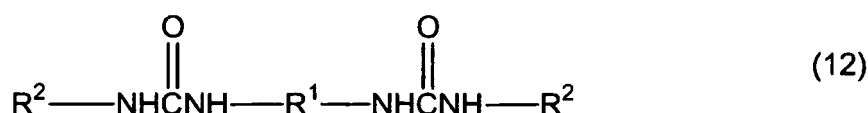
and arylalkyl. More specifically, there may be mentioned straight-chain or branched alkyl groups such as hexyl, heptyl, octyl, nonyl, decyl, undecyl, dodecyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl, octadecyl, nonadecyl and eicosyl; straight-chain or branched alkenyl groups such as hexenyl, heptenyl, octenyl, nonenyl, decenyl, undecenyl, dodecenyl, tetradecenyl, pentadecenyl, hexadecenyl, heptadecenyl, octadecenyl, nonadecenyl and eicosenyl; cyclohexyl groups; alkylcyclohexyl groups such as methylcyclohexyl, dimethylcyclohexyl, ethylcyclohexyl, diethylcyclohexyl, propylcyclohexyl, isopropylcyclohexyl, 1-methyl-3-propylcyclohexyl, butylcyclohexyl, amylcyclohexyl, amylmethylcyclohexyl, hexylcyclohexyl, heptylcyclohexyl, octylcyclohexyl, nonylcyclohexyl, decylcyclohexyl, undecylcyclohexyl, dodecylcyclohexyl, tridecylcyclohexyl and tetradecylcyclohexyl; aryl groups such as phenyl and naphthyl; alkylaryl groups such as tolyl, ethylphenyl, xylol, propylphenyl, cumenyl, methylphenyl, ethylphenyl, dimethylphenyl and propylphenyl; and arylalkyl groups such as benzyl, methylbenzyl and ethylbenzyl, among which alkyl, cycloalkyl, alkylcycloalkyl, aryl and alkylaryl groups are preferred from the viewpoint of heat resistance and anti-acoustic properties.

**[0034]** A compound represented by general formula (1) may be obtained, for example, by reacting a diisocyanate represented by  $\text{OCN-R}^1\text{-NCO}$  with a compound represented by  $\text{R}^2\text{NH}_2$ ,  $\text{R}^3\text{R}^4\text{NH}$  or  $\text{R}^5\text{OH}$  or a mixture thereof, in a base oil at 10-200°C.  $\text{R}^1$ ,  $\text{R}^2$ ,  $\text{R}^3$ ,  $\text{R}^4$  and  $\text{R}^5$  in the formulas representing the starting material compounds have the same respective definitions as  $\text{R}^1$ ,  $\text{R}^2$ ,  $\text{R}^3$ ,  $\text{R}^4$  and  $\text{R}^5$  of the compounds represented by general formula (1).

**[0035]** From the standpoint of heat resistance, particularly preferred compounds among those represented by general formula (1) are compounds wherein A and B are  $\text{NHR}^2$ , i.e. compounds represented by general formula (12) below. In general formula (12),  $\text{R}^1$  is a divalent organic group and  $\text{R}^2$  is a monovalent organic group, and they are equivalent to  $\text{R}^1$  and  $\text{R}^2$  mentioned above.

**[0036]**

### [Chemical Formula 11]



The thickener content is preferably at least 2 % by mass and more preferably at least 5 % by mass based on the total amount of the composition. If the thickener content is less than 2 % by mass the effect of adding the thickener will be insufficient, making it difficult to satisfactorily produce a grease from the grease composition. The thickener content is preferably not greater than 30 % by mass and more preferably not greater than 20 % by mass based on the total amount of the composition. If the thickener content is greater than 30 % by mass the grease composition will become excessively hard, making it difficult to obtain satisfactory lubricating performance.

**[0037]** The grease composition for constant velocity joints according to the invention also comprises as essential components carbon black with a mean particle size of not greater than 500 nm and an organic molybdenum compound, in addition to the aforementioned lubricating base oil and thickener.

**[0038]** Carbon black consists of black particles with a diameter of about 3-500 nm obtained by momentarily (for a few milliseconds) heating crude hydrocarbons (oil, gas, etc.) at high temperature (for example, 300-1800°C and preferably 800-1800°C) for conversion to carbon, and it is distinguished from graphite. For example, the crystal structure of graphite consists of hexagonal (or trigonal polygonal) flat sheets, whereas carbon black consists of unit particles of a type of amorphous carbon with fine crystals aggregated in a complex manner, and the fine crystals have a random layer structure with aggregation of several layers of aromatic planar molecules with average diameters of 20-30 Å. Carbon black also forms a structure with the unit particles linking together into chains, and acidic functional groups may be present on the surfaces of the particles. When graphite is used instead of carbon black, insufficient anti-flaking, anti-seizure and anti-wear properties are exhibited.

**[0039]** As mentioned above, the mean particle size of the carbon black used for the invention is preferably not greater than 500 nm and more preferably not greater than 100 nm. If the mean particle size of the carbon black is greater than 500 nm, insufficient anti-flaking, anti-seizure and anti-wear properties are exhibited. There is no particular restriction on the lower limit for the mean particle size, but from the standpoint of production ease and availability, it will normally be at least 10 nm and preferably at least 15 nm. Here, the "mean particle size" is the mean particle size (mean diameter) of the unit particles of the carbon black, and it is the average value of measurement with an electron microscope.

**[0040]** The process for producing the carbon black used for the invention is not particularly restricted so long as the mean particle size is within the aforementioned range. As typical production processes there may be mentioned the furnace process, acetylene process, lamp process, thermal process and channel process.

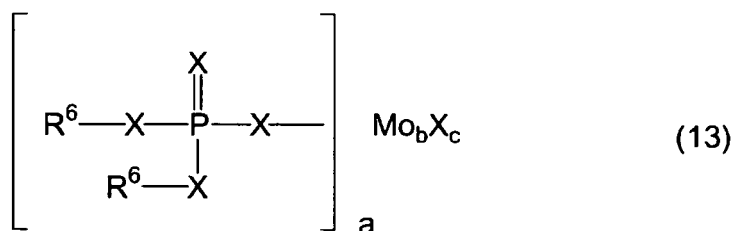
**[0041]** The carbon black content is preferably at least 0.05 % by mass and more preferably at least 0.1 % by mass

based on the total amount of the composition. If the carbon black content is below this lower limit, the effect of improving the anti-flaking, anti-seizure and anti-wear by addition of the carbon black will tend to be inadequate. The carbon black content is preferably at least not greater than 15 % by mass and more preferably not greater than 10 % by mass based on the total amount of the composition. If the carbon black content is above this upper limit, no further effect of improving the anti-flaking, anti-seizure and anti-wear will be achieved corresponding to the increased content.

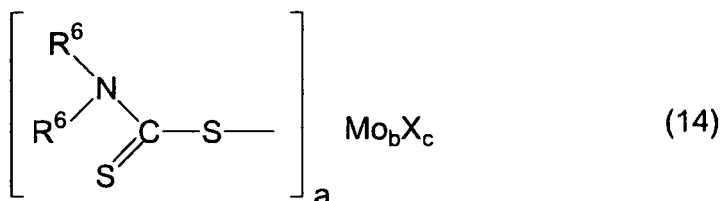
**[0042]** As examples of organic molybdenum compounds to be used for the invention there may be mentioned the phosphoric acid or thiophosphoric acid ester derivatives represented by general formula (13) below, and the dithiocarbamic acid ester derivatives represented by general formula (14) below.

**[0043]**

### [Chemical Formula 12]



### [Chemical Formula 13]



In general formulas (13) and (14), each  $\text{R}^6$  may be the same or different and represents a C1 or greater hydrocarbon group, each X may be the same or different and represents oxygen or sulfur, and a, b and c each represent an integer of 1-6.

**[0044]** As examples of hydrocarbon groups represented by  $\text{R}^6$  in general formulas (13) and (14) there may be mentioned C1-24 alkyl, C5-7 cycloalkyl, C6-11 alkylcycloalkyl, C6-18 aryl, C7-24 alkylaryl and C7-12 arylalkyl.

**[0045]** As the aforementioned alkyl groups there may be mentioned, specifically, methyl, ethyl, propyl (including all branched isomers), butyl (including all branched isomers), pentyl (including all branched isomers), hexyl (including all branched isomers), heptyl (including all branched isomers), octyl (including all branched isomers), nonyl (including all branched isomers), decyl (including all branched isomers), undecyl (including all branched isomers), dodecyl (including all branched isomers), tridecyl (including all branched isomers), tetradecyl (including all branched isomers), pentadecyl (including all branched isomers), hexadecyl (including all branched isomers), heptadecyl (including all branched isomers), octadecyl (including all branched isomers), nonadecyl (including all branched isomers), eicosyl (including all branched isomers), heneicosyl (including all branched isomers), docosyl (including all branched isomers), tricosyl (including all branched isomers) and tetracosyl (including all branched isomers). As the aforementioned cycloalkyl groups there may be mentioned, specifically, cyclopentyl, cyclohexyl and cycloheptyl.

**[0046]** As the aforementioned alkylcycloalkyl groups there may be mentioned, specifically, methylcyclopentyl (including all substituted isomers), ethylcyclopentyl (including all substituted isomers), dimethylcyclopentyl (including all substituted isomers), propylcyclopentyl (including all branched isomers and substituted isomers), methylethylcyclopentyl (including all substituted isomers), trimethylcyclopentyl (including all substituted isomers), butylcyclopentyl (including all branched isomers and substituted isomers), methylpropylcyclopentyl (including all branched isomers and substituted isomers), diethylcyclopentyl (including all substituted isomers), dimethylethylcyclopentyl (including all substituted isomers), methylcyclohexyl (including all substituted isomers), ethylcyclohexyl (including all substituted isomers), dimethylcyclohexyl



(including all substituted isomers), propylcyclohexyl (including all branched isomers and substituted isomers), methyl-ethylcyclohexyl (including all substituted isomers), trimethylcyclohexyl (including all substituted isomers), butylcyclohexyl (including all branched isomers and substituted isomers), methylpropylcyclohexyl (including all branched isomers and substituted isomers), diethylcyclohexyl (including all substituted isomers), dimethylethylcyclohexyl (including all substituted isomers), methylcycloheptyl (including all substituted isomers), ethylcycloheptyl (including all substituted isomers), dimethylcycloheptyl (including all substituted isomers), propylcycloheptyl (including all branched isomers and substituted isomers), methylethylcycloheptyl (including all substituted isomers), trimethylcycloheptyl (including all substituted isomers), butylcycloheptyl (including all branched isomers and substituted isomers), methylpropylcycloheptyl (including all branched isomers and substituted isomers), diethylcycloheptyl (including all substituted isomers) and dimethylethylcycloheptyl (including all substituted isomers).

**[0047]** As the aforementioned aryl groups there may be mentioned, specifically, phenyl and naphthyl.

**[0048]** As the aforementioned alkylaryl groups there may be mentioned, specifically, tolyl (including all substituted isomers), xylyl (including all substituted isomers), ethylphenyl (including all substituted isomers), propylphenyl (including all branched isomers and substituted isomers), methylethylphenyl (including all substituted isomers), trimethylphenyl (including all substituted isomers), butylphenyl (including all branched isomers and substituted isomers), methylpropylphenyl (including all branched isomers and substituted isomers), diethylphenyl (including all substituted isomers), dimethylethylphenyl (including all substituted isomers), pentylphenyl (including all branched isomers and substituted isomers), hexylphenyl (including all branched isomers and substituted isomers), heptylphenyl (including all branched isomers and substituted isomers), octylphenyl (including all branched isomers and substituted isomers), nonylphenyl (including all branched isomers and substituted isomers), decylphenyl (including all branched isomers and substituted isomers), undecylphenyl (including all branched isomers and substituted isomers), dodecylphenyl (including all branched isomers and substituted isomers), tridecylphenyl (including all branched isomers and substituted isomers), tetradecylphenyl (including all branched isomers and substituted isomers), pentadecylphenyl (including all branched isomers and substituted isomers), hexadecylphenyl (including all branched isomers and substituted isomers), heptadecylphenyl (including all branched isomers and substituted isomers) and octadecylphenyl (including all branched isomers and substituted isomers).

**[0049]** As examples of the aforementioned arylalkyl groups there may be mentioned benzyl, phenethyl, phenylpropyl (including all branched isomers) and phenylbutyl (including all branched isomers).

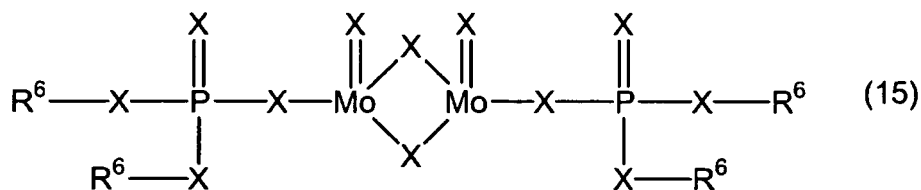
**[0050]** As specific compounds represented by general formulas (13) and (14) above there may be mentioned molybdenum phosphate, molybdenum thiophosphate, molybdenum dithiophosphate and molybdenum dithiocarbamate.

**[0051]** The phosphoric acid or thiophosphoric acid ester derivatives represented by general formula (13) and the dithiocarbamic acid ester derivatives represented by general formula (14) are usually obtained by reacting a phosphoric acid ester, thiophosphoric acid ester or dithiocarbamic acid ester with an inorganic molybdenum compound (molybdenum trioxide, molybdic acid or its salt, etc.), together with a sulfur source if necessary.

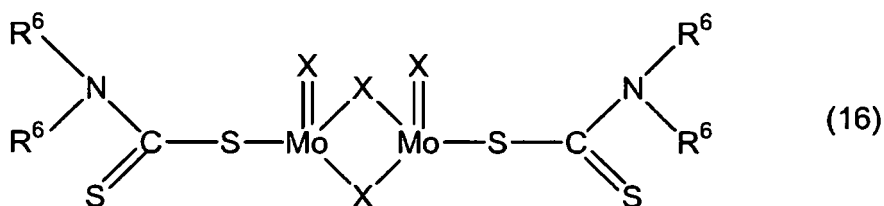
**[0052]** Molybdenum can adopt different valence states, and therefore the aforementioned reaction generally yields a mixture of compounds. The most typical compounds among these are compounds represented by the following formulas (15) and (16).

**[0053]**

#### [Chemical Formula 14]



[Chemical Formula 15]



(wherein  $R^6$  and  $X$  have the same definitions as  $R^6$  and  $X$  in formula (13)).

According to the invention, any one of the compounds represented by general formulas (13) and (14) above may be used alone as an organic molybdenum compound or both may be used in combination, but from the standpoint of thermal stability it is preferred to use a compound represented by general formula (14).

The organic molybdenum compound content is preferably at least 0.1 % by mass and more preferably at least 0.5 % by mass based on the total amount of the composition. If the organic molybdenum compound content is not at least 0.1 % by mass, the anti-flaking, anti-seizure and anti-wear of the grease will not be adequate. The upper limit is 20 % by mass and preferably 10 % by mass. If the content exceeds 20 % by mass, no further improvement in anti-flaking, anti-seizure and anti-wear will be obtained corresponding to the greater amount.

**[0054]** The grease composition for constant velocity joints according to the invention comprises the lubricating base oil, the thickener, the carbon black with a mean particle size of not greater than 500 nm and the organic molybdenum compound as described above, but it also preferably further contains at least one selected from among sulfur-based extreme pressure agents, phosphorus-based extreme pressure agents and zinc-based extreme pressure agents.

**[0055]** As sulfur-based extreme pressure agents there may be mentioned dihydrocarbyl polysulfides, sulfurized esters, sulfurized mineral oils, thiazole compounds and thiadiazole compounds.

**[0056]** Dihydrocarbyl polysulfides are sulfur-based compounds generally known as polysulfides or sulfurized olefins, and specifically they are represented by the following general formula (17).

[0057]



In general formula (17), R<sup>7</sup> and R<sup>8</sup> may be the same or different and each represents C3-20 straight-chain or branched alkyl, C6-20 aryl, C6-20 alkylaryl or C6-20 arylalkyl, and x represents an integer of 2-6 and preferably 2-5.

**[0058]** As alkyl groups represented by R<sup>7</sup> and R<sup>8</sup> above there may be mentioned, specifically, n-propyl, isopropyl, n-butyl, isobutyl, sec-butyl, tert-butyl, straight-chain or branched pentyl, straight-chain or branched hexyl, straight-chain or branched heptyl, straight-chain or branched octyl, straight-chain or branched nonyl, straight-chain or branched decyl, straight-chain or branched undecyl, straight-chain or branched dodecyl, straight-chain or branched tridecyl, straight-chain or branched tetradecyl, straight-chain or branched pentadecyl, straight-chain or branched hexadecyl, straight-chain or branched heptadecyl, straight-chain or branched octadecyl, straight-chain or branched nonadecyl and straight-chain or branched eicosyl.

**[0059]** As aryl groups represented by R<sup>7</sup> and R<sup>8</sup> there may be mentioned, specifically, phenyl and naphthyl.

**[0060]** As alkylaryl groups represented by R<sup>7</sup> and R<sup>8</sup> there may be mentioned, specifically, tolyl (including all structural isomers), ethylphenyl (including all structural isomers), straight-chain or branched propylphenyl (including all structural isomers), straight-chain or branched butylphenyl (including all structural isomers), straight-chain or branched pentylphenyl (including all structural isomers), straight-chain or branched hexylphenyl (including all structural isomers), straight-chain or branched heptylphenyl (including all structural isomers), straight-chain or branched octylphenyl (including all structural isomers), straight-chain or branched nonylphenyl (including all structural isomers), straight-chain or branched decylphenyl (including all structural isomers), straight-chain or branched undecylphenyl (including all structural isomers), straight-chain or branched dodecylphenyl (including all structural isomers), xylyl (including all structural isomers), ethylmethylphenyl (including all structural isomers), diethylphenyl (including all structural isomers), di(straight-chain or branched) propylphenyl (including all structural isomers), di(straight-chain or branched) butylphenyl (including all structural isomers), methylnaphthyl (including all structural isomers), ethylnaphthyl (including all structural isomers), straight-chain or branched propylnaphthyl (including all structural isomers), straight-chain or branched butylnaphthyl (including all structural isomers), dimethylnaphthyl (including all structural isomers), ethylmethylnaphthyl (including all structural isomers), diethylnaphthyl (including all structural isomers), di(straight-chain or branched) propylnaphthyl (including all structural isomers) and di(straight-chain or branched) butylnaphthyl (including all structural isomers).

**[0061]** As arylalkyl groups represented by  $R^7$  and  $R^8$  there may be mentioned, specifically, benzyl, phenylethyl (including all isomers) and phenylpropyl (including all isomers).

**[0062]**  $R^7$  and  $R^8$  are both preferably C3-18 alkyl, C6-8 aryl, C7-8 alkylaryl or C7-8 arylalkyl derived from propylene, 1-butene or isobutylene.

**[0063]** Specifically, as preferred alkyl groups there may be mentioned isopropyl, isopropyl, branched hexyl derived from propylene dimer (including all branched isomers), branched nonyl derived from propylene trimer (including all branched isomers), branched dodecyl derived from propylene tetramer (including all branched isomers), branched pentadecyl derived from propylene pentamer (including all branched isomers), branched octadecyl derived from propylene hexamer (including all branched isomers), sec-butyl, tert-butyl, branched octyl derived from 1-butene dimer (including all branched isomers), branched octyl derived from isobutylene dimer (including all branched isomers), branched dodecyl derived from 1-butene trimer (including all branched isomers), branched dodecyl derived from isobutylene trimer (including all branched isomers), branched hexadecyl derived from 1-butene tetramer (including all branched isomers) and branched hexadecyl derived from isobutylene tetramer (including all branched isomers). Phenyl may be mentioned as a preferred aryl group. As preferred alkylaryl groups there may be mentioned tolyl (including all structural isomers), ethylphenyl (including all structural isomers) and xylyl (including all structural isomers). As preferred arylalkyl groups there may be mentioned benzyl and phenethyl (including all isomers).

**[0064]** From the standpoint of achieving superior anti-flaking and anti-seizure, more preferably  $R^7$  and  $R^8$  each separately represent a C3-18 branched alkyl group derived from ethylene or propylene, and most preferably a C6-15 branched alkyl group derived from ethylene or propylene.

**[0065]** The dihydrocarbyl polysulfide used may be one with any sulfur content, but from the viewpoint of anti-flaking and anti-seizure, it is preferred to use one with a sulfur content of 10-55 % by mass and preferably 20-50 % by mass.

**[0066]** As specific examples of sulfurized esters there may be mentioned esters obtained by using desired methods for sulfidization of animal and vegetable oils such as beef tallow, lard, fish oil, rapeseed oil and soybean oil; unsaturated fatty acid esters obtained by reacting unsaturated fatty acids (including oleic acid, linoleic acid and fatty acids extracted from the aforementioned animal and vegetable oils) with various alcohols; and mixtures thereof, which have been sulfurized by any desired process.

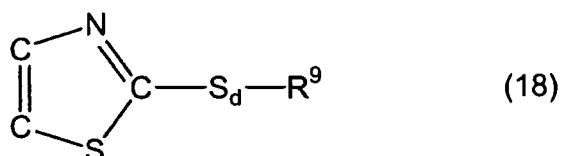
**[0067]** Although the sulfurized ester may have any desired sulfur content, from the standpoint of anti-flaking and anti-seizure the sulfur content is usually 2-40 % by mass and preferably 5-35 % by mass.

**[0068]** Sulfurized mineral oils are obtained by dissolving elemental sulfur in mineral oils. There are no particular restrictions on mineral oils to be used for the invention, and specifically there may be mentioned the mineral oil-based lubricating base oils mentioned above as examples of lubricating base oils. The elemental sulfur may be used in any of various forms such as bulk, powder or molten liquid forms, but using it in powder or molten liquid form is preferred as it allows efficient dissolution in the base oil. Using elemental sulfur in molten liquid form is advantageous in that the liquids will mix together allowing the dissolution process to be accomplished in a very short time, but this requires the handling temperature to be above the melting point of elemental sulfur and therefore necessitates special equipment such as heating devices, while handling in such high-temperature environments is dangerous and difficult. In contrast, elemental sulfur powder is inexpensive and easy to manage while its dissolution time is sufficiently short, and it is therefore particularly preferred. There are no particular restrictions on the sulfur content of the sulfurized mineral oil, but for most purposes it is preferably 0.05-1.0 % by mass and more preferably 0.1-0.5 % by mass based on the total amount of the sulfurized mineral oil.

**[0069]** As thiazole compounds there are preferably used compounds represented by the following general formulas (18) and (19).

**[0070]**

#### [Chemical Formula 16]



## 10



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[0072]

## 30



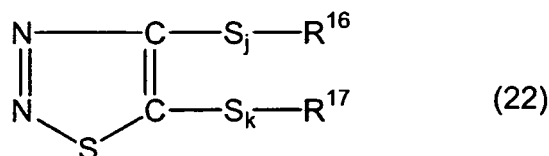
## 40



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## [Chemical Formula 20]



[In general formulas (20), (21) and (22),  $\text{R}^{12}$ ,  $\text{R}^{13}$ ,  $\text{R}^{14}$ ,  $\text{R}^{15}$ ,  $\text{R}^{16}$  and  $\text{R}^{17}$  may be the same or different and each represents hydrogen or a C1-20 hydrocarbon group, and c, d, e, f, g and h may be the same or different and each represents an integer of 0-8.] As mentioned above,  $\text{R}^{12}$ ,  $\text{R}^{13}$ ,  $\text{R}^{14}$ ,  $\text{R}^{15}$ ,  $\text{R}^{16}$  and  $\text{R}^{17}$  in general formulas (20)-(22) above each represent hydrogen or a C1-20 hydrocarbon group, but  $\text{R}^{12}$ ,  $\text{R}^{13}$ ,  $\text{R}^{14}$ ,  $\text{R}^{15}$ ,  $\text{R}^{16}$  and  $\text{R}^{17}$  are preferably each hydrogen or a C1-18 hydrocarbon group and more preferably hydrogen or a C1-12 hydrocarbon group. Also as mentioned above, c, d, e, f, g and h in general formulas (7)-(9) each represent an integer of 0-3, but c, d, e, f, g and h are preferably each an integer of 0-2. As specific examples of such thiadiazole compounds there may be mentioned 2,5-bis(n-hexyldithio)-1,3,4-thiadiazole, 2,5-bis(n-octyldithio)-1,3,4-thiadiazole, 2,5-bis(n-nonyldithio)-1,3,4-thiadiazole, 2,5-bis(1,1,3,3-tetramethylbutyldithio)-1,3,4-thiadiazole, 3,5-bis(n-hexyldithio)-1,2,4-thiadiazole, 3,5-bis(n-octyldithio)-1,2,4-thiadiazole, 3,5-bis(n-nonyldithio)-1,2,4-thiadiazole, 3,5-bis(1,1,3,3-tetramethylbutyldithio)-1,2,4-thiadiazole, 4,5-bis(n-hexyldithio)-1,2,3-thiadiazole, 4,5-bis(n-octyldithio)-1,2,3-thiadiazole, 4,5-bis(n-nonyldithio)-1,2,3-thiadiazole and 4,5-bis(1,1,3,3-tetramethylbutyldithio)-1,2,3-thiadiazole.

**[0073]** As sulfur-based extreme pressure agents to be used for the invention there are particularly preferred dihydrocarbyl polysulfides and sulfurized esters among those mentioned above, from the viewpoint of anti-flaking, anti-seizure and anti-wear.

**[0074]** When a sulfur-based extreme pressure agent is included in the grease composition for constant velocity joints of the invention, the content is not particularly restricted but is preferably 0.05-20 % by mass, more preferably 0.1-15 % by mass and even more preferably 0.5-10 % by mass based on the total composition.

**[0075]** As phosphorus-based additives there are preferred one or more selected from among phosphoric acid esters, acidic phosphoric acid esters, acidic phosphoric acid ester amine salts, phosphorous acid esters and phosphorothionates.

**[0076]** Among these phosphorus-based additives, the phosphoric acid esters, acidic phosphoric acid esters, acidic phosphoric acid ester amine salts and phosphorous acid esters are esters of phosphoric acid or phosphorous acid with alkanols or polyether-type alcohols, or derivatives thereof.

**[0077]** More specifically, as phosphoric acid esters there may be mentioned tributyl phosphate, triphenyl phosphate, trihexyl phosphate, triheptyl phosphate, trioctyl phosphate, trinonyl phosphate, tridecyl phosphate, triundecyl phosphate, tridodecyl phosphate, tritridecyl phosphate, tritetradecyl phosphate, tripentadecyl phosphate, trihexadecyl phosphate, triheptadecyl phosphate, trioctadecyl phosphate, trioleyl phosphate, triphenyl phosphate, tricresyl phosphate, trixylenyl phosphate, cresyldiphenyl phosphate and xylenyldiphenyl phosphate;

as acidic phosphoric acid esters there may be mentioned monobutyl acid phosphate, monopentyl acid phosphate, monohexyl acid phosphate, monoheptyl acid phosphate, monooctyl acid phosphate, monononyl acid phosphate, monodecyl acid phosphate, monoundecyl acid phosphate, monododecyl acid phosphate, monotridecyl acid phosphate, monotetradecyl acid phosphate, monopentadecyl acid phosphate, monoheptadecyl acid phosphate, monooctadecyl acid phosphate, monooleyl acid phosphate, dibutyl acid phosphate, dipentyl acid phosphate, dihexyl acid phosphate, diheptyl acid phosphate, dioctyl acid phosphate, dinonyl acid phosphate, didecyl acid phosphate, diundecyl acid phosphate, didodecyl acid phosphate, ditridecyl acid phosphate, ditetradecyl acid phosphate, dipentadecyl acid phosphate, dihexadecyl acid phosphate, diheptadecyl acid phosphate, dioctadecyl acid phosphate and dioleyl acid phosphate;

as acidic phosphoric acid ester amine salts there may be mentioned salts of amines, such as methylamine, ethylamine, propylamine, butylamine, pentylamine, hexylamine, heptylamine, octylamine, dimethylamine, diethylamine, dipropylamine, dibutylamine, dipentylamine, dihexylamine, diheptylamine, dioctylamine, trimethylamine, triethylamine, tripropylamine, tributylamine, tripentylamine, trihexylamine, triheptylamine and trioctylamine, with the aforementioned acidic phosphoric acid esters;

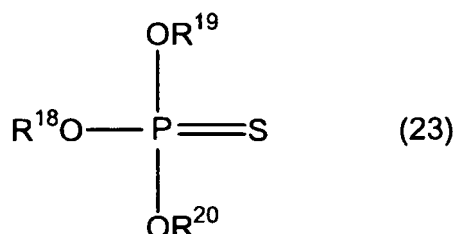
as chlorinated phosphoric acid esters there may be mentioned tris-dichloropropyl phosphate, tris-chloroethyl phosphate, tris-chlorophenyl phosphate and polyoxyalkylene-bis[di(chloroalkyl)] phosphate;

and as phosphorous acid esters there may be mentioned dibutyl phosphite, dipentyl phosphite, dihexyl phosphite, diheptyl phosphite, dioctyl phosphite, dinonyl phosphite, didecyl phosphite, diundecyl phosphite, didodecyl phosphite, dioleyl phosphite, diphenyl phosphite, dicresyl phosphite, tributyl phosphite, tripentyl phosphite, trihexyl phosphite, tri-

heptyl phosphite, trioctyl phosphite, trinonyl phosphite, tridecyl phosphite, triundecyl phosphite, tridodecyl phosphite, trioleyl phosphite, triphenyl phosphite and tricresyl phosphite. These compounds may also be used in admixture.

**[0078]** As phosphorothionates there are preferably used compounds represented by the following general formula (23).  
**[0079]**

### [Chemical Formula 21]



[In this formula, R<sup>18</sup>, R<sup>19</sup> and R<sup>20</sup> may be the same or different, and each represents C1-24 hydrocarbon group.]

As C1-24 hydrocarbon groups represented by R<sup>18</sup>, R<sup>19</sup> and R<sup>20</sup> there may be mentioned, specifically, alkyl, cycloalkyl, alkenyl, alkylcycloalkyl, aryl, alkylaryl and arylalkyl.

**[0080]** As examples of alkyl groups there may be mentioned alkyl groups such as methyl, ethyl, propyl, butyl, pentyl, hexyl, heptyl, octyl, nonyl, decyl, undecyl, dodecyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl and octadecyl (where the alkyl groups may be straight-chain or branched).

**[0081]** As examples of cycloalkyl groups there may be mentioned C5-7 cycloalkyl groups such as cyclopentyl, cyclohexyl and cycloheptyl. As examples of the aforementioned alkylcycloalkyl groups there may be mentioned C6-11 alkylcycloalkyl groups such as methylcyclopentyl, dimethylcyclopentyl, methylethylcyclopentyl, diethylcyclopentyl, methylcyclohexyl, dimethylcyclohexyl, methylethylcyclohexyl, diethylcyclohexyl, methylcycloheptyl, dimethylcycloheptyl, methylethylcycloheptyl and diethylcycloheptyl (with any positions of substitution of the alkyl groups on the cycloalkyl groups).

**[0082]** As examples of alkenyl groups there may be mentioned alkenyl groups such as butenyl, pentenyl, hexenyl, heptenyl, octenyl, nonenyl, decenyl, undecenyl, dodecenyl, tridecenyl, tetradecenyl, pentadecenyl, hexadecenyl, heptadecenyl and octadecenyl (where the alkenyl groups may be straight-chain or branched, and the double bonds may be at any positions).

**[0083]** As examples of aryl groups there may be mentioned aryl groups such as phenyl and naphthyl. As examples of the aforementioned alkylaryl groups there may be mentioned C7-18 alkylaryl groups such as tolyl, xylyl, ethylphenyl, propylphenyl, butylphenyl, pentylphenyl, hexylphenyl, heptylphenyl, octylphenyl, nonylphenyl, decylphenyl, undecylphenyl and dodecylphenyl (where the alkyl groups may be straight-chain or branched and substituted at any positions on the aryl groups).

**[0084]** As examples of arylalkyl groups there may be mentioned C7-12 arylalkyl groups such as benzyl, phenylethyl, phenylpropyl, phenylbutyl, phenylpentyl and phenylhexyl (where the alkyl groups may be straight-chain or branched).

**[0085]** The C1-24 hydrocarbon groups represented by R<sup>18</sup>, R<sup>19</sup> and R<sup>20</sup> are preferably alkyl, aryl or alkylaryl, and more preferably C4-18 alkyl, C7-24 alkylaryl or phenyl.

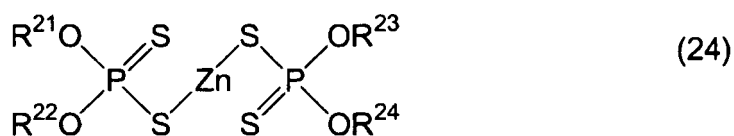
**[0086]** As phosphorothionates represented by general formula (23) there may be mentioned, specifically, tributyl phosphorothionate, tripentyl phosphorothionate, trihexyl phosphorothionate, triheptyl phosphorothionate, trioctyl phosphorothionate, trinonyl phosphorothionate, tridecyl phosphorothionate, triundecyl phosphorothionate, tridodecyl phosphorothionate, tritridecyl phosphorothionate, tritetradecyl phosphorothionate, tripentadecyl phosphorothionate, trihexadecyl phosphorothionate, triheptadecyl phosphorothionate, trioctadecyl phosphorothionate, trioleyl phosphorothionate, triphenyl phosphorothionate, tricresyl phosphorothionate, trixylenyl phosphorothionate, cresyldiphenyl phosphorothionate, xylenyldiphenyl phosphorothionate, tris(n-propylphenyl) phosphorothionate, tris(isopropylphenyl) phosphorothionate, tris(n-butylphenyl) phosphorothionate, tris(isobutylphenyl) phosphorothionate, tris(s-butylphenyl) phosphorothionate and tris(t-butylphenyl) phosphorothionate. These compounds may also be used in admixture.

**[0087]** When a phosphorus-based extreme pressure agent is included in the grease composition for constant velocity joints of the invention, the content is not particularly restricted but is preferably 0.01-15 % by mass, more preferably 0.05-10 % by mass and even more preferably 0.1-5 % by mass based on the total composition.

**[0088]** As zinc-based extreme pressure agents there may be mentioned the zinc dithiophosphate compounds represented by general formula (24) below, the zinc dithiocarbamate compounds represented by general formula (25) below and zinc salts of the phosphorus compounds represented by general formulas (26) and (27) below.

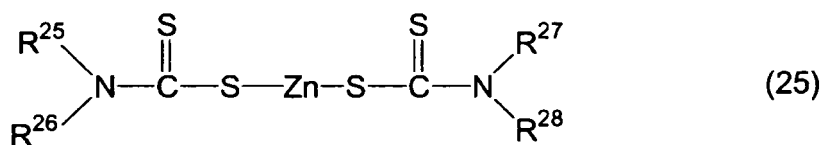
[0089]

[Chemical Formula 22]



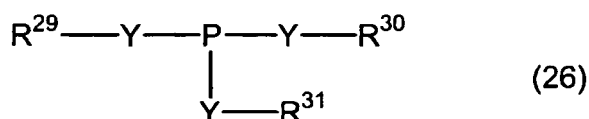
(wherein R<sup>21</sup>, R<sup>22</sup>, R<sup>23</sup> and R<sup>24</sup> may be the same or different and each represents a C1 or greater hydrocarbon group)

[Chemical Formula 23]



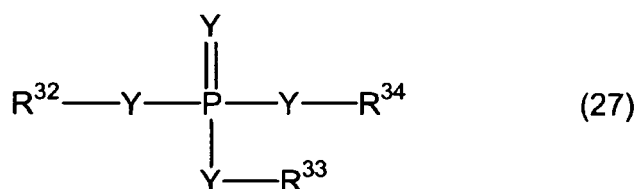
(wherein R<sup>25</sup>, R<sup>26</sup>, R<sup>27</sup> and R<sup>28</sup> may be the same or different and each represents a C1 or greater hydrocarbon group)

[Chemical Formula 24]



(wherein each Y represents oxygen or sulfur, at least two of the three Ys are oxygen, and R<sup>29</sup>, R<sup>30</sup> and R<sup>31</sup> may be the same or different and each represents hydrogen or a C1-30 hydrocarbon group)

[Chemical Formula 25]



(wherein each Y represents oxygen or sulfur, at least three of the four Y<sub>s</sub> are oxygen, and R<sup>32</sup>, R<sup>33</sup> and R<sup>34</sup> may be the same or different and each represents hydrogen or a C1-30 hydrocarbon group)

[0090] As examples of hydrocarbon groups represented by R<sup>21</sup>-R<sup>28</sup> in general formulas (24) and (25) there may be

mentioned C1-24 alkyl, C5-7 cycloalkyl, C6-11 alkylcycloalkyl, C6-18 aryl, C7-24 alkylaryl and C7-12 arylalkyl groups.

**[0091]** As alkyl groups there may be mentioned, specifically, methyl, ethyl, propyl (including all branched isomers), butyl (including all branched isomers), pentyl (including all branched isomers), hexyl (including all branched isomers), heptyl (including all branched isomers), octyl (including all branched isomers), nonyl (including all branched isomers), decyl (including all branched isomers), undecyl (including all branched isomers), dodecyl (including all branched isomers), tridecyl (including all branched isomers), tetradecyl (including all branched isomers), pentadecyl (including all branched isomers), hexadecyl (including all branched isomers), heptadecyl (including all branched isomers), octadecyl (including all branched isomers), nonadecyl (including all branched isomers), eicosyl (including all branched isomers), heneicosyl (including all branched isomers), docosyl (including all branched isomers), tricosyl (including all branched isomers) and tetracosyl (including all branched isomers).

**[0092]** As cycloalkyl groups there may be mentioned, specifically, cyclopentyl, cyclohexyl and cycloheptyl.

**[0093]** As alkylcycloalkyl groups there may be mentioned, specifically, methylcyclopentyl (including all substituted isomers), ethylcyclopentyl (including all substituted isomers), dimethylcyclopentyl (including all substituted isomers), propylcyclopentyl (including all branched isomers and substituted isomers), methylethylcyclopentyl (including all substituted isomers), trimethylcyclopentyl (including all substituted isomers), butylcyclopentyl (including all branched isomers and substituted isomers), methylpropylcyclopentyl (including all branched isomers and substituted isomers), diethylcyclopentyl (including all substituted isomers), dimethylethylcyclopentyl (including all substituted isomers), methylcyclohexyl (including all substituted isomers), ethylcyclohexyl (including all substituted isomers), dimethylcyclohexyl (including all substituted isomers), propylcyclohexyl (including all branched isomers and substituted isomers), methylethylcyclohexyl (including all substituted isomers), trimethylcyclohexyl (including all substituted isomers), butylcyclohexyl (including all branched isomers and substituted isomers), methylpropylcyclohexyl (including all branched isomers and substituted isomers), diethylcyclohexyl (including all substituted isomers), dimethylethylcyclohexyl (including all substituted isomers), methylcycloheptyl (including all substituted isomers), ethylcycloheptyl (including all substituted isomers), dimethylcycloheptyl (including all substituted isomers), propylcycloheptyl (including all branched isomers and substituted isomers), methylethylcycloheptyl (including all substituted isomers), trimethylcycloheptyl (including all substituted isomers), butylcycloheptyl (including all branched isomers and substituted isomers), methylpropylcycloheptyl (including all branched isomers and substituted isomers), diethylcycloheptyl (including all substituted isomers) and dimethylethylcycloheptyl (including all substituted isomers).

**[0094]** As examples of aryl groups there may be mentioned phenyl and naphthyl.

**[0095]** As examples of alkylaryl groups there may be mentioned tolyl (including all substituted isomers), xylyl (including all substituted isomers), ethylphenyl (including all substituted isomers), propylphenyl (including all branched isomers and substituted isomers), methylethylphenyl (including all substituted isomers), trimethylphenyl (including all substituted isomers), butylphenyl (including all branched isomers and substituted isomers), methylpropylphenyl (including all branched isomers and substituted isomers), diethylphenyl (including all substituted isomers), dimethylethylphenyl (including all substituted isomers), pentylphenyl (including all branched isomers and substituted isomers), hexylphenyl (including all branched isomers and substituted isomers), heptylphenyl (including all branched isomers and substituted isomers), octylphenyl (including all branched isomers and substituted isomers), nonylphenyl (including all branched isomers and substituted isomers), decylphenyl (including all branched isomers and substituted isomers), undecylphenyl (including all branched isomers and substituted isomers), dodecylphenyl (including all branched isomers and substituted isomers), tridecylphenyl (including all branched isomers and substituted isomers), tetradecylphenyl (including all branched isomers and substituted isomers), pentadecylphenyl (including all branched isomers and substituted isomers), hexadecylphenyl (including all branched isomers and substituted isomers), heptadecylphenyl (including all branched isomers and substituted isomers) and octadecylphenyl (including all branched isomers and substituted isomers).

**[0096]** As examples of arylalkyl groups there may be mentioned benzyl, phenethyl, phenylpropyl (including all branched isomers) and phenylbutyl (including all branched isomers).

**[0097]** As specific C1-30 hydrocarbon groups represent by  $R^{29}$ - $R^{34}$  for zinc salts of the phosphorus compounds represented by general formulas (26) and (27) above, there may be mentioned alkyl, cycloalkyl, alkenyl, alkylcycloalkyl, aryl, alkylaryl and arylalkyl groups.

**[0098]** As examples the aforementioned alkyl groups there may be mentioned alkyl groups such as methyl, ethyl, propyl, butyl, pentyl, hexyl, heptyl, octyl, nonyl, decyl, undecyl, dodecyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl and octadecyl (where the alkyl groups may be straight-chain or branched).

**[0099]** As examples of the aforementioned cycloalkyl groups there may be mentioned C5-7 cycloalkyl groups such as cyclopentyl, cyclohexyl and cycloheptyl. As examples of the aforementioned alkylcycloalkyl groups there may be mentioned C6-11 alkylcycloalkyl groups such as methylcyclopentyl, dimethylcyclopentyl, methylethylcyclopentyl, diethylcyclopentyl, methylcyclohexyl, dimethylcyclohexyl, methylethylcyclohexyl, diethylcyclohexyl, methylcycloheptyl, dimethylcycloheptyl, methylethylcycloheptyl and diethylcycloheptyl (with any positions of substitution of the alkyl groups on the cycloalkyl groups).

**[0100]** As examples of the aforementioned alkenyl groups there may be mentioned alkenyl groups such as butenyl,



pentenyl, hexenyl, heptenyl, octenyl, nonenyl, decenyl, undecenyl, dodecenyl, tridecenyl, tetradecenyl, pentadecenyl, hexadecenyl, heptadecenyl and octadecenyl (where the alkenyl groups may be straight-chain or branched, and the double bonds may be at any positions).

**[0101]** As examples of the aforementioned aryl groups there may be mentioned aryl groups such as phenyl and naphthyl. As examples of the aforementioned alkylaryl groups there may be mentioned C7-18 alkylaryl groups such as tolyl, xylol, ethylphenyl, propylphenyl, butylphenyl, pentylphenyl, hexylphenyl, heptylphenyl, octylphenyl, nonylphenyl, decylphenyl, undecylphenyl and dodecylphenyl (where the alkyl groups may be straight-chain or branched and substituted at any positions on the aryl groups).

**[0102]** As examples of the aforementioned arylalkyl groups there may be mentioned C7-12 arylalkyl groups such as benzyl, phenylethyl, phenylpropyl, phenylbutyl, phenylpentyl and phenylhexyl (where the alkyl groups may be straight-chain or branched).

**[0103]** The C1-30 hydrocarbon groups represented by  $R^{29}$ - $R^{34}$  are preferably C1-30 alkyl groups or C6-24 aryl groups, and are more preferably C3-18 alkyl groups and even more preferably C4-12 alkyl groups.

**[0104]**  $R^{29}$ ,  $R^{30}$  and  $R^{31}$  may be the same or different and each represents hydrogen or one of the aforementioned hydrocarbon groups, but preferably one to three from among  $R^{29}$ ,  $R^{30}$  and  $R^{31}$  are the aforementioned hydrocarbon groups, more preferably one or two are the aforementioned hydrocarbon groups, and even more preferably two are the aforementioned hydrocarbon groups.

**[0105]** Also,  $R^{32}$ ,  $R^{33}$  and  $R^{34}$  may be the same or different and each represents hydrogen or one of the aforementioned hydrocarbon groups, but preferably one to three from among  $R^{32}$ ,  $R^{33}$  and  $R^{34}$  are the aforementioned hydrocarbon groups, more preferably one or two are the aforementioned hydrocarbon groups, and even more preferably two are the aforementioned hydrocarbon groups.

**[0106]** In the phosphorus compounds represented by general formula (26), at least two of the three Ys must be oxygen, but preferably all of the Ys are oxygen.

**[0107]** In the phosphorus compounds represented by general formula (27), at least three of the four Ys must be oxygen, but preferably all of the Ys are oxygen.

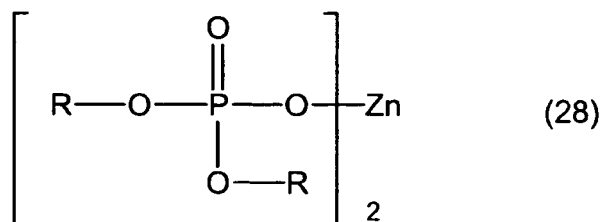
**[0108]** As examples of phosphorus compounds represented by general formula (26) there may be mentioned phosphorous acid and monothiophosphorous acid; phosphorous acid monoesters and monothiophosphorous acid monoesters having one of the aforementioned C1-30 hydrocarbon groups; phosphorous acid diesters and monothiophosphorous acid diesters having two of the aforementioned C1-30 hydrocarbon groups; phosphorous acid triesters and monothiophosphorous acid triesters having three of the aforementioned C1-30 hydrocarbon groups; and mixtures thereof. Among these, phosphorous acid monoesters and phosphorous acid diesters are preferred, and phosphorous acid diesters are especially preferred.

**[0109]** As examples of phosphorus compounds represented by general formula (27) there may be mentioned phosphoric acid and monothiophosphoric acid; phosphoric acid monoesters and monothiophosphoric acid monoesters having one of the aforementioned C1-30 hydrocarbon groups; phosphoric acid diesters and monothiophosphoric acid diesters having two of the aforementioned C1-30 hydrocarbon groups; phosphoric acid triesters and monothiophosphoric acid triesters having three of the aforementioned C1-30 hydrocarbon groups; and mixtures thereof. Among these, phosphoric acid monoesters and phosphoric acid diesters are preferred, and phosphoric acid diesters are especially preferred.

**[0110]** Zinc salts of phosphorus compounds represented by general formulas (26) and (27) will differ in structure depending on the number of OH groups or SH groups of the phosphorus compound, and therefore no restrictions are placed on the structure. For example, when 1 mole of zinc oxide is reacted with 2 moles of a phosphoric acid diester (one OH group), a compound with the structure represented by the following formula (28) may be obtained as the main component, although polymerized molecules may also be present (where R in the formula represents a C1-30 hydrocarbon group).

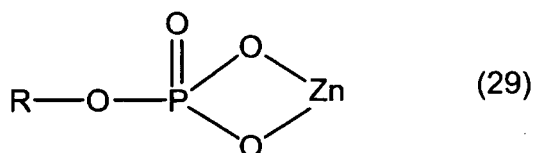
**[0111]**

## [Chemical Formula 26]



Also, for example, when 1 mole of zinc oxide is reacted with 1 mole of a phosphoric acid monoester (two OH groups), a compound having the structure represented by the following formula (29) may be obtained as the main component, although polymerized molecules may also be present (where R in the formula represents a C1-30 hydrocarbon group).  
[0112]

## [Chemical Formula 27]



Moreover, the grease composition for constant velocity joints according to the invention preferably also further contains at least one selected from among complexes of fatty acid salts and carbonates, wherein the fatty acid is overbased by the carbonate (hereinafter this will be referred to as "carbonate-dispersed overbased fatty acid salt"), and organic acid salts.

[0113] The carbonate-dispersed overbased fatty acid salt has a carbonate dispersed in the fatty acid salt, with the fatty acid salt being overbased by the carbonate.

[0114] The fatty acid salt in the carbonate-dispersed overbased fatty acid salt may have a straight-chain or branched fatty acid. The fatty acid may be saturated or unsaturated, but from the viewpoint of solubility in the base oil it is preferably an unsaturated fatty acid. There is no particular restriction on the number of unsaturated bonds, but the preferred number is one. The number of carbon atoms in the fatty acid is also not particularly restricted, but C10-25 fatty acids are preferred from the viewpoint of dispersibility of the carbonate fine particles.

[0115] As specific examples of preferred fatty acids to be used in the carbonate-dispersed overbased fatty acid salt of the invention there may be mentioned oleic acid (C18, one unsaturated bond), erucic acid (C22, one unsaturated bond), linoleic acid (C18, two unsaturated bonds) and linolenic acid (C18, three unsaturated bonds), among which oleic acid is most preferred.

[0116] The fatty acid salt in the carbonate-dispersed overbased fatty acid salt may be an alkali metal salt or alkaline earth metal salt of any of the aforementioned fatty acids, but salts of alkaline earth metals such as magnesium, barium and calcium are preferred, with calcium being more preferred.

[0117] The carbonate in the carbonate-dispersed overbased fatty acid salt may be an alkali metal carbonate or alkaline earth metal carbonate, and specifically there may be mentioned carbonates of lithium, sodium, potassium, magnesium, calcium and barium, although carbonates of alkaline earth metals are preferred and calcium carbonate is more preferred.

[0118] The carbonate is present as fine particles in the carbonate-dispersed overbased fatty acid salt. There are no particular restrictions on the mean particle size thereof, but from the viewpoint of conferring sufficient performance for anti-flaking and anti-seizure, the mean particle size is preferably at least 50 nm, more preferably at least 100 nm, even more preferably at least 300 nm, yet more preferably at least 500 nm, even yet more preferably at least 1000 nm and most preferably at least 2000 nm. Here, "mean particle size" refers to the mean particle size calculated by the Marquadt method with measurement in a dynamic light scattering particle distribution system.

[0119] There are no particular restrictions on the mixing ratio of the fatty acid salt and carbonate in the carbonate-

dispersed overbased fatty acid salt of the invention, but from the viewpoint of further improving the anti-flaking and anti-seizure, the carbonate content is preferably at least 10 parts by mass, more preferably at least 20 parts by mass, even more preferably at least 30 parts by mass, yet more preferably at least 40 parts by mass and most preferably at least 50 parts by mass, with respect to 100 parts by mass of the fatty acid salt. From the standpoint of solubility in the lubricating base oil, the carbonate content is preferably not greater than 1000 parts by mass, more preferably not greater than 500 parts by mass, even more preferably not greater than 400 parts by mass, yet more preferably not greater than 300 parts by mass and most preferably not greater than 200 parts by mass, with respect to 100 parts by mass of the fatty acid salt.

**[0120]** Any process may be used to produce the carbonate-dispersed overbased fatty acid salt, and as a specific example, the fatty acid salt may be dissolved in a carrier oil, and then carbon dioxide gas blown in with a system containing an alkali metal base or alkaline earth metal base. The carrier oil used here may be one of those mentioned as the lubricating base oil for the grease composition for constant velocity joints of the invention. As alkali metal and alkaline earth metal bases there may be mentioned hydroxides and oxides, and more specifically calcium hydroxide, calcium oxide, magnesium oxide, barium oxide and the like.

**[0121]** The carbonate-dispersed overbased fatty acid salt used for the invention is generally obtained as a solution in the carrier oil, and from the viewpoint of solubility in the lubricating base oil the carrier oil is preferably used in an amount of at least 10 parts by mass, more preferably at least 15 parts by mass, even more preferably 20 parts by mass and most preferably 25 parts by mass with respect to 100 parts by mass as the total of the fatty acid salt and carbonate. Also, the carrier oil is preferably used at not greater than 1000 parts by mass, more preferably not greater than 700 parts by mass, even more preferably not greater than 500 parts by mass and most preferably not greater than 400 parts by mass with respect to 100 parts by mass as the total of the fatty acid salt and carbonate.

**[0122]** A methanol compound may be added to the reaction system during production of the carbonate-dispersed overbased fatty acid salt in order to promote production of carbonate fine particles.

**[0123]** The carbonate-dispersed overbased fatty acid salt used for the invention may also be an overbased mixture of a fatty acid salt with another organic acid salt such as a sulfonate.

**[0124]** The content of the carbonate-dispersed overbased fatty acid salt of the invention is preferably not less than 0.05 % by mass and more preferably not less than 0.1 % by mass based on the total composition. If the content of the carbonate-dispersed overbased fatty acid salt is not at least 0.05 % by mass, an insufficient improving effect will tend to be obtained for the anti-flaking and anti-seizure by addition of the carbonate-dispersed overbased fatty acid salt. The content of the carbonate-dispersed overbased fatty acid salt is preferably not greater than 10 % by mass and more preferably not greater than 5.0 % by mass based on the total composition. If the content of the carbonate-dispersed overbased fatty acid salt exceeds 10 % by mass, no further effect of improving the anti-flaking and anti-seizure will be achieved corresponding to the increased content. Here, the content of the carbonate-dispersed overbased fatty acid salt is the total of the fatty acid salt content and the carbonate content, without the carrier oil or other components.

**[0125]** The base number of the carbonate-dispersed overbased fatty acid salt of the invention is not particularly restricted, but from the standpoint of more excellent anti-flaking and anti-seizure, it is usually at least 50 mgKOH/g, preferably at least 100 mgKOH/g, more preferably at least 150 mgKOH/g, even more preferably at least 200 mgKOH/g and most preferably at least 250 mgKOH/g, when dissolved in the carrier oil. There is no particular restriction on the upper limit, but it will generally be not greater than 600 mgKOH/g. The base value referred to here is the base value (mgKOH/g) determined by the hydrochloric acid method, based on JIS K 2501 "Petroleum Products and Lubricants - Test Method for Neutralization", Section 6.

**[0126]** As organic acid salts there are preferably used sulfonates, phenates and salicylates, as well as mixtures thereof. As cation components for these organic acid salts there may be mentioned alkali metals such as sodium and potassium; alkaline earth metals such as magnesium, calcium and barium; and amines such as ammonia, alkylamines with C1-3 alkyl groups (monomethylamine, dimethylamine, trimethylamine, monoethylamine, diethylamine, triethylamine, monopropylamine, dipropylamine, tripropylamine and the like), and alkanolamines with C1-3 alkanol groups (monomethanolamine, dimethanolamine, trimethanolamine, monoethanolamine, diethanolamine, triethanolamine, monopropanolamine, dipropanolamine, tripropanolamine and the like). Preferred among these are alkali metals and alkaline earth metals, among which calcium is particularly preferred. Using an alkali metal or alkaline earth metal as the cation component of the organic acid salt will tend to produce even higher lubricity.

**[0127]** The base value of the organic acid salt is preferably 50-500 mgKOH/g and more preferably 100-450 mgKOH/g. If the base value of the organic acid salt is less than 100 mgKOH/g the lubricity-enhancing effect of the organic acid salt addition will tend to be unsatisfactory, while organic acid salts with a base value of greater than 500 mgKOH/g are also not preferred because they are generally very difficult to produce and obtain. The base value referred to here is the base value [mgKOH/g] determined by the perchlorate method, with measurement according to JIS K 2501 "Petroleum Products and Lubricants - Determination of Neutralization Number", Section 7.

**[0128]** The content of the organic acid salt is preferably 0.1-30 % by mass, more preferably 0.5-25 % by mass and even more preferably 1-20 % by mass based on the total amount of the composition. If the organic acid salt content is less than this lower limit, the improving effect on the anti-flaking and anti-seizure by addition of the organic acid salt will

tend to be inadequate, while if it exceeds the upper limit, the stability of the grease composition will be lowered and precipitates will tend to be produced.

**[0129]** The sulfonate used may be one produced by any desired process. For example, there may be used an alkali metal salt, alkaline earth metal salt or amine salt of an alkylaromatic sulfonic acid compound obtained by sulfonation of an alkylaromatic compound with a molecular weight of 100-1500 and preferably 200-700, or a mixture thereof. As the alkylaromatic sulfonic acid compounds referred to here, there may be mentioned synthetic sulfonic acids including sulfonated alkylaromatic compounds of lubricating oil fractions of common mineral oils, petroleum sulfonic acids such as "mahogany acid" yielded as a by-product of white oil production, sulfonated products of alkylbenzenes with straight-chain or branched alkyl groups, which are by-products in production plants for alkylbenzenes used as starting materials for detergents and are obtained by alkylation of benzene with polyolefins, or sulfonated alkylnaphthalenes such as dinonylnaphthalene. There may also be mentioned, specifically, neutral sulfonates obtained by reacting the aforementioned alkylaromatic sulfonic acid with an alkali metal salt (alkali metal oxide, hydroxide or the like), an alkaline earth metal salt (alkaline earth metal oxide, hydroxide or the like) or one of the aforementioned amines (ammonia, alkylamine, alkanolamine, etc.); basic sulfonates obtained by heating a neutral sulfonate with an excess of an alkali metal base, alkaline earth metal base or amine in the presence of water; "carbonated overbased sulfonates" obtained by reacting a neutral sulfonate with an alkali metal base, alkaline earth metal base or amine in the presence of carbon dioxide gas; "borated overbased sulfonates" produced by reacting a neutral sulfonate with an alkali metal base, alkaline earth metal base or amine and a boric acid compound such as boric acid or boric anhydride, or by reacting a carbonated overbased sulfonate with a boric acid compound such as boric acid or boric anhydride; and mixtures of these compounds.

**[0130]** As phenates there may be mentioned, specifically, neutral phenates obtained by reacting an alkylphenol having one or two C4-20 alkyl groups with an alkali metal salt (alkali metal oxide, hydroxide or the like), an alkaline earth metal salt (alkaline earth metal oxide, hydroxide or the like) or one of the aforementioned amines (ammonia, alkylamine, alkanolamine, etc.) in the presence or in the absence of elemental sulfur; basic phenates obtained by heating a neutral phenate with an excess of an alkali metal base, alkaline earth metal base or amine in the presence of water; "carbonated overbased phenates" obtained by reacting a neutral phenate with an alkali metal base, alkaline earth metal base or amine in the presence of carbon dioxide gas; "borated overbased phenates" produced by reacting a neutral phenate with an alkali metal base, alkaline earth metal base or amine and a boric acid compound such as boric acid or boric anhydride, or by reacting a carbonated overbased phenate with a boric acid compound such as boric acid or boric anhydride; and mixtures of these compounds.

**[0131]** As salicylates there may be mentioned, specifically, neutral salicylates obtained by reacting an alkylsalicylic acid having one or two C4-20 alkyl groups with an alkali metal salt (alkali metal oxide, hydroxide or the like), an alkaline earth metal salt (alkaline earth metal oxide, hydroxide or the like) or one of the aforementioned amines (ammonia, alkylamine, alkanolamine, etc.) in the presence or in the absence of elemental sulfur; basic salicylates obtained by heating a neutral salicylate with an excess of an alkali metal base, alkaline earth metal base or amine in the presence of water; "carbonated overbased salicylates" obtained by reacting a neutral salicylate with an alkali metal base, alkaline earth metal base or amine in the presence of carbon dioxide gas; "borated overbased salicylates" produced by reacting a neutral salicylate with an alkali metal base, alkaline earth metal base or amine and a boric acid compound such as boric acid or boric anhydride, or by reacting a carbonated overbased salicylate with a boric acid compound such as boric acid or boric anhydride; and mixtures of these compounds.

**[0132]** The worked penetration of the grease composition for constant velocity joints according to the invention is preferably 220 or greater and more preferably 265 or greater. If the worked penetration is not at least 220, the grease will be excessively hard and the effect of the invention may not be satisfactorily exhibited. Also, the worked penetration is preferably not greater than 430 and more preferably not greater than 400. If the worked penetration exceeds 430, the grease will be excessively soft and it may be difficult for the grease composition to fill constant velocity joints. Here, "worked penetration" refers to the penetration immediately after 60 working passes, as measured according to JIS K2220, "Grease", Section 5.3 "Penetration Test Method".

**[0133]** The grease composition for constant velocity joints of the invention may also contain solid lubricants, antioxidants, oiliness agents, rust preventing agents, viscosity index improvers and the like, so long as the properties of the composition are not impaired.

**[0134]** As specific examples of solid lubricants there may be mentioned boron nitride, fluorinated graphite, polytetrafluoroethylene, molybdenum disulfide, antimony sulfide, alkali (earth) metal borates and the like.

**[0135]** As specific antioxidants there may be mentioned phenol-based compounds such as 2,6-di-*t*-butylphenol and 2,6-di-*t*-butyl-*p*-cresol; amine-based compounds such as dialkyldiphenylamine, phenyl- $\alpha$ -naphthylamine and *p*-alkylphenyl- $\alpha$ -naphthylamine; sulfur-based compounds; phenothiazine-based compounds and the like.

**[0136]** As oiliness agents there may be mentioned, specifically, amines such as lauryl amine, myristyl amine, palmityl amine, stearylamine and oleylamine; higher alcohols such as lauryl alcohol, myristyl alcohol, palmityl alcohol, stearyl alcohol and oleyl alcohol; higher fatty acids such as lauric acid, myristic acid, palmitic acid, stearic acid and oleic acid, fatty acid esters such as methyl laurate, methyl myristate, methyl palmitate, methyl stearate and methyl oleate; amides

such as lauryl amide, myristyl amide, palmityl amide, stearyl amide and oleyl amide; and other fats and oils.

**[0137]** As rust preventing agents there may be mentioned, specifically, metal soaps; polyhydric alcohol partial esters such as sorbitan fatty acid esters; amines; phosphoric acid; phosphoric acid salts; and the like.

**[0138]** As viscosity index improvers there may be mentioned, specifically, polymethacrylate, polyisobutylene, polystyrene and the like.

**[0139]** For preparation of a grease composition for constant velocity joints according to the invention, for example, a lubricating base oil may be combined with a thickener, carbon black having a mean particle size of not greater than 500 nm, an organic molybdenum compound and if necessary other additives, and the mixture stirred and passed through a roll mill or the like. Alternatively, it may be produced by pre-adding the starting component for a thickener to the lubricating base oil and melting and stirring the mixture to prepare the thickener in the lubricating base oil, and then adding the carbon black having a mean particle size of not greater than 500 nm, the organic molybdenum compound and if necessary other additives, stirring the mixture and passing it through a roll mill or the like.

**[0140]** The grease composition for constant velocity joints of the invention having the construction described above exhibits excellent anti-flaking, anti-seizure, anti-wear and low-friction properties, and can contribute to a high degree of increased performance and extended life of constant velocity joints. There is no restriction on the constant velocity joints to which the grease composition for constant velocity joints of the invention is applied, and for example, there may be mentioned fixed constant velocity joints such as Barfield joints, Rzeppa joints and undercutting free joints, and slide type constant velocity joints such as double offset joints, tripod joints and cross groove joints.

## Examples

**[0141]** The present invention will now be explained in further detail by examples and comparative examples, with the understanding that the invention is in no way limited by the examples.

[Examples 1-7 and Comparative Examples 1-4]

**[0142]** For Examples 1-6 and Comparative Examples 1-4, a solvent-refined paraffin-based mineral oil with a kinematic viscosity of 126 mm<sup>2</sup>/s at 40°C was used as the lubricating base oil, diphenylmethane-4,4'-diisocyanate was heated to dissolution in the base oil, and the amine and alcohol listed in Tables 1 to 3 were then heated to dissolution in the same base oil. Next, the different additives listed in Table 1 were added to the resulting gel-like substance, and after stirring the mixture it was passed through a roll mill to obtain a grease composition.

**[0143]** In Example 7, lithium 12-hydroxystearate was added to the lubricating base oil as the thickener instead of the diisocyanate, alcohol and amine. Next, the different additives listed in Table 2 were added, and after stirring the mixture it was passed through a roll mill to obtain a grease composition.

**[0144]** In Tables 1 to 3, the dihydrocarbyl polysulfide is sulfurized polyisobutylene (sulfur content: 45 % by mass), the sulfurized oil is sulfurized lard (sulfur content: 30 % by mass) and the antioxidant is an amine-based antioxidant (phenyl- $\alpha$ -naphthylamine).

[Bench durability test]

**[0145]** The grease compositions of Examples 1-7 and Comparative Examples 1-3 were subjected to the following bench durability test.

**[0146]** In consideration of the traveling pattern of an automobile, a commercially available #82-size Birfield joint was used under conditions where 1 cycle was a mode with modified rpm, torque and operating angle, and the number of cycles until seizing of the joint or flaking at sites was evaluated. The results are shown in Tables 1 to 3.

**[0147]**

[Table 1]

	Example			
	1	2	3	4
Lubricant base oil [% by mass]	78.0	86.0	86.5	84.0
Thickener [% by mass]	16.0	8.0	8.0	8.0

(continued)

		Example			
		1	2	3	4
5	Thickener raw materials (molar ratio)	Diphenylmethane 4,4'-diisocyanate			
		Cyclohexylamine			
		Octadecyl alcohol			
10	Carbon black (mean particle size: 40 nm) [% by mass]	2.0	1.0	-	1.0
	Carbon black (mean particle size: 100 nm) [% by mass]	-	-	1.0	-
15	Molybdenum dithiocarbamate [% by mass]	2.0	1.0	2.0	2.0
	Molybdenum dithiophosphate [% by mass]	-	3.0	-	-
	Dihydrocarbyl polysulfide [% by mass]	1.0	-	1.0	1.0
20	Sulfurized oil [% by mass]	-	-	-	-
	Tricresyl phosphate [% by mass]	-	-	0.5	-
	Zinc dithiophosphate [% by mass]	-	-	-	3.0
25	Overbased calcium sulfonate [% by mass]	-	-	-	-
	Graphite [% by mass]	-	-	-	-
	Antioxidant [% by mass]	1.0	1.0	1.0	1.0
	Worked penetration	280	330	320	330
	Bench durability test [cycles]	450	400	500	500

[0148]

[Table 2]

		Example		
		5	6	7
35	Lubricant base oil [% by mass]	85.5	87.0	84.5
	Thickener [% by mass]	6.5	7.0	10.0
40	Thickener raw materials (molar ratio)	Diphenylmethane 4,4'-diisocyanate		
		Cyclohexylamine		
		Octadecyl alcohol		
45	Carbon black (mean particle size: 40 nm) [% by mass]	1.0	1.0	
	Carbon black (mean particle size: 100 nm) [% by mass]	-	-	1.0
	Molybdenum dithiocarbamate [% by mass]	1.0	2.0	2.0
50	Molybdenum dithiophosphate [% by mass]	3.0	-	-
	Dihydrocarbyl polysulfide [% by mass]	-	1.0	1.0
	Sulfurized oil [% by mass]	2.0	-	-
55	Tricresyl phosphate [% by mass]	-	-	0.5
	Zinc dithiophosphate [% by mass]	-	-	-
	Overbased calcium sulfonate [% by mass]	-	1.0	-
	Graphite [% by mass]	-	-	-

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(continued)

	Example		
	5	6	7
Antioxidant [% by mass]	1.0	1.0	1.0
Worked penetration	370	350	330
Bench durability test [cycles]	450	600	300

[0149]

[Table 3]

		Comp. Ex.			
		1	2	3	4
Lubricant base oil [% by mass]		80.0	90.0	89.5	84.0
Thickener [% by mass]		16.0	8.0	8.0	8.0
Thickener raw materials (molar ratio)	Diphenylmethane 4,4'-diisocyanate	1	5	5	5
	Cyclohexylamine	2	8	8	8
	Octadecyl alcohol	-	2	2	2
Carbon black (mean particle size: 40 nm) [% by mass]		-	1.0	-	-
Carbon black (mean particle size: 100 nm) [% by mass]		-	-	-	-
Molybdenum dithiocarbamate [% by mass]		2.0	-	-	2.0
Molybdenum dithiophosphate [% by mass]		-	-	-	-
Dihydrocarbyl polysulfide [% by mass]		1.0	-	1.0	1.0
Sulfurized oil [% by mass]		-	-	-	-
Tricresyl phosphate [% by mass]		-	-	0.5	-
Zinc dithiophosphate [% by mass]		-	-	-	3.0
Overbased calcium sulfonate [% by mass]		-	-	-	-
Graphite (mean particle size: 3 μm) [% by mass]		-	-	-	1.0
Antioxidant [% by mass]		1.0	1.0	1.0	1.0
Worked penetration		285	320	330	300
Bench durability test [cycles]		60	20	60	100

## Examples 8-11

[0150] For Examples 8 to 11, the solvent-refined paraffin-based mineral oil and hydrogenation-refined naphthene-based mineral oil listed in Table 4 were used together as the lubricating base oil, diphenylmethane-4,4'-diisocyanate was heated to dissolution in the base oil, and the amine and alcohol listed in Table 5 were then heated to dissolution in the same base oil. Next, the different additives listed in Table 5 were added to the resulting gel-like substance, and after stirring each mixture it was passed through a roll mill to obtain the grease compositions listed in Table 5.

[0151]

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[Table 4]

		Solvent-refined paraffin-based mineral oil	Hydrogenation-refined naphthene-based mineral oil
Kinematic viscosity [mm <sup>2</sup> /s]	40°C	175	238
	100°C	16	12.4
Viscosity index		96	-41
Ring analysis	%C <sub>A</sub>	7.36	13.9
	%C <sub>N</sub>	26.22	45.9
	%C <sub>P</sub>	66.42	40.2
Aniline point [°C]		113.4	81.5
Pour point [°C]		-16	-15
Sulfur content [% by mass]		0.546	0
Nitrogen content [wt ppm]		104	60

In Table 5, the dihydrocarbyl polysulfide is sulfurized polyisobutylene (sulfur content: 45 % by mass).

[Boot material immersion test]

**[0152]** The grease compositions of Examples 8 to 11 were subjected to the following boot material immersion test.

**[0153]** Following the procedure of JIS K 6258, a chloroprene-based rubber material was immersed in the grease composition and held at 120°C for 500 hours, and the weight change of the rubber material, volume change, tensile strength change and tensile elongation change before and after immersion were determined. The results are shown in Table 5.

**[0154]** [Bench durability test]The grease compositions of Examples 8 to 11 were subjected to the following bench durability test.

**[0155]** In consideration of the traveling pattern of an automobile, a commercially available #95-size tripod joint was used under conditions where 1 cycle was a mode with modified rpm, torque and operating angle, and the time until seizing of the joint or flaking at sites was evaluated. The results are shown in Table 5.

**[0156]**

[Table 5]

		Example			
		8	9	10	11
Solvent-refined paraffin-based mineral oil [% by mass]		76.0	66.5	58.0	76.0
Hydrogenation-refined naphthene-based mineral oil [% by mass]		10.0	20.0	30.0	10.0
Thickener [% by mass]		8.0	8.0	8.0	8.0
Thickener raw materials (molar ratio)	Diphenylmethane 4,4'-diisocyanate	5	5	5	5
	Cyclohexylamine	8	8	8	8
	Octadecyl alcohol	2	2	2	2
Carbon black (mean particle size: 40 nm)[% by mass]		0.5	1.0	1.0	2.0
Molybdenum dithiocarbamate [% by mass]		3.0	3.0	3.0	3.0
Molybdenum dithiophosphate [% by mass]		-	-	1.0	1.0
Tricresyl phosphate [% by mass]		0.5	0.5	-	0.5
Dihydrocarbyl polysulfide [% by mass]		2.0	1.0	-	1.0



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(continued)

		Example			
		8	9	10	11
Boot material immersion	Strength change [%]	-7	-1	-4	-16
	test Elongation change [%]	-15	-10	-12	-18
Bench durability test [cycles]		900	1000	1000	1100

## Claims

1. A grease composition for constant velocity joints **characterized by** comprising a lubricating base oil, a thickener, carbon black with a mean particle size of not greater than 500 nm, and an organic molybdenum compound.
2. The grease composition for constant velocity joints according to claim 1, **characterized by** further comprising at least one from among sulfur-based extreme pressure agents, phosphorus-based extreme pressure agents and zinc-based extreme pressure agents.
3. The grease composition for constant velocity joints according to claim 1 or 2, **characterized by** further comprising at least one selected from among complexes of fatty acid salts and carbonates, wherein said fatty acids are overbased by the carbonates, and organic acid salts.

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2005/002820

<b>A. CLASSIFICATION OF SUBJECT MATTER</b> Int.Cl. <sup>7</sup> C10M141/10, 163/00// (C10M141/10, 125:02, 135:18, 137:10) (C10M163/00, 125:02, 135:18, 137:10, 159:20), C10N10:04, 10:12, 20:06, 30:06, 40:04, 50:10 According to International Patent Classification (IPC) or to both national classification and IPC														
<b>B. FIELDS SEARCHED</b> Minimum documentation searched (classification system followed by classification symbols) Int.Cl. <sup>7</sup> C10M141/10, 125/02, 135/18, 137/10, 159/20, 163/00, C10N10:04, 10:12, 20:06, 30:06, 40:04, 50:10 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 1996-2005 Kokai Jitsuyo Shinan Koho 1971-2004 Toroku Jitsuyo Shinan Koho 1994-2005 Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)														
<b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b> <table border="1"> <thead> <tr> <th>Category*</th> <th>Citation of document, with indication, where appropriate, of the relevant passages</th> <th>Relevant to claim No.</th> </tr> </thead> <tbody> <tr> <td>Y</td> <td>JP 2003-155491 A (Kabushiki Kaisha Matsumura Sekiyu Kenkyusho et al.), 30 May, 2003 (30.05.03), &amp; CN 1424388 A &amp; EP 1314774 A1 &amp; US 2003/0139302 A1</td> <td>1-3</td> </tr> <tr> <td>Y</td> <td>JP 2002-195277 A (NSK Ltd.), 10 July, 2002 (10.07.02), &amp; US 2002/0076125 A1 &amp; US 6652148 B2</td> <td>1-3</td> </tr> <tr> <td>Y</td> <td>JP 2002-250351 A (NSK Ltd.), 06 September, 2002 (06.09.02), (Family: none)</td> <td>1-3</td> </tr> </tbody> </table>			Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.	Y	JP 2003-155491 A (Kabushiki Kaisha Matsumura Sekiyu Kenkyusho et al.), 30 May, 2003 (30.05.03), & CN 1424388 A & EP 1314774 A1 & US 2003/0139302 A1	1-3	Y	JP 2002-195277 A (NSK Ltd.), 10 July, 2002 (10.07.02), & US 2002/0076125 A1 & US 6652148 B2	1-3	Y	JP 2002-250351 A (NSK Ltd.), 06 September, 2002 (06.09.02), (Family: none)	1-3
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