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438-8510 (JP). **SHIBATA, Takaaki** [JP/JP]; 1578 Higashikaizuka, Iwata-shi, Shizuoka 438-8510 (JP).

(74) Agents: **KAWAGUCHI, Yoshio** et al.; Kawaguti & Partners, Jochi Kioizaka Bldg., 7-1, Kioi-cho, Chiyoda-ku, Tokyo 102-0094 (JP).

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(71) Applicants (for all designated States except US): **Showa Shell Sekiyu K.K.** [JP/JP]; 3-2, Daiba 2-chome, Minato-ku, Tokyo 135-8074 (JP). **NTN Corporation** [JP/JP]; 3-17, Kyomachibori 1-chome, Nishi-ku, Osaka-shi, Osaka 550-0003 (JP).

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(72) Inventors; and

(75) Inventors/Applicants (for US only): **OZAKI, Takahiro** [JP/JP]; 3-2, Daiba 2-chome, Minato-ku, Tokyo 135-8074 (JP). **NUMAZAWA, Kouichi** [JP/JP]; 3-2, Daiba 2-chome, Minato-ku, Tokyo 135-8074 (JP). **TAKABE, Shinichi** [JP/JP]; 1578 Higashikaizuka, Iwata-shi, Shizuoka

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(54) Title: GREASE COMPOSITION FOR CONSTANT VELOCITY JOINTS

(57) Abstract: The present invention provides a grease composition for use in constant velocity joints, having improved wear resistance and endurance characteristics. The grease is made by incorporating in a urea grease (A) a molybdenum dialkyldithiocarbamate, (B) a molybdenum dithiophosphate, (C) 2- (4-morpholinylthio) benzothiazole and (D) a calcium sulphonate. The blended amount of (A) + (B) is made not more than 10% by weight, and that of (C) and (D) not more than 3% by weight respectively. Also, a grease for use in constant velocity joints is made by further adding (E) zinc dithiophosphate to the aforementioned components (A) + (B) + (C) + (D) in a blended amount of not more than 3% by weight.

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DESCRIPTION

GREASE COMPOSITION FOR CONSTANT VELOCITY JOINTS

Technical Field

The present invention relates to a grease composition for constant velocity joints suitable for use at the lubrication points of constant velocity joints in automobiles.

Background Art

Hitherto, lithium soap greases which contain molybdenum disulphide and extreme-pressure lithium soap greases which contain sulphur-phosphorus type additives have been used as greases for constant velocity joints. In recent years, low-wear and temperature-resistant greases for high-temperature use in which urea compounds or lithium complex soaps are used as the thickener and where organomolybdenum compounds are added have since become commonly used.

In particular, because ball-type constant velocity joints are subject to a complex rolling-sliding action and are lubricated under high surface pressure, the parts constituting the constant velocity joint, namely the outer race, inner race, ball and cage, are repeatedly subject to stresses, so that wear occurs and flaking (a peeling phenomenon) is generated.

A grease which reduces wear in constant velocity joints, which inhibits the flaking due to metal fatigue and which has excellent durability has been proposed, and

this effect cited, by using a molybdenum dialkyldithiocarbamate and a molybdenum dithiophosphate together in a urea grease and adding 10% by weight, see JP-B 5-79280 (1993).

Disclosure of the Invention

Problem to be solved by the Invention

The present invention provides a grease for use in constant velocity joints which improves on the aforementioned greases, which reduces wear in constant velocity joints, which inhibits the flaking due to metal fatigue and which further has excellent durability.

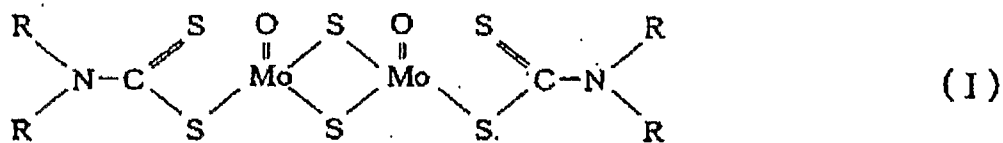
Means for solving Problem

For the present invention, durability tests were carried out by adding various additives to a low-wear urea grease in which (A) a molybdenum dialkyldithiocarbamate and (B) a molybdenum dithiophosphate were used together, and by using wear-resistance tests by means of an SRV test rig and actual constant velocity joints. Flaking-resistance performance was evaluated, and as a result perfection of the present invention was achieved.

In other words, the present invention provides a grease for use in constant velocity joints which incorporates in a urea grease (A) a molybdenum dialkyldithiocarbamate as represented by the undermentioned formula I, (B) a molybdenum dithiophosphate as represented by the undermentioned formula II, (C) 2-(4-morpholinylthio)benzothiazole as

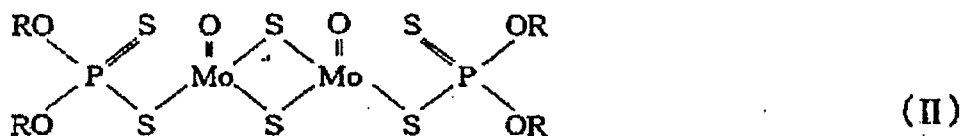
represented by the undermentioned formula III and (D) a calcium sulphonate.

Chemical structure 1



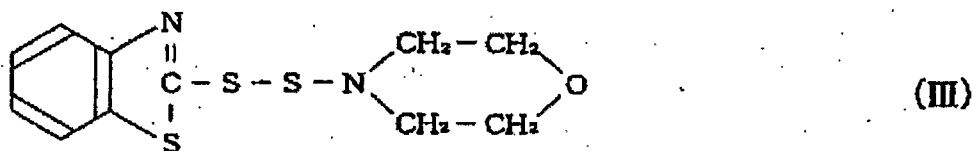
(where R denotes an alkyl group of 1 ~ 24 carbons)

Chemical structure 2



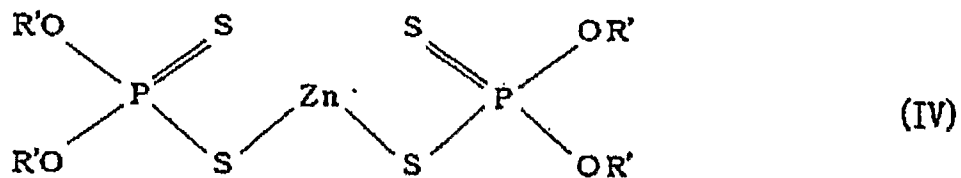
(where R is a primary or secondary alkyl group or an aryl group)

Chemical structure 3



The blending is such that the total of the aforementioned (A) and (B) is not more than 10% by weight, (C) is not more than 3% by weight and (D) is not more than 3% by weight.

In a preferred embodiment according to the present invention a grease for use in constant velocity joints is provided by further incorporating not more than 3% by weight of (E) a zinc dithiophosphate as represented by the undermentioned formula IV.

Chemical structure 4

(where R' is a primary or secondary alkyl group or an aryl group)

In a further aspect the present invention provides the use of the grease composition in constant velocity joints, preferably in automobiles.

Effect of the Invention

In the present invention, it is inferred - without intending to be limited to any specific theory - that the urea compound adheres to the metal surfaces, iron oxide or iron sulphide is formed in the lower layer thereof, making a solid film, and so it is possible to offer a grease for use in constant velocity joints which reduces wear in the constant velocity joints and which also inhibits the phenomenon of flaking due to the accompanying metal fatigue, and which has excellent durability.

Best Mode for Carrying Out the Invention

For the base oil of the invention it is possible to use e.g. mineral oils, plant oils and synthetic oils such as ester oils, ether oils and hydrocarbon oils, or mixtures thereof.

For the thickener added to this base oil it is possible to use urea compounds such as monoureas, diureas, triureas and tetraureas. For example, there are diurea compounds obtained by reaction of diisocyanates and monoamines, and tetraurea compounds obtained by reaction of diisocyanates, monoamines and diamines.

As specific examples of diisocyanates, mention may be made of diphenylmethane diisocyanate, tolylene diisocyanate, bitolyene diisocyanate and naphthylene diisocyanate. Also, as examples of monoamines, mention may be made of octylamine, dodecylamine, stearylamine, oleylamine, aniline, *p*-toluidine and cyclohexylamine. As examples of diamines, mention may be made of ethylenediamine, propanediamine, butanediamine and phenylenediamine.

The molybdenum dialkyldithiocarbamates as represented by aforementioned formula (I) and which are the aforementioned constituent (A) have alkyl groups of 1 ~ 24 carbons as their R, and preferably have alkyl groups of 3 ~ 18 carbons. As specific examples, mention may be made of sulphurised molybdenum diethyldithiocarbamate, sulphurised molybdenum dipropyldithiocarbamate, sulphurised molybdenum dibutyldithiocarbamate, sulphurised molybdenum dipentyldithiocarbamate, sulphurised molybdenum dihexyldithiocarbamate, sulphurised molybdenum dioctyldithiocarbamate, sulphurised molybdenum didecyldithiocarbamate, sulphurised molybdenum didodecyl dithiocarbamate, sulphurised molybdenum di(butylphenyl)dithiocarbamate, sulphurised molybdenum di(nonylphenyl)dithiocarbamate, sulphurised oxymolybdenum diethyldithiocarbamate, sulphurised oxymolybdenum dipropyldithiocarbamate, sulphurised oxymolybdenum

dibutyldithiocarbamate, sulphurised oxymolybdenum
dipentyldithiocarbamate, sulphurised oxymolybdenum
dihexyldithiocarbamate, sulphurised oxymolybdenum
dioctyldithiocarbamate, sulphurised oxymolybdenum
didecyldithiocarbamate, sulphurised oxymolybdenum
didodecyldithiocarbamate, sulphurised oxymolybdenum
di(butylphenyl)dithiocarbamate, sulphurised oxymolybdenum
di(nonylphenyl)dithiocarbamate, and mixtures thereof.

As examples of the molybdenum dithiophosphates as represented by aforementioned formula (II) and which are the aforementioned constituent (B) mention may be made of sulphurised molybdenum diethyldithiophosphate, sulphurised molybdenum dipropyldithiophosphate, sulphurised molybdenum dibutyldithiophosphate, sulphurised molybdenum dipentyldithiophosphate, sulphurised molybdenum dihexyldithiophosphate, sulphurised molybdenum dioctyldithiophosphate, sulphurised molybdenum didecyldithiophosphate, sulphurised molybdenum didodecyldithiophosphate, sulphurised molybdenum di(butylphenyl)dithiophosphate, sulphurised molybdenum di(nonylphenyl) dithiophosphate, sulphurised oxymolybdenum diethyldithiophosphate, sulphurised oxymolybdenum dipropyldithiophosphate, sulphurised oxymolybdenum dibutyldithiophosphate, sulphurised oxymolybdenum dipentyldithiophosphate, sulphurised oxymolybdenum dihexyldithiophosphate, sulphurised oxymolybdenum dioctyldithiophosphate, sulphurised oxymolybdenum didecyldithiophosphate, sulphurised oxymolybdenum didodecyldithiophosphate, sulphurised oxymolybdenum di(butylphenyl)dithiophosphate, sulphurised oxymolybdenum di(nonylphenyl) dithiophosphate, and mixtures thereof.

The aforementioned component (C) is 2-(4-morpholinylthio)benzothiazole as represented by the aforementioned formula (III), and it is a slightly yellow-white powder of molecular weight 284.

In ordinary applications it is the substance used as a vulcanisation accelerator of vulcanised rubber such as tires, tubes and footwear.

The aforementioned component (D) is an ordinary calcium sulphonate. Examples are the calcium salt of petroleum sulphonic acid, the calcium salt of an alkyl aromatic sulphonic acid, the overbased calcium salt of petroleum sulphonic acid and the overbased calcium salt of an alkyl aromatic sulphonic acid. It is possible to use these singly or in mixtures.

This component (D) functions as a dispersant, controls wear resistance and further contributes an improvement in durability.

The zinc dithiophosphates as represented by the aforementioned formula (IV) and which are the aforementioned component (E) are zinc dialkyl dithiophosphates or zinc diaryldithiophosphates.

In the aforementioned formula (IV), R' denotes a primary or secondary alkyl group or an aryl group. For example, it may be a methyl group, ethyl group, propyl group, isopropyl group, butyl group, secondary butyl group, isobutyl group, pentyl group, 4-methylpentyl group, hexyl group, 2-ethylhexyl group, heptyl group, octyl group, nonyl group, decyl group, isodecyl group, dodecyl group, tetradecyl group, hexadecyl group, octadecyl group, eicosyl group, docosyl group, tetracosyl group, cyclopentyl group, cyclohexyl group, methylcyclohexyl group, ethylcyclohexyl group, dimethylcyclohexyl group, cycloheptyl group, phenyl group, tolyl group, xylyl group,

ethyl phenyl group, propylphenyl group, butylphenyl group, pentylphenyl group, hexylphenyl group, heptylphenyl group, octylphenyl group, nonylphenyl group, decylphenyl group, dodecylphenyl group, tetradecylphenyl group, hexadecylphenyl group, octadecylphenyl, benzyl group or a phenethyl group.

Each of these R' may be the same or different.

As specific examples of the primary alkyl zinc dithiophosphates of the aforementioned component (E) mention may be made of zinc diisopropyl dithiophosphate, zinc diisobutyl dithiophosphate and zinc diisodecyl dithiophosphate.

Also, as specific examples of the secondary alkyl zinc dithiophosphates mention may be made of mono or di-sec-pentyl zinc dithiophosphate and mono or di-4-methyl-2-pentyl zinc dithiophosphate.

As specific examples of aryl zinc dithiophosphates mention may be made of zinc di-para-dodecylphenol dithiophosphate, zinc diheptylphenol dithiophosphate and zinc di-para-nonylphenol dithiophosphate.

In the grease composition for constant velocity joints of the present invention, the amount of molybdenum dithiocarbamate which is component (A) and molybdenum dithiophosphate which is component (B) blended into the urea grease may be made such that it is not more than 10% by weight.

If the amount of the aforementioned components (A) and (B) in the urea grease is greater than mentioned above, the effect is the same or may even be the reverse effect. Normally, an amount of 1 ~ 3% by weight of each of component (A) and component (B) is preferred.

Also, the amount of 2-(4-morpholinylthio)-benzothiazole which is component (C) in the grease is not

more than 3% by weight. If it exceeds this, the effect is the same or may even be the reverse effect. Normally, 0.1 ~ 1% by weight is preferred.

The amount of calcium sulphonate which is component (D) in the grease may be made not more than 3% by weight. If it exceeds this, the effect is the same or may even be the reverse effect. Normally, 0.1 ~ 1% by weight is preferred.

In the case where a zinc dithiophosphate which is component (E) is included in addition to the aforementioned components (A) + (B) + (C) + (D), even if the blended amount of each of components (A), (B), (C) and (D) is reduced as much as possible, excellent wear resistance and anti-flaking performance can be obtained.

The amount of this zinc dithiophosphate which is component (E) in the grease should be made not more than 3% by weight. If it exceeds this, the effect is the same or may even be the reverse effect. Normally, an excellent effect may be expected if it is made to be co-present preferably in the amount of 0.5 ~ 1% by weight.

It is possible at the same time to incorporate as appropriate into this grease composition for constant velocity joints, in addition to the aforementioned components, anti-oxidants, rust preventatives, extreme-pressure agents anti-wear agents and other additives.

Examples

The undermentioned materials were prepared in order to make the examples and comparative examples.

1. Diurea grease

The diurea grease was obtained by reacting 295.2 g diphenylmethane-4,4'-diisocyanate and 304.8 g octylamine

in 5400 g of a refined mineral oil with a kinetic viscosity at 100°C of approximately 15 mm²/s and evenly dispersing the diurea compound produced. The content of the diurea compound was 10% by weight. The penetration of this diurea grease (25°C, 60W) (JIS K2220) was 283.

2. Tetraurea grease

The tetraurea grease was obtained by reacting 382.7 g diphenylmethane-4,4'-diisocyanate, 411.4 g octylamine and 46 g ethylenediamine in 5160 g of a refined mineral oil with a kinetic viscosity at 100°C of approximately 15 mm²/s and evenly dispersing the tetraurea compound produced. The content of the tetraurea compound was 14% by weight. The penetration of this tetraurea grease (25°C, 60W) (JIS K2220) was 285.

3. Molybdenum dialkyl dithiocarbamate (MoDTC):

"Sakuralube 600" (trade name, made by Asahi Denka Kogyo Ltd.) was used.

4. Molybdenum dithiophosphate (MoDTP):

4-1. MoDTP (1) : "Sakuralube 300" (trade name, made by Asahi Denka Kogyo Ltd.) was used.

4-2. MoDTP (2) : "Molyvan L" (trade name, made by Vanderbilt Ltd.) was used.

5. 2-(4-morpholinyl)dithio)benzothiazole

6. Zinc dithiophosphate (ZnDTP) :

6-1. ZnDTP (1) : "Lubrizol 1395" (trade name, made by Lubrizol Ltd.) was used.

6-2. ZnDTP (2) : "Lubrizol 1371" (trade name, made by Lubrizol Ltd.) was used.

7. Calcium sulphonate: Calcium salt of petroleum sulphonic acid.

Examples 1 ~ 10

Using the aforementioned materials, and on the basis of the materials and blends shown in Tables 1 and 2, each

material was added to the diurea grease or tetraurea grease, and after mixing and kneading in a three-roll mill and finishing to a uniform consistency, the constant velocity joint greases of Examples 1 ~ 10 were obtained.

Comparative Examples 1 ~ 10

Using the aforementioned materials, and on the basis of the materials and blends shown in Tables 3 and 4, each material was added to the diurea grease or tetraurea grease in the same way as for the aforementioned Examples, and after mixing and kneading in a three-roll mill and finishing to a uniform consistency, the constant velocity joint greases of Comparative Examples 1 ~ 10 were obtained.

Evaluation tests

The following tests were carried out in order to evaluate the performance of the constant velocity joint greases of the examples and comparative examples.

1. Wear resistance tests (wear resistance tests using an SRV test rig)

Test method: in accordance with ASTM D5707

Test conditions: • Load 300 N
• Frequency 50 Hz
• Stroke 2.7 mm
• Temperature 50°C
• Test time 960 min

Evaluation criterion: Depth of wear of test specimen disk (μm)

2. Tests of anti-flaking performance (actual durability test)

Test method: A Birfield-type fixed joint (BJ) and a double offset plunging-type joint (DOJ) were packed with the test specimens, and their anti-flaking performance was evaluated by means of an actual test rig.

Test conditions: · Speed 500 rpm
 · Angle 6 degrees
 · Torque 600 N · m
 · Test time 300 hr

Degree of joint flaking (evaluation): (O) Very good

O Good

Δ Rather poor

3. The results of the aforementioned tests are shown in Tables 1 ~ 4.

In the case of Comparative Examples 1 ~ 4 and 6 ~ 9, since the results of the durability tests were "rather poor", the wear resistance test using the SRV test rig was omitted and not carried out.

Evaluation and Discussion

As is clear from the examples shown in Tables 1 and 2, all the constant velocity joint greases of the Examples had a low degree of wear and excellent wear resistance in the wear resistance tests using an SRV test rig. In addition, both the BJ and DOJ in the durability tests obtained ratings as "very good" or "good", and excellent results were also obtained in respect of anti-flaking performance.

Also, as shown in Examples 4, 5, 9 and 10, if a ZnDTP is also used, satisfactory test results can be obtained while reducing the total amount of the additives.

On the other hand, as is clear from the Comparative Examples shown in Tables 3 and 4, in the case of Comparative Examples 1 ~ 4 and 6 ~ 9, both the BJ and DOJ were "rather poor" in the durability tests, and satisfactory results were not obtained in respect of anti-flaking performance.

In the case of Comparative Examples 5 and 10, satisfactory results were exhibited for the BJ and DOJ in

the durability tests, and anti-flaking performance was good, but wear resistance was as much as some seven times worse than in the aforementioned Examples and these results would not sustain a practical application.

Table 1

Example		1	2	3	4	5
Composition	I Diurea grease	94	93	95	95.5	96
	II Tetraurea grease					
	Mo-DTC	2	2	2	1	1
	Mo-DTP (1)	1		1		1
	Mo-DTP (2)		2		1	
	2-(4-morpholinyldithio)benzothiazole	2	2	1	0.5	0.5
	Zn-DTP (1)				1	
	Zn-DTP (2)					0.5
	Calcium sulphonate	1	1	1	1	1
	Total wt. %	100	100	100	100	100
Test results	SRV test	6	6	5	4	3
	Durability test	Depth of wear (μm)				
		BJ				
		DOJ				
		0	(0)	0	(0)	(0)

Table 2

Example		6	7	8	9	10
Composition	I Diurea grease					
	II Tetraurea grease	94	95	94	95.5	96
	Mo-DTC	2	2	2	1	1
	Mo-DTP (1)	1		1		0.5
	Mo-DTP (2)		1	1	1	0.5
	2-(4-morpholinyldithio)benzothiazole	2	1	1	0.5	0.5
	Zn-DTP (1)					0.5
	Zn-DTP (2)				1	0.5
	Calcium sulphonate	1	1	1	1	0.5
	Total wt. %	100	100	100	100	100
Test results	SRV test	5	5	4	3	5
	Durability test	Depth of wear (μm)				
		BJ				
		DOJ				
		0	0	0	(O)	(O)

Table 3

Comparative Example		1	2	3	4	5
Composition	I Diurea grease	92	92	92	93	94
	II Tetraurea grease					
	Mo-DTC	5	5	3	3	2
	Mo-DTP (1)	3		5		1
	Mo-DTP (2)		3		2	
	2-(4-morpholinyldithio)benzothiazole					2
	Zn-DTP (1)				2	
	Zn-DTP (2)					1
	Calcium sulphonate					
	Total wt. %	100	100	100	100	100
Test results	SRV test	Not measured	Not measured	Not measured	Not measured	35
	Durability test	Δ	Δ	Δ	Δ	(0)
		Δ	Δ	Δ	Δ	0

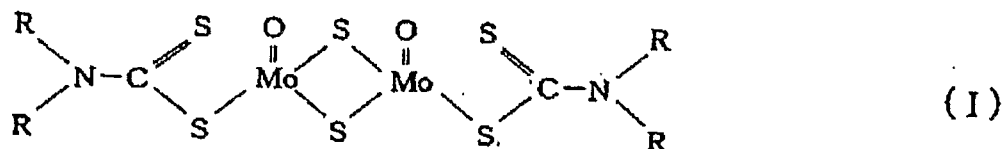
Table 4

Comparative Example		6	7	8	9	10
Composition	I Diurea grease					
	II Tetraurea grease	93	93	93	94	93
	Mo-DTC	5	5	3	2	1
	Mo-DTP (1)	2				1
	Mo-DTP (2)		2	2	2	1
	2-(4-morpholinyldithio)benzothiazole					2
	Zn-DTP (1)			2	1	1
	Zn-DTP (2)				1	1
	Calcium sulphonate					
	Total wt. %	100	100	100	100	100
Test results	SRV test	Not measured	Not measured	Not measured	Not measured	33
	Durability test	Δ	Δ	Δ	Δ	(O)
		Δ	Δ	Δ	Δ	(O)

C L A I M S

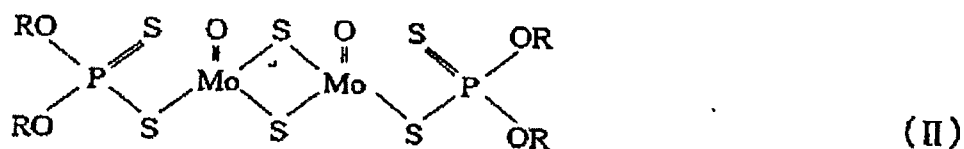
1. Grease composition for use in constant velocity joints, wherein a urea grease constituted from a base oil and a urea compound is blended with:

(A) a molybdenum dialkyldithiocarbamate as represented by the undermentioned formula (I)



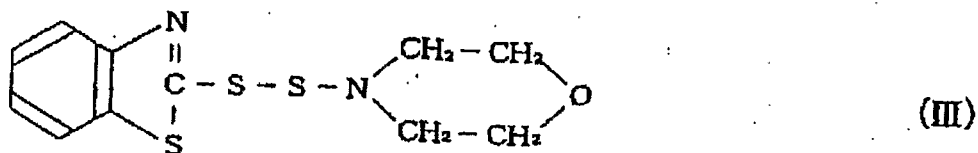
(where R denotes an alkyl group of 1 ~ 24 carbons) and

(B) at least one kind of a molybdenum dialkyldithiophosphate or a molybdenum diaryldithiophosphate as represented by the undermentioned formula (II)



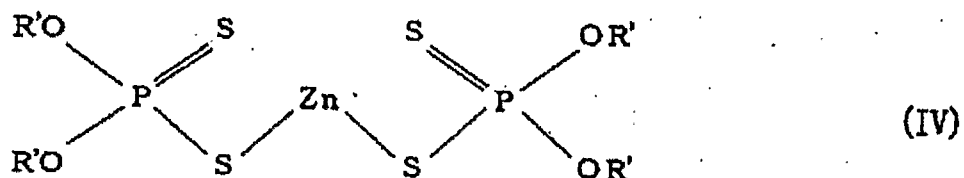
(where R is a primary or secondary alkyl group or an aryl group), the blended amount of (A) and (B) being not more than 10% by weight, and

(C) not more than 3% by weight of 2-(4-morpholinyldithio)benzothiazole as represented by the undermentioned formula (III)



and (D) not more than 3% by weight of a calcium sulphonate.

2. A grease composition for use in constant velocity joints in accordance with Claim 1 characterised in that at least one kind of a zinc dialkyldithiophosphate or a zinc diaryldithiophosphate as represented by the undermentioned formula IV



(where R' is a primary or secondary alky group or an aryl group) is further incorporated in the amount of not more than 3% by weight.

3. Use of the grease composition according to claim 1 or 2 in constant velocity joints.

INTERNATIONALSEARCHREPORT

International application No.

PCT/JP2007/059992

A. CLASSIFICATION OF SUBJECT MATTER

Int.Cl. See extra sheet

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Int.Cl. C10M 169/06, 115/08, 135/10, 135/18, 135/36, 137/10, 159/24,
C10N 10/04, 10/12, 30/06, 40/04, 50/10, F16D 3/20-3/229

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Published examined utility model applications of Japan 1922-1996
Published unexamined utility model applications of Japan 1971-2007
Registered utility model specifications of Japan 1996-2007
Published registered utility model applications of Japan 1994-2007

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP 2001-11482 A (KYODO YUSHI CO., LTD. et al.), 2001.01.16 &DE 10031648 A1 &FR 2795737 A1 &KR 2001072539 A &US 6355602 B1	1-3
Y	JP 9-324190 A (KYODO YUSHI CO., LTD. et al.), 1997.12.16 &DE 19723960 A1 &US 6037314 A	1-3

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

* Special categories of cited documents:

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Date of the actual completion of the international search

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Name and mailing address of the ISA/JP

Japan Patent Office

3-4-3, Kasumigaseki, Chiyoda-ku, Tokyo 100-8915, Japan

Authorized officer

YAMAMOTO, Masahiro

Telephone No. +81-3-3581-1101 Ext. 3483

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INTERNATIONALSEARCHREPORT

International application No.

PCT/JP2007/059992

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP 10-140174 A (NSK LTD.) , 1998.05.26 (Family:none)	1-3
Y	JP 58-61189 A (HITACHI, LTD.) , 1983.04.12 (Family:none)	1-3

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2007/059992

CLASSIFICATION OF SUBJECT MATTER

C10M169/06 (2006.01) i, F16D3/20 (2006.01) i, C10M115/08 (2006.01) n,
C10M135/10 (2006.01) n, C10M135/18 (2006.01) n, C10M135/36 (2006.01) n,
C10M137/10 (2006.01) n, C10M159/24 (2006.01) n, C10N10/04 (2006.01) n,
C10N10/12 (2006.01) n, C10N30/06 (2006.01) n, C10N40/04 (2006.01) n,
C10N50/10 (2006.01) n