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(54) **Traction fluid lubricants.**

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FR-A- 1 343 967
FR-A- 1 541 833
US-A- 2 914 550
US-A- 2 995 590
US-A- 4 141 851

(73) Proprietor : **SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V.**
Carel van Bylandtlaan 30
NL-2596 HR Den Haag (NL)

(72) Inventor : **Dare-Edwards, Martin Philip**
55 Moorside Avenue
Parkgate South Wirral (GB)

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Description

The present process relates to the use of certain organo-silicon compounds as lubricants, in particular their use in traction drives.

These lubricants can be used in a variety of engineering applications, being of particular value in traction drives. Traction is broadly defined as the adhesive friction of a body on a surface on which it moves. A traction drive is a device in which torque is transmitted from an input element to an output element through nominal point or line contact typically with a rolling action by virtue of the traction between the contacting elements. While traction elements are commonly spoken of as being in contact, it is generally accepted that a fluid film is present therebetween. Almost all traction drives require fluids to remove heat, to prevent wear at the contact surfaces and to lubricate bearings and other moving parts associated with the drive. Thus, instead of metal to metal rolling contact there is a film of fluid introduced into the contact zone and interposed between the metal elements. The nature of this fluid determines to a large extent the limits in performance and the capacity of the drive. Most traction drives are designed to operate with a traction fluid which preferably has a coefficient of traction above about 0.06, a viscosity in the range of about 4-20,000 mPa.s over a temperature range of 40°C to -20°C and good thermal and oxidative stability. The fluid should also be noncorrosive to common materials of construction and have good load-bearing and low wear-rate properties.

Mineral base oils are rather unsatisfactory lubricants for traction drives since in general their traction (friction) coefficient is low, which means that for any given load applied to the gears the maximal tangential force that may be transmitted by the friction wheels is low.

FR-A-1 343 967 discloses the use of an aryl aliphatic-oxy silane containing 2 or 3 aryl groups and an aliphatic-oxy group containing 6 or more carbon atoms, as a functional fluid, e.g. a heat-transfer medium, hydraulic fluid or lubricant. The preferred silanes are the diaryl dialkoxy silanes.

FR-A-1 541 833 discloses, as traction fluids, organic compounds

(1) having about 12 to about 70 carbon atoms, up to 8 of which can be replaced by atoms other than carbon atoms and can be selected from such atoms as oxygen, nitrogen, phosphorus and silicon, and

(2) containing

(a) at least 1 saturated carbon atom-containing ring having at least 6 member atoms, or

(b) an acyclic structure in which there are at least 3 quaternary carbon atoms, and

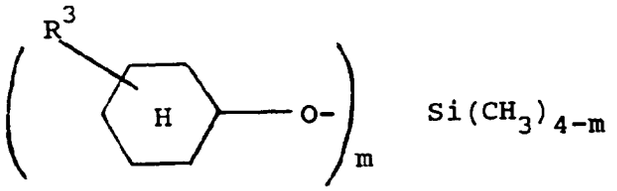
(3) having a coefficient of traction of at least 0.06.

It has now been found that certain organosilicon compounds constitute good lubricants and traction fluids. Accordingly, the present invention provides the use as lubricants, and especially as traction fluids, of organosilicon compounds of the general formula I

$$(R^1O)_mSiR^2_{4-m} \qquad I$$

wherein m is 2, 3 or 4; each group R^1 represents a cyclohexyl group optionally substituted by an alkyl group which contains 1-6 carbon atoms; and R^2 represents an alkyl group of up to 4 carbon atoms, with the proviso that when each group R^1 represents an unsubstituted cyclohexyl group, then m is 2 or 3, the said organo-silicon compounds being liquid at ambient temperature. The alkyl groups, whether present as grouping R^2 or as substituent(s) in group R^1, may be linear or branched and groups having up to 4 carbon atoms provide a particularly suitable combination of physical properties and material costs.

Preferred silicone esters are those of formula II:



II

wherein m is 2, 3 or 4; and R^3 represents H or methyl,
with the proviso that when R^3 represents H, then m is 2 or 3.

These organo-silicon compounds are known compounds, and therefore may be prepared by known procedures, such as the reaction between the appropriate alcohol and a chlorosilane:-

$$Cl_mSiR^2_{4-m} + mR^1OH = R^2_{4-m}Si(OR^1)_m$$

Naturally, the optimal reaction conditions depend to some extent on the precise nature of R^1 and R^2, and in the (preferred) case where R^1 is cyclohexyl and R^2 is methyl it was found convenient to carry out the reaction in an organic solvent, such as dichloromethane, in the presence of a base, such as pyridine or triethylamine,

to remove the hydrogen chloride generated, and at a temperature not exceeding 25°C. As is normal with chlorosilane reactions, the process should desirably be carried out with dry reagents and under an inert atmosphere (e.g. nitrogen) to minimize hydrolysis of the reagents. Purification of the products was conveniently achieved by use of a thin-film evaporator.

It has been found that the viscosity characteristics of the above organo-silicon compounds are very suitable for use in e.g. friction wheel gears (traction drives) in which application they may be admixed with conventional grease thickeners. Such thickeners can be of any number of materials commonly used to thicken mineral oils to lubricating viscosity, including both organic and inorganic compositions such as metallic soaps, synthetic polymers, organosiloxanes, clays, bentonite, and colloidal silica. Suitably, the viscosity properties of compounds to be used in traction drives are such that the compounds are operable between -30 and 150°C. To achieve this it is advantageous that the ester compounds in the lubricants according to the invention have a viscosity of at most 1000, preferably 250mm^2/s at 40°C and at least 1, preferably 3mm^2/s at 100°C.

The compounds can be used as lubricants in various engineering applications. Since they show excellent lubricating performance in traction drives, the invention in particular provides the use of these organo-silicon compounds as traction fluids, and also the operation of a traction drive wherein such compounds form the traction fluid.

The organo-silicon compounds of the present invention can be used per se as lubricants. They can be mixed with other lubricants such as mineral or synthetic oils, and various additives can be added, such as VI-improvers, pour point depressants, dispersants, detergents, anti-oxidants and the like. A mixture that can be of particular interest for traction fluid applications is a blend with a polyolefin, in particular a polyalpha olefin, especially polyisobutylene, since the presence of the polymer can usefully enhance the traction coefficient of the fluid blend. The molecular weight of such polyolefin blend components is conveniently in the range 500-10,000, a specific example of a suitable polyisobutylene being "Hyvis", and the proportion of polyolefin may vary from zero to 70 by weight.

The following Examples illustrate the preparation of representative compounds used in the present invention, together with their frictional properties.

Example 1

Dimethyldi(cyclohexyloxy)silane

A solution of cyclohexanol (1240g, 12.4m) and triethylamine (1250.0g, 12.4m) in dichloromethane (6L) was stirred at 0-5°C in an atmosphere of dry nitrogen. Dimethyldichlorosilane (800g, 6.2m) was added dropwise over 3-4 hours, maintaining the temperature of the reaction mixture at below 10°C throughout this period. On completion of the addition, the mixture was stirred at room temperature for a further 18 hours and then the white precipitate of triethylamine hydrochloride filtered and washed with dichloromethane (500ml). The combined organic filtrates were washed with water (2 x 2L) and the solvent removed by evaporation under reduced pressure at 30°C to give a pale-yellow oil (1750g). Traces of residual solvent and unreacted alcohol were removed by evaporation in a KDL-4 thin-film evaporator at 40°C (1.0mm Hg pressure) (133.3 Pa). The oily residue was then distilled using the same apparatus at 120°C (0.4mm Hg) (53.3 Pa) to give dimethyldi(cyclohexyloxy)silane (1200g, 75.5%, 97.6% w/w by GLC analysis, using an area percent calculation), as a colourless oil.

Example 2

Methyltri(cyclohexyloxy)silane

A solution of cyclohexanol (1240g, 12.4m) and triethylamine (1273.0g, 12.4m) in dichloromethane (6L) was stirred at 0-5°C in an atmosphere of dry nitrogen. Methyltrichlorosilane (620.0g, 4.15m) was added dropwise over 3-4 hours, maintaining the temperature of the reaction mixture at below 10°C throughout this period. On completion of the addition, the mixture was stirred at room temperature for a further 18 hours and then the white precipitate of triethylamine hydrochloride filtered and washed with dichloromethane (500ml). The combined organic filtrates were washed with water (2 x 2L) and the solvent removed by evaporation under reduced pressure at 30°C to give a pale-yellow oil (1543.4g). Traces of residual solvent and unreacted alcohol were removed by evaporation in a KDL-4 thin-film evaporator at 40°C (1.0mm Hg pressure) (133.3 Pa). The oily residue was then distilled using the same apparatus at 160°C (0.2mm Hg) (26.7 Pa) to give methyltri(cyclohexyloxy)- silane (1116.0g, 79.0%, 96.5% w/w by GLC analysis using an area percent calculation), as a colourless oil.

Example 3

Dimethyldi(methylcyclohexyloxy)silane

A solution of methylcyclohexanol (1400.0 g, 12.3m) and triethylamine (1250.0g, 12.3m) in dichloromethane (6L) was stirred at 0-5°C in an atmosphere of dry nitrogen. Dimethyldichlorosilane (800.0g, 6.2m) was added dropwise over 3-4 hours, maintaining the temperature of the reaction mixture at below 10°C throughout this period. On completion of the addition, the mixture was stirred at room temperature for a further 18 hours and then the white precipitate of triethylamine hydrochloride filtered and washed with dichloromethane (500ml). The combined organic filtrates were washed with water (2 x 2L) and the solvent removed by evaporation under reduced pressure at 30°C to give a pale-yellow oil (1624g). Traces of residual solvent and unreacted alcohol were removed by evaporation in a KDL-4 thin-film evaporator at 40°C (1.0mm Hg pressure) (133.3 Pa). The oily residue was then distilled using the same apparatus at 120°C (0.4mm Hg) (53.3 Pa) to give dimethyldi(methylcyclohexyloxy)silane (1340g, 76.0%, 99% w/w as a mixture of isomers by GLC analysis using an area percent calculation), as a colourless oil.

Example 4

Methyltri(methylcyclohexyloxy)silane

A solution of methylcyclohexanol (1260.0g, 11.0m) and triethylamine (1130.0g, 11.0m) in dichloromethane (6L) was stirred at 0-5°C in an atmosphere of dry nitrogen. Methyltrichlorosilane (550.0g, 3.68m) was added dropwise over 3-4 hours, maintaining the temperature of the reaction mixture at below 10°C throughout this period. On completion of the addition, the mixture was stirred at room temperature for a further 18 hours and then the white precipitate of triethylamine hydrochloride filtered and washed with dichloromethane (500ml). The combined organic filtrates were washed with water (2 x 2L) and the solvent removed by evaporation under reduced pressure at 30°C to give a pale-yellow oil (1202g). Traces of residual solvent and unreacted alcohol were removed by evaporation in a KDL-4 thin-film evaporator at 40°C (1.0mm Hg pressure) (133.3 Pa). The oily residue was then distilled using the same apparatus at 180°C (0.4mm Hg) (53.3 Pa) to give methyltri(methylcyclohexyloxy)silane (1025.1g, 72.8%, 99.3% w/w as a mixture of isomers by GLC analysis using an area percent calculation), as a colourless oil.

Example 5

Tetra(methylcyclohexyloxy)silane

A solution of methylcyclohexanol (1560g, 13.7m) and triethylamine (1380g, 13.6m) in dichloromethane (6L) was stirred at 0-5°C in an atmosphere of dry nitrogen. Silicon tetrachloride (577.0g, 3.4m) was added dropwise over 3-4 hours, maintaining the temperature of the reaction mixture at below 10°C throughout this period. On completion of the addition, the mixture was stirred at room temperature for a further 18 hours and then the white precipitate of triethylamine hydrochloride filtered and washed with dichloromethane (500ml). The combined organic filtrates were washed with water (2 x 2L) and the solvent removed by evaporation under reduced pressure at 30°C to give a yellow oil (1595.0g). Traces of residual solvent and unreacted alcohol were removed by evaporation in a KDL-4 thin-film evaporator at 40°C (1.0mm Hg pressure) (133 Pa). The oily residue was then distilled using the same apparatus at 185°C (0.4mm Hg) (53.3 Pa) to give tetra(methylcyclohexyloxy)silane (1190.3g, 73.5%, 99.6% w/w as a mixture of isomers by GLC analysis using an area percent calculation), as a colourless to pale-yellow oil.

Example 6

Friction coefficient measurement

All friction measurements were performed on a two-disc machine. Hardened steel discs are fixed on the ends of two shafts so as to make tangential contact with each other. Radial forces may be applied to press the discs together with loads of 0-200 kgf. Each disc is driven by an electric motor. The speeds of rotation of the two discs are different, such that there is a slip.

Between the electric motor and the shaft carrying the lower test specimen, a measuring device is fitted which indicates the transmitted friction torque. The measuring device is a gear dynamometer with a pendulum

which is swung out of its vertical balanced position when power is transmitted, the sine of the angle of inclination being a measure of the torque. The torque measurement is pre-calibrated through the design and dimensions of the instrument. The friction coefficient is defined by the torque measured divided by the product of the radial force times the radius of the lower disc.

Both discs used had a diameter of 50.0mm, the upper disc having a width of 3mm, the lower one having a width of 10mm. The top shaft speed was 606rpm, and the mean tangential (or surface) velocity was 1.48 ms^{-1}. The slip employed was 9.1%.

All experiments were run at ambient temperature (21°C±2°C). The friction readings are provided at loadings equivalent to Hertzian stresses of 0.69, 0.97, 1.19 and 1.38 GPa.

The friction coefficients of the compounds are indicated in the following Table. For the compound of Example I, whose m.pt. is 48-50°C, these coefficients were determined on a supercooled fluid at 21(±2)°C.

The kinematic viscosity properties of the compounds are also included in this Table.

Tetra(cyclohexyloxy)silane was also synthesised, but the foregoing frictional properties could not be measured since the material was solid to 71°C.

Table

	Compound of Example:-				
	1	2	3	4	5
Kinematic Visc. cSt 40°C	4.130	15.01	4.061	12.18	52.53
Kinematic Visc. cSt 100°C	1.479	3.173	1.431	2.800	5.934
Viscosity Index	73	54	78	55	22
Density g cm^{-3} 40°C	0.9333	0.9772	0.9080	0.9427	0.9635
Density g cm^{-3} 100°C	0.8845	0.9330	0.8622	0.9013	0.9215
Traction Coefficient at					
0.69 GPa	0.063	0.075	0.068	0.081	0.089
0.97 GPa	0.078	0.090	0.080	0.093	0.103
1.19 GPa	0.084	0.098	0.083	0.097	0.109
1.38 GPa	0.086	0.104	0.085	0.100	0.110

Claims

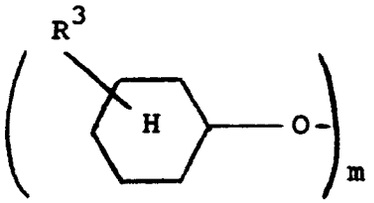
Claims for the following Contracting States : BE, DE, FR, GB, IT, NL

1. Use as lubricants of organo-silicon compounds of the general formula I:-

$$(R^1O)_mSiR^2_{4-m} \qquad I$$

wherein m is 2, 3 or 4; each group R^1 represents a cyclohexyl group optionally substituted by an alkyl group which contains 1-6 carbon atoms; and R^2 represents an alkyl group of up to 4 carbon atoms, with the proviso that when each group R^1 represents an unsubstituted cyclohexyl group, then m is 2 or 3, the said organo-silicon compounds being liquid at ambient temperature.

2. Use according to claim 1 wherein the organo-silicon compound is of formula II:



$$Si(CH_3)_{4-m} \qquad II$$

wherein R^3 represents H or methyl and m is 2, 3 or 4, with the proviso that when R^3 represents H, then m is 2 or 3.

3. Use according to claim 1 or 2 wherein the organo-silicon compound has a kinematic viscosity of at most 1000 mm^2/s at 40°C and at least 1mm^2/s at 100°C.

4. Lubricant composition which contains as the major component an organo-silicon compound as defined in any one of claims 1-3.

5. Use as a traction fluid of an organo-silicon compound as defined in any one of claims 1-3 or a composition as defined in claim 4.

6. Method of operating a traction drive wherein the traction fluid is an organo-silicon compound as defined in any one of claims 1-3 or a composition as defined in claim 4.

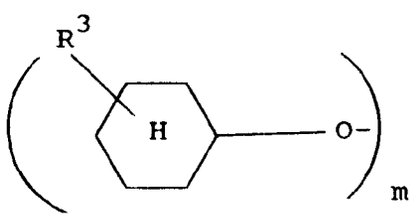
Claims for the following Contracting State : ES

1. Use as lubricants of organo-silicon compounds of the general formula I:-

$$(R^1O)_mSiR^2_{4-m} \qquad I$$

wherein m is 2, 3 or 4; each group R^1 represents a cyclohexyl group optionally substituted by an alkyl group which contains 1-6 carbon atoms; and R^2 represents an alkyl group of up to 4 carbon atoms, with the proviso that when each group R^1 represents an unsubstituted cyclohexyl group, then m is 2 or 3, the said organo-silicon compounds being liquid at ambient temperature.

2. Use according to claim 1 wherein the organo-silicon compound is of formula II:



$$Si(CH_3)_{4-m} \qquad II$$

wherein R^3 represents H or methyl and m is 2, 3 or 4, with the proviso that when R^3 represents H, then m is 2 or 3.

3. Use according to claim 1 or 2 wherein the organo-silicon compound has a kinematic viscosity of at most

1000 mm^2/s at 40°C and at least 1mm^2/s at 100°C.

4. Use as a traction fluid of an organo-silicon compound as defined in any one of claims 1-3 or a lubricant composition which contains as the major component an organo-silicon compound as defined in any one of claims 1-3.

5. Method of operating a traction drive wherein the traction fluid is an organo-silicon compound as defined in any one of claims 1-3 or a composition as defined in claim 4.

Patentansprüche

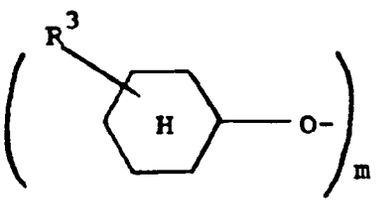
Patentansprüche für folgende Vertragsstaaten : BE, DE, FR, GB, IT, NL

1. Verwendung von Organosiliciumverbindungen der nachstehenden Formel I als Schmiermittel

$$(R^1O)_mSiR^2_{4-m} \qquad I$$

in welcher m den Wert 2, 3 oder 4 hat, jede Gruppe R^1 eine Cyclohexylgruppe bedeutet, welche gegebenenfalls durch eine Alkylgruppe mit 1 bis 6 Kohlenstoffatomen substituiert ist, und R^2 eine Alkylgruppe mit bis zu 4 Kohlenstoffatomen darstellt, mit der Maßgabe, daß für den Fall, daß jede Gruppe R^1 eine nicht substituierte Cyclohexylgruppe ist, m den Wert 2 oder 3 aufweist, wobei die besagten Organosiliciumverbindungen bei Umgebungstemperatur flüssig sind.

2. Vewendung nach Anspruch 1, wobei die Organosiliciumverbindung der nachstehenden Formel II entspricht



II

in welcher R^3 Wasserstoff oder Methyl ist und m den Wert 2, 3 oder 4 hat mit der Maßgabe, daß für den Fall, daß R^3 Wasserstoff bedeutet, m den Wert 2 oder 3 hat.

3. Verwendung nach Anspruch 1 oder 2, wobei die Organosiliciumverbindung eine kinematische Viskosität von höchstens 1000 mm^2/s bei 40°C und mindestens von 1 mm^2/s bei 100°C aufweist.

4. Schmiermittelzusammensetzung, welche als Hauptkomponente eine Organosiliciumverbindung, wie in irgendeinem der Ansprüche 1 bis 3 definiert, enthält.

5. Verwendung einer Organosiliciumverbindung, wie in irgendeinem der Ansprüche 1 bis 3 definiert, oder eine Zusammensetzung wie in Anspruch 4 definiert, als Schmierflüssigkeit für Reibgetriebe.

6. Methode zum Betrieb eines Reibgetriebes, in welcher die Getriebeschmierflüssigkeit eine Organosiliciumverbindung, wie in irgendeinem der Ansprüche 1 bis 3 definiert, oder eine Zusammensetzung, wie in Anspruch 4 definiert, ist.

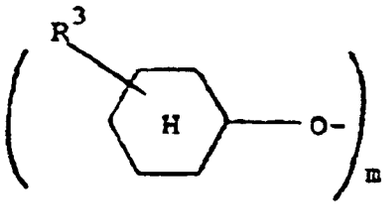
Patentansprüche für folgenden Vertragsstaat : ES

1. Verwendung von Organosiliciumverbindungen der nachsteheden Formel I als Schmiermittel

$$(R^1O)_mSiR^2_{4-m} \qquad I$$

in welcher m den Wert 2, 3 oder 4 hat, jede Gruppe R^1 eine Cyclohexylgruppe bedeutet, welche gegebenenfalls durch eine Alkylgruppe mit 1 bis 6 Kohlenstoffatomen substituiert ist, und R^2 eine Alkylgruppe mit bis zu 4 Kohlenstoffatomen darstellt, mit der Maßgabe, daß für den Fall, daß jede Gruppe R^1 eine nicht substituierte Cyclohexylgruppe ist, m den Wert 2 oder 3 aufweist, wobei die besagten Organosiliciumverbindungen bei Umgebungstemperatur flüssig sind.

2. Vewendung nach Anspruch 1, wobei die Organosiliciumverbindung der nachstehenden Formel II entspricht



II

in welcher R^3 Wasserstoff oder Methyl ist und m den Wert 2, 3 oder 4 hat mit der Maßgabe, daß für den Fall, daß R^3 Wasserstoff bedeutet, m den Wert 2 oder 3 hat.

3. Verwendung nach Anspruch 1 oder 2, wobei die Organosiliciumverbindung eine kinematische Viskosität von höchstens 1000 mm^2/s bei 40°C und mindestens von 1 mm^2/s bei 100°C aufweist.

4. Verwendung einer Organosiliciumverbindung, wie in irgendeinem der Ansprüche 1 bis 3 definiert, oder einer Schmiermittelzusammensetzung, welche als Hauptkomponente eine Organosiliciumverbindung , wie in irgendeinem der Ansprüche 1 bis 3 definiert, enthält, als Schmierflüssigkeit für Reibgetriebe.

5. Methode zum Betrieb eines Reibgetriebes, in welcher die Getriebeschmierflüssigkeit eine Organosiliciumverbindung, wie in irgendeinem der Ansprüche 1 bis 3 definiert, oder eine Zusammensetzung, wie in Anspruch 4 definiert, ist.

Revendications

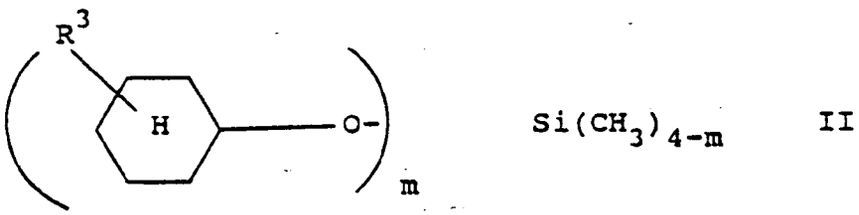
Revendications pour les Etats contractants suivants : BE, DE, FR, GB, IT, NL

1. Utilisation comme lubrifiants de composés organo-silicium de la formule générale I :

$$(R^1O)_mSiR^2_{4-m} \qquad I$$

où m est 2, 3 ou 4 ; chaque groupe R^1 représente un groupe cyclohexyle éventuellement substitué par un groupe alcoyle qui contient 1-6 atomes de carbone ; et R^2 représente un groupe alcoyle ayant jusqu'à 4 atomes de carbone, avec la condition que quand chaque groupe R^1 représente un groupe cyclohexyle non substitué, alors m est 2 ou 3, ces composés organo-silicium étant liquides à la température ambiante.

2. Utilisation selon la revendication 1, où le composé organo-silicium est de formule II :



où R^3 représente H ou un groupe méthyle et m est 2, 3 ou 4, avec la condition que quand R^3 représente H, alors m est 2 ou 3.

3. Utilisation selon la revendication 1 ou 2, où le composé organosilicium a une viscosité cinématique d'au maximum 1000 mm^2/s à 40°C et d'au moins 1 mm^2/s à 100°C.

4. Composition lubrifiante qui contient comme constituant majeur un composé organo-silicium tel que défini dans l'une quelconque des revendications 1-3.

5. Utilisation comme fluide de traction d'un composé organo-silicium tel que défini dans l'une quelconque des revendications 1-3 ou d'une composition telle que définie dans la revendication 4.

6. Méthode pour faire fonctionner une transmission à traction, où le fluide de traction est un composé or-

gano-silicium tel que défini dans l'une quelconque des revendications 1-3 ou une composition telle que définie dans la revendication 4.

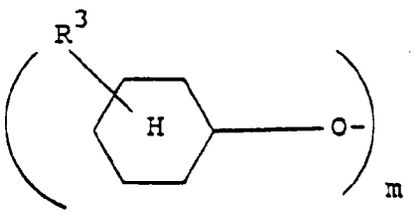
Revendications pour l'Etat contractant suivant : ES

1. Utilisation comme lubrifiants de composés organo-silicium de la formule générale I :

$$(R^1O)_mSiR^2_{4-m} \qquad I$$

où m est 2, 3 ou 4 ; chaque groupe R^1 représente un groupe cyclohexyle éventuellement substitué par un groupe alcoyle qui contient 1-6 atomes de carbone ; et R^2 représente un groupe alcoyle ayant jusqu'à 4 atomes de carbone, avec la condition que quand chaque groupe R^1 représente un groupe cyclohexyle non substitué, alors m est 2 ou 3, ces composés organosilicium étant liquides à la température ambiante.

2. Utilisation selon la revendication 1, où le composé organo-silicium est de formule II :



$$Si(CH_3)_{4-m} \qquad II$$

où R^3 représente H ou un groupe méthyle et m est 2, 3 ou 4, avec la condition que quand R^3 représente H, alors m est 2 ou 3.

3. Utilisation selon la revendication 1 ou 2, où le composé organo-silicium a une viscosité cinématique d'au maximum 1000 mm^2/s à 40°C et d'au moins 1 mm^2/s à 100°C.

4. Utilisation comme fluide de traction d'un composé organo-silicium tel que défini dans l'une quelconque des revendications 1-3 ou d'une composition lubrifiante qui contient comme constituant majeur un composé organo-silicium tel que défini dans l'une quelconque des revendications 1-3.

5. Méthode pour faire fonctionner une transmission à traction, où le fluide de traction est un composé organo-silicium tel que défini dans l'une quelconque des revendications 1-3 ou une composition telle que définie dans la revendication 4.