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- as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii))
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(54) Title: SILICONE HYDRAULIC FLUIDS

(57) Abstract: The present invention relates to a hydraulic fluid composition comprising (A) at least one polydiorganosiloxane; (B) a chlorendate diester; and (C) a lubricant compound which is an antimony or lead compound of a dialkylphosphorodithioate.

SILICONE HYDRAULIC FLUIDS

CROSS-REFERENCE TO RELATED APPLICATIONS

5 [0001] This application claims the benefit of US Application No. 61/352617 as filed on 8 June, 2010.

BACKGROUND OF THE INVENTION

10 [0002] The present invention relates to a hydraulic fluid composition comprising (A) at least one polydiorganosiloxane; (B) a chlorendate diester; and (C) a lubricant compound which is an antimony or lead compound of a dialkyldithiocarbamate or dialkylphosphorodithioate.

15 [0003] Hydraulic systems, in which power is transferred from one place to another, are used extensively in industrial equipment, farm equipment, transportation equipment, and the like. Illustrative of such equipment are lifters, jacks, elevators, mills, presses, and braking, power steering and suspension systems for vehicles.

20 [0004] High pressures and temperatures, which are frequently present in hydraulic systems, place high demands on the thermal and oxidative stability of the fluid used as the hydraulic medium. In addition, the lubricity of the hydraulic fluid is especially important with hydraulic systems wherein a pump is used to pressurize or move the hydraulic fluid from one place to another.

25 [0005] Polydiorganosiloxanes have been recognized as having exceptional thermal and oxidative stability, compatibility with seal materials, and high viscosity indices, said properties making them potentially useful as hydraulic fluids. Unfortunately, the generally low surface tension of polydiorganosiloxanes tends to cause them to have marginal lubricity on metals. As a result, additives improving the lubricity of polydiorganosiloxanes have been sought.

30 [0006] Although the use of certain silicone materials in hydraulic fluid compositions have been widely accepted, there still exists a need for a silicone hydraulic fluid composition which has better stability to settling at room temperature and at lower and higher

temperatures . There is also a need for a hydraulic fluid having improved performance over a wider temperature range and higher oxidation stability.

5 BRIEF SUMMARY OF THE INVENTION

[0007] The present invention relates to a hydraulic fluid composition comprising (A) at least one polydiorganosiloxane; (B) a chlorendate diester; and (C) a lubricant compound which is an antimony or lead compound of a dialkyldithiocarbamate or dialkylphosphorodithioate.

10 [0008] The present invention further relates to a hydraulic fluid composition comprising (A) at least two polydiorganosiloxanes; (B) a chlorendate diester; and (C) a lubricant compound which is an antimony or lead compound of a dialkyldithiocarbamate or dialkylphosphorodithioate.

15 [0009] In yet another aspect of the present invention, a method is provided for transmitting power from one place to another place using the polydiorganosiloxane hydraulic fluid compositions of this invention.

[0010] It is an object of the present invention to provide improved polydiorganosiloxane hydraulic fluid compositions. Another object is to provide polydiorganosiloxane hydraulic fluid compositions having good lubricating properties. A further object is to provide a
20 hydraulic fluid which has settling stability over a wide range of temperature. Another object is to provide an improved process for transmitting power from one place to another place via a hydraulic fluid.

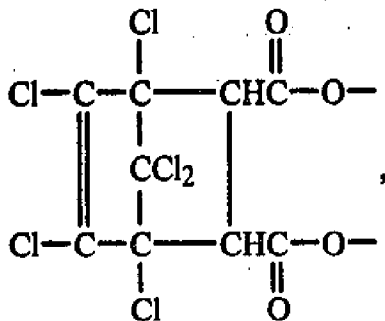
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DETAILED DESCRIPTION OF THE INVENTION

[0011] The present invention relates to a composition comprising

(A) 2 to 99 parts of at least one polydiorganosiloxane selected from (A1) 0 to 99 parts
 5 of a polydiorganosiloxane having a formula $R'R_2Si-O(Me_2SiO)_wSiR_2R'$ and a viscosity of $1.00 \times 10^{-6} \text{ m}^2/\text{second}$ to $1.00 \times 10^{-4} \text{ m}^2/\text{second}$ at 25°C and (A2) 0 to 99 parts having a formula $R'R_2Si-O(Me_2SiO)_x(MeR^aSiO)_ySiR_2R'$ and a viscosity of $1.00 \times 10^{-5} \text{ m}^2/\text{second}$ to $1.00 \times 10^{-4} \text{ m}^2/\text{second}$ at 25°C , wherein Me represents the methyl group, each R represents a monovalent group selected from hydrocarbon groups containing from 1 to 6 carbon atoms,
 10 and halogenated hydrocarbon groups containing from 1 to 6 carbon atoms, each R' represents a group selected from R groups, the hydride group, and the hydroxy group, w has an average value from 1 to 70, each R^a represents an aryl hydrocarbon group having from 6 to 10 carbon atoms, x has an average value from 5 to 50, and y has an average value from 0.5 to 6;

(B) 0.9 to 60 parts by weight of a chlorendate diester having the formula
 15 $R''O_2CQCO_2R''$, wherein $-O_2C-QCO_2-$ represents the chlorendate residue,



and each R'' represents a group selected from alkyl groups containing from 4 to 10 carbon atoms and the tetrahydrofurfuryl group; and

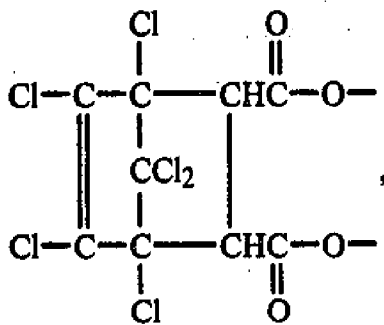
(C) 0.1 to 38 parts by weight of a lubricant compound selected from N,N-
 20 dialkyldithiocarbamates of lead and antimony and dialkylphosphorodithioates of lead and antimony, the total parts of (A) plus (B) plus (C) being 100 parts by weight.

[0012] In another embodiment, the present invention is a composition comprising

(A) 2 to 99 parts of at least two polydiorganosiloxanes selected from (A1) 1 to 99 parts
 25 of a polydiorganosiloxane having a formula $R'R_2Si-O(Me_2SiO)_wSiR_2R'$ and a viscosity of $1.00 \times 10^{-6} \text{ m}^2/\text{second}$ to $1.00 \times 10^{-4} \text{ m}^2/\text{second}$ at 25°C and (A2) 1 to 99 parts of a

polydiorganosiloxane having a formula $R'R_2Si-O(Me_2SiO)_x(MeR^aSiO)_ySiR_2R'$, wherein Me represents the methyl group, each R represents a monovalent group selected from hydrocarbon groups containing from 1 to 6 carbon atoms, and halogenated hydrocarbon groups containing from 1 to 6 carbon atoms, each R' represents a group selected from R groups, the hydride group, and the hydroxy group, w has an average value from 1 to 70, each R^a represents an aryl hydrocarbon group having from 6 to 10 carbon atoms, x has an average value from 5 to 50, and y has an average value from 0.5 to 6;

(B) 0.9 to 60 parts by weight of a chlorendate diester having the formula $R''O_2CQCO_2R''$, wherein $-O_2C-QCO_2-$ represents the chlorendate residue,



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and each R'' represents a group selected from alkyl groups containing from 4 to 10 carbon atoms, and the tetrahydrofurfuryl group; and

(C) 0.1 to 38 parts by weight of a lubricant compound selected from N,N-

15 dialkyldithiocarbamates of lead and antimony and dialkylphosphorodithioates of lead and antimony, the total parts of (A) plus (B) plus (C) being 100 parts by weight.

[0013] The at least one polydiorganosiloxane, component (A) in the compositions of the present invention is selected from (A1) a polydiorganosiloxane having a formula $R'R_2Si-O(Me_2SiO)_zSiR_2R'$ and (A2) a polydiorganosiloxane having a formula

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$R'R_2Si-O(Me_2SiO)_x(MeR^aSiO)_ySiR_2R'$, wherein Me represents the methyl group, each R represents a monovalent group selected from hydrocarbon groups containing from 1 to 6 carbon atoms, and halogenated hydrocarbon groups containing from 1 to 6 carbon atoms, each R' represents a group selected from R groups, the hydride group, and the hydroxy group, R^a represents an aryl hydrocarbon group having from 6 to 10 carbon atoms, w has an average

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value from 1 to 70, x has an average value from 5 to 50, and y has an average value from 0.5 to 6.

[0014] Examples of suitable R hydrocarbon groups containing from 1 to 6 carbon atoms include methyl, ethyl, propyl, isobutyl, pentyl, isopentyl, neopentyl, hexyl, vinyl, and allyl.

5 Halogenated hydrocarbon groups consist of hydrocarbon groups as hereinabove delineated in which one or more of the hydrogen atoms has been replaced by a halogen atom, such as fluorine, chlorine, or bromine. Examples of halogenated hydrocarbon groups include chloromethyl, 3 chloropropyl, and 3,3,3 trifluoropropyl. Alternatively, each R is a hydrocarbon group containing 1 to 4 carbon atoms. Alternatively, each R is methyl .

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[0015] R' represents a group selected from R groups, the hydride group, and the hydroxy group. The R groups are as described above. Alternatively, each R' is an R group. Alternatively, each R' is methyl.

15 **[0016]** Examples of suitable R^a hydrocarbon groups containing from 6 to 10 carbon atoms are phenyl, tolyl, mesityl, and naphthyl. Alternatively, each R^a is phenyl.

[0017] Subscript w of Component (A1) has an average value from 1 to 70, alternatively 3 to 42, alternatively 5 to 35.

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[0018] Subscript x of Component (A2) has an average value from 5 to 50, alternatively 11 to 43, alternatively 11 to 37.

25 **[0019]** Subscript y of Component (A2) has an average value from 0.5 to 6, alternatively 3 to 5, alternatively 3 to 4.

[0020] The viscosity of polydiorganosiloxane component (A1) is from 1.00×10^{-6} m²/second to 1.00×10^{-4} m²/second (1 to 100 centistokes) at 25° C. Alternatively, the viscosity of polydiorganosiloxane component (A1) is from 2.00×10^{-6} m²/second to 5.00×10^{-5} m²/second at 25° C (2 to 50 centistokes). Alternatively, the viscosity of

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polydiorganosiloxane component (A1) is from 5.00×10^{-6} m²/second to 3.00×10^{-5} m²/second at 25° C. (5 to 30 centistokes).

5 [0021] The viscosity of polydiorganosiloxane component (A2) is from 1.00×10^{-5} m²/second to 1.00×10^{-4} m²/second (10 to 100 centistokes) at 25° C. Alternatively, the viscosity of polydiorganosiloxane component (A2) is from 2.00×10^{-5} m²/second to 7.50×10^{-5} m²/second at 25° C. (20 to 75 centistokes). Alternatively, the viscosity of polydiorganosiloxane component (A2) is from 2.00×10^{-5} m²/second to 5.00×10^{-5} m²/second at 25° C. (20 to 50 centistokes)

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[0022] Component (A) may be present in the composition in amounts from 2 to 99 parts based on 100 parts of components (A) plus (B) plus (C). Alternatively, Component (A) is present in amounts from 10 to 97 parts on the same basis. Alternatively, Component (A) is present in amounts from 20 to 96 parts on the same basis. Alternatively, Component (A) is present in amounts from 30 to 95 parts on the same basis. Whether Component (A) is at least one or at least two polydiorganosiloxanes, total parts Component (A) equals parts by weight Component (A1) plus parts by weight Component (A2).

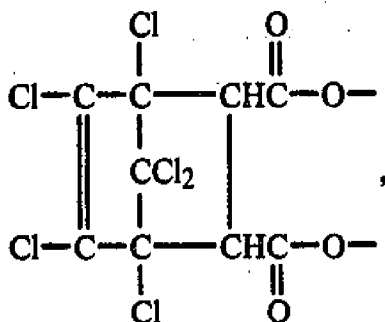
20 [0023] Component (A1) may be present in the composition in amounts from 0 to 99 parts based on 100 parts of components (A) plus (B) plus (C). Alternatively, Component (A1) is present in amounts from 1 to 99 parts on the same basis. Alternatively, Component (A1) is present in amounts from 10 to 67 parts on the same basis. Alternatively, Component (A1) is present in amounts from 20 to 50 parts on the same basis. Alternatively, Component (A1) is present in amounts from 30 to 45 parts on the basis.

30 [0024] Component (A2) may be present in the composition in amounts from 0 to 99 parts based on 100 parts of components (A) plus (B) plus (C). Alternatively, Component (A2) is present in amounts from 1 to 99 parts on the same basis. Alternatively, Component (A2) is present in amounts from 30 to 87 parts on the same basis. Alternatively, Component (A2) is present in amounts from 35 to 76 parts on the same basis. Alternatively, Component (A2) is present in amounts from 50 to 65 parts on the basis.

[0025] Suitable methods for synthesis of polydiorganosiloxanes for use as component (A1) and/or (A2) in the compositions of the present invention are well known. Illustrative of suitable synthetic methods is the cohydrolysis and subsequent condensation of appropriately selected diorganodialkoxysilanes or diorganodichlorosilanes, along with desired amounts of $R'R_2SiX$ species, wherein R' and R are as defined above and X represents a hydrolyzable group, such as a halo group, such as chloro, fluoro or bromo; or an alkoxy group, such as methoxy, or ethoxy. Another suitable synthetic method is the acid or base catalyzed equilibration of diorganocyclosiloxanes and an $R'R_2SiX$ species as defined above.

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[0026] The chlorendate diester, component (B) in the compositions of the present invention, has the formula $R''O_2CQCO_2R''$, wherein $-O_2C-QCO_2-$ represents the chlorendate residue:



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and wherein each R'' is independently selected from alkyl groups containing from 4 to 10 carbon atoms, and the tetrahydrofurfuryl group. Examples of said alkyl groups are butyl, pentyl, hexyl, heptyl, octyl, 2-ethylhexyl, nonyl, decyl, and the like. Alternatively, each R'' is butyl or 2-ethylhexyl.

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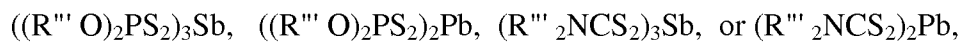
[0027] Chlorendate diesters are well-known materials; many are commercially available. The preferred chlorendate diesters are commercially available from, for example, Norquay Technology Chester PA.

[0028] Component (B) may be present in the composition in amounts from 0.9 to 60 parts based on 100 parts of components (A) plus (B) plus (C). Alternatively, Component (B) is

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present in amounts from 3 to 40 parts on the same basis. Alternatively, Component (B) is present in amounts from 3.7 to 30 parts on the same basis. Alternatively, Component (A) is present in amounts from 4.5 to 10 parts on the same basis.

5 [0029] Component (C) in the compositions of the present invention, is selected from the lead and antimony compounds of N,N-dialkyldithiocarbamates and the lead and antimony compounds of dialkyl phosphorodithioates. These compounds have the general formulae:



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wherein each R''' is independently selected from alkyl groups having from 3 to 10 carbon atoms. Examples of alkyl groups having from 3 to 10 carbon atoms include propyl, butyl, 2-ethylhexyl, pentyl, hexyl, heptyl, nonyl, decyl, and the like. Alternatively, each R''' is propyl or 2-ethylhexyl. Alternatively, each R''' is propyl.

15

[0030] The compounds that are used as component (C) in the compositions of the present invention are well-known materials in the lubricant art; many are commercially available. The preferred lubricant compounds are commercially available from, for example, the Vanderbilt Company of Norwalk, CT.

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[0031] Component (C) may be present in the composition in amounts from 0.1 to 38 parts based on 100 parts of components (A) plus (B) plus (C). Alternatively, Component (C) is present in amounts from 0.2 to 20 parts on the same basis. Alternatively, Component (B) is present in amounts from 0.3 to 15 parts on the same basis. Alternatively, Component (A) is present in amounts from 0.5 to 10 parts on the same basis.

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[0032] Although the present composition can include other ingredients, in certain embodiments a block copolymer consisting of, on average, at least one block of polydimethylsiloxane covalently bonded through one of its terminal units to a terminal unit of at least one block of polybutadiene or hydrogenated polybutadiene may not be present.

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[0033] The polydiorganosiloxane hydraulic fluid of the present invention are prepared by mixing appropriately selected quantities of components (A), (B), and (C) together. Mixing can be accomplished by heating said components together, such as heating said components together at a temperature of about 70°C, and holding said components together at that temperature for a period of time from about 1 minute to about 30 minutes in duration. Alternatively, mixing can be accomplished by agitation of said components together. For example, said components can be mixed together by means of a high shear mixer, such as an Eppenbach.RTM. mixer. Of course, mixing can be accomplished by heating and agitating. Agitation, with or without heating, is a preferred method for preparation of the polydiorganosiloxane hydraulic fluids of the present invention.

[0034] Small amounts of non-essential components, such as colorants, spray flammability resistance additives, fire retardants, and the viscosity control additives can be added to the polydiorganosiloxane hydraulic fluid compositions of the present invention. Examples of such non-essential additives include dyes, to make the hydraulic fluid more readily identifiable, and highly brominated compounds to reduce flammability.

[0035] The polydiorganosiloxane hydraulic fluid compositions of the present invention are stable hydraulic fluids of excellent lubricity. Accordingly, it is a further object of this invention to provide, in a process of transmitting power from one place to another place, the improvement which comprises using as the hydraulic fluid the composition comprising components (A), (B), and (C) as described above. Said process of transmitting power from one place to another place via a hydraulic fluid is accomplished through use of a hydraulic system.

[0036] While hydraulic systems vary from highly complex control systems to simple presses, they can be characterized as comprising 6 main elements in fluid communication: (1) an hydraulic fluid; (2) a reservoir in which to store said fluid; (3) means to generate pressure in said fluid, such as a pump or the like; (4) piping, to transmit the generated pressure through said fluid; (5) means to convert said pressure into power at a place removed from the place at

which the pressure was generated, such as an hydraulic motor, actuator, cylinder, ram, jack, or the like; and (6) pressure control means, such as control valves, relief valves and the like.

5 [0037] In order to utilize the hydraulic principle, of course, it is necessary for the fluid to be within an enclosed volume able to sustain elevated pressures. Some leakage of fluid is inevitable, and can be tolerated so long as elevated pressures can be sustained.

10 [0038] The polydiorganosiloxane hydraulic fluid compositions of the present invention can be used as the hydraulic fluid in a system for transmitting power from one place to another place as hereinabove described. Advantageously, the hydraulic fluid compositions of the present invention can be used as the hydraulic fluid in hydraulic systems wherein the fluid is exposed to extremes of temperature and high pressure.

15 [0039] The following examples are disclosed to further describe, and teach how to practice, the present invention. These examples are not to be construed as limiting the present invention, which is properly delineated by the appended claims. All parts and percentages are by weight unless otherwise stated. Viscosity values were measured in centistokes at 25°C., and converted to m^2/second by multiplying by $1.00 \times 10^{-6} \text{ m}^2/\text{second}/\text{centistoke}$, and rounding the result of said multiplication to three significant figures.

20 [0040] Test Procedures

[0041] Viscosity was determined by the general procedure set forth in ASTM D-445.

25 [0042] Lubricity: Lubricity was determined by the general procedure set forth in ASTM D-4172. Standard 1.27 cm (1/2 inch) AISI-E-52100 chrome alloy steel balls were thoroughly cleaned and placed in a Roxana Four Ball Tester with the appropriate amount of fluid to be tested. Testing conditions were: 1200 r.p.m., 40 kg load, 75°C. Testing was one hour in duration. Results of this testing are reported herein as average scar diameter, determined by microscopic examination of the balls at the conclusion of testing. Results are reported in mm, 30 and are reproducible within a range of approximately +/- 0.1%.

EXAMPLE 1

[0043] A hydraulic fluid of the present invention was prepared by thoroughly mixing 56 parts of dimethyl, phenylmethyl, trimethyl-terminated siloxane having a viscosity of 5.00×10^{-5} m²/second measured at 25°C, 37 parts dimethyl, trimethylsiloxy-terminated siloxane having a viscosity of 1.00×10^{-5} m²/second measured at 25°C, 5.7 parts di-2-ethylhexyl chlorendate (DEHC) and 0.65 parts antimony tris(o,o-dipropylphosphorodithioate). A low shear mixing apparatus such as a KitchenAide Mixer was utilized until the mixture acquired a uniform appearance and no settling was noted. The mixture exhibited an almost clear appearance. Components and amounts as well as viscosity and average wear scar diameters are displayed in Table 1.

TABLE I						
Composition, parts						
Example	Di-methyl silicone	Phenyl-methyl silicone	DEHC	Dithiophosphate	Viscosity, m ² /sec, ASTM D-445	Average scar diameter, mm ASTM D 4172
1	37	56	5.7	0.65	2.7×10^{-5}	1.36

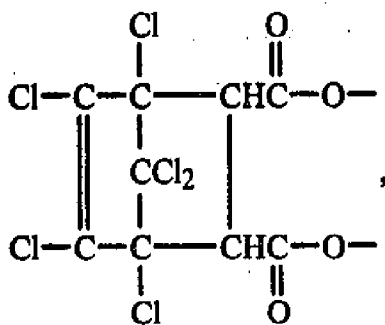
CLAIMS

1. A composition comprising:

5 (A) 2 to 99 parts of at least one polydiorganosiloxane selected from
 (A1) 0 to 99 parts of a polydiorganosiloxane having a formula
 $R'R_2Si-O(Me_2SiO)_wSiR_2R'$ having a viscosity of $1.00 \times 10^{-6} \text{ m}^2/\text{s}$ to
 $1.00 \times 10^{-4} \text{ m}^2/\text{second}$ at 25°C , and
 (A2) 0 to 99 parts having a formula $R'R_2Si-O(Me_2SiO)_x(MeR^aSiO)_ySiR_2R'$
 10 having a viscosity of $1.00 \times 10^{-5} \text{ m}^2/\text{s}$ to $1.00 \times 10^{-4} \text{ m}^2/\text{s}$ at 25°C ,
 wherein Me represents the methyl group, each R represents a monovalent group
 selected from hydrocarbon groups containing from 1 to 6 carbon atoms, and
 halogenated hydrocarbon groups containing from 1 to 6 carbon atoms, each R'
 represents a group selected from R groups, the hydride group, and the hydroxy group,
 15 w has an average value from 1 to 70, each R^a represents an aryl hydrocarbon group
 having from 6 to 10 carbon atoms, x has an average value from 5 to 50,
 and y has an average value from 0.5 to 6;

(B) 0.9 to 60 parts by weight of a chlorendate diester having the formula

20 $R''O_2CQCO_2R''$, wherein $-O_2C-QCO_2-$ represents the chlorendate residue,



each R'' represents a group selected from alkyl groups containing from 4 to 10
 carbon atoms and the tetrahydrofurfuryl group; and

25 (C) 0.1 to 38 parts by weight of a lubricant compound selected from N,N-
 dialkyldithiocarbamates of lead and antimony and dialkylphosphorodithioates of lead and
 antimony, the total parts of (A) plus (B) plus (C) being 100 parts by weight.

2. The composition of claim 1 wherein component (A) comprises at least two polydiorganosiloxanes selected from (A1) 1 to 99 parts of a polydiorganosiloxane having a formula $R'R_2Si-O(Me_2SiO)_wSiR_2R'$ and a viscosity of 1.00×10^{-6} m²/s to 1.00×10^{-4} m²/second at 25° C and (A2) 1 to 99 parts of a polydiorganosiloxane having a formula $R'R_2Si-O(Me_2SiO)_x(MeR^aSiO)_ySiR_2R'$, wherein Me represents the methyl group, each R represents a monovalent group selected from hydrocarbon groups containing from 1 to 6 carbon atoms, and halogenated hydrocarbon groups containing from 1 to 6 carbon atoms, each R' represents a group selected from R groups, the hydride group, and the hydroxy group, w has an average value from 1 to 70, each R^a represents an aryl hydrocarbon group having from 6 to 10 carbon atoms, x has an average value from 5 to 50, and y has an average value from 0.5 to 6
3. The composition of Claim 1 or 2 where R is methyl, R' is methyl, R^a is phenyl, R'' is 2-ethylhexyl, R''' is propyl, w has an average value from 3 to 35, x has an average value of from 11 to 37, y has an average value of 3 to 4, 30 to 40 parts of Component (A1) having a viscosity of 5.00×10^{-6} m²/second to 3.00×10^{-5} m²/second at 25° C, 50 to 60 parts of Component (A2) having a viscosity of 2.00×10^{-5} m²/second to 5.00×10^{-5} m²/second at 25° C, 4.5 to 10 parts of Component (B), 0.5 to 10 parts of Component (C) where component (C) is a dipropylphosphorodithioate.
4. In a process of transmitting power from one place to another place via a hydraulic fluid, the improvement which comprises using as the hydraulic fluid the composition of claim 1, 2 or 3.

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2011/037647

A. CLASSIFICATION OF SUBJECT MATTER
 INV. C10M169/04
 ADD.
 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)
 C10M

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

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
 EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	GB 1 535 265 A (DOW CORNING) 13 December 1978 (1978-12-13)	1,4
Y	claim 1; examples 1,2,5,6,7,8 -----	2,3
X	EP 0 108 369 A2 (DOW CORNING [US]) 16 May 1984 (1984-05-16)	1,4
Y	examples, claim 1 -----	2,3
Y	EP 0 805 185 A2 (DOW CORNING [US]) 5 November 1997 (1997-11-05) page 3, line 14 - line 56 -----	2,3
Y	GB 2 086 922 A (WACKER CHEMIE GMBH) 19 May 1982 (1982-05-19) claim 1 -----	2,3

Further documents are listed in the continuation of Box C.

See patent family annex.

* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
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