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(54) Silicone fluids for viscous couplings.

(57) An organopolysiloxane, containing a C1-18, optionally halogenated, hydrocarbon group, is used as the fluid in a viscous coupling, with as additive:

a) a phosphorus type anti-wear agent (27 formulae are given), e.g. a triaryl phosphate, optionally also with a sulfur or zinc dithiophosphate agent, each in amount of 0.01 - 5 wt %; or

b) a metal deactivator, e.g. benzotriazole or other heterocylic compound or an organic acid, and or a corrosion inhibitor, e.g. a stearate, each in amount of 0.001 to 1 wt %.

An antioxidant may also be added, e.g. an amine, in amount of 0.001 to 5 wt %.

The fluids are exemplified as used in a coupling having 111 metal discs, with 25 to 50 rpm difference in speeds of rotation, at 130° C for up to 100 hours; they have improved heat-resistance, and so suffer little change in viscosity or torque in use.

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SILICONE FLUIDS FOR VISCOUS COUPLINGS

The present invention relates to a fluid for use in viscous couplings and which has high durability.

In a fluid coupling (also called a viscous coupling), a plurality of inner plates movably disposed on a driving shaft and a plurality of outer plates fixed on a driven shaft with predetermined spacings are combined together alternately and are accommodated in a housing, which contains the viscous operating fluid for torque transmission. Under such arrangement, shearing force, i.e. shear torque, is generated in said groups of plates due to the difference of the revolutions between the driving shaft and the driven shaft, in order to transmit torque to the driven shaft.

In recent years, organopolysiloxane oils, such as dimethylpolysiloxane or methylphenylpolysiloxane, have been used as the hydraulic fluid or the operating fluid for fluid couplings.

A dimethylpolysiloxane (also called dimethyl-silicone oil) of high viscosity index has been widely used, but it is difficult to maintain stable torque transmission ability for a long period under severe operating condition at high temperature. This is mainly due to the low thermal stability of this fluid at high temperature. Because the operating conditions are becoming increasingly severe in various usages including in viscous couplings, it is desirable to improve the thermal stability of the silicone oil, which constitutes the main component of dimethyl-silicone.

To prevent oxidation or gelation, antioxidants, such as iron octanoate, phenylamine derivatives or ferrocene derivatives, have been added to an organopolysiloxane oil. But although a certain level of gelation preventive effect can be obtained at high temperature when these antioxidants are added, the viscosity increases when the viscous coupling is used continuously.

The object of this invention is to provide a fluid for viscous couplings, which has good resistance to thermal decomposition and gelation and has high stability.

In one embodiment a fluid for viscous couplings according to the present invention is characterized in that an organopolysiloxane is the base oil and a phosphorus type anti-wear agent is added to it.

In the conventional type of fluid for viscous couplings, the quality of antioxidants has been improved in order to prevent the thickening effect caused by thermal deterioration during the operation at high temperature. But when antioxidant is added to the fluid and it is used in a viscous coupling, the viscosity is still increased.

The present inventors have considered that this problem cannot be solved simply by the improvement of the effect of an antioxidant and have found that the metallic contact between disks of a viscous coupling exerts a very strong influence; it appears that the fresh surface of the metal disk exposed due to metallic contact acts to catalyse the deterioration of the organopolysiloxane and increases deterioration of the fluid.

By adding anti-wear agent to the fluid, a film of this agent is formed on the fresh metal surface of metal and the catalytic effect is thus prevented. This substantially prevents the thickening of the fluid.

With the fluid for viscous coupling according to the present invention, it is possible to increase the heat-resistant property of the fluid and to improve its durability by adding antioxidants together with the anti-wear agent.

It is desirable to also include in the aforesaid fluid for viscous coupling of this invention an anti-wear agent of the sulphur and/or zinc dithiophosphate type. A phosphorus, sulphur and zinc dithiophosphate type of anti-wear agent each has a certain effect when each is added alone to the fluid for viscous coupling. In this invention, however, an agent of the phosphorus, sulphur and/or zinc dithiophosphate type are admixed and blended together, and this gives a cumulative effect to form a film on the new metal surface and to suppress catalytic action thereby, thus almost completely eliminating the thickening of the fluid. This provides a better effect than when a phosphorus type agent is used alone.

The phosphorus type, sulphur type and zinc dithiophosphate type anti-wear agents give an adsorption effect on the metal in a specific temperature range according to their respective thermal stability. It appears that various friction and wear conditions occur in the viscous coupling itself during the operation and that the environmental temperature also widely differs. By this invention, the anti-wear agents with different absorption property are combined to cope with such conditions.

By optionally adding an antioxidant to the fluid in addition to these anti-wear agents, it is possible to increase the heat-resistant property and to improve the durability of the fluid.

In another embodiment of the fluid of this invention, an organopolysiloxane is again used as base oil and a metal deactivator and/or corrosion inhibitor is added.

Although a metal deactivator and/or corrosion inhibitor has lower solubility in the coupling fluid than the anti-wear agent, these substances can prevent increase of viscosity of the fluid when they are added in small quantity. This increases further the heat-resistance and improves the durability of the fluid. The combined use therewith of an anti-wear agent and/or antioxidants is also desirable, so as to increase the heat-resistance and durability of the fluid.

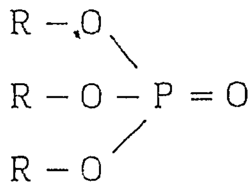
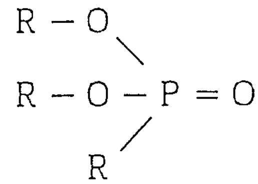
An organopolysiloxane, which is the base oil of the fluid according to this invention, has the following formula:

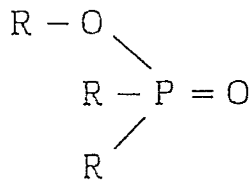
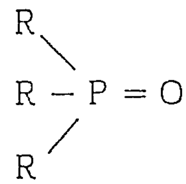
$$R-\overset{R}{\underset{R}{\overset{|}{\underset{|}{Si}}}}-O-(-\overset{R}{\underset{R}{\overset{|}{\underset{|}{Si}}}}-O-)_n-\overset{R}{\underset{R}{\overset{|}{\underset{|}{Si}}}}-R$$

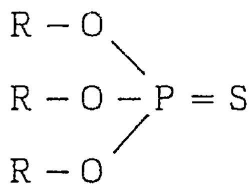
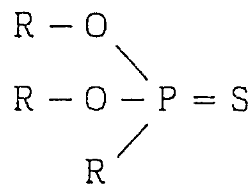
wherein R represents the same or different, optionally halogenated, hydrocarbon group having 1 - 18 carbon atoms, and n is an integral number of 1 - 300.

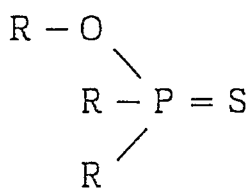
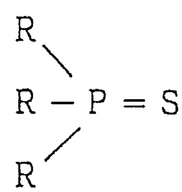
Examples of the group R are an alkyl group such as a methyl, ethyl, n-propyl, i-propyl, n-butyl, i-butyl, t- butyl, n-pentyl, neopentyl, hexyl, heptyl, octyl, decyl or octadecyl group, an aryl group such as a phenyl or naphthyl group, an aralkyl group such as a benzyl, 1-phenylethyl or 2-phenylethyl group, an alallyl group such as an o-,m-, p-diphenyl group, or a halogenated hydrocarbon group such as an o-, m-, p-chlorphenyl group, o-, m-, p-bromphenyl group, 3,3,3-trifluoropropyl group, 1,1,1,3,3,-3-hexafluoro-2-propyl group, heptafluorisopropyl group or heptafluor-n-propyl group. Particularly, it is preferable to use as R a fluorinated hydrocarbon group having 1 - 8 carbon atoms other than an aliphatic unsaturated group; or, a mixture of a methylpolysiloxane and phenylpolysiloxane.

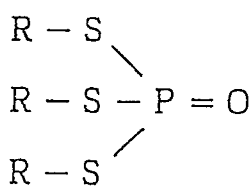
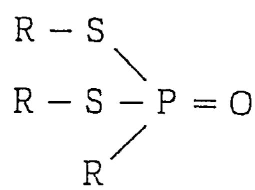
In the first embodiment, there is added to the organopolysiloxane one or more of a phosphorus type anti-wear agent. Examples thereof are those of one of the following formulae (1) - (27) wherein R may be hydrogen, or an alkyl, aryl or benzyl group. R may be the same or the different.

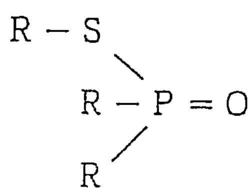
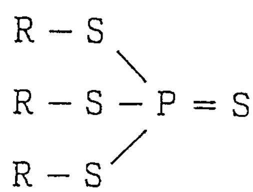
(1)  (2) 

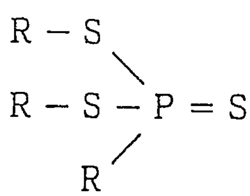
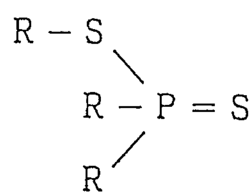
(3)  (4) 

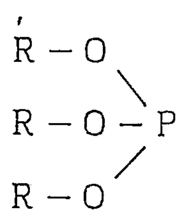
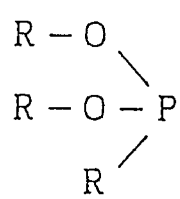
(5)  (6) 

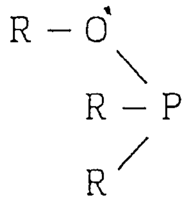
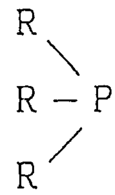
(7)  (8) 

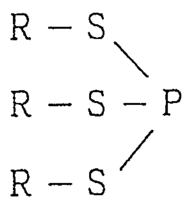
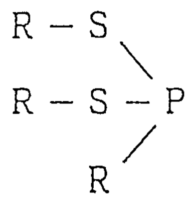
(9)  (10) 

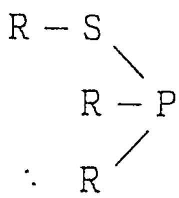
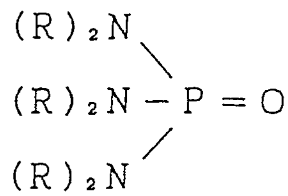
(11)  (12) 

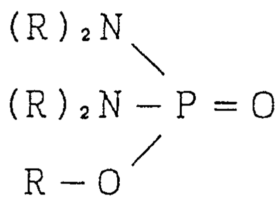
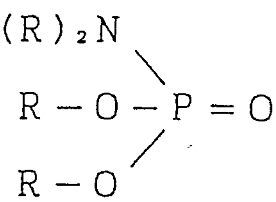
(13)  (14) 

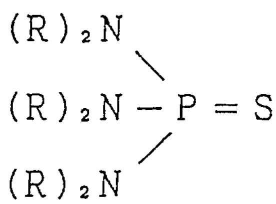
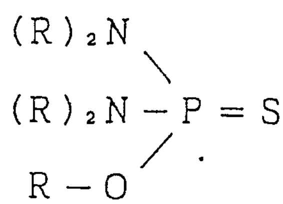
(15)  (16) 

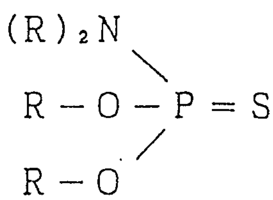
(17)  (18) 

(19)  (20) 

(21)  (22) 

(23)  (24) 

(25)  (26) 

(27) 

In the following, actual compounds are given for some of the formulae, namely:

Formula (1), triaryl phosphates, e.g. benzyldiphenyl phosphate, allydiphenylphosphate, triphenyl phosphate, tricresyl phosphate, ethyldiphenyl phosphate, tributyl phosphate, dibutyl phosphate, cresyldiphenyl phosphate, dicresylphenyl phosphate, ethylphenyldiphenyl phosphate, diethylphenylphenyl phosphate, propylphenyldiphenyl phosphate, dipropylphenylphenyl phosphate, triethylphenyl phosphate, tripropylphenyl phosphate, butylphenyldiphenyl phosphate, dibutylphenylphenyl phosphate, tributylphenyl phosphate, a propyl phenyl phenyl phosphate mixture, butyl phenyl phenyl phosphate mixture, or an acid phosphate such as lauryl acid phosphate, stearyl acid phosphate or di-2-ethylhexyl phosphate.

Formula (2), e.g. di-n-butylhexyl phosphate.

Formula (3), e.g. n-butyl-n-dioctyl phosphate.

Formula (5), triaryl phosphoro-thionates, e. g. triphenyl phosphoro-thionate and alkylaryl phosphoro-thionates

Formula (15), e.g. triisopropyl phosphite and diisopropyl phosphite.

Formula (19), e.g. trilauryl thiophosphite.

Formula (22), e.g. hexamethyl phosphoric triamide.

Formula (24), e.g. dibutyl phosphoroamidate.

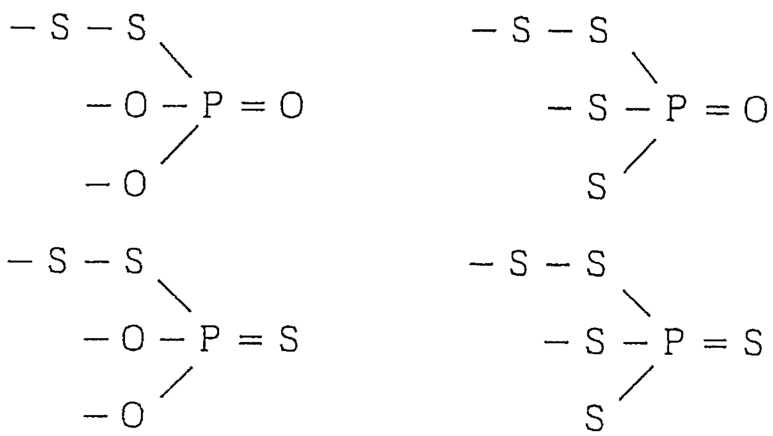
Among these compounds, the effects are particularly conspicuous in the cases of the compounds with excellent thermal stability having the structure of triaryl phosphate or triaryl phosphoro-thionate.

Examples of the sulfur type and/or zinc dithiophosphate type anti-wear agent which may be added now follow.

As the sulfur type of anti-wear agent, a sulfide such as diphenyl sulfide, diphenyl disulfide, dibenzyl disulfide, di-n-butyl sulfide, di-n-butyl disulfide, di-tert-butyl disulfide, di-tert-dodecyl sulfide or di-tert-dodecyl trisulfide, a sulfurized oil such as sulfurized sperm oil or sulfurized dipentene, or a thiocarbonate such as xanthic disulfide; and as a zinc dithiophosphate anti-wear agent a primary alkyl zinc dithiophosphate, secondary alkyl zinc dithiophosphate, alkyl-aryl zinc dithiophosphate or aryl zinc dithiophosphate.

It is preferable to use each type of anti-wear agent (phosphorus type, sulfur type and/or zinc dithiophosphate) in an amount of 0.01 - 5 wt% of the organopolysiloxane, more preferably 0.1 - 3 wt%. The ratio of phosphorus type anti-wear agent to total anti-wear agents is preferably 5-95 wt%.

Instead of adding a mixture of phosphorus type and sulfur type anti-wear agents, a compound may be used containing both phosphorus and sulfur, such as having at least one of the following formulae:



e.g. benzyl (di-n-pentyl phosphoryl) bisulfide.

It is preferable to use these agents in amount of 0.01 - 5 wt % to the organopolysiloxane, and more preferably, 0.1 - 3 wt %.

In another embodiment of the fluid for viscous coupling of this invention, a metal deactivator and/or corrosion inhibitor is added to the organopolysiloxane, alone or together with one or more of the above anti-wear agents.

As the metal deactivator, benzotriazole, benzothiazole, thiadiazole, triazole, dithiocarbamate, indazole, or derivatives of any of these, or organic carboxylic acids including dibasic acids such as adipic acid, sebacic acid or dodecane dioic acid, or monobasic acids such as stearic acid, oleic acid or lauric acid, or amine salts of these compounds may be used.

As suitable corrosion inhibitors, there are isostearate, n-octadecylammonium stearate, a long chain aliphatic diamine e.g. DUOMEEN-T diorate, lead naphthenate, sorbitan oleate, pentaerythrite oleate, oleyl sarcosine, alkyl succinic acid, alkenyl succinic acid, and derivatives of these compounds.

Prefered amounts of the metal deactivator and corrosion inhibitors are each 0.001 - 1.0 wt % of the organopolysiloxane, more preferably 0.01 - 0.5 wt %. When the added quantity exceeds 1.0 wt %, it is not desirable because precipitation increases. If it is less than 0.001 wt %, there is no effect.

The durability of the fluid can be increased by adding antioxidant when an phosphorus anti-wear agent is added alone, or when a phosphorus type anti-wear agent and sulfur type anti-wear agent and/or zinc dithiophosphate type anti-wear agent are together added, or when a metal deactivator and/or corrosion inhibitor is added alone or together with the above anti-wear agents.

Suitable antioxidants are amine type antioxidants such as dioctyldiphenylamine, phenyl-α-naphthylamine, alkyldiphenylamine, N-nitrosodiphenylamine, phenothiazine, N,N′-dinaphthyl-p-phenylenediamine, acridine, N-methylphenothiazine, N-ethylphenothiazine, dipyrizylamine and diphenylamine, phenol type antioxidants such as 2,6-di-t-butylparacresol, 4,4′-methylenebis (2,6-di-t-butylphenol) and 2,6-di-t-butylphenol, or organic metal compound type antioxidants such as an organic iron salt such as iron octoate, ferrocene or iron naphthoate, an organic cerium salt such as cerium naphthoate or cerium toluate, and an organic zirconium salt such as including zirconium octoate. The above antioxidants may be used alone or in a combination of two or more compounds to provide cumulative effects.

Preferred amounts of the antioxidants are 0.001 -5 wt % of the organopolysiloxane, more preferably 0.01 - 2 wt %.

The invention will now be illustrated by reference to specific examples. Viscosities were measured at 25° C.

Example 1

To dimethylisilicone (viscosity 50,000 mm^2/s) tricresyl phosphate was added in the percentages shown below (none in control samples) as phosphorus type anti-wear agent; some samples (a) also contained 1.0 wt % of diphenylamine as antioxidant. Each fluid for viscous coupling thus prepared was filled into a viscous coupling having 111 disks at 25° C and at a filling degree of 85 vol %. The difference in speeds of rotation was 50 rpm.

The viscous coupling was placed in a bath kept at a constant temperature of 130° C and was operated for 50 hours.

After the operation, the viscosity change and torque change were measured; the results are given in the table below

To evaluate the heat-resistant property of the anti-wear agent, a hot tube coking test was performed, and the temperature at which the specimen was gelated or blocked by coking in the glass tube was measured at every 10° C. The lowest temperature is also shown in the table below.

Added quantity of anti-wear agent (wt %)	Viscosity change (%)	Torque change (%)	Blocking temperature (° C)
(a) with diphenylamine antioxidant			
2.0	-5	-5	330
1.0	+1	0	330
0.5	+5	+5	330
0	Measurement not achievable	Measurement not achievable*	330
(b) without antioxidant			
2.0	-3	-3	290
1.0	+1	+1	290
0.5	+6	+5	290
0	Measurement not achievable	Measurement not achievable	290

Example 2

Example 1 was repeated, but with various amounts of tricresyl phosphate (A) and triphenyl phosphorothionate (B) as phosphorus type anti-wear agents in the weight percentage shown below. The four fluids thus prepared were tested as in Example 1; the results are given in the table below:

(A) Added quantity	(B) Added quantity	Viscosity change (%)	Torque change (%)	Blocking temperature (°C)
a) with antioxidant in the fluid				
0	1.0	+2	+1	330
0.5	0.5	+1	0	330
b) without antioxidant				
0	1.0	+1	+1	290
0.5	0.5	+3	+3	290

Examples 1 and 2 show that satisfactory fluids can be prepared even when antioxidant is not added.

Comparative example1

To dimethylsilicone (viscosity 50000 mm^2/s) 1.0 wt % of diphenylamine was added as antioxidant (in samples (a)) and dibenzyl disulfide was added as sulfur type anti-wear agent in the percentage shown below. The fluids thus prepared were tested by as in the Embodiment 1 and the results were:

Added quantity of anti-wear agent (wt %)	Viscosity change(%)	Torque change(%)	Blocking temperature (°C)
(a) with antioxidant			
1.0	-2	-2	300*
0.5	+5	+7	310*
0	Measurement not achievable	Measurement not achievable	320
(b) without antioxidant			
2.0	-3	-4	250*
1.0	+1	0	250*
0.5	+7	+7	260*
0	Measurement not achievable	measurement not achievable	290

* Accompanied by coking.

As is evident from this comparative example, both sulfur type and phosphorus type agents have almost the same torque stability as anti-wear agents in the fluid. However, because the heat-resistance of the additive itself is inferior to that of the organopolysiloxane, the coking phenomenon occurs, in which black decomposed products of the additive are generated in the hot tube coking test, and the thermal stability of the fluid is thus reduced by the addition of such agent.

Examples 3 to 5

To dimethylsilicone (viscosity 100,000mm^2/s) one of three specified phosphates was added as phosphorus type anti-wear agent in the percentages given below (none for a control sample). Each fluid for viscous coupling thus prepared was used in a coupling as in Example 1 but the rotating speed difference was 25 rpm and the bath temperature was 170 °C, with the following results:

Ex No	Agent	Added quantity of anti-wear agent (wt %)	Viscosity change(%)	Torque change(%)
3	tricresyl phosphate	2.0	-6	-7
		1.0	-3	-3
		0.5	0	-1
	None	0	Measurement not achievable	Measurement not achievable
4	triphenyl phosphate	2.0	-5	-5
		1.0	0	0
		0.5	+2	+1
5	triphenyl phosphorothionate	2.0	-7	-7
		1.0	-5	-3
		0.5	0	+1

Comparative Examples 2 and 3

Example 3 was repeated but using dibenzyl disulfide and a polysulfide respectively as sulfur type anti-wear agents instead of the phosphorus type anti-wear agent tricresyl phosphate. The results are:

Cp. Ex. No	Agent	Addeded quantity of anti-wear agent (wt %)	Viscosity change (%)	Torque change (%)
2	Dibenzyl disulfide	2.0	-20	-35
		1.0	-10	-22
		0.5	-8	-17
	None	0	Measurement not achievable	Measurement not achievable
3	polysulfide	2.0	-22	-25
		1.0	-15	-20
		0.5	-10	-12

It is evident from these comparative examples that both phosphorus type and sulfur type agents exhibit excellent durability in viscosity change and torque change of the fluid for viscous coupling when the temperature is relatively low as in the Examples 1 and 2 and Comparative Example 1, whereas the phosphorus type shows the higher durability at high temperature. This is attributable to the fact that, because a sulfur type anti-wear agent was a lower heat-resistant property, the reaction with dimethylsilicone or with the plates in the viscous coupling proceeded excessively at high temperature, which the phosphorus type anti-wear agent was higher heat-resistant property.

As for the odor of the coupling fluids that of Example 3 is odorless and did not have a strong sulfur odor as did the fluids of the comparative examples. A phosphorus type of anti-wear agent is thus more convenient in use than a sulfur type anti-wear agent.

Example 6 and Comparative Examples 4 and 5

To dimethylsilicone (viscosity 50,000 mm^2/s) there was added a phosphorus type anti-wear agent according to the invention or one of two sulfur type anti-wear agents (for comparison) each in the percentages below. Each of the six fluids thus prepared was filled into an autoclave at 25 °C at a filling degree of 80 vol %. After replacing the remaining air with nitrogen, it was placed at 200 °C in a thermostat for 24 hours.

After the test, the viscosity change was measured, and the results were:

Ex No	Agent	Added quantity of anti-wear agent (wt%)	Viscosity change (%)
6	triphenyl phosphate	2.0	-1
		1.0	±0
Comp 4	dibenzyl disulfide	2.0	-27
		1.0	-18
Comp 5	a polysulfide	2.0	-17
		1.0	-12

From the above comparative Examples 4 - 5, it is seen that the sulfur type anti-wear agents having lower heat-resistant property were deteriorated, and this apparently induced the viscosity decrease and the deterioration of the dimethylsilcone.

By contrast, the phosphorus type agent in Example 6 was stable to dimethylsilicone and this may be attributed to the high heat-resistant property of thus agent.

Example 7

To dimethylsilicone (viscosity 100,000 mm^2/s) of tricresyl phosphate (phosphorus type) and dibenzyl disulfide (sulfur type) were added together and separately (for comparison) in the percentages below as anti-wear agents. In a series (a) of samples, 1.0 % of diphenylamine was also added as antioxidant. The fluid for viscous coupling thus prepared was filled into a viscous coupling having 111 iron disks at 25 °C and at a filling degree of 85 vol %. The rotating speed difference was 35 rpm.

The viscous coupling was maintained in a bath kept at constant temperature of 130 °C and was operated for 100 hours.

After the operation, viscosity change and torque change were measured. The results are given in the table below together with the results of the iron quantity, measured as wear fragment quantity.

Added quantity of phosphorus type (wt %)	Added quantity of sulfur type (wt %)	Viscosity change (%)	Torque change (%)	Wear fragment iron (ppm)
(a) with antioxidant				
0.5	0	+5	+5	450
0	0.5	+7	+5	480
0.25	0.25	+1	0	120
(b) without antioxidant				
0.5	0	+5	+5	470
0	0.5	+5	+5	430
0.25	0.25	+1	+1	130

It is evident from the results that the viscosity change and torque change as well as amount of wear fragment iron are increased more in the case where two anti-wear agents are simultaneously added to the fluid for viscous coupling than when only one of the anti-wear agents is added: and that the results are simular even without antioxidant

In a coupling fluid to which both of the above phosphorus type and sulfur type anti-wear agents were added, 0.20 wt % di-sec-butyl zinc dithiophosphate (zinc dithiophosphate type) was added. The fluid thus prepared was tested by the same procedure as above. As the result, the viscosity change was +1%, torque change was 0%, and wear fragment iron quantity was 140 ppm. Thus, an excellent fluid for viscous coupling can be obtained by combining phosphorus type, sulfur type and zinc dithiophosphate type anti-wear agents.

Example 8

In the specimen of Example 7, sulfurized sperm oil was added in the percentages given below as sulfur type anti-wear agent instead of dibenzyl disulfide (sulfur type) anti-wear agent. The fluid thus prepared was tested as in Example 7 and the results were:

Added quantity of phosphorus type (wt %)	Added quantity of sulfur type (wt %)	Viscosity change (%)	Torque change (%)	Wear fragment iron (ppm)
0	0.5	+5	+7	450
0.25	0.25	+3	+3	200

Also when a sulfurized olefin was used instead of the sulfurized sperm oil, similar results were obtained.

Example 9

In the specimen of Example 7, an aminedibutyl phosphonate (phosphorus type agent) anti-wear agent was added in the percentages given below instead of tricresyl phosphate (phosphorus type) anti-wear agent. The fluid was tested as in Example 7, and the results were:

Added quantity of phosphorus type (wt %)	Added quantity of sulfur type (wt %)	Viscosity change (%)	Torque change (%)	Wear fragment iron (ppm)
0.5	0	+7	+5	450
0.25	0.25	+1	+1	200

Example 10

In the specimen of Example 7, di-sec-butyl zinc dithiophosphate (zinc dithiophosphate type amount) was added in the percentages below instead of dibenzyl disulfide (sulfur type). The fluid thus prepared was tested as in Example 7, and the results were:

Added quantity of phosphorus typ (wt %)	Added quantity of zinc thiophosphate (wt %)	Viscosity change (%)	Torque change (%)	Wear fragment iron (ppm)
0	0.5	+8	+7	350
0.25	0.25	+3	+3	250

Example 11

In the fluid of Example 7, triphenyl phosphorothionate (phosphorus type) anti-wear agent was added in the percentage as given below instead of tricresyl phosphate (phosphorus type) anti-wear agent. The fluid prepared was tested as in Example 7, and the results were:

Added quantity of phosphorus type (wt %)	Added quantity of sulfur type (wt %)	Viscosity change (%)	Torque change (%)	Wear fragment iron (ppm)
0.5	0	+5	+3	350
0.25	0.25	+1	0	130

Example 12

To dimethylsilicone (viscosity 50,000 mm²/s) 0.5 wt % of phenyl-α-naphthylamine was added as antioxidant and benzothiazole was added as metal deactivator, and triphenyl phosphate as anti-wear agent in the percentages given below. The fluid thus prepared was filled into a viscous coupling having 111 disks at 25° C and at a filling degree of 85 vol %. The rotating speed difference was 50 rpm.

The viscous coupling was placed in a bath at constant temperature of 130° C and was operated for 100 hours. After the operation, viscosity change and torque change were measured; the results were:

Anti-wear agent (wt %)	Metal deactivator (wt %)	Viscosity change (%)	Torque change (%)
0	0	Measurement not achievable*	Measurement not achievable*
0	0.1	+10	+10
0	0.4	+8	+7
0	0.8	+5	+5
0.5	0.1	+2	+2

* Stopped before the expiration of 100 hours due to sudden increase of torque.

Example 13

To dimethylsilicone (viscosity 50,000 mm²/s) 1.0 wt % of diphenylamine was added as anti-oxidant, benzotriazole was added as metal deactivator, and tricresyl phosphate was added as anti-wear agent, and in the percentages given below in one series (a) of samples 1.0 wt % of diphenylamine was added as antioxidant. Each fluid was tested as in Example 12 with the results:

Anti-wear agent (wt %)	Metal deactivator (wt %)	Viscosity change (%)	Torque change (%)
(a) with antioxidant			
0	0	Measurement not achievable*	Measurement not achievable*
0	0.1	+8%	+8%
0	0.4	+5%	+5%
0	0.8	+3%	+3%
0.5	0.1	±0	±0
(b) without antioxidant			
0	0	Measurement not achievable	Measurement not achievable
0	0.1	+10%	+10%
0	0.4	+7%	+5%
0	0.8	+5%	+4%
0.5	0.1	+2%	±0%

* Measurement stopped before the expiration of 100 hours due to sudden increase in torque.

Example 14

In each specimen of Example 13, a corrosion inhibitor n-octadecyl ammonium stearate was added in the percentage given below instead of the metal deactivator. Each fluid sample was tested as in Example 13, and the results are below. In the table, the added quantity of the anti-wear agent is not given.

Added quantity of corrosion inhibitor (wt%)	Viscosity change (%)	Torque change (%)
0	Measurement not achievable	Measurement not achievable
0.1	+12%	+12%
0.4	+8%	+10%
0.8	+4%	+5%
0.1	+3%	+3%
(b) without antioxidant		
0	Measurement not achievable	Measurement not achievable
0.1	+14%	+14%
0.4	+10%	+10%
0.8	+5%	+6%
0.1	+3%	+3%

Example 15

Example 13, was repeated but the amount of the metal deactivator was 0.1 wt % and the corrosion inhibitor 0.2 wt %. The viscosity change was ±0%, and torque change was +3%.

Claims

1. A fluid for viscous coupling, comprising an organopolysiloxane as base oil and a phosphorus type anti-wear agent as additive.
2. A fluid as claimed in Claim 1, which also contains a sulfur type anti-wear agent and/or zinc dithiophosphate type anti-wear agent.
3. A fluid for viscous coupling, comprising an organopolysiloxane as base oil and a metal deactivator as additive.
4. A fluid for viscous coupling, comprising an organopolsiloxane as base oil and a corrosion inhibitor as additive.
5. A fluid as claimed in Claim 4, which also contains a metal deactivator.
6. A fluid as claimed in Claim 3,4 or 5, which also contains an anti-wear agent.
7. A fluid as claimed in Claim 6, wherein said anti-wear agent is as defined in Claim 1 or 2.
8. A fluid as claimed in any preceding claim which also contains an antioxidant.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 90 30 5069

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim
Y	GB-A-2 206 887 (COSMO OIL) * Claim 1; page 2, line 25 - page 3, line 14 *	1-8
Y	FR-A-2 455 079 (OLIN CORP.) * Page 2, lines 8-19; page 12, lines 1-15; page 9, line 22 - page 11, line 35 *	1-8
X	US-A-3 532 730 (A.L. CULPEPPER) * Column 3, line 71 - column 4, line 9 *	1,4,6-8
X	US-A-4 744 915 (UNION CARBIDE) * Column 2, lines 62-68 *	4
X	GB-A- 695 308 (DOW CORNING) * Page 1, lines 53-85; page 3, lines 17-27 *	3-6
A	GB-A-1 296 163 (DOW CORNING) * Page 2, lines 46-100; page 1, lines 49-57 *	2,6,7
A	GB-A-2 203 750 (DOW CORNING) * Page 5, lines 25-30 *	8

CLASSIFICATION OF THE APPLICATION (Int. Cl.5)

C 10 M 169/04 //
C 10 N 40/04
(C 10 M 169/04
C 10 M 107:50
C 10 M 133:04
C 10 M 133:12
C 10 M 135:28
C 10 M 135:36
C 10 M 137:00)

TECHNICAL FIELDS SEARCHED (Int. Cl.5)

C 10 M

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	06-08-1990	HILGENGA K.J.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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