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(54) **Siloxane-polyalphaolefin hydraulic fluid.**

(57) There is provided a homogeneous blend of a dimethylsiloxane/ alkylmethylsiloxane copolymer with a hydrogenated polyalphaolefin based on oligomers of decene-1. The blend serves as a base fluid which has a viscosity-temperature profile and flash point suitable for use in a -54°C. to 135°C. fire-resistant hydraulic fluid having particular utility in military aircraft hydraulic systems.

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SILOXANE-POLYALPHAOLEFIN HYDRAULIC FLUID

The present invention relates to hydraulic fluid compositions which may be used over a wide temperature range and yet exhibit relatively high flash points. More particularly, the fluids of this invention consist essentially of a dimethylsiloxane/alkylmethylsiloxane copolymer blended with a synthetic hydrogenated polyalphaolefin. This invention further relates to a method of employing said compositions in a hydraulic system.

Certain silicone fluids are known for their superior resistance to shear, thermal and oxidative degradation. Moreover, unlike typical hydrocarbon lubricating oils or hydraulic fluid, they maintain a relatively flat viscosity-temperature profile. On the other hand, these silicones generally do not possess the high degree of lubricity in steel-to-steel contact that is exemplified by hydrocarbons such as mineral oil, a property vital in many hydraulic applications. This problem can be resolved to some extent by the incorporation of certain lubricity additives in the silicone "base fluid."

In an effort to simultaneously capitalize on the key advantages of both of these fluid types, several workers have combined them in various lubricant compositions. Thus. for example, in GB-A-1,224,885, Mackenzie discloses compositions comprising mineral oil and, as a viscosity index improver therefor, a homopolymeric diorganopolysiloxane in which the majority of the organo groups are methyl groups. In the siloxane polymers of these compositions, the remaining organo groups may be selected from alkyl, alkaryl or aralkyl groups having from 6 to 30 carbon atoms in amount sufficient to render them soluble in the mineral oil.

Morro et al., in US-A-4,059,534, teach a lubricating oil composition comprising 1% to 50% by weight of a polydimethylsiloxane having a viscosity of 100,000 to 1,000,000 cS at 100°F., the remainder of the mixture being a hydrocarbon oil having a viscosity of less than 40,000 cS at -65°F. which may be selected from alkenes, isoparaffins and naphthenics. A major contribution to the art asserted by this disclosure is the dissolution of high viscosity polydimethylsiloxane in the hydrocarbon oil to provide a lubricating composition having improved viscosity-temperature properties in which the siloxane component does not separate from the mixture at low temperatures.

EP-A-0 177 825 , published April 16, 1986, describes lubricating oil preparations comprising silicone copolymers containing organomethylsiloxane units wherein the organo group is selected from alkyl groups having 1 to 7 carbon atoms or the phenyl radical and alkylmethylsiloxane units wherein the alkyl group can have from 6 to 16 carbon atoms.

These copolymers have the further restrictions in that they must have a pour point below -15°C. and must be miscible with mineral oil, synthetic lubricating oils and the lubricity additives commonly employed in such oils.

More recently, the need for a fire-resistant military aircraft hydraulic fluid having a wide operating temperature range was documented by Gschwender et al. in Lubrication Engineering, 42, 485-490 (1986). On the one hand. it was observed that although mineral oil-based hydraulic fluid (MIL-H-5606) has been extensively used in military aircraft and is considered operational down to -54°C., this fluid suffers from two major drawbacks. A relatively low flash point of approximately 100 - 110°C. makes this fluid dangerously susceptible to facile ignition and high rate of propagation once a fire starts. Additionally, this fluid has a less than desirable shear stability, particularly in view of design trends toward higher hydraulic pressures (and associated higher shear rates) in modern Air Force aircraft. On the other hand, experience with synthetic hydrogenated polyalphaolefin fluids (MIL-H-83282) has been good in that these fluids have high (>205°C.) flash points and are relatively shear stable. Unfortunately, hydraulic fluids based on such polyalphaolefins (hereinafter PAO) either exhibit too high a viscosity at low temperatures for use in arctic operation and are thus generally only serviceable down to about -46°C. or they have too low a flash point (i.e., when low molecular weight PAO is used).

Thus, Gschwender et al. present a list of the target properties for a so-called "Low-Temperature MIL-H-83282" fluid which combines aspects of both MIL specifications and includes viscosity limitations as follows:

Temperature (°C.)	Viscosity (cS)
-54	2500 (maximum)
-40	500 (maximum)
100	3.5 (minimum)
135	1.5 (minimum)

It was further recognized that the high flash point of the MIL-H-83282 fluid would not be compatible with the above low temperature viscosities and compromise target values of at least 163°C. and 191°C. were set for the flash point and fire point, respectively. Additionally, a major requirement of a successful candidate fluid was that this fluid be compatible with the existing military fluids (i.e., MIL-H5606 and MIL-H-83282) so as to make "drain-and-fill" replacement in current hydraulic systems possible.

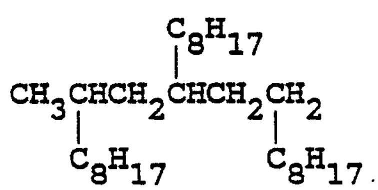
None of the above patent art disclosures described above teach a mixture of a dimethylsiloxane/alkylmethylsiloxane copolymer with a polyalphaolefin to form a base fluid composition which combines the viscosity-temperature profile and flash point required of a candidate suitable for use in a -54°C. to 135°C. fire-resistant hydraulic fluid.

It has now been discovered that the above-described low temperature viscosities can be achieved by blending certain dimethylsiloxane/alkylmethylsiloxane copolymers with hydrogenated polyalphaolefins (PAO) based on oligomers of decene-1. Unexpectedly, this can be accomplished while still maintaining a flash point of at least 163°C. as well as other desirable features of both the dimethylsiloxane/alkylmethylsiloxane copolymer and PAO fluids for use in hydraulic applications. Moreover, the blends so formed are compatible with the existing military fluids (i.e., MIL-H5606 and MIL-H-83282) so that "drain-and-fill" replacement in current hydraulic systems is possible.

This invention, therefore, relates to a homogeneous hydraulic fluid composition, consisting essentially of: (A) about 40 to 90 parts by weight of a dimethylsiloxane/alkylmethylsiloxane copolymer represented by the average formula

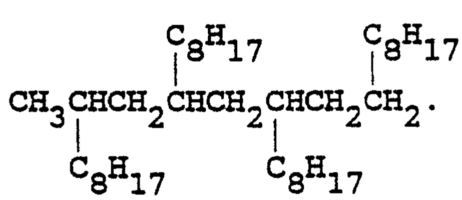
$$(R')_3SiO(Me_2SiO)_x(\underset{R}{MeSiO})_ySi(R')_3$$

wherein Me denotes the methyl group, R′ is independently selected from alkyl radicals having 1 to 10 carbon atoms, R denotes an alkyl group having from 4 to 10 carbon atoms, x has an average value of about 5 to 15. y has a value of about 0.5 to 4 and the ratio y/x is at most 0.3; and (B) about 10 to 60 parts by weight of a synthetic hydrogenated polyalphaolefin represented by the formula



said blend having a total of 100 parts by weight of said components (A) and (B).

This invention further relates to similar compositions wherein component (B) is represented by the formula



This invention still further relates to similar compositions wherein component (B) is selected from the dimer, trimer or tetramer of decene-1, and said homogeneous hydraulic fluid has a viscosity of at most

2500 cS at -54°C., a viscosity of at least 3.5 cS at 100°C. and an open cup flash point of at least 163°C.

This invention also relates to a process for transmitting force in a hydraulic system, the improvement comprising using the above-described compositions as a hydraulic fluid in said system.

The present invention relates to hydraulic fluid compositions consisting essentially of a homogeneous blend of (A) a dimethylsiloxane/alkylmethylsiloxane copolymer and (B) a synthetic hydrogenated polyalphaolefin.

Component (A) of the present invention is a copolymer which may be represented by the average formula

$$(R')_3SiO(Me_2SiO)_x(\underset{R}{MeSiO})_ySi(R')_3,$$

wherein Me denotes the methyl radical and R is a linear or branched alkyl group having 4 to 10 carbon atoms, such as n-pentyl, isopentyl, n-hexyl, n-heptyl, n-octyl, diisobutyl, n-nonyl or n-decyl. Straight chain alkyl groups are preferred over branched structures. It is further preferred that R is selected from the n-hexyl or n-octyl radicals. The R' groups on the terminal silicon atoms are identical or different short chain alkyl groups having from one to 10 carbon atoms, such as methyl, ethyl, propyl, butyl, hexyl, octyl or decyl, with methyl being preferred.

The values of x in the above formula may range from about 5 to 15, preferably from 6 to 12. The value of y may correspondingly range from about 0.5 to 4, preferably from 0.5 to 3. The value of y is, however, restricted such that the ratio y/x is at most 0.3. It is preferred that, in the above general formula, x and y are chosen such that the viscosity of the copolymer fluid ranges from about 5 to 50 centistokes (cS) at 25°C. Preferably. the viscosity is about 10 - 20 cS. Highly preferred embodiments of the invention result when R is n-hexyl, R' is methyl, x is about 6 and y is about 1 or when R is n-octyl, R' is methyl, x is about 8 and y is about 0.5.

The copolymers of the present invention may be prepared by methods which are well known in the art. For example, they may be synthesized by the cohydrolysis, and subsequent condensation, of the appropriate diorganodialkoxysilanes or diorganodichlorosilanes. In this approach, a desired quantity of an end-capping reactant, $(R')^3SiZ$, is included in the reaction mixture to regulate the molecular weight of the product (i.e., more end-capping reactant yields a lower molecular weight product). The R' groups of said end-capping reactant are the same as those set forth above for component (A). The Z group is a hydrolyzable species such as halide (e.g., chlorine, fluorine or bromine) or alkoxy (e.g., methoxy, ethoxy).

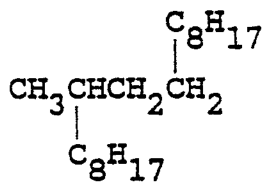
Another procedure which may be followed in the synthesis of the subject copolymers is the acid or base catalyzed equilibration of diorganocyclosiloxanes in the presence of the aforementioned end-capping reactant.

Yet another, and preferred, method comprises the steps of preparing an SiH-functional copolymer intermediate of the general formula

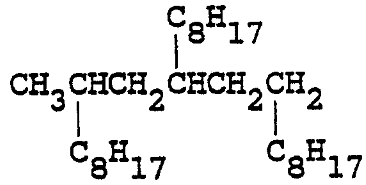
$$(R')_3SiO(Me_2SiO)_x(\underset{H}{MeSiO})_ySi(R')_3,$$

wherein Me represents the methyl radical and R' has been previously defined. These intermediates are well known in the art and may be prepared by the acid equilibration of a commercially available polymethylhydrogensiloxane with cyclic dimethylsiloxanes in various ratios. Molecular weight of the intermediate may be controlled by incorporating the proper level of an endcapping monomer, such as hexamethyldisiloxane. The SiH-functional intermediate is then reacted with about a 25% excess of the desired alkene to chemically add the latter to the SiH groups of the former. This addition reaction is catalyzed by platinum complexes known in the art.

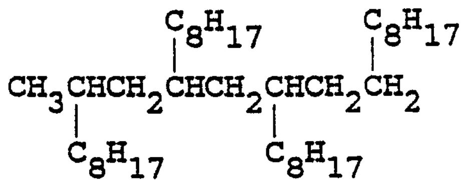
Component (B) of the present invention is at least one synthetic hydrogenated polyalphaolefin (PAO) selected from the dimer, trimer or tetramer of decene-1, which may be represented by the following formulae:



(dimer)



(trimer)



(tetramer).

In the preferred embodiment of this invention. component (B) is the PAO trimer. It is, however, possible to achieve the high flash point and viscosity-temperature profile needed to meet the requirements of the Low-Temperature MIL-H-83282 target by blending component (A) with mixtures of the above dimer, trimer and tetramer. as may be determined by routine experimentation.

The PAO fluids are well known in the art and can be prepared from the monomer decene-1 by catalytic polymerization and hydrogenation. The interested reader is directed, for example, to methods outlined by Cupples et al. in the October 1981 government report number AFWAL-TR-81-4109 (Materials Laboratory, Air Force Wright Aeronautical Laboratories, Wright-Patterson Air Force Base). These fluids are commercially available from the Gulf Oil Chemicals Co., Houston, Texas, under the trade name SYNFLUID.

The compositions of the present invention may be prepared by simply blending components (A) and (B) to form a homogeneous mixture therebetween. The method of mixing is not critical and any of the commonly employed methods known in the art may be used. The blend so formed is referred to as a "base fluid" in that it is ordinarily combined with lubricity additives and other adjuvants, described infra, to form a fully formulated hydraulic fluid.

When component (B) is the decene trimer, from about 40 to 90 parts by weight of component (A) is blended with about 60 to 10 parts of component (B) to form 100 parts of the compositions of the present invention. It is preferred, however, that from about 40 to 60 parts by weight of component (A) is blended with about 60 to 40 parts of the decene trimer. Most preferably, equal weights of the decene trimer and the dimethylsiloxane/alkylmethylsiloxane copolymer fluid of component (A) are employed wherein the dimethylsiloxane/alkylmethylsiloxane copolymer component is delineated by the preferred ranges described above.

When component (B) is the decene tetramer, from about 80 to 90 parts by weight of component (A) is blended with about 20 to 10 parts of component (B) to form 100 parts of the compositions of the present invention.

Alternatively, the compositions of the present invention may be defined in terms of the key physical properties required by the Low-Temperature MIL-H-83282 target. In this case, 100 parts by weight of the blend consists of about 10 to 90 parts by weight of component (A) and about 90 to 10 parts by weight of at least one synthetic hydrogenated polyalphaolefin selected from the dimer, trimer or tetramer of decene-1, such that the homogeneous blend has a viscosity of at most 2500 cS at -54°C., a viscosity of at least 3.5 cS at 100°C. and an open cup flash point of at least 163°C. Preferably, about 40 to 60 parts by weight of component (A) is blended with about 60 to 40 parts of component (B). These properties may readily be obtained through routine experimentation by those skilled in the art in light of the above disclosure.

As mentioned above, various additives are generally incorporated into the base fluids of this invention to prepare completely formulated hydraulic fluids. Antiwear, or lubricity, additives such as tricresyl phosphate, dibutylchlorendate and antimony dialkyl-dithiocarbamate are preferably incorporated at about 1 to 10 percent by weight.

Rubber swell additives, such as di(2-ethylhexyl) sebecate or tributyl phosphate, may be incorporated at about 1 to 5% by weight. These are typically employed with polydimethylsiloxanes since these base fluids tend to shrink rubber components by leaching out plasticizers from the latter. Generally, less swell additive is needed as alkyl content, or size of the alkyl group, of component (A) is increased or a greater amount of component (B) is utilized.

Antioxidants such as p, p′ dioctyldiphenylamine and butylated hydroxytoluene at concentrations of about 0.1 to 0.5% by weight are generally required to protect the alkyl groups from oxidation at elevated operating temperatures. Dyes may also be incorporated at low levels, mainly for identification purposes.

Other, non-essential additives, such as fire retardants, defoamers and corrosion inhibitors may also find utility in combination with the base fluids of the present invention.

Increasing the alkylmethylsiloxane content of component (A) towards 100% resulted in polymers which were unsatisfactory in that these base fluids could not be formulated to conform to the viscosity requirements of the Low-Temperature MIL-H-83282 target. Furthermore, as alkyl content is increased, swell of any rubber components in the hydraulic system also increases, an undesirable consequence in some systems.

Those skilled in the art will recognize that, although some of the requirements of the Low-Temperature MIL-H-83282 target can be met by varying molecular weight of the base fluid composition and/or formulating with various additives, the key properties addressed above are not usually amenable to such manipulation. Thus, for example, if a base fluid (i.e., the siloxane copolymer-PAO blend of the instant invention) does not simultaneously exhibit the required viscosity-temperature profile and a sufficiently high flash point, compounding that base fluid into a completely formulated hydraulic fluid for testing will generally not result in meeting the Low-Temperature MIL-H-83282 target properties. Accordingly, those base fluid compositions which did not pass these key property requirements were judged incompatible with the Low-Temperature MIL-H-83282 target..

This invention also relates to an improved process for transmitting hydraulic force (pressure) in a hydraulic system, wherein the hydraulic fluid consists essentially of one of the siloxane copolymer-PAO blends described above. In the improved process, the fluid-containing portions of the hydraulic components of said hydraulic system are filled with the compositions of this invention, as disclosed above. Examples of hydraulic components envisioned herein include pumps, fluid coupling devices, such as diaphragms, hydraulic line means, valves and hydraulic actuating means, such as hydraulic motors, pistons and servo-valves.

The hydraulic systems which benefit most from the use of the hydraulic base fluids of this invention are those which are required to operate between the temperature extremes of -54°C. and 135°C. and simultaneously require improved fire resistance over systems using mineral oil as the hydraulic fluid. The process of the present invention is, therefore, most appropriately employed in conjunction with military aircraft hydraulic systems. Other hydraulic systems wherein the process of the present invention may be advantageously employed include those of civilian aircraft, military transport and combat vehicles for land and sea, space vehicles and machinery for use in arctic regions or other cold environments.

The following examples are presented to further illustrate the compositions of this invention, but are not to be construed as limiting the invention, which is delineated in the appended claims. All parts and percentages in the examples are on a weight basis unless indicated to the contrary.

Example 1

To a flask, equipped with a stirrer, thermometer, condenser, addition funnel and means for purging with dry nitrogen gas, there was added 316 grams of hexene-1 and 0.1 ml of a chloroplatinic acid complex of divinyltetramethyldisiloxane diluted with dimethylvinylsiloxy endblocked polydimethylsiloxane to provide 0.7 weight percent platinum (prepared according to Example 1 of U.S. Patent No. 3.419,593 to Willing). To this mixture there was added, over a period of 2 hours, 800 grams of a siloxane copolymer of the following average composition:

$Me^3SiO(MeHSiO)_{4.4}(Me^2SiO)_{9.8}SiMe^3$

wherein Me denotes the methyl radical. The reaction was exothermic and the contents were cooled so as to keep the temperature between 25 and 50°C. The resulting copolymer had the calculated average structure

$$Me_3SiO(Me_2SiO)_{9.8}(\underset{\underset{C_6H_{13}}{|}}{MeSiO})_{4.4}SiMe_3$$

Example 2

A mixture of 400 grams of the copolymer prepared in Example 1, above, 500 grams of a siloxane having the average formula

$Me^3SiO(Me^2SiO)_{4.7}SiMe^3$,

100 grams of a mixture of dimethylcyclosiloxanes having 3, 4 and 5 dimethylsiloxy units and 1 gram of KOH was stirred and heated to about 160°C. under a nitrogen atmosphere. About 20 ml of volatile products were removed by distillation. The remaining mixture was further reacted at 160°C. for 4 hours, after which it was cooled to room temperature. One gram of trimethylchlorosilane was added and the resulting combination stirred for an additional 30 minutes. The product was pressure-filtered to remove the KCl formed during neutralization of the base catalyst. Finally, the product was stripped to a pot temperature of 250°C. (208°C. vapor temperature) at 20 mm Hg. The residue weighed 579 grams, had a specific gravity of 0.927 grams per cc, a viscosity of 11.55 cS at 25°C. and a calculated average structure

$$Me_3SiO(Me_2SiO)_{6.26}(\underset{\underset{C_6H_{13}}{|}}{MeSiO})_{1.05}SiMe_3.$$

Example 3

A homogeneous blend was prepared by mixing equal portions of the fluid prepared in Example 2 and a 4 cS SYNFLUID (Gulf Oil Chemicals Co., Houston, TX). The latter fluid is described as a trimer of decene having the formula

$$CH_3\underset{\underset{C_8H_{17}}{|}}{CH}CH_2\overset{\overset{C_8H_{17}}{|}}{C}HCH_2\underset{\underset{C_8H_{17}}{|}}{CH_2}.$$

The blend was subjected to measurements of viscosity at various temperatures as well as open-cup flash point and fire point, the results being reported in Table 1, below.

Example 4

A siloxane copolymer having the average structure

$$Me_3SiO(Me_2SiO)_{7.5}(\underset{\underset{C_8H_{17}}{|}}{MeSiO})_{0.5}SiMe_3$$

was prepared according to the method described in Example 1, above. This fluid was blended in equal amount with 4 cS SYNFLUID as in Example 3 and the blend viscosity and flash point determined. These results also appear in Table 1.

(Comparative) Example 5

A solution of 538 grams of octene-1 and 0.05 ml of a 0.1 molar solution of a chloroplatinic acid in isopropyl alcohol was heated to 120°C. under a nitrogen atmosphere. To this mixture there was added. over a period of 40 minutes, 480 grams of a siloxane polymer having the following average composition:

$Me^3SiO(MeHSiO)_{1.3}SiMe^3$

wherein Me denotes the methyl radical. The reaction produced an exotherm to about 130°C. and this temperature was maintained for 80 minutes to complete the reaction. The product was stripped to a pot temperature of 240°C. (191°C. vapor temperature) at 10 mm Hg and then pressure-filtered. This homopolymer had a viscosity of 14.1 cS at 25°C. and a calculated average structure

$$Me_3SiO(\underset{\substack{|\\ C_8H_{17}}}{MeSiO})_3SiMe_3.$$

This fluid was blended with an equal amount of the decene trimer (4 cS SYNFLUID), as described in Example 3, above. Viscosity and flash point test results for this blend are also reported in Table 1.

As can be seen from Table 1, only the compositions of the present invention simultaneously satisfy the viscosity and flash point requirements of the Air Force Low-Temperature MIL-H-83282 target.

TABLE 1

Fluid	Viscosity (cS) at -54°C.	at -40°C.	at 25°C.	at 100°C.	at 135°C.	Open-Cup Flash Point (°C.)	Fire Point (°C.)
Example 3	1527	431	17.6	4.24	2.20	207	238
Example 4	1919	482	18.0	3.63	2.55	215	243
(Comparative) Example 5	---	1007	19.9	3.5	---	---	---
Decene dimer (2cS SYNFLUID)*	1200	262	---	1.70	---	160	182
Decene trimer (4cS SYNFLUID)*	13300	2420	---	3.87	---	219	249
LOW-TEMPERATURE MIL-H-83282: TARGET PROPERTIES	2500 (max.)	500 (max.)	---	3.5 (min.)	1.5 (min.)	163 (min.)	191 (min.)

* A product of Gulf Chemicals Co., Houston, Texas.

Claims

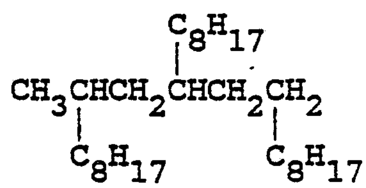
1. A homogeneous blend consisting essentially of:

(A) about 40 to 90 parts by weight of a siloxane copolymer represented by the average formula

$$(R')_3SiO(Me_2SiO)_x(\underset{R}{MeSiO})_ySi(R')_3$$

wherein Me denotes the methyl group, R' is independently selected from alkyl radicals having 1 to 10 carbon atoms, R denotes an alkyl group having from 4 to 10 carbon atoms, x is about 5 to 15, y is about 0.5 to 4 and the ratio y/x is at most 0.3; and

(B) about 60 to 10 parts by weight of a synthetic hydrogenated polyalphaolefin represented by the formula



said blend having a total of 100 parts by weight of said components (A) and (B).

2. The composition of claim 1, wherein 40 to 60 parts by weight of component (A) are blended with 60 to 40 parts by weight of component (B).

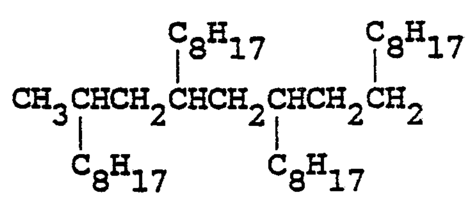
3. A homogeneous blend consisting essentially of:

(A) about 80 to 90 parts by weight of a dimethylsiloxane/alkylmethylsiloxane copolymer represented by the average formula

$$(R')_3SiO(Me_2SiO)_x(\underset{R}{MeSiO})_ySi(R')_3$$

wherein Me denotes the methyl group, R' is independently selected from alkyl radicals having 1 to 10 carbon atoms, R denotes an alkyl group having from 4 to 10 carbon atoms, x is of about 5 to 15, y is about 0.5 to 4 and the ratio y/x is at most 0.3; and

(B) about 20 to 10 parts by weight of a synthetic hydrogenated polyalphaolefin represented by the formula



said blend having a total of 100 parts by weight of said components (A) and (B).

4. A homogeneous blend consisting essentially of:

(A) about 10 to 90 parts by weight of a siloxane copolymer represented by the average formula

$$(R')_3SiO(Me_2SiO)_x(\underset{R}{MeSiO})_ySi(R')_3$$

wherein Me denotes the methyl group, R' is independently selected from alkyl radicals having 1 to 10 carbon atoms, R denotes an alkyl group having from 4 to 10 carbon atoms, x is about 5 to 15, y is about

0.5 to 4 and the ratio y/x is at most 0.3: and

(B) about 90 to 10 parts by weight of at least one synthetic hydrogenated polyalphaolefin selected from the dimer, trimer or tetramer of decene-1, said blend having a total of 100 parts by weight of said components (A) and (B) and a viscosity of at most 2500 cS at -54°C., a viscosity of at least 3.5 cS at 100°C, and an open cup flash point of at least 163°C.

5. In a process for transmitting force in a hydraulic system, the improvement comprising using the composition of claim 1 as a hydraulic fluid in said system.

6. In a process for transmitting force in a hydraulic system, the improvement comprising using the composition of claim 3 as a hydraulic fluid in said system.

7. In a process for transmitting force in a hydraulic system, the improvement comprising using the composition of claim 4 as a hydraulic fluid in said system.