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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 : **C10M 169/04, 171/00 // (C10M 169/04, 101:02, 105:36, 105:38, 107:02, 129:10, 133:12, 133:56, 155:02), C10N 30:18, 40:04, 60:14**	**A1**	**(11) International Publication Number:** **WO 97/04051** **(43) International Publication Date:** 6 February 1997 (06.02.97)

(21) International Application Number: PCT/US96/11737

(22) International Filing Date: 15 July 1996 (15.07.96)

(30) Priority Data:
502,953 17 July 1995 (17.07.95) US

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(81) Designated States: AU, CA, JP, SG, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

Published
With international search report.

(54) Title: POWER TRANSMITTING FLUIDS WITH IMPROVED RESISTANCE TO FOAMING

(57) Abstract

This invention concerns power transmitting fluids, particularly automatic transmission fluids, containing synthetic or partially synthetic lubricating oils that have superior foam suppression properties due to the presence of a fluorosilicone antifoamant.

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POWER TRANSMITTING FLUIDS WITH IMPROVED RESISTANCE TO FOAMING

BACKGROUND OF THE INVENTION

This invention relates to compositions and methods of improving the properties of power transmitting fluids, particularly to obtaining automatic transmission fluids with superior resistance to foaming.

The continuing advances made by automobile manufacturers to improve vehicle performance and reliability place increased demands on the lubricants used in the vehicle engine and drive train. This is especially true of automatic transmission fluids (ATF's). Over the last ten years changing specifications have reduced the allowable low temperature viscosities for ATF's as well as increased the requirements for oxidation resistance and wear protection. These more severe requirements resulted in numerous changes in additive technology as well as caused reformulation of ATF's to incorporate more oxidatively stable base oils with improved low temperature viscosities. These trends have resulted in formulation of partially synthetic or fully synthetic to meet these more severe requirements.

One aspect of ATF performance that is often overlooked is foam suppression. This includes the fluid's resistance to foam formation as well as its ability to break or dissipate foam, if formed. Many of the changes in fluid composition and transmission design have aggravated this problem and heightened the need for improved antifoamants. Fluids formulated with less viscous, more volatile base stocks, required for improved low temperature performance, tend to produce more foam, and more stable foams than ATF's formulated with the heavier base oils of previous generations. As vehicle transmissions become smaller and operate at higher temperatures and pressures, the transmissions tend to generate more foam. Many of the rotating elements in the transmission are now operating at speeds greater than 5,000 rpm, which provides an enormous potential for increased foam generation through churning and aeration.

We have now found that a particular class of antifoamants described generally as fluorosilicones (e.g., perfluorinated polyalkylsiloxanes) are very effective and superior antifoamants for power transmitting fluids, especially partially or fully synthetic ATF's. These antifoamants are effective in treating foam generated by air entrainment or by physical churning, and they are effective at very low concentrations in the finished fluid.

SUMMARY OF THE INVENTION

This invention relates to a power transmitting fluid comprising:

(a) a synthetic lubricating oil;

(b) a fluorosilicone antifoamant; and

(c) optionally, a natural lubricating oil.

This invention also concerns a method for providing power transmitting fluids having superior foam suppression.

A particular advantage of this invention is that the compositions disclosed pass the ATF foam suppression requirements of both the Ford Motor Company and General Motors Corporation.

DETAILED DESCRIPTION OF THE INVENTION

This invention describes the use of fluorinated silicones of varying molecular weights to suppress foam formation in power transmitting fluids, especially automatic transmission fluids. Examples of other types of power transmitting fluids included within the scope of this invention are gear oils, hydraulic fluids, heavy duty hydraulic fluids, industrial oils, power steering fluids, pump oils, tractor fluids, universal tractor fluids and the like. These power transmitting fluids can be formulated with a variety of performance additives and in a variety of base oils.

Lubricating Oils

Lubricating oils contemplated for use in this invention are derived from synthetic lubricating oils or mixtures of synthetic lubricating oils and natural lubricating oils. Suitable lubricating oils include base stocks obtained by isomerization of synthetic wax and slack wax, as well as base stocks produced by hydrocracking (rather than solvent extracting) the aromatic and polar components of the crude. In general, both the natural and synthetic lubricating oils will each have a kinematic viscosities ranging from about 1 to about 40 mm^2/s (cSt) at 100°C, although typical applications will require each oil to have a viscosity ranging from about 2 to about 8 mm^2/s (cSt) at 100°C.

Synthetic lubricating oils include hydrocarbon oils and halo-substituted hydrocarbon oils such as oligomerized, polymerized, and interpolymerized olefins [e.g., polybutylenes, polypropylenes, propylene, isobutylene copolymers, chlorinated polylactenes, poly(1-hexenes), poly(1-octenes), poly-(1-decenes), etc., and mixtures thereof]; alkylbenzenes [e.g., dodecylbenzenes, tetradecylbenzenes, dinonyl-benzenes, di(2-ethylhexyl)benzene, etc.]; polyphenyls [e.g., biphenyls, terphenyls, alkylated polyphenyls, etc.]; and alkylated diphenyl ethers, alkylated diphenyl sulfides, as well as their derivatives, analogs, and homologs thereof, and the like. The preferred oils from this class of synthetic oils are oligomers of α-olefins, particularly oligomers of 1-decene.

Synthetic lubricating oils also include alkylene oxide polymers, interpolymers, copolymers, and derivatives thereof where the terminal hydroxyl groups have been modified by esterification, etherification, etc. This class of synthetic oils is exemplified by polyoxyalkylene polymers prepared by polymerization of ethylene oxide or propylene oxide; the alkyl and aryl ethers of these polyoxyalkylene polymers (e.g., methyl-polyisopropylene glycol ether having an average molecular weight of 1000, diphenyl ether of polypropylene glycol having a molecular weight of 1000 - 1500); and mono- and polycarboxylic esters thereof (e.g., the acetic acid esters, mixed C_3-C_8 fatty acid esters, and C_{12} oxo acid diester of tetraethylene glycol).

Another suitable class of synthetic lubricating oils comprises the esters of dicarboxylic acids (e.g., phthalic acid, succinic acid, alkyl succinic acids and alkenyl succinic acids, maleic acid, azelaic acid, subric acid, sebasic acid, fumaric acid, adipic acid, linoleic acid dimer, malonic acid, alkylmalonic acids, alkenyl malonic acids, etc.) with a variety of alcohols (e.g., butyl alcohol, hexyl alcohol, dodecyl alcohol, 2-ethylhexyl alcohol, ethylene glycol, diethylene glycol monoethers, propylene glycol, etc.). Specific examples of these esters include dibutyl adipate, di(2-ethylhexyl) sebacate, di-n-hexyl fumarate, dioctyl sebacate, diisooctyl azelate, diisodecyl azelate, dioctyl isothalate, didecyl phthalate, dieicosyl sebacate, the 2-ethylhexyl diester of linoleic acid dimer, and the complex ester formed by reacting one mole of sebasic acid with two moles of tetraethylene glycol and two moles of 2-ethylhexanoic acid, and the like. A preferred type of oil from this class of synthetic oils are adipates of C_4 to C_{12} alcohols.

Esters useful as synthetic lubricating oils also include those made from C_5 to C_{12} monocarboxylic acids and polyols and polyol ethers such as neopentyl glycol, trimethylolpropane pentaerythritol, dipentaerythritol, tripentaerythritol, and the like.

Silicon-based oils (such as the polyalkyl-, polyaryl-, polyalkoxy-, or polyaryloxy-siloxane oils and silicate oils) comprise another useful class of synthetic lubricating oils. These oils include tetra-ethyl silicate, tetraisopropyl silicate, tetra-(2-ethylhexyl) silicate, tetra-(4-methyl-2-ethylhexyl) silicate, tetra-(p-tert-butylphenyl) silicate, hexa-(4-methyl-2-pentoxy)-disiloxane, poly(methyl)-siloxanes and poly(methylphenyl) siloxanes, and the like. Other synthetic lubricating oils include liquid esters of phosphorus-containing acids (e.g., tricresyl phosphate, trioctyl phosphate, and diethyl ester of decylphosphonic acid), polymeric tetra-hydrofurans, poly-α-olefins, and the like.

Natural lubricating oils include animal oils, vegetable oils (e.g., castor oil and lard oil), petroleum oils, mineral oils, and oils derived from coal or shale. The preferred natural lubricating oil is mineral oil.

The mineral oils useful in this invention include all common mineral oil base stocks. This would include oils that are naphthenic or paraffinic in chemical structure. The oils may be refined by conventional methodology using acid, alkali, and clay or other agents such as aluminum chloride, or they may be extracted oils produced, for example, by solvent extraction with solvents such as phenol, sulfur dioxide, furfural, dichlordiethyl ether, etc. They may also be hydrotreated or hydrofined, dewaxed by chilling or by catalytic processing, or hydrocracked. The mineral oil may be produced from natural crude sources or be composed of isomerized wax materials or residues of other refining processes.

Typically the mineral oils will have kinematic viscosities of from 2.0 to 8.0 mm^2/s (cSt) at 100°C. The preferred mineral oils have kinematic viscosities of from 2 to 6 mm^2/s (cSt), and most preferred are those mineral oils with viscosities of 3 to 5 mm^2/s (cSt) at 100°C.

When the power transmitting fluid is a mixture of synthetic lubricating oil and natural lubricating oil (i.e., partially synthetic), the fluid will contain 1 to 80 weight percent, preferably from about 10 to 75 weight percent, and most preferably from about 10 to about 50 weight percent synthetic lubricating oil.

The lubricating oils may be derived from refined, rerefined oils, or mixtures thereof. Unrefined oils are obtained directly from a natural source or synthetic source (e.g., coal, shale, or tar sands bitumen) without further purification or treatment. Examples of unrefined oils include a shale oil obtained directly from a retorting operation, a petroleum oil obtained directly from distillation, or an ester oil obtained directly from an esterification process, each of which is then used without further treatment. Refined oils are similar to the unrefined oils except that refined oils have been treated in one or more purification steps to improve one or more properties. Suitable purification techniques include distillation, hydrotreating, dewaxing, solvent extraction, acid or base extraction, filtration, and percolation, all of which are known to those skilled in the art. Rerefined oils are obtained by treating used oils in processes similar to those used to obtain the refined oils. These rerefined oils are also known as reclaimed or reprocessed oils and are often additionally processed by techniques for removal of spent additives and oil breakdown products.

Antifoamants

Not all antifoamants are capable of providing the superior foam suppression as are the compositions of this invention. Two conventional antifoamants that do not provide the foam suppression of this invention are polysiloxanes and polyacrylates, and are referred to as dispersible and soluble antifoamants, respectively. The siloxane type antifoamants are hydrocarbyl silicone oils, typically polydimethyl siloxanes with varying molecular weights. These silicone fluids are characterized by their viscosities, which range from about 10,000 to about 60,000 centipoise (cP) at 25°C. Silicone type antifoamants do not dissolve in lubricating oils, but are dispersed in the oil as fine droplets. Typical treat rates in the finished lubricating oils are from 5 to 100 ppm. A well known polydimethylsiloxane antifoamant is manufactured by Dow Corning Company and sold as DC-200.

The polyacrylate type antifoamants are polymers or copolymers containing acrylic acids, acrylic esters, methacrylic acids or methacrylic esters in the backbone. These acrylic groups are used to attach sidechains of varying lengths which give the materials their surface activity and, thus, their performance as antifoamants. The polyacrylate type antifoamants are soluble in the lubricating oil, and are therefore called soluble antifoamants. They are typically used at concentrations of 500 to 5,000 ppm in the finished oil. A well known additive of this type is DF-283 sold by R.T. Vanderbilt Company.

In contrast, the antifoamants of this invention are described as fluorosilicones, which are typically polymers or copolymers of perfluorinated alkyl siloxanes. A common monomer in these fluorosilicones is 3,3,3-trifluoropropylmethyl siloxane. The fluorosilicones are polymerized to various molecular weights and are characterized by their viscosities at 25°C. Typical fluorosilicones have viscosities from about 150 to about 10,000, preferably from about 200 to about 5,000, and more preferably from about 200 to about 500 mm^2/s (cSt) at 25°C. The most preferred fluorosilicone for use in this invention is poly(3,3,3-trifluoropropylmethylsiloxane), polymerized to have a 25°C viscosity of about 300 mm^2/s (cSt). One commercial fluorosilicone satisfying these requirements is FS 1265, 300 mm^2/s (cSt), made and sold by the Dow Corning Company. FS 1265 contributes from 1 to 15 ppm of elemental silicon when FS 1265 is present in a fluid at a concentration ranging from approximately 5 to 50 ppm.

The fluorosilicone antifoamant may be added to the lubricating oil by a variety of methods including direct dispersion, dilution in a "carrier" solvent, or pre-dispersion in an additive package that is subsequently dissolved in the lubricating oil. Treat rates of the fluorosilicones based on active ingredient, are no greater than 100 ppm in the finished fluid. Preferably, the treat rate is less than 50 ppm and, most preferably, ranges from 10 to 25 ppm.

Other additives known in the art may be present in the power transmitting fluid including dispersants, antiwear agents, antioxidants, corrosion inhibitors, detergents, extreme pressure additives, friction modifiers, and the like. They are typically disclosed in, for example, "Lubricant Additives" by C. V. Smalheer and R. Kennedy Smith, 1967, pp. 1-11 and U.S. Patent 4,105,571.

Representative amounts of these additives are summarized as follows:

Additive	(Broad) Wt.%	(Preferred) Wt.%
Corrosion Inhibitor	0.01 - 3	0.02 - 1
Antioxidants	0.01 - 5	0.2 - 3
Dispersants	0.10 - 10	2 - 5
Friction Modifiers	0.01 - 5	0.1 - 3
Detergents	0.01 - 6	0.01 - 3
Antiwear Agents	0.001- 5	0.2 - 3
Seal Swellants	0.1 - 8	0.5 - 5

Suitable dispersants include hydrocarbyl succinimides, hydrocarbyl succinamides, mixed ester/amides of hydrocarbyl-substituted succinic acid, hydroxyesters of hydrocarbyl-substituted succinic acid, and Mannich condensation products of hydrocarbyl-substituted phenols, formaldehyde and polyamines. Mixtures of such dispersants can also be used.

The preferred dispersants are the alkenyl succinimides. These include acyclic hydrocarbyl substituted succinimides formed with various amines or amine derivatives such as are widely disclosed in the patent literature. Use of alkenyl succinimides which have been treated with an inorganic acid of phosphorus (or an anhydride thereof) and a boronating agent are also suitable for use in the compositions of this invention as they are much more compatible with elastomeric seals made from such substances as fluoroelastomers and silicon-containing elastomers. Polyisobutenyl succinimides formed from polyisobutenyl succinic anhydride and an alkylene polyamine such as triethylene tetramine or tetraethylene pentamine wherein the polyisobutenyl substituent is derived from polyisobutene having a number average molecular weight in the range of 500 to 5000 (preferably 800 to 2500) are particularly suitable. Dispersants may be post-treated by any reagents known to one skilled in the art. (see, e.g., U.S. Pat. Nos. 3,254,025, 3,502,677, and 4,857,214).

Suitable antioxidants are amine-type and phenolic antioxidants. Examples of the amine-type antioxidants include phenyl alpha naphthylamine, phenyl beta naphthylamine, diphenylamine, bis- alkylated diphenyl amines (e.g., p,p'-bis(alkylphenyl)amines wherein the alkyl groups contain from 8 to 12 carbon atoms each). Phenolic antioxidants include sterically hindered phenols (e.g., 2,6-di-tert-butylphenol, 4-methyl-2,6-di-tert-butylphenol, etc.) and bis-phenols (e.g., 4,4'- methylenebis(2,6-di-tert-butylphenol), etc.) and the like.

Suitable friction modifiers are composed of two distinct parts, a polar head group and an oleophilic tail group. The polar head groups cause the molecule to be adsorbed onto the friction surfaces. These groups can consist of but not be limited to: amines, mono and diethoxylated amines, carboxylic acids, amides, imides, alcohols, phenols, thiols, sulfonic acids, phosphites, phosphates, esters and combinations thereof. The oleophilic groups are typically alkyl groups, normally linear alkyl. They range in carbon number from C_8 to C_{30}, preferably C_{12} to C_{20}. They may be saturated or unsaturated, they may contain hetero atoms such as N or S as long as they do not adversely effect their function as a friction modifier.

Examples of friction modifiers suitable for use in this invention are: oleamide, tallow amine, diethoxylated tallow amine, N,N-bis(2-hydroxyethyl)-octadecyl amine, N,N-bis(2-hydroxyethyl)-stearyloxypropylamine, oleic acid, N-2-hydroxylethyl,N-(N',N'-bis(2-hydroxylethyl)ethylamine)-stearylamine, the diamide produced from isostearic acid and tetraethylene pentamine, and the like.

The additive concentrates of this invention will contain the fluorosilicone antifoamant and other desired additives in a natural and/or synthetic lubricating oil, in relative proportions such that by adding the concentrate to a larger amount of synthetic or mixture of a suitable natural and synthetic oil, the resulting fluid will contain each of the ingredients in the desired concentration. Thus, the concentrate may contain a synthetic oil as the lubricating oil if the desired final composition contains a lesser amount of synthetic oil relative to the natural oil. The concentrate typically will contain between 25 to 100, preferably from 65 to 95, most preferably from 75 to 90 weight percent of the antifoamant with other desired additives, and synthetic or natural/synthetic (partial synthetic) lubricating oil.

The following examples are given as specific illustrations of the claimed invention. It should be understood, however, that the invention is not limited to the specific details set forth in the examples. All parts and percentages are by weight unless otherwise specified.

In these examples, two foam tests were used to evaluate the foam control capabilities of automatic transmission fluids. The first test is the ASTM D-892 test as modified by Ford Motor Company for inclusion in their MERCON® Automatic Transmission Fluid Specification for Service, dated September 1, 1992 (Section 3.9). In this test, the fluid is aerated in a tall graduated cylinder using a fine diffuser suspended in a column of ATF maintained at a certain test temperature. The test has 4 distinct 5 minute aeration sequences of varying temperature and air flow rates. At the end of each aeration sequence, the volume of foam is reported in milliliters (ml's) and reported again at the end of a 1 minute settling period. The data is given in the format 50/0, which means 50 ml's of foam present after aeration, and none present after 1 minute of settling. Ford requires that at no time there be more than 100 ml's of foam, and that after the 1 minute settling there is no visible foam remaining. Therefore, to pass a fluid must have a result of less than 100/0.

The second foam test is defined in the General Motors DEXRON®-III Specification, GM-6297M, April 1993, Appendix A. The GM test uses a pump and impeller to aerate and churn the fluid. The test is first run at 95°C and the level of foam reported in millimeters (mm's). The apparatus is then heated to 135°C and the level of foam again reported in mm's, as well as the time in seconds for the foam to collapse after stopping the agitation. GM requires that no foam be present at 95°C, that no more than 5 mm of foam be present at 135°C, and that the foam, if present at 135°C, collapses in less than 15 seconds.

EXAMPLE 1

Twelve (12) partial synthetic automatic transmission fluids were blended according to the formulations shown in Table 1. Each fluid contained 8.0 weight percent of an additive package having conventional amounts of a succinimide dispersant, antioxidants, antiwear agents, friction modifiers, a corrosion inhibitor, and a diluent oil. Each formulation also contained varying amounts of the 3 types of antifoamants for comparison: (1) a polydimethyl siloxane, DC-200 (12,500 mm^2/s (cSt) at 25°C) from Dow Corning Company; (2) a polymethacrylate, DF-283 from R.T. Vanderbilt Company; and (3) a fluorosilicone, FS 1265 (300 mm^2/s (cSt) at 25°C) from Dow Corning Company. The 12 blends used two different types of natural lubricating oil base stocks: (1) a conventional solvent extracted neutral oil from Exxon (≈3.1 mm^2/s (cSt) at 100°C) and (2) hydrotreated and deep dewaxed base stocks from Petro-Canada (60 N ≈ 2.7 mm^2/s (cSt) at 100°C; 80 N ≈ 3.4 mm^2/s (cSt) at 100°C). Each blend also contained varying amounts of a mixture of two synthetic oils: a poly-α-olefin having a kinematic viscosity of approximately 4 mm^2/s (cSt) at 100°C and di-isooctyl adipate.

The 12 partially synthetic ATF blends were tested by both the Ford and GM foam test methods. The results of this testing are also shown in Table 1, with numbers in **BOLD-FACED/SHADED** type indicating a failing result. This data show that the polydimethyl siloxane based antifoamant (DC-200 12,500) is better at controlling foam in the GM test than the polymethacrylate, especially at 93°C. Blends 1 and 2 show a particular problem with the polydimethyl siloxane antifoamants. In the GM test, increasing treat rate of silicone antifoamant (Blend 2 compared to Blend 1) gives better results at 135°C; Blend 1 fails whereas Blend 2 passes. However, in Blend 2, the same increase in concentration gave poorer results in the fourth sequence of the Ford test, i.e., the increased concentration causes the borderline Blend 1 to actually fail the test. The same concentration effect can be seen in Blends 7 and 8 containing the Petro-Canada base stock. The polydimethyl siloxane based antifoamants are very poor in controlling foam in Sequence 4 of the Ford test. None of the fluids treated with DC-200 (Blends 1, 2, 7 and 8) passed both foam tests.

The acrylate based antifoamant, DF-283, is better at controlling foam in the Ford test, especially in Sequence 4. However, it has very poor control of foam at 93°C in the GM test. Every blend made using the DF-283 (Blends 3, 4, 9, and 10) fail the GM foam test at 93°C. It is also clear from Blends 3 and 4 that increasing the concentration of DF-283 leads to poorer results in the GM test at 135°C. None of the blends treated with the DF-283 acrylate based anti-foamant passed both foam tests.

In contrast, the blends treated with the fluorosilicone, FS 1265, (Blends 5, 6, 11, and 12) give essentially no foam, in any test, at any condition. Blends 5, 6, 11, and 12 are the only blends that pass both foam tests.

EXAMPLE 2

Three fully synthetic ATF's were blended and treated with the same three antifoamants described in Example 1 above. The compositions of the fluids are given in Table 2. Table 2 also contains the results of the foam testing performed by both the GM and Ford methods, failing results are again shown in **BOLD-FACED/SHADED** type. The results obtained show the superiority of the fluorosilicone antifoamant when compared to those obtained using either of the conventional antifoamants.

The performance of the three antifoamants in the fully synthetic ATF's was analogous to their performance in the partially synthetic fluids. Blend 13 in Table 2, the fluid treated with the polydimethyl siloxane, DC-200, again performed very poorly in the Ford test, failing Sequences 2 and 4. Blend 13 passed the General Motors requirements easily.

Blend 14 contained the polymethacrylates antifoamant, DF-283. Although Blend 14 passed Sequences 2 and 4 of the Ford test, it failed

Sequences 1 and 3. Blend 14 completely failed the General Motors foam test.

Blend 15, which was treated with the fluorosilicone based antifoamant of the invention, FS 1265, passed all of the requirements of both the Ford and General Motors test, giving no observable foam in any sequence of either test. This clearly demonstrates the superiority of the fluorosilicone based antifoamants when compared to either type of conventional antifoamant.

The principles, preferred embodiments, and modes of operation of the present invention have been described in the foregoing specification. The invention which is intended to be protected herein, however, is not to be construed as limited to the particular forms disclosed, since these are to regarded as illustrative rather than restrictive. Variations and changes may be made by those skilled in the art without departing from the spirit of the invention.

Table 1

PARTIALLY SYNTHETIC ATF'S

BLENDS:	1	2	3	4	5	6	7	8	9	10	11	12	LIMITS
Additive Package	8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00	
PAO-4	32.40	32.40	32.40	32.40	32.40	32.40	12.83	12.83	12.83	12.83	12.83	12.83	
Di-isooctyl adipate	10.00	10.00	10.00	10.00	10.00	10.00	2.33	2.33	2.33	2.33	2.33	2.33	
TLA 5012	1.00	1.00	1.00	1.00	1.00	1.00	1.10	1.10	1.10	1.10	1.10	1.10	
EXXON Solvent 75 Neutral	48.60	48.60	48.60	48.60	48.60	48.60	-	-	-	-	-	-	
PETRO-CANADA 60 Neutral	-	-	-	-	-	-	60.57	60.57	60.57	60.57	60.57	60.57	
PETRO-CANADA 80 Neutral	-	-	-	-	-	-	15.17	15.17	15.17	15.17	15.17	15.17	
ANTI-FOAMANT, PPM FLUID													
DC-200 12,500 *	15	36	-	-	-	-	15	36	-	-	-	-	
VANLUBE DF-283 **	-	-	80	160	-	-	-	-	80	160	-	-	
DOW CORNING FS 1265 ***	-	-	-	-	18	36	-	-	-	-	18	36	
TEST RESULTS													
FORD METHOD													
Seq. 1, Vol., ml's	0/0	0/0	45/0	55/0	50/0	0/0	0/0	0/0	70/0	55/0	25/0	40/0	100/0
Seq. 2, Vol., ml's	25/0	35/0	0/0	0/0	0/0	0/0	30/0	30/0	0/0	0/0	0/0	0/0	100/0
Seq. 3, Vol., ml's	0/0	0/0	45/0	40/0	0/0	0/0	0/0	0/0	65/0	60/0	0/0	0/0	100/0
Seq. 4, Vol., ml's	90/0	100/0	0/0	0/0	0/0	0/0	90/0	100/0	0/0	0/0	0/0	0/0	100/0
GM METHOD													
95 C, mm (Foam Height)	0	0	3	5	0	0	0	0	7	6	0	0	0
135 C, mm (Foam Height)	8	4	7	12	0	0	10	13	11	12	0	0	5
135 C, sec. (Collapse Time)	8	10	8	7	****	****	5	15	6	8	****	****	15

Notes:

*DC-200 is a registered trademark of Dow Corning Company

**VANLUBE is a registered trademark of R. T. Vanderbilt Company

***FS 1265 is a registered trademark of Dow Corning Company

****No collapse time measurable as no foam was formed.

Table 2

FULLY SYNTHETIC ATF'S

BLENDS:	13	14	15	
Additive Package	8.00	8.00	8.00	
PAO-4	77.00	77.00	77.00	L
Di-isooctyl adipate	14.00	14.00	14.00	I
TLA 5012	1.00	1.00	1.00	M
				I
ANTI-FOAMANT, PPM FLUID				T
DC-200 12,500 *	16	-	-	S
VANLUBE DF-283 **	-	80	-	
DOW CORNING FS 1265 ***	-	-	18	
TEST RESULTS				
FORD METHOD				
Seq. 1, Vol., ml's	0/0	40/20	0/0	100/0
Seq. 2, Vol., ml's	25/7	0/0	0/0	100/0
Seq. 3, Vol., ml's	0/0	40/23	0/0	100/0
Seq. 4, Vol., ml's	85/11	0/0	0/0	100/0
GM METHOD				
95 C, mm (Foam Height)	0	5	0	0
135 C, mm (Foam Height)	0	9	0	5
135 C, sec. (Collapse Time)	****	4	**	15

* DC-200 is a registered trademark of Dow Corning Company

** VANLUBE is a registered trademark of R.T. Vanderbilt Company

*** FS1265 is a registered trademark of Dow Corning Company

****No collapse time measurable as no foam was formed.

CLAIMS:

1. A power transmitting fluid comprising:

(a) a synthetic lubricating oil;

(b) a fluorosilicone antifoamant; and,

(c) optionally, a natural lubricating oil.

2. The composition of claim 1 where the synthetic oil is a poly-α-olefin, monoester, diester, polyolester, or mixtures thereof.

3. The composition of claim 2, where the natural lubricating oil is a mineral oil.

4. The composition of claim 3, where the fluid is an automatic transmission fluid.

5. The composition of claim 4 further comprising a borated or non-borated succinimide dispersant, a phenolic or amine antioxidant, and a friction modifier such that the sum of the dispersant, antioxidant, and friction modifier is from 2.0 to 11.0 weight percent of the composition.

6. The composition of claim 1, where the antifoamant is present in an amount no greater than 100 ppm.

7. The composition of claim 1, where the antifoamant contributes from 1 to 30 ppm of elemental silicon to the fluid.

8. A method for suppressing the formation of foam in a partially synthetic or fully synthetic power transmitting fluid which comprises adding a fluorosilicone polymer to the fluid.

INTERNATIONAL SEARCH REPORT

national Application No
PCT/US 96/11737

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C10M169/04 C10M171/00 //(C10M169/04,101:02,105:36,105:38,
107:02,129:10,133:12,133:56,155:02),C10N30:18,C10N40:04,C10N60:14

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)
IPC 6 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)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US,A,3 933 659 (R.E.LYLE) 20 January 1976 see column 1, line 10 - line 13 see column 11, line 32 - line 33 see column 12, line 10 - line 14 see column 12, line 41 - line 43 see column 13, line 44 - column 14, line 20 ---	1-8
A	PATENT ABSTRACTS OF JAPAN vol. 017, no. 567 (C-1120), 14 October 1993 & JP,A,05 163496 (MITSUBISHI OIL CO LTD;OTHERS: 01), 29 June 1993, see abstract ---	1-5
A	US,A,5 360 562 (D.R. CHRISOPE) 1 November 1994 see column 13, line 35 - line 60 ---	1,2,4,5

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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

° Special categories of cited documents :

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

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

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Date of the actual completion of the international search 16 September 1996	Date of mailing of the international search report 03.10.96
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer Hilgenga, K

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP,A,0 620 267 (IDEMITSU KOSAN COMPANY LIMITED) 19 October 1994 see page 4, line 8 - line 9 see page 4, line 14 - line 15 see page 5, line 53 - line 54 see page 7 - page 8; examples 1-4 ---	1-5
A	PATENT ABSTRACTS OF JAPAN vol. 008, no. 222 (C-246), 9 October 1984 & JP,A,59 105091 (NISHINIHON TSUUSHIYOU KK), 18 June 1984, see abstract ---	1-3,6-8
A	DATABASE WPI Section Ch, Week 8430 Derwent Publications Ltd., London, GB; Class A97, AN 84-185816 XP002013422 & JP,A,59 105 092 (NISHINIPPON TSUSHO) , 18 June 1984 see abstract -----	1-3,6-8

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INTERNATIONAL SEARCH REPORT

Information on patent family members

ational Application No
PCT/US 96/11737

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A-3933659	20-01-76	CA-A- 1060878	21-08-79
		DE-A- 2530378	22-01-76
		FR-A- 2277882	06-02-76
		GB-A- 1522962	31-08-78
		GB-A- 1522963	31-08-78
		GB-A- 1522961	31-08-78
		JP-A- 51031369	17-03-76
US-A-5360562	01-11-94	US-A- 5089156	18-02-92
		AU-B- 635160	11-03-93
		AU-A- 8559291	16-04-92
		CA-A- 2051551	11-04-92
		DE-D- 69103717	06-10-94
		DE-T- 69103717	22-12-94
		EP-A- 0480644	15-04-92
		JP-A- 4264198	18-09-92
EP-A-620267	19-10-94	JP-A- 6293892	21-10-94
		CA-A- 2120888	10-10-94
		US-A- 5460741	24-10-95