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p-DIETHYLAMINOPHENYL SILANES

Harry M. Schiefer and Donald R. Weyenberg, Midland, Mich., assignors to Dow Corning Corporation, Midland, Mich., a corporation of Michigan

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5 Claims. (Cl. 260—448.2)

This application is a division of application Ser. No. 350,543, filed Mar. 9, 1964, which is a continuation-in-part of applicants' copending application Ser. No. 167,171, filed Jan. 18, 1962, now abandoned.

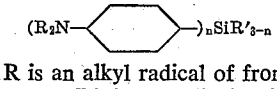
This invention relates to improved high temperature lubricants. Applicants have discovered a method for rendering high temperature lubricants, in particular certain ester lubricants, extremely resistant to oxidation at high temperatures, such as those found in aircraft jet engines.

The bearings of the jet and turbo-jet engines are lubricated by pumping into the bearings a lubricant coming from a closed circuit sump. With the development of more powerful engines, greater difficulty has been experienced in maintaining the stability of the lubricant under the higher temperatures encountered in these engines. While many lubricants will work satisfactorily at a temperature of 100 to 150° C., these lubricants at a temperature of 200° C. will form a large amount of sludge, increase in viscosity and become very acidic. This results in the corrosion of metals, particularly those being lubricated, and may result in the clogging of the lubricating system.

The best lubricants for jet engines have been found to be esters of alkanoic acids and polyhydric alcohols which have at least two methylol groups bonded to a quaternary carbon atom, such as 1,2,2-trimethylolpropane. Even though these compounds have a high degree of oxidative and heat stability, they are not stable in jet engines when the sump temperatures are in excess of 170° C. and the temperature at some of the bearings is in excess of 315° C.

It is an object of the present invention to improve the oxidative and heat stability of high temperature lubricating fluids. In particular, it is an object to improve the oxidative and heat stability of the polyhydric alcohol ester lubricating fluids. Another object is to provide high temperature lubricating fluids which are resistant to sludge formation.

These objects are obtained by a composition of matter comprising a mixture of (1) a high temperature organic lubricating fluid and (2) from 0.1 to 5 percent by weight based upon the weight of (1) of a compound of the formula



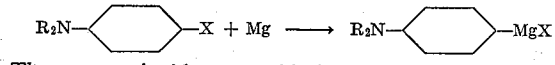
wherein each R is an alkyl radical of from 1 to 10 inclusive carbon atoms, R′ is a radical selected from the group consisting of alkyl radicals of from 1 to 10 inclusive carbon atoms, phenyl, vinyl, hydrogen atoms and —OR‴ radicals where R‴ is an alkyl radical containing from 1 to 4 carbon atoms, R″ is a radical selected from the group consisting of phenyl and —OR‴ radicals where R‴ is defined above and n is an integar of from 1 to 3 inclusive.

The antioxidant (2) is present in an amount from 0.1 to 5 percent by weight based upon the weight of the lubricating fluid. However, the preferred range of the antioxidant is from 0.5 to 3 percent by weight. There is generally very little improvement in the stability of the lubricating fluid when more than 2 percent of the antioxidant is added. R in the organosilicon antioxidant is an alkyl radical of from 1 to 10 inclusive carbon atoms.

Examples of suitable R radicals are methyl, ethyl, propyl, tert-butyl, octyl and decyl. R′ is an alkyl radical of from 1 to 10 inclusive carbon atoms, phenyl, vinyl, hydrogen atoms or —OR‴ radicals where R‴ is an alkyl radical containing from 1 to 4 carbon atoms. Since n is an integer of from 1 to 3 inclusive, there can be from 1 to 3 dialkylaminophenyl radicals per molecule. R″ is either a phenyl radical or an alkoxy radical, —OR‴, in which R‴ is a monovalent alkyl radical containing from 1 to 4 carbon atoms, such as methyl, ethyl, propyl, butyl or isopropyl radicals. R″ is preferably a phenyl radical.

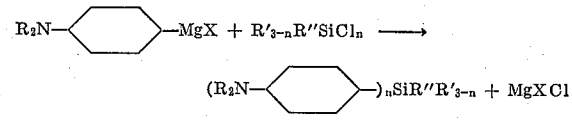
The antioxidants are prepared by first preparing a Grignard reagent and then reacting this reagent with a chlorosilane. The first reaction is represented by the equation:

(1)



The new antioxidants of this invention are obtained by reacting the above Grignard reagent with a chlorosilane in accordance with the following equation:

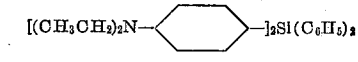
(2)

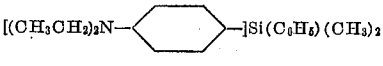


R, R′, R″ and n are defined above. X is any halogen atom. Alternatively, these antioxidants can be prepared by first preparing a lithium reagent by the reaction of a p-halogeno-N,N-dialkylaniline with lithium. The lithium reagent is then reacted with the proper chlorosilane.

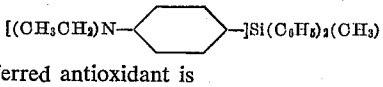
Tetrahydrofuran is a suitable solvent for both reactions. Reaction 1 can be conducted at a temperature slightly above room temperature. Reaction 2 is best carried out at reflux for a substantial period of time. The product can then be separated from the metal salts produced in reaction 2.

Although all of the antioxidants disclosed above are useful, the best antioxidants are those where R is ethyl, R″ is phenyl, R′ is either phenyl or methyl, preferably phenyl and n is an integer of from 1 to 2 inclusive. Examples of such compounds are

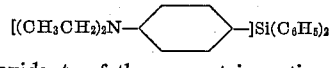




and



The preferred antioxidant is



The antioxidants of the present invention besides providing anti-oxidant properties and stabilizing the viscosity and the acidity of the high temperature lubricating fluids, particularly the esters of alkanoic acids, also have an unexpected property of preventing sludge formation in the high temperature lubricating fluids. Antioxidants which contain only alkyl radicals attached to the silicon atom of the silane other than the dialkylaminophenyl radicals, do not prevent sludge formation.

Although these antioxidants are useful in stabilizing high temperature organic lubricating fluids in general, the preferred lubricating fluids are esters of alkanoic acids and of polyhydric alcohols containing at least two methylol radicals bonded to a quaternary carbon atom. The

alkanoic acid must have at least 5 carbon atoms and preferably from 5 to 8 carbon atoms. Although the straight-chained alkanoic acids are preferred, the branched alkanic acids can also be used. Examples of suitable alkanoic acids are pentanoic, hexanoic, heptanoic and octanoic and 2,3-dimethylpentanoic acids. The polyhydric alcohols can also be esterified with a mixture of alkanoic acids.

The polyhydric alcohol contains at least two methylol groups on a quaternary carbon atom and can contain as many as four methylol groups on a quaternary carbon atom. The term "quaternary carbon atom" means that this carbon atom is bonded to four other carbon atoms. Thus, the carbon atom beta to the hydroxyl radical in these alcohols is not bonded to any hydrogen atoms. Esters of such alcohols have a higher resistance to oxidation than those that have a hydrogen atom on the carbon atom which is beta to the hydroxyl radical. Other examples of suitable polyhydric alcohols are 1,2,2-trimethylolpropane, 1,1,1-trimethylolethane, neopentylglycol, 2-butyl-2-ethyl-1,3-propanediol, and 2,2,4-trimethyl-1,3-pentanediol. The preferred ester is the triheptanoate of trimethylolpropane.

These esters are prepared by the reaction of the polyhydric alcohol with a slight excess of alkanoic acid. Although it is not necessary to use a catalyst, a suitable catalyst, such as p-toluene sulfonic acid, benzene sulfonic acid, zinc and lead salts can be employed. This reaction is preferably conducted at a temperature between 180° and 240° C. for a period between 6 and 14 hours. Water is eliminated by evaporation during the course of the reaction, preferably in the presence of an azeotropic agent, such as a fluid hydrocarbon.

Another type of suitable high temperature lubricating fluid are the esters of tert-alkylcarbinols and dicarboxylic acids. These carbinols also have a methylol radical on a quaternary carbon atom. Hence, there are no hydrogen atoms on the carbon atoms beta to the hydroxyl radical. Preferably the carbinol has from 5 to 12 inclusive carbon atoms. Examples of such carbinols are 2,2,4-trimethyl-1-pentanol, and 1-methyl-cyclohexylmethanol. Preferably the dicarboxylic acid has from 4 to 12 carbon atoms. Examples of suitable dicarboxylic acids are glutaric, adipic, suberic and sebacic acids. Sebacic and adipic acids are preferred. Specific examples of such esters are bis(2,2,4-trimethylpentyl)sebacate, bis(1-methylcyclohexyl)-methyl)sebacate and bis(1-methylcyclohexylmethyl) adipate. Methods for preparing these esters are described in "High-Temperature Lubricating Fluids," Blake, Edwards, Hammann, Reichard, Wright Air Development Command TR 54–532 Pt. 2 (January 1956).

The antioxidants of this invention are used in the same manner in these fluids as in the esters of alkanoic acids and polyhydric alcohols. The heat and oxidative stability of these fluids are also increased when the antioxidants of this invention are added to them.

The oxidative and heat stability of mixtures of lubricating fluids is also improved with the antioxidants of this invention. For example, the oxidative and heat stability of a mixture of a silicone lubricating fluid and one of the previously described esters is improved by the antioxidants of this invention.

Applicants have also discovered that a mixture of one of the dialkylaminophenylsilane antioxidants and a conventional amine antioxidant prevents viscosity increase and acidification in high temperature lubricating oils better than either of the compounds alone, even when the compounds are used in equivalent quantities. This is true of both classes of the esters disclosed above. The preferred lubricating fluids are the esters of polyhydric alcohols. Examples of some of the conventional antioxidants which can be used in combination with the previously described organosilicon antioxidants are; phenyl-α-naphthylamine, phenothiazine, phenyl-β-naphthylamine, n-ethyl-1-naphthylamine, 1-naphthylamine and dioctyldiphenylamine. Although a synergistic effect is obtained when any of these amines are used in combination with the organosilicon antioxidant, the best results are obtained with a combination of phenyl-α-naphthylamine and one of the organosilicon antioxidants previously described. For example, a triheptanoate of 1,2,2-trimethylolpropane which has been stabilized with a mixture of one percent phenyl-α-naphthylamine and one percent (p-diethylaminophenyl)triphenylsilane can resist a temperature of 218° C. for a period of time in excess of 30 hours. The synergistic effect obtained with these mixtures of antioxidants is believed to be due to their complementary characteristics. It is belived that the organosilicon compound functions at its best as an antioxidant at the highest temperatures and that the organic amine provides the basic stability at lower temperatures. However, the applicants do not wish to be bound by this particular theory. The mixture of antioxidants is used in the same range of weight limitations as set forth above for the organosilicon antioxidant, i.e. from 0.1 to 5 percent by weight based upon the weight of the lubricating fluid. It is preferable that from 0.5 to 3 percent by weight of the mixture be employed. The synergistic effect obtained by this mixture is obtained when from 20 to 80 percent by weight based on the weight of the antioxidant mixture is the organic amine and the remainder of the mixture is the organosilicon antioxidant. However, the best results are obtained when from 35 to 65 percent of the mixture is the organic amine and the remainder is the organosilicon antioxidant.

Both the organosilicon antioxidant and/or conventional antioxidants are merely added to the high temperature lubricating fluid. In some cases it may be desirable to heat the lubricating fluid in order to dissolve the antioxidant. When the mixture of antioxidants is used, they can be mixed together and the mixture added or they can be added separately to the lubricant.

The following examples are exemplary of the best method for the preparation of the compounds of this invention. However, other methods can be employed. In each case the reaction was carried out in a nitrogen atmosphere.

Example 1

50 ml. of tetrahydrofuran was added to a 500 ml. 3-necked flask equipped with condenser, stirrer, and dropping funnel. 10 g. (0.412 g. atom) of magnesium turnings was then added. 68.0 g. (0.298 mole) of p-bromo-N,N-diethylaniline was dissolved in 100 ml. of tetrahydrofuran. The reaction was primed twice by placing a few pieces of magnesium in a test tube with a few ml. of tetrahydrofuran and p-bromo-N,N-diethylaniline. Once the reaction had started in the test tube, the contents were dumped into the flask. The p-bromo-N,N-diethylaniline in tetrahydrofuran was then added over a 90 minute period. The pot temperature was maintained at about 30° to 40° C. during this time. The contents were then stirred for 45 minutes at room temperature. 51.2 g. (0.22 mole) of $(C_6H_5)_2(CH_3)SiCl$ was then dissolved in 50 ml. of tetrahydrofuran and the solution was added to the reaction mixture over a 30 minute period. The pot temperature was maintained at 30 to 35° C. during this time. The mixture was then refluxed for 7 hours and then stirred at room temperature for 11 hours.

A solution of 42.72 g. of NH_4Cl and 16 g. of NaOH in 800 ml. of H_2O was prepared and placed in a beaker containing ice. The reaction mixture was poured into this solution. The organic layer was separated and distilled at a reduced pressure of 0.20–0.22 mm. of Hg. 50.4 g. (65.4% yield) of a yellow liquid boiling at 202 to 208° C. was obtained. The yellow liquid crystallized to give a tan solid with a melting point of 68 to 70° C. The tan solid was recrystallized from ethanol and dried. 40.3 g. of (p-diethylaminophenyl)diphenylmethylsilane, a white solid with a melting point of 73° C., was obtained.

Example 2

The following organosilicon compounds were prepared in accordance with the procedure of Example 1. Any modifications of the procedure of Example 1 are set forth in the following paragraphs.

A. The compound (p-diethylaminophenyl)triphenylsilane was prepared by the reaction of 68.43 g. (0.30 mole) p-bromo-N,N-diethylaniline with 10 g. (0.41 g.-atom) of magnesium and 88.35 g. (0.30 mole) of triphenylchlorosilane. The mixture was refluxed for 19 hours and then hydrolyzed. The crude product was recrystallized from diethylether to yield 56.2 g. (45.2% yield) of the pure product, (p-diethylaminophenyl)triphenylsilane.

B. A compound bis(p-diethylaminophenyl)diphenylsilane was prepared by the reaction of 68 g. (0.298 mole) of p-bromo-N,N-diethylaniline with 6.25 g. (0.90 g.-atom) of lithium wire and 28.6 g. (0.113 mole) of diphenyldichlorosilane in diethylether. The ether was replaced by tetrahydrofuran and the reaction refluxed for 8 hours and then hydrolyzed. The crude product was recrystallized from a commercial aliphatic solvent to give 22.65 g. (41.9% yield of theoretical) of the pure product, bis(p-diethylaminophenyl)diphenylsilane.

C. The compound (p-diethylaminophenyl-phenyldimethylsilane was obtained by the reaction of 68 g. (0.298 mole) of p-bromo-N,N-diethylaniline with 10 g. (0.412 g.-atom) of magnesium turnings and 37.55 g. (0.22 mole) of phenyldimethylchlorosilane. The mixture was refluxed for seven and one-half hours and then hydrolyzed. The crude product was separated by distillation at reduced pressure (0.25 to 0.36 mm./Hg). Redistillation of the crude product at reduced pressure (0.1 mm./Hg) gave 32.2 g. (51.6% yield of theoretical) of the pure product, (p-diethylaminophenyl)phenyldimethylsilane.

D. The compound (p-diethylaminophenyl)diphenylvinylsilane was prepared by the reaction of 5.3 (0.22 g-atom) magnesium turnings, 46.0 g. (0.22 mole) diphenylvinylchlorosilane, and 45.6 g. (0.20 mole) p-bromo-N,N-diethylaniline. The diphenylvinylchlorosilane and the p-bromo-N,N-diethylaniline was mixed with 50 ml. of tetrahydrofuran and added to a mixture of the magnesium and 250 ml. of tetrahydrofuran over a two hour period. The temperature was maintained at 45° C. during the addition and the material was then refluxed for 45 minutes. The product was hydrolyzed and recrystallized from ethanol and then hexane. The melting point of the (p-diethylaminophenyl)diphenylvinylsilane was 67 to 70° C.

E. The compound (p-diethylaminophenyl)diphenylsilane was prepared by reacting 5.28 g. (0.22 mole) of magnesium turnings, 43.8 g. (0.20 mole) of diphenylchlorosilane and 47.9 g. (0.21 mole) of p-bromo-N,N-diethylaniline. The diphenylchlorosilane and the p-bromo-N,N-diethylaniline were mixed with 50 ml. tetrahydrofuran and added over a two hour period to the magnesium mixed with 100 ml. of tetrahydrofuran. The temperature during this period was 45° C. and the material was then refluxed for one hour. The product was hydrolyzed and recrystallized from hexane. The melting point of (p-diethylaminophenyl)diphenylsilane was 58 to 59° C.

F. The compound (p-diethylaminophenyl)methyldimethoxysilane was prepared by reacting 24.3 (1 g.-atom) of magnesium with 228 g. (2 moles) of p-bromo-N,N-diethylaniline. The Grignard product is then aded to 272 g. (2 moles) of methyltrimethoxysilane over a 40 minute period. The reactions were conducted in tetrahydrofuran. The temperature during reaction was 55° C. The tetrahydrofuran was stripped off and the product was replaced with toluene. The final product was distilled at 2 mm. of Hg and 125–130° C. The (p-diethylaminophenyl) methyldimethoxysilane was obtained in 39.4 yield.

Example 3

The antioxidant properties of the compounds prepared in the preceding examples when used alone or in combination with phenyl-α-naphthylamine in a lubricating fluid are measured in the following tables. The lubricating fluid in which these antioxidant properties were tested is the triheptanote of 1,2,2-trimethylolpropane. The antioxidant to be tested was merely added to the lubricating fluid in the quantity set forth in the following tables. It was necessary in some cases to heat the antioxidant-containing lubricating fluid in order to obtain a homogeneous solution.

In Table I, the stability of these lubricating fluids was tested by heating the fluid at 218° C. and then measuring the properties at the expiration of certain time periods. Air was bubbled through the lubricating fluid at a rate of 8 liters of dry air per hour per 20 g. of lubricating fluid. The air was bubbled into the fluid through a 1/16 in. (inside diameter) tube. At the end of various time periods the viscosity of the lubricating fluid was tested at 99° C. The acid number of the fluid was also checked at this time in accordance with ASTM D–974–58T (1958). The concentration of antioxidant is expressed in weight percent based upon the weight of the lubricating fluid. The percent increase in viscosity in each case is based upon the viscosity of the lubricating fluid prior to heating. The increase in acid number is merely the difference between the acid number prior to heating and the acid number after heating for a certain period of time.

TABLE I

Antioxidant	Time in hours	Visc. in cs. at 99° C.	Percent Increase in Viscosity	Acid No.	Increase in Acid Number
(1) None	0	3.53		0.017	
	24	10.05	185	23.1	23.1
	30	15.28	333	25.5	25.5
(2) 1% phenyl-α-naphthylamine	0	3.60		0.014	
	24	5.99	66.4	18.6	18.6
	30	7.19	100.0	19.3	19.3
(3) 2% phenyl-α-naphthylamine	0	3.66		0.02	
	24	4.85	32.5	7.2	7.2
(4) 1% (p-diethylaminophenyl)-triphenylsilane (Ex. 2A) and 1% phenyl-α-naphthylamine.	0	3.52		0.10	
	22	3.72	5.7	0.39	0.3
	30	3.68	4.5	0.45	0.4
(5) 1% (p-diethylaminophenyl)methyldiphenylsilane (Ex. 1) and 1% phenyl-α-naphthylamine.	0	3.65		0.10	
	24			0.50	0.40
(6) 0.5% bis(p-diethylaminophenyl)diphenylsilane (Ex. 2B) and 0.5% phenyl-α-naphthylamine.	0	3.52		0.056	
	30	4.74	35	12.7	12.6

The lubricating fluids tested in Table II were tested in the same manner as those tested in Table I except that the air was bubbled into the sample at a rate of 1 liter per hour per 20 g. sample. The viscosity and acid number were determined after 24 hours at 232° C. In Table II, the lubricating fluid without any additive had, prior to heating, a viscosity of 3.47 at 99° C. and an acid number of 0.28.

TABLE II

Additive	Visc. in cs. at 99° C.	Percent Viscosity Increase	Acid No	Acid No. Increase
(1) None	4.39	26.5	22.2	22.0
(2) 0.5% phenyl-α-naphthylamine	4.2	25.0	11.2	11.0
(3) 0.5% (p-diethylaminophenyl) triphenylsilane (Ex. 2A) and 0.5% phenyl-α-naphthylamine.	3.93	13.3	9.10	8.9
(4) 0.5% (p-diethylaminophenyl) triphenylsilane (Ex. 2A) and 0.5% phenyl-α-naphthylamine.	3.82	10.1	3.14	2.96
(5) 0.5% (p-diethylaminophenyl)diphenylmethylsilane (Ex. 1).	4.17	18.1	19.6	19.1
(6) 0.5% (p-diethylaminophenyl) methyldiphenylsilane (Ex. 1) and 0.5% phenyl-α-naphthylamine.	4.18	18.8	9.5	9.0
(7) 0.5% (p-diethylaminophenyl) phenyldimethylsilane (Ex. 2C).	4.11	16.6	16.8	16.4
(8) 0.5% (p-diethylaminophenyl)phenyldimethylsilane (Ex. 2C) and 0.5% phenyl-α-naphthylamine.	3.75	3.3	4.06	3.6

Example 4

The following products are obtained when the following reactants are reacted in accordance with the procedure of Example 1. When these products are added to a triheptanoate of 1,2,2-trimethylolpropane lubricating fluid at the following weight concentrations based upon the weight of the lubricating fluid, the oxidative and heat stability of the lubricating fluid is increased.

TABLE III

Grignard Reagent	Silane	Product	Concentration in lubricating fluid
(1) 0.30 mole $(C_8H_{17})_2N$—⟨⟩—MgX	0.15 mole $(C_6H_5)(CH_2{=}CH)SiCl_2$	$[(C_8H_{17})_2N$—⟨⟩—$]_2Si(C_6H_5)(CH{=}CH_2)$	1.0
(2) 0.45 mole $(C_4H_9)_2N$—⟨⟩—MgX	0.15 mole $(C_6H_5)SiCl_3$	$[(C_4H_9)_2N$—⟨⟩—$]_3Si(C_6H_5)$	0.5
(3) 0.30 mole $(C_3H_7)_2N$—⟨⟩—MgX	0.15 mole $(C_6H_5)Si(OC_3H_7)_3$	$[(C_3H_7)_2N$—⟨⟩—$]_2Si(C_6H_5)(OC_3H_7)$	0.2
(4) 0.15 mole $(C_6H_{11})_2N$—⟨⟩—MgX	0.15 mole $(C_6H_5)(C_6H_{13})_2SiCl$	$(C_6H_{11})_2N$—⟨⟩—$Si(C_6H_{13})_2(C_6H_5)$	1.5
(5) 0.15 mole $(C_{10}H_{21})_2N$—⟨⟩—MgX	0.15 mole $(C_6H_5)_2(C_2H_5)SiCl$	$(C_{10}H_{21})_2N$—⟨⟩—$Si(C_6H_5)_2(C_2H_5)$	2.0
(6) 0.30 mole $(C_2H_5)_2N$—⟨⟩—MgX	0.15 mole $(CH_2{=}CH)Si(OC_2H_5)_3$	$[(C_2H_5)_2N$—⟨⟩—$]_2Si(OC_2H_5)(CH_2{=}CH)$	5.0
(7) 0.15 mole $(C_2H_5)_2N$—⟨⟩—MgX	0.15 mole $(C_6H_5)_2(C_{10}H_{21})SiCl$	$(C_2H_5)_2N$—⟨⟩—$Si(C_6H_5)_2(C_{10}H_{21})$	0.8
(8) 0.30 mole $(C_2H_5)_2N$—⟨⟩—MgX	0.15 mole $(C_6H_5)(C_4H_9)SiCl_2$	$[(C_2H_5)_2N$—⟨⟩—$]_2Si(C_6H_5)(C_4H_9)$	2.5

Example 5

Comparable results are obtained when any of the following high temperature lubricating fluids are substituted for the triheptanoate of 1,2,2-trimethylolpropane used in Example 3.

A. triheptanoate of 1,1,1-trimethylolethane.
B. tripentanoate of 1,2,2-trimethylolpropane.
C. tetraheptanoate of pentacrythriotol.
D. diheptanoate of 2-butyl-2-ethyl-1,3-propylenediol.
E. dioctanoate of 2,2,4-trimethyl-1,3-pentanediol.
F. diheptanoate of neopentylglycol.
G. bis(2,2,4-trimethylpentyl)sebacate.
H. bis(1-methylcyclohexylmethyl)sebacate.
I. bis(1-methylcyclohexylmethyl)adipate.

Example 6

The heat and oxidative stability of triheptanoate of 1,2,2-trimethylolpropane lubricating fluid is increased when any of the following combinations of antioxidants are added to the fluid. All percentages are weight percentages based upon the weight of the lubricating fluid.

A. 2.5 percent phenyl-α-naphthylamine and 2.5 percent bis(p-diethylaminophenyl)diphenylsilane.

B. 1.5 percent phenyl-β-naphthylamine and 1.5 percent (p-diethylaminophenyl)triphenylsilane.

C. 0.5 percent phenothiazine and 0.5 percent (p-diethylaminophenyl)diphenylsilane.

D. 0.5 percent n-ethyl-1-naphthylamine and 1 percent (p-diethylaminophenyl)methyldimethoxysilane.

E. 1.5 percent 1-naphthylamine and 0.5 percent (p-diethylaminophenyl)diphenylvinylsilane.

F. 1 percent dioctyldiphenylamine and 1 percent bis(p-dipropylaminophenyl)phenylpropoxysilane.

Example 7

The oxidative and heat stability of a high temperature lubricating fluid composed of 50 percent by weight of the

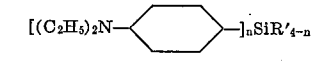
triheptanoate of 1,2,2-trimethylolpropane and 50 percent by weight of a silicone lubricating fluid composed of 70 mol percent trimethylsilyl-endblocked dimethylsiloxane and 30 mol percent phenylmethylsiloxane is improved when any of the following antioxidants are added at the following weight concentrations based upon the weight of the lubricating fluid.

A. 1 percent (p-diethylaminophenyl)triphenylsilane.

B. A mixture of 0.5 percent phenyl-α-naphthylamine and 0.5 percent (p-diethylaminophenyl)triphenylsilane.

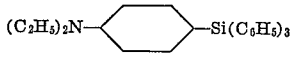
We claim:

1. A silane of the formula

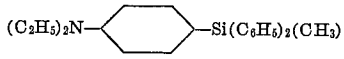


in which *n* is an integer from 1 to 2 inclusive, at least one R′ is phenyl and any remaining R′ groups are methyl radicals.

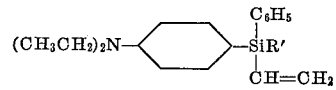
2. A silane of the formula



3. A silane of the formula

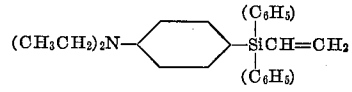


4. A silane of the formula



wherein R′ is a radical selected from the group consisting of alkyl radicals of from 1 to 10 inclusive carbon atoms, phenyl, vinyl, hydrogen atoms and —OR‴ radicals where R‴ is an alkyl radical containing from 1 to 4 carbon atoms.

5. A silane of the formula



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TOBIAS E. LEVOW, *Primary Examiner.*

J. G. LEVITT, P. F. SHAVER, *Assistant Examiner.*