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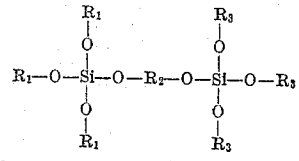
SILICON ESTERS

Andrew D. Abbott, Ross, and Robert O. Bolt, San Rafael, Calif., assignors to California Research Corporation, San Francisco, Calif., a corporation of Delaware

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6 Claims. (Cl. 260—448.8)

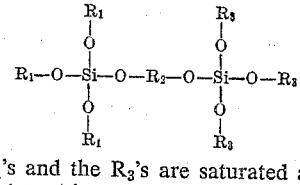
This invention relates to the preparation of novel silicon ester compounds. These compounds have excellent viscosity and temperature-viscosity characteristics and are well adapted for employment as hydraulic fluids and for other lubricant purposes.

The compounds of this invention are of the type having the general formula:

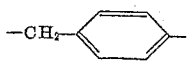


where the R_1's and the R_3's are saturated aliphatic radicals of from 1 to 16 carbon atoms each and R_2 is a divalent hydrocarbon radical of from 2 to 18 carbon atoms, at least two of which are in a chain between the adjacent oxygen atoms, which is free of aliphatic unsaturation.

Certain preferred compounds of this invention within the above description are those of the types having the general formula



where the R_1's and the R_3's are saturated aliphatic radicals of from 1 to 16 carbon atoms each and R_2 is a divalent hydrocarbon radical of from 3 to 18 carbon atoms, at least three of which are in a chain between the adjacent oxygen atoms, which is free of aliphatic unsaturation. The various R_1's may represent the same or different radicals, and the same is true with respect to the R_3 radicals. Representative R_1 and R_3 radicals are methyl, ethyl, isopropyl, sec.-butyl, tert.-butyl, amyl, isoamyl, tert.-amyl, cyclopentyl, cyclohexyl, 2-ethylbutyl, 2-ethylhexyl, decyl, dodecyl, and the like, while representative R_2 radicals are 1,3-propylene, 1,3- and 1,4-butylene, 1,3-, 1,5- and 2,4-pentylene, 1,4- and 2,6-hexylene, 1,8-octylene, 1,10-decylene, 1,3- and 1,4-cyclohexylene, 1,3- and 1,4-phenylene,
and 3,5-tolylene.



and 3,5-tolylene.

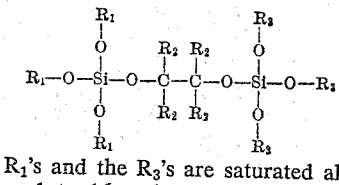
The silicate esters of the above type according to this invention have a low pour point and good viscosity and viscosity index properties, coupled with low volatility even at high temperatures. Thus, the present compounds are adapted to be used at temperatures even below −65° F., while their viscosity at 210° F. is unusually good, i.e., from about 1.5 to 10 cs. The ASTM slope of the viscosity-temperature curves of the present compounds is from about 0.61 to 0.70 as measured from 100° to 210° F., while relatively low slope values are indicative of correspondingly high viscosity-index qualities.

In view of the foregoing properties, and since the present compounds of the above type are somewhat combustion resistant, it is obvious that said compounds are well adapted to be used as synthetic lubricants, and

particularly as hydraulic fluids. However, in those applications where it is important that the silicate ester be highly resistant to deterioration in the presence of water, those compounds should be chosen wherein at least one each of said R_1 and R_2 radicals in the structural formula given above is a secondary, tertiary, or branched-chain primary alkyl radical of from 3 to 16 carbon atoms, and more preferably, all the R_1 and R_3 radicals are of this character. As will be indicated in Table I given below, the silicon esters of the present invention which incorporate such preferred groups have greatly superior hydrolytic stability charatceristics. It is also preferred that R_2 be a straight or branched-chain alkylene group of from 3 to 18 carbon atoms, at least three of which are in a chain between the adjacent oxygen atoms. The following compounds are representative of those which fall into the preferred category:

1,3-bis[tri-(2-butoxy)siloxy]propane
1,4-bis[tri(2-butoxy)siloxy]butane
1,4-bis(di-tert.-butoxy-2-ethylbutoxy siloxy)butane
2-(dimethoxy tert.-butoxy siloxy)-5-(dipropoxy-2-butoxy siloxy)-hexane
1,4-bis[tri(2-butoxy)siloxy]pentane
2,4-bis[tri(2-butoxy)siloxy]pentane
1,6-bis[tri(2-butoxy)siloxy]hexane
1,6-bis[di(2-butoxy)(tert.-butoxy)siloxy]hexane
1,4-bis[tri(2-ethyl-1-butoxy)siloxy]butane
2,4-bis[tri(2-ethyl-1-butoxy)siloxy]pentane
2,5-bis[tri(2-ethyl-1-butoxy)siloxy]hexane
1,5-bis[tri(2-butoxy)siloxy]-2-methylpentane
2,4-bis[tri(2-butoxy)siloxy]-2-methylpentane
1,10-bis[tri(2-butoxy)siloxy]decane
1-tri(2-ethylhexoxy)siloxy-7-tri-(tert.-butoxy)-siloxy-4-isobutylheptane
1-(dipropoxy-2-butoxy siloxy)-4-(triisopropoxy siloxy)-pentane

Still other compounds of this invention within the above general formula can be described as the 1,2-bis-[tri(alkoxy)siloxy]alkanes and are of the type having the general formula



where the R_1's and the R_3's are saturated aliphatic radicals of from 1 to 16 carbon atoms each and the R_2's, which may be the same or different, are hydrogen atoms or alkyl groups of from 1 to 4 carbon atoms each. The various R_1's may represent the same or different radicals, and the same is true with respect to the R_3 radicals. Representative R_1 and R_3 radicals are methyl, ethyl, isopropyl, sec.-butyl, tert.-butyl, amyl, isoamyl, tert.-amyl, cyclopentyl, cyclohexyl, 2-ethylbutyl, 2-ethylhexyl, decyl, dodecyl, and the like, while representative R_2 radicals, in addition to the hydrogen atom, are methyl, ethyl, n-propyl, isopropyl, n-butyl, sec.-butyl and tert.-butyl. Thus, exemplary compounds of this type coming within the scope of this invention are:

1,2-bis[tri(ethoxy)siloxy]ethane
1-[tri(isopropoxy)siloxy]-2-[tri(2-butoxy)-siloxy]-propane
1-[dimethoxy)(ethoxy)siloxy]-2-[tri-isopropyoxy]-ethane
2,3-bis[tri(2-butoxy)siloxy]pentane
2,3-bis[tri(2-ethyl-1-butoxy)siloxy]-4-tert.-butyl pentane
2,3-bis[tri(tert.-butoxy)siloxy]butane
2,3-bis[di(tert.-butoxy)(ethoxy)siloxy]butane
1-[tri(2-butoxy)siloxy]-2-[tri-(tert.-butoxy)-siloxy]-propane

1,2-bis[tri(tert.-butoxy)siloxy]ethane
1,2-bis[tri(tert.-amoxy)siloxy]ethane

The silicate esters of the above type according to this invention also have a low pour point and good viscosity and viscosity index properties, coupled with low volatility even at high temperatures. Thus, the present compounds are adapted to be used at temperatures as low as −65° F., while their viscosity at 210° F. is unusually good, i.e., from about 2.2 to 3.0 cs. The ASTM slope of the viscosity-temperature curves of the present compounds is from about 0.65 to 0.70 as measured from 100° to 210° F., which relatively low slope values are indicative of correspondingly high viscosity-index qualities.

In view of the foregoing properties, and since the present compounds of the above type are also highly combustion resistant, it is obvious that said compounds are useful as synthetic lubricants, and particularly as hydraulic fluids. However, in those applications where it is important that the silicate ester be highly resistant to deterioration in the presence of water, those compounds should be chosen wherein at least one each of said R_1 and R_3 radicals in the structural formula given above is a secondary, tertiary, or branched-chain primary alkyl radical of from 3 to 16 carbon atoms, preferably a tertiary alkyl radical of from 4 to 16 carbon atoms, and more preferably, all the R_1 and R_3 radicals are of this character. As will be indicated in Table II given below, the silicon esters of the present invention which incorporate such preferred groups have greatly superior hydrolytic stability characteristics. The following compounds are representative of those which fall into the preferred category:

1,2-bis[tri(tert.-butoxy)siloxy]ethane
1,2-bis[tri(tert.-amoxy)siloxy]ethane
1,2-bis[tri(1-ethyl-1-methyl pentoxy)-siloxy]ethane
1,2-bis[di(tert.-butoxy)(2-butoxy)siloxy]-ethane
1-[tri(tert.-butoxy)siloxy]-2-[di(ethoxy)-tert.-butoxy) siloxy]ethane
1-[tri(tert.-butoxy)siloxy]-2-[tri(tert.-amoxy)siloxy] propane
1,2-bis[tri(tert.-butoxy)siloxy]propane
1,2-bis[tri(tert.-butoxy)siloxy]butane
2,3-bis[tri(tert.-butoxy)siloxy]butane
2,3-bis[tri(1,1,3-trimethylbutoxy)siloxy]pentane
1,2-bis[tri(tert.-butoxy)siloxy]pentane
1,2-bis[tri(tert.-butoxy)siloxy]hexane
3,4-bis[tri(tert.-butoxy)siloxy]hexane

The ester compounds of this invention can readily be prepared by the application of conventional methods of organic syntheses. As a general rule, however, the first step is to react the desired primary, secondary and/or tertiary alcohol with silicon tetrachloride in the molar proportions required to form the trialkoxymonochlorosilane. With primary and secondary alcohols this reaction readily takes place even at temperatures below 0° F., while with tertiary alcohols, the recommended procedure is to add the alcohol along with pyridine to a cooled solution of $SiCl_4$ in toluene (preferably maintained below 0° F. to minimize loss of $SiCl_4$ by evaporation), with the reaction then being completed by refluxing the mixture. When the R_1 and R_3 groups are to be the same, the ester end product can then be formed by condensing the trialkoxymonochlorosilane with the desired glycol (preferably in the presence of pyridine, ammonia or other acid acceptor). When the R_3 groups are to be different from the R_1 groups, a possible procedure is to first react one-half of the hydroxy groups of the glycol with one of the trialkoxychlorosilane compounds, with the remaining portion of said hydroxy groups then being condensed with other trialkoxychlorosilane derivative. However, since mixtures of esters of the type described herein are highly useful, the preferred practice when employing different trialkoxychlorosilane compounds is to react the same simultaneously with the glycol. The mixture of di-(trialkoxysiloxy)alkanes (assuming an alkylene glycol reactant, for example) formed in this manner can either be used as such, or it can be separated into two or more of its component fractions, with each separated portion then being used for hydraulic purposes or otherwise.

While, as indicated above, the silicon esters of this invention can be used alone for hydraulic fluid and other purposes, good results are also obtained with compositions wherein the present esters are blended with a minor percentage of other additive compounds. Thus, the oxidative stability of the present esters can be increased by the use of from 0.1 to 10% by weight of an anti-oxidant, e.g., phenyl-α-naphthylamine, p-hydroxydiphenylamine, tertiary butylcatechol or the like. Improved anti-wear, or extreme pressure, qualities can be imparted by the addition of from 0.1 to 10% of a material of the type of tricresyl phosphate, for example. Further, the viscosity characteristics of the present esters can be improved by the addition of from 0.1 to 10% of a polymeric viscosity index improver such as, for example, a polymethacrylate (Acryloid), a silicon such as dimethyl-, diethyl- or methyl phenyl silicone, or an alkoxypolysiloxane. The composition can also be blended with minor amounts of other synthetic oils, such as the poly-1,2-oxyalkylene glycols and the ether and/or ester derivatives thereof. Thus, use of from 1 to 20% of a polypropylene glycol (mole weight about 800) imparts improved stability characteristics to compositions prepared from the present silicon esters and a conventional viscosity index improver of the type noted above. As rust inhibitors, there can be added from 0.1 to 10% of any one of several compounds, including metal sulfonates such as zinc dipolypropene-benzene sulfonate, sodium white oil-benzene sulfonate, and the like. Particularly attractive hydraulic fluids are those which incorporate a total of from 70 to 98% by weight of the present silicon esters, together with 0.1 to 10% each of a viscosity index improver, a rust inhibitor and (optionally) an anti-wear agent.

The examples given below illustrate methods by which the compounds of the preferred type according to the present invention as described above can be prepared, Example I giving a typical preparation of a trialkoxymonochlorosilane intermediate.

EXAMPLE I

Tri-(2-Butoxy)Chlorosilane

In this operation 1 mole of $SiCl_4$ was charged to a flask and cooled to −15° C. To the flask was then slowly introduced, with stirring, 3.2 moles of 2-butanol, while maintaining the temperature at −10° C. When the addition of the alcohol was complete, the temperature was allowed to rise to about 20° C. and the agitation continued for three hours, with nitrogen being bubbled through the mixture to carry off the HCl formed during the reaction. The crude product was then distilled in vacuo, with the tri-(2-butoxy)chlorosilane being recovered in 78% yield as the fraction boiling at 74–80° C. at 2.5 mm. Hg.

EXAMPLE II

2,4-Bis[Tri(2-Ethyl-1-Butoxy)Siloxy]Pentane

In this operation, 0.184 mole 2-4-pentanediol, 0.51 mole pyridine and 225 ml. mixed xylenes were charged to a flask. To this solution 0.41 mole of tri(2-ethyl-1-butoxy)chlorosilane was added slowly with agitation while the temperature of the reaction mixture was maintained at −15° C. The resulting mixture was then heated to 100° C. for about one hour, cooled and filtered. The filter cake was twice reslurried with benzene and filtered. The crude product was washed with water and distilled in vacuo, the desired 2,4-bis[tri(2-ethyl-1-butoxy)siloxy] pentane product being recovered in 95% yield as the fraction boiling at 241–245° C. at 1 mm. Hg.

EXAMPLE III

1,4-Bis[Tri(2-Butoxy)Siloxy]Butane

In preparing the above compound, there were charged

to a flask 0.62 mole of tri(2-butoxy)chlorosilane and 75 ml. of mixed xylenes. To this mixture was slowly added 0.275 mole of 1,4-butanediol and 0.75 mole pyridine, the reaction temperature being maintained at about 20° C. The mixture was then refluxed for two hours, after which the product was cooled, filtered, and the filter cake washed with xylene to separate any portion of the desired product remaining in the solid pyridine hydrochloride. The filtrate and the xylene washings were then combined and distilled, with the 1,4-bis[tri(2-butoxy)-siloxy] butane being recovered in 94% of the theoretical yield as the fraction boiling at about 185–190° C. at 1 mm. Hg.

EXAMPLE IV

1,4-Bis[Tri(2-Ethyl-1-Butoxy)Siloxy]Butane

This operation was conducted using the same general method and conditions as described in Example III above, there being employed 0.41 mole of tri(2-ethyl-1-butoxy) chlorosilane, 0.184 mole of 1,4-butanediol, 0.51 mole pyridine, and 225 ml. of mixed xylenes. The desired product compound had a boiling point of 255–257 at 1 mm. Hg.

EXAMPLE V

1,4-Bis[Mixed Tri(2-Ethyl-1-Butoxy)Siloxy and Tri(2-Butoxy)Soloxy]Butane

In carrying out this operation, 0.2 mole of tri(2-butoxy)chlorosilane and 0.2 mole of tri(2-ethyl-1-butoxy)chlorosilane are charged to a flask along with 180 ml. of mixed xylenes. To the flask was then slowly added a mixture of 0.184 mole of 1,4-butanediol, 0.51 mole of pyridine and 50 ml. of mixed xylenes, the temperature of the reaction mixture being held at about 23° C. during the addition period. The mixture was then refluxed for two hours, cooled and filtered. The filtrate was then distilled, with the desired mixture of 1,4-bis [tri(2-ethyl - 1 - butoxy)siloxy]butane, 1,4-bis[tri(2-butoxy)siloxy]butane being recovered in 85% yield as the fraction boiling between about 185° C. and 260° C. at 1 mm. Hg.

The data given in Table I which follows show the physical properties of a number of different silicon esters coming within the scope of the present invention according to a preferred embodiment as illustrated by the above examples. The items in the table are self-explanatory with the exception of the column marked "Hydrolytic Stability." Hydrolytic stability was measured by contact—the compounds with refluxing distilled water and observing for hazing or precipitation as evidence of decomposition. The time required for decomposition to become evident was taken as the measure of hydrolytic stability and is so reported in the table to follow.

In further illustration of still other compounds of the present invention and methods by which they are prepared, the following additional examples are given, Example VI being included as a typical preparation of a tri-(tert.-alkoxy)monochlorosilane.

EXAMPLE VI

Tri-(Tert.-Butoxy)Chlorosilane

In this operation 340 grams of silicon tetrachloride was placed in a flask, and to this liquid was then slowly added, over a two-hour period, a mixture of 450 grams of tert.-butanol, 720 grams of pyridine and 300 ml. of mixed xylenes, the reaction mixture being maintained at a temperature of about 0° C. during this period. The contents of the flask were then heated to 100° C. for 4 hours, after which the mixture was combined with 500 ml. benzene, filtered, and stripped of its low boiling components under reduced pressure (50 mm. Hg). The residue was now fractionated, and 190 grams of the desired tri-(tert.-butoxy)chlorosilane product was recovered as the portion boiling at about 46.7° C. at 1 mm. Hg.

EXAMPLE VII

1,2-Bis[Tri-(Tert.-Butoxy)Siloxy]Ethane

To a mixture of 75 grams of tri-(tert.-butoxy)chlorosilane and 40 ml. of xylene was added dropwise a mixture of 7 grams of ethylene glycol and 25 grams of pyridine. During this addition period the temperature rose to 62° C. The mixture was now heated at 100° C. for 4.5 hours, after which it was cooled, filtered, and distilled in vacuo to yield 35 grams of the desired 1,2-bis[tri-(tert.-butoxy)siloxy]ethane product as the portion boiling at 148.9° C. at 1 mm. Hg. This product had a refractive index (n^{20}/D) of 1.4074, a density (D 20/4) of 0.9361, and a viscosity at 100 and 210° F. of 19.7 cs. and 3.96 cs., respectively.

EXAMPLE VIII

1,2-Bis[Tri-(2-Butoxy)Siloxy]Ethane

In this operation, 300 grams of tri-(2-butoxy)chlorosilane and 100 grams of xylene were placed in a flask, and to the contents of the flask was slowly added a mixture of 30 grams of ethylene glycol and 100 grams of pyridine. The resulting mixture was then refluxed for 3 hours, after which it was cooled, filtered and distilled in vacuo. The desired 1,2-bis[tri-(2-butoxy)siloxy]ethane product was recovered in 50% yield as the portion boiling at 147–148° C. at 1 mm. Hg. It had a density (D 20/4) of 0.9523, a refractive index (n^{20}/D) of 1.4172, viscosities at −65° F., 100° F. and 210° F. of 480 cs., 6.5 cs. and 2.37 cs., and an ASTM slope (−65 to 210° F.) of 0.65.

TABLE I.—SUMMARY OF PHYSICAL PROPERTIES

Compound	Boiling Pt., ° C., at 1 mm. Hg	Density 20/4	Refractive Index n^{20}/D	Viscosity—Centistokes at ° F.				ASTM Slope		Hydrolytic Stability (Hrs.)
				−65	−40	100	210	−65 to 210° F.	100 to 210° F.	
1,4-Bis[tri(2-butoxy)-siloxy]butane	188	0.9340	1.4184	407	127	5.92	2.18	0.66	0.67	168
2,4-Bis[tri(2-butoxy)-siloxy]pentane	174	0.9285	1.4193	1,701	-------	7.94	2.56	-------	0.69	68
1,4-Bis[tri(n-butoxy)-siloxy]butane	180	-------	-------	-------	-------	-------	-------	-------	-------	<0.5
1,3-Bis[tri(2-butoxy)-siloxy]propane	163	0.9449	-------	347	-------	-------	2.22	0.68	-------	40
1,5-Bis[tri(n-propoxy)-siloxy]pentane	155	-------	-------	-------	-------	-------	-------	-------	-------	<0.5
2,4-Bis[tri(2-ethyl-1-butoxy)siloxy]pentane	241-245	0.9223	1.4349	3,680	572	11.1	3.45	0.66	0.62	[1] 48
2,5-Bis[tri(2-ethyl-1-butoxy)siloxy]hexane	274	0.9226	1.4359	2,790	616	11.8	3.66	0.63	0.61	[1] >240
1,6-Bis[tri(2-butoxy)-siloxy]hexane	197	0.9305	1.4208	673	184	6.88	2.44	0.66	0.65	168
1,5-Bis[tri(2-butoxy)-siloxy]-2-methylpentane	182	0.9329	1.4212	1,506	392	9.35	2.95	0.65	0.65	[1] >400
2,4-Bis[tri(2-butoxy)-siloxy]-2-methylpentane	177	-------	-------	2,113	481	9.55	2.98	0.67	0.66	-------
1,10-Bis[tri(2-butoxy)-siloxy]decane	227	0.9229	1.4258	1,530	382	9.96	3.24	0.63	0.62	[1] 48
1,3-Bis[tri(2-butoxy)-siloxy]benzene	203	0.9749	1.4431	1,154	280	7.54	2.55	0.68	0.66	72
1,4-Bis[tri(2-butoxy)-siloxy]benzene	215	0.9757	1.4430	1,377	310	8.47	2.83	0.66	0.64	>120
1,3-Bis[tri(2-propoxy)-siloxy]benzene	156	0.9857	1.4338	320	96	4.63	1.74	0.72	-------	>120
1,4-Bis[tri(2-propoxy)-siloxy]benzene	152	0.9858	1.4329	-------	-------	5.38	1.96	-------	0.70	66

[1] With 1% phenyl-α-naphthylamine antioxidant.

EXAMPLE IX

1,2-Bis[Tri-(2-Butoxy)Siloxy]Propane

In carrying out this preparation, 400 grams of tri(2-butoxy)chlorosilane and 100 grams of xylene were placed in a flask, and to this mixture was slowly added 120 grams of pyridine and 47 grams of 1,2-propylene glycol, the temperature rising to 70° C. during the addition. The mixture was then refluxed for 10 hours, cooled, filtered and distilled in vacuo, 122 grams of the desired 1,2-bis[tri(2-butoxy)siloxy]propane product being recovered as the portion boiling at 162° C. at 1 mm. Hg. This compound has a density (D 20/4) of 0.9380, a refractive index (n^{20}/D) of 1.4158, viscosities at −65° F., 100° F. and 210° F. of 476 cs., 5.88 cs. and 2.22 cs., and an ASTM slope (−65 to 210° F.) of 0.66.

EXAMPLE X

2,3-Bis[Tri-(2-Butoxy)Siloxy]Butane

In this operation there was followed the same general procedure as described in Example IX except that there was employed 2,3-butanediol instead of the propylene glycol. The desired product (recovered in 44% yield) had a boiling point of 162° C. at 1 mm. Hg., a density (D 20/4) of 0.9368, a refractive index (n^{20}/D) of 1.4180, viscosities at −65° F., 100° F. and 210° F. of 1897 cs., 9.61 cs. and 2.84 cs., and an ASTM slope (−65 to 210° F.) of 0.68.

In Table II below a comparison is made as regards the hydrolytic stability of various of the silicate esters of the above type coming within the scope of this invention. This determination was made, as in the previous test described above, by contacting the compounds with refluxing distilled water and observing for hazing or precipitation as evidence of decomposition. The time required for decomposition to become evident was taken as the measure of hydrolytic stability.

TABLE II.—HYDROLYTIC STABILITY

Compound	Hydrolytic Stability (Hours)
1,2-Bis[tri(2-ethyl-1-butoxy)-siloxy]ethane	0.5
1,2-Bis[tri(tert.-butoxy)siloxy]-ethane	48
1,2-Bis[tri(2-butoxy)siloxy]ethane	0.5
1,2-Bis[tri(2-butoxy)siloxy]propane	24
2,3-Bis[tri(2-butoxy)siloxy]butane	24
1,2-Bis[tri(tert.-butoxy)siloxy]-propane	57
1,2-Bis[di(2-butoxy)(tert.-butoxy)-siloxy]butane	45
1,2-Bis[di(2-butoxy)(tert.-butoxy)-siloxy]hexane	38
1,2-Bis[tri(tert.-butoxy)siloxy]hexane	55
1,2-Bis[tri(tert.-amoxy)siloxy]ethane	45
1,2-Bis[tri(2-ethyl-2-pentoxy)siloxy]-ethane	51

This application is a continuation-in-part of Abbott and Bolt application Serial No. 352,596, filed May 1, 1953, now abandoned.

We claim:

1. Silicon esters of the group consisting of
   1,4-bis[tri(2-butoxy)siloxy]butane,
   2,4-bis[tri(2-butoxy)siloxy]pentane,
   1,6-bis[tri(2-butoxy)siloxy]hexane,
   1,4-bis[tri(2-butoxy)siloxy]benzene, and
   1,3-bis[tri(2-propoxy)siloxy]benzene.
2. 1,4-bis[tri(2-butoxy)siloxy]butane.
3. 2,4-bis[tri(2-butoxy)siloxy]pentane.
4. 1,6-bis[tri(2-butoxy)siloxy]hexane.
5. 1,4-bis[tri(2-butoxy)siloxy]benzene.
6. 1,3-bis[tri(2-propoxy)siloxy]benzene.

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