Acyl Iodides in Organic Synthesis:
IX. Cleavage of the Si–O–C and Si–O–Si Moieties

M. G. Voronkov, A. A. Trukhina, L. I. Belousova, G. A. Kuznetsova, and N. N. Vlasova

Favorskii Irkutsk Institute of Chemistry, Siberian Division, Russian Academy of Sciences,
ul. Favorskogo 1, Irkutsk, 664033 Russia
*e-mail: voronkov@irioch.irk.ru*

Abstract—Reactions of acyl iodides RCOI (R = Me, Ph) with organosilicon compounds involve cleavage of the Si–O–C and Si–O–Si fragments. Acetyl iodide reacts with alkyl(alkoxy)silanes with evolution of heat, and cleavage of the Si–O bond results in the formation of oligo- or polysiloxanes, alkyl iodides, and alkyl acetates. 1,3-Diacetoxytetramethyldisiloxane is formed in the reaction of acetyl iodide with dimethoxy(dimethyl)silane. Acyl iodides readily react with 1-ethoxysilatrane to give 1-acyloxysilatranes as a result of cleavage of the C–O bond. The reaction of acetyl iodide with hexaethyldisiloxane yields triethylsilyl acetate and triethyliodosilane, while in the reaction with octamethyltrisiloxane iodo(trimethyl)silane and dimethyl(trimethylsiloxy)silyl acetate are obtained.

We previously showed that reactions of trialkyl-iodosilanes R_3SiI (R = Me, Et) with alkoxysilanes [1–4] and oligo- and poly(peralkoxysiloxanes) [1, 5, 6] involve cleavage of the Si–O–C moiety and that their reactions with hexaalkyldisiloxanes [1, 7, 8] are accompanied by cleavage of the Si–O–Si fragment (Scheme 1).

Scheme 1.
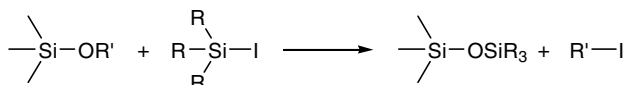
It is known that acyl halides RCOX (X = F, Cl) [9–11] are also capable of splitting the Si–O–C and Si–O–Si moieties in alkoxysilanes and cyclosiloxanes. However, these reactions generally require fairly severe conditions (e.g., increased pressure) and the presence of catalysts ($FeCl_3$, pyridine, etc.) and lead to the formation of the corresponding organosilicon halides and acyloxy derivatives. The Si–O–C fragment
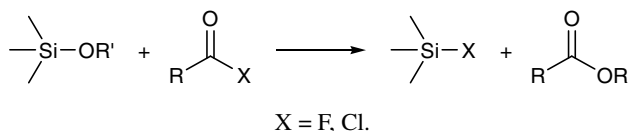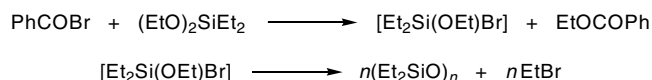
is cleaved by acyl halides much more readily than the Si–O–Si fragment (Scheme 2).

Unlike the above reactions, acyl bromides RCOBr (R = Me, Ph) react with alkyl(alkoxy)silanes of the general formula $R_{4-n}Si(OR')_n$, (n = 2–4) to give polysiloxanes [12]. Presumably, labile alkyl(alkoxy)bromosilanes are formed as intermediates (Scheme 3).
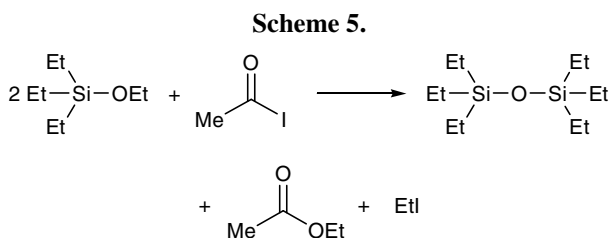
Scheme 3.

In continuation of our studies on acyl iodides as equivalents of iodo(trimethyl)silane in deoxygenation and iodination of oxygen-containing organic and heteroelement compounds [12–17], we examined reactions of acetyl iodide with alkyl(alkoxy)silanes $R_{4-n}Si(OR')_n$ (R, R' = Me, Et; n = 1–4) and 1-ethoxy-silatrane $EtOSi(OCH_2CH_2)_3N$. In contrast to RCOX

Scheme 2.


X = F, Cl.

Scheme 4.

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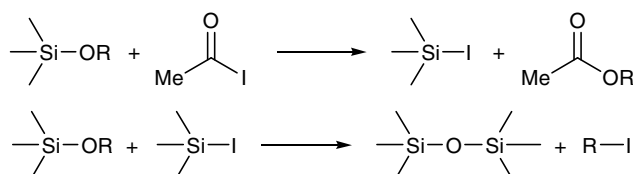
(X = F, Cl, Br), acetyl iodide (X = I) readily reacted with alkyl(alkoxy)silanes according to Scheme 4. The reactions involved cleavage of the Si–O bond and were accompanied by heat evolution.

The first step of the process is formation of quite reactive alkyl(alkoxy)iodosilanes, and their subsequent reactions with initial alkyl(alkoxy)silanes give oligo- and polysiloxanes. The absence of acetoxysilanes among the products indicates that just the Si–O rather than C–O bond is cleaved. Thus the behavior of acetyl iodide differs from that of iodo(trimethyl)silane which reacts at the C–O bond of alkyl(alkoxy)silanes [3, 4, 6]. The final products in the reaction of acetyl iodide with ethoxy(triethyl)silane ($n = 1$) were ethyl acetate, hexaethyldisiloxane, and ethyl iodide (Scheme 5).
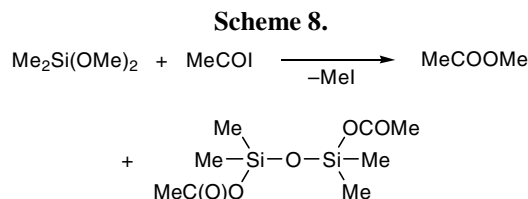
Scheme 5.

The reaction of triethoxy(methyl)silane with acetyl iodide gave polymethylsilsesquioxane (MeSiO$_{1.5}$)$_n$ together with ethyl acetate and EtI (Scheme 6).
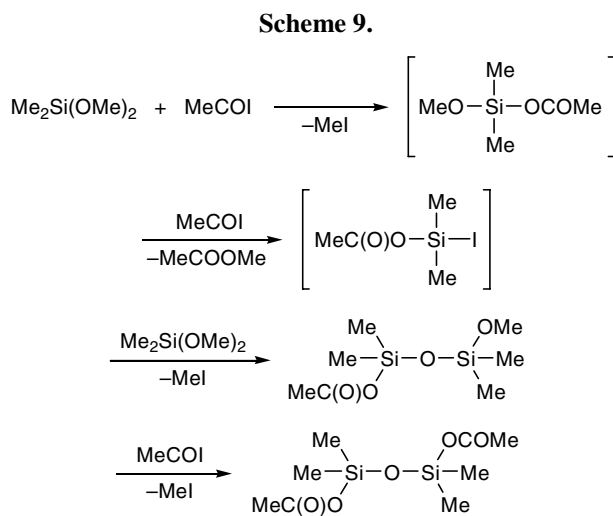
Scheme 6.
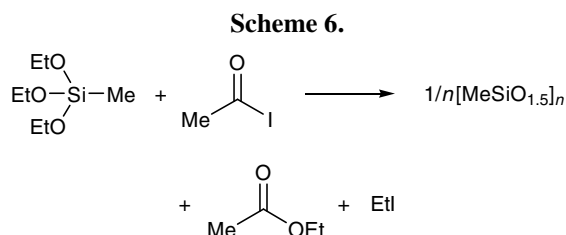
Tetraethoxysilane reacts with acetyl iodide to form different products, depending on the reactant ratio. The reaction with equimolar amounts of the reactants leads to the formation of ethyl acetate, ethyl iodide, and octaethoxytrisiloxane, while in the reaction with 4 equiv of acetyl iodide, the silicon-containing product is silicon(IV) oxide SiO$_2$ (Scheme 7).
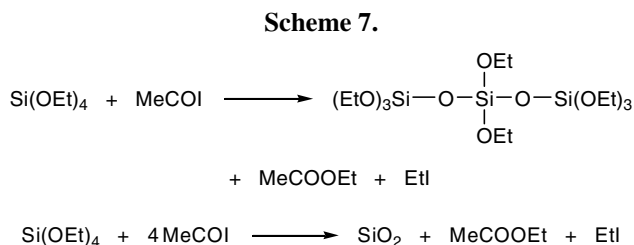
Scheme 7.

By contrast, 1,3-diacetoxy(tetramethyl)disiloxane was obtained in the reaction of acetyl iodide with dimethoxy(dimethyl)silane (Scheme 8).

Scheme 8.

This result suggests that acetyl iodide acts as both iodinating and acylating agent toward dimethoxy(dimethyl)silane; here, both Si–O and C–O bonds in the substrate are cleaved (Scheme 9).

Scheme 9.

We also found that acyl iodides readily react with 1-ethoxysilatrane in toluene to give the corresponding 1-acyloxysilatranes (Scheme 10).
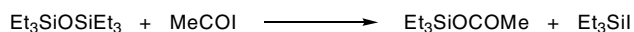
Scheme 10.

R = Me, Ph.
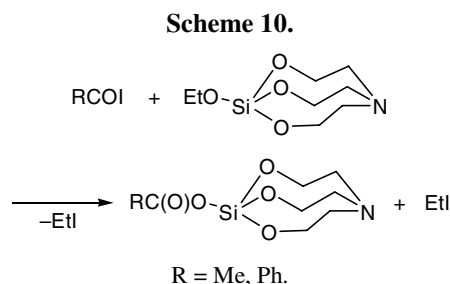
The reaction of acetyl iodide with 1-ethoxysilatrane was exothermic (the mixture spontaneously warmed up to 40°C). After keeping for 24 h at room temperature, ~60% of 1-acetoxysilatrane was formed. Benzoyl iodide reacted with 1-ethoxysilatrane without heat evolution; after 24 h at room temperature, the yield of

1-benzoyloxysilatrane was 70%. This reaction may be regarded as a new method for the preparation of 1-acyloxysilatranes. The latter were synthesized for the first time in two ways [18, 19]: by dehydrocondensation of silatrane with carboxylic acids in the presence of $ZnCl_2$ and by reaction of 1-ethoxysilatrane with carboxylic acids in the presence of Lewis acids. Several years later, Frye et al. [20] described the synthesis of 1-benzoyloxysilatrane in 90% yield from the same compounds in 1,2-dichlorobenzene at 150–200°C. 1-Acetoxysilatrane was obtained in two ways: by reaction of 1-ethoxysilatrane with acetic anhydride and by reaction of acetyl chloride with silatrane in the presence of triethylamine [20]. 1-Acetoxysilatrane was also synthesized in 60% yield by reaction of 1-iodosilatrane with ethyl acetate [21].

Acetyl iodide is also capable of cleaving the disiloxane Si–O–Si fragment but only at elevated temperature. Cleavage of that fragment by the action of acyl chlorides and acyl fluorides was studied previously. For example, the procedure for the preparation of oligo-α,ω-dichloropermethylsiloxanes via reaction of octamethylcyclotetrasiloxanes or linear polydimethylsiloxanes with excess benzoyl, phthaloyl, or isophthaloyl chloride in the presence of $FeCl_3$ was covered by patent [22].
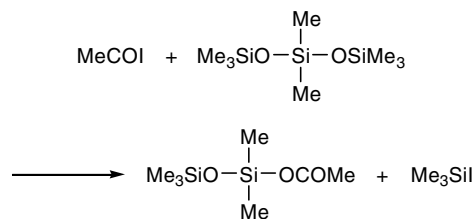
Octamethylcyclotetrasiloxane failed to react with acetyl chloride at a molar ratio of 1:2 on heating for 5 h at 120°C [9, 23]. Only when the reaction was carried out under more severe conditions, namely in a steel high-pressure reactor at 230°C or in the presence of anhydrous iron(III) chloride as catalyst under atmospheric pressure at 120°C, quantitative cleavage of $(Me_2SiO)_4$ with acetyl chloride was observed with formation of α,ω-diacetoxyoligo(dimethylsiloxanes) $MeCOO[Me_2SiO]_nCOOMe$ ($n = 2, 4$), dichloro(dimethyl)silane, 1,3-dichlorotetramethyldisiloxane, and hexamethylcyclotrisiloxane. Acetyl fluoride reacted with octamethylcyclotetrasiloxane at 250–260°C in a high-pressure reactor at a lower rate than did acetyl chloride, and the products were α,ω-diacetoxyoligo(dimethylsiloxanes) $MeCOOMe_2SiO[Me_2SiO]_nCOOMe$ ($n = 0$–2), 1,3-difluorotetramethyldisiloxane, difluoro(dimethyl)silane, and acetic anhydride. The reaction of acetyl iodide with hexaethyldisiloxane gave triethylsilyl acetate and iodo(triethyl)silane (Scheme 11).

Iodo(trimethyl)silane and dimethyl(trimethylsiloxy)silyl acetate were formed (yield ~80%) in the

Scheme 11.

$$Et_3SiOSiEt_3 \; + \; MeCOI \longrightarrow Et_3SiOCOMe \; + \; Et_3SiI$$

reaction of acetyl iodide with octamethyltrisiloxane (Scheme 12).

Scheme 12.

Unlike the reactions with acyl chlorides and fluorides (RCOX; X = Cl, F) [9], acetyl iodide readily reacted with octamethylcyclotetrasiloxane on slight heating under solvent-free conditions in the absence of a catalyst. The process was accompanied by opening of the tetrasiloxane ring with formation of open-chain 1,15-diacetoxyhexadecamethyloctasiloxane (yield 54%; Scheme 13).

Scheme 13.
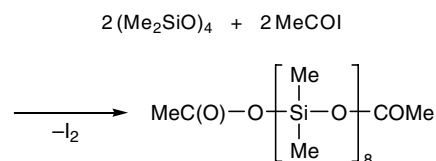
No 1-acetoxy-7-iodooctamethyltetrasiloxane expected to be formed as primary product in this reaction was detected. An analogous pattern was observed in the reaction of octamethylcyclotetrasiloxane with acetyl fluoride and acetyl chloride [9, 23]. The mechanism of the process remains so far unclear, and studies in this line are in progress.

The only product of the reaction of acetyl iodide with tetramethyl(divinyl)disiloxane at a ratio of 1:1 was dimethyl(vinyl)silyl acetate (Scheme 14).

Scheme 14.
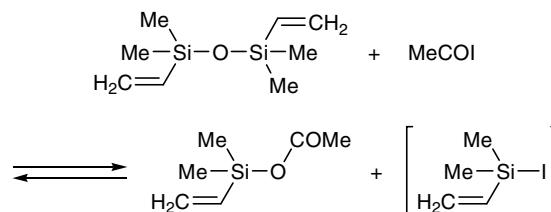
In addition, the initial reactants were detected in the reaction mixture which was difficult to separate. The other expected product, iodo(dimethyl)(vinyl)silane, was not identified. Probable reasons are reversibility of

the reaction and low hydrolytic stability of iodo(dimethyl)(vinyl)silane.
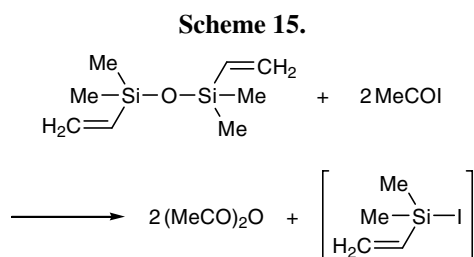
The reaction of tetramethyl(divinyl)disiloxane with 2 equiv of acetyl iodide, apart from the above compounds, gave a considerable amount of acetic anhydride as a result of cleavage of the Si–O bond in the Si–OC(O)CH$_3$ fragment of dimethyl(vinyl)silyl acetate by the action of acetyl iodide (Scheme 15).

Scheme 15.

Acetyl iodide reacted in a similar way with carboxylic acid esters [24]. Benzoyl iodide failed to react with tetramethyl(divinyl)disiloxane at 100–110°C.

EXPERIMENTAL

The IR spectra were recorded on a UR-20 spectrometer from samples prepared as thin films. The ^{1}H and ^{29}Si NMR spectra were measured on a Bruker DPX-400 spectrometer (400 MHz for ^{1}H) from solutions in chloroform-d using hexamethyldisiloxane as internal reference. Chromatographic analysis was performed on an LKhM-8M instrument equipped with a thermal conductivity detector; carrier gas helium; 2-m×4-mm column; oven temperature programming mode. The mass spectra (electron impact, 70 eV) were obtained using an HP 5971A mass-selective detector coupled with an HP 5890 gas chromatograph (Ultra-2 column, 5% of phenylmethylsilicone; injector temperature 250°C; oven temperature programming from 70 to 280°C at a rate of 20 deg/min).

Initial acyl iodides were prepared by reaction of the corresponding acyl chlorides with anhydrous sodium iodide according to the procedure reported in [25].

Reaction of acetyl iodide with ethoxy(triethyl)-silane. Acetyl iodide, 5.31 g (0.031 mol), was mixed with ethoxy(triethyl)silane, 4.99 g (0.031 mol), and the mixture spontaneously warmed up to 50°C. When the exothermic reaction was complete, distillation of the mixture gave a fraction boiling at 72–77°C; according to the GLC data (comparison with authentic samples), it contained 47% of ethyl iodide, 52% of ethyl acetate,

and 5.4 g (44%) of hexaethyldisiloxane, bp 85–92°C (10 mm); published data [26]: bp 62°C (2 mm).

Reaction of acetyl iodide with dimethoxy(dimethyl)silane. Acetyl iodide, 10.62 g (0.062 mol), was mixed with dimethoxy(dimethyl)silane, 3.75 g (0.031 mol), and the mixture spontaneously warmed up to 60°C. Distillation of the mixture gave a fraction boiling in the temperature range from 42 to 67°C, which contained 34% of methyl iodide and 46% of methyl acetate (GLC data), and 5.6 g (72%) of tetramethyldisiloxane-1,3-diyl diacetate with bp 95–100°C (10 mm). IR spectrum, ν, cm^{-1}: 3040 (C–H), 1720 (C=O), 1000–1100 (Si–O–Si). ^{1}H NMR spectrum, δ, ppm: 2.01 s (6H, MeCO), 0.06 s (12H, Me$_2$Si). ^{29}Si NMR spectrum: δ$_{Si}$ –7.4 ppm. Mass spectrum, m/z (I_{rel}, %): 43 (100) [MeCO]$^+$, 59 (3) [MeCOO]$^+$, 89 (6) [MeCOOSi]$^+$, 119 (5) [Me$_2$SiCOOMe]$^+$, 133 (34) [Me$_2$SiCOOMeO]$^+$, 163 (1) [Me$_2$SiCOOMeOSi]$^+$, 253 (1) [M]$^+$. Found, %: C 37.78; H 7.56; Si 22.51. C$_8$H$_{18}$O$_5$Si$_2$. Calculated, %: C 38.40; H 7.20; Si 22.40.

Reaction of acetyl iodide with triethoxy(methyl)-silane. Acetyl iodide, 10.62 g (0.062 mol), was mixed with triethoxy(methyl)silane, 3.64 g (0.021 mol), and the mixture spontaneously warmed up to 60°C. When the exothermic reaction was complete, distillation of the mixture gave a fraction boiling at 72–77°C, which contained (GLC) 42% of ethyl iodide and 56% of ethyl acetate. The tarry still residue contained polymethylsilsesquioxane (MeSiO$_{1.5}$)$_n$. Found, %: C 15.76; H 3.99; Si 39.38. CH$_3$O$_{1.5}$Si. Calculated, %: C 17.91; H 4.47; Si 41.79.

Reaction of acetyl iodide with tetraethoxysilane at a ratio of 1:1. Distillation of the reaction mixture obtained under analogous conditions from 10.62 g (0.062 mol) of acetyl iodide and 11.9 (0.062 mol) of tetraethoxysilane gave a fraction with bp 72–77°C, which contained (GLC) 42% of ethyl iodide and 56% of ethyl acetate, and 4.46 g (60%) of octaethoxytrisiloxane, bp 115–122°C (27 mm). IR spectrum, ν, cm^{-1}: 3020–3100 (C–H), 1050 (Si–O–Si). ^{1}H NMR spectrum, δ, ppm: 3.8 (OCH$_2$), 1.1 (C**H**$_3$CH$_2$O). ^{29}Si NMR spectrum: δ$_{Si}$ 9.8 ppm. Mass spectrum, m/z (I_{rel}, %): 41 (14) [OEt]$^+$, 167 (18) [(EtO)$_3$Si]$^+$, 183 (41) [(EtO)$_3$SiO]$^+$, 387 (9) [M – (EtO)$_2$Si]$^+$, 401 (100) [M – EtOSi]$^+$, 431 (36) [M – (OEt)O]$^+$. Found, %: C 40.42; H 8.23; Si 18.04. C$_{16}$H$_{40}$O$_{10}$Si$_3$. Calculated, %: C 40.34; H 8.40; Si 17.65.

Reaction of acetyl iodide with tetraethoxysilane at a ratio of 4:1. Distillation of reaction mixture

obtained under similar conditions from 10.62 g (0.062 mol) of acetyl iodide and 2.97 g (0.016 mol) of tetraethoxysilane gave a fraction with bp 72–77°C, which contained (GLC) 64% of ethyl iodide and 32% of ethyl acetate. The still residue was silicon dioxide. Calculated, %: Si 46.66. SiO$_2$. Found, %: Si 44.04.

Reaction of acetyl iodide with 1-ethoxysilatrane. Acetyl iodide, 2.9 g (0.017 mol), was added dropwise to a solution of 3.73 g (0.017 mol) of 1-ethoxysilatrane in 50 ml of toluene. The mixture spontaneously warmed up to 40°C and was left to stand for 24 h at room temperature. The precipitate was filtered off and recrystallized from methylene chloride–heptane (1:1). Yield of 2,8,9-trioxa-5-aza-1-silabicyclo[3.3.3]undecan-1-yl acetate 2.52 g (63%), mp 170–171°C; published data [19]: mp 173–174°C. According to the GLC data, the filtrate contained ethyl iodide.

Reaction of benzoyl iodide with 1-ethoxysilatrane. Benzoyl iodide, 2.32 g (0.01 mol), was added dropwise to a solution of 2.19 g (0.01 mol) of 1-ethoxysilatrane in 50 ml of toluene under stirring at room temperature. The mixture was kept for 24 h at room temperature, and the precipitate was filtered off and recrystallized from xylene. Yield of 2,8,9-trioxa-5-aza-1-silabicyclo[3.3.3]undecan-1-yl benzoate 2.23 g (76.4%), mp 220–222°C; published data [19]: mp 221–222°C. The filtrate contained ethyl iodide (GLC).

Reaction of acetyl iodide with hexaethyldisiloxane. A mixture of 5.31 g (0.031 mol) of acetyl iodide and 7.63 g (0.031 mol) of hexaethyldisiloxane was heated for 6 h at 90°C. Distillation of the mixture gave a fraction with bp 80–85°C (20 mm), which contained (according to the ^{29}Si NMR data) 40% of triethyliodosilane and 50% of triethylsilyl acetate. ^{29}Si NMR spectrum, δ_{Si}, ppm: 25.09 (Et$_3$SiO), 13.01 (Et$_3$SiI).

Reaction of acetyl iodide with tetramethyl(divinyl)disiloxane at a ratio of 1:1. A mixture of 8.5 g (0.05 mol) of acetyl iodide and 9.3 g (0.05 mol) of tetramethyl(divinyl)disiloxane was heated for 3.5 h at 60–65°C and for 3.5 h at 105–110°C. Distillation of the mixture gave 5.8 g (55%) of dimethyl(vinyl)silyl acetate, bp 145–150°C, n_D^{20} = 1.4100. IR spectrum, ν, cm^{-1}: 3050 (C–H), 1710 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.9 s (3H, MeCO), –0.016 s (6H, Me$_2$Si), 5.59 d.d and 5.78 d.d (2H, CH$_2$=), 5.96 d.d (1H, CH=). ^{29}Si NMR spectrum: δ_{Si} 9.8 ppm. Found, %: C 49.71; H 8.38; Si 20.08. C$_6$H$_{12}$SiO$_2$. Calculated, %: C 49.96; H 8.39; Si 19.47.

Reaction of acetyl iodide with tetramethyl(divinyl)disiloxane at a ratio of 2:1. A mixture of 8.5 g (0.05 mol) of acetyl iodide and 4.6 g (0.025 mol) of tetramethyl(divinyl)disiloxane was heated for 7 h under reflux. Distillation of the mixture gave 2.1 g (84%) of acetic anhydride (identified by GLC), bp 138–139°C; published data [27]: bp 140°C.

Reaction of acetyl iodide with octamethyltrisiloxane. A mixture of 3.69 g (0.02 mol) of acetyl iodide and 5.13 g (0.02 mol) of octamethyltrisiloxane was heated for 6 h at 105–110°C. Distillation of the mixture gave 3.2 g (74%) of iodotrimethylsilane, bp 101–103°C (published data [2]: bp 106°C) and 3.9 g (87%) of dimethyl(trimethylsiloxy)silyl acetate, bp 170–172°C, n_D^{20} = 1.3900. IR spectrum, ν, cm^{-1}: 3070 (C–H), 1710 (C=O), 1050 (SiOSi). ^{1}H NMR spectrum, δ, ppm: 2.05 (MeCO), 0.09–0.02 (Me$_2$Si). ^{29}Si NMR spectrum, δ_{Si}, ppm: 6.95 (Me$_3$Si), 21.6 (Me$_2$Si). Found, %: C 39.06; H 9.51; Si 28.34. C$_7$H$_{15}$Si$_2$O$_3$. Calculated, %: C 40.74; H 8.79; Si 27.21.

Reaction of acetyl iodide with octamethylcyclotetrasiloxane. A mixture of 5.31 g (0.031 mol) of acetyl iodide and 9.18 g (0.031 mol) of octamethylcyclotetrasiloxane was heated for 3 h at 90°C. Distillation of the mixture gave 11.62 g (54%) of hexadecamethyloctasiloxane-1,8-diyl diacetate, bp 92–95°C (10 mm). IR spectrum, ν, cm^{-1}: 3040 (C–H), 1715 (C=O), 1250 (SiMe$_2$), 1000–1080 (Si–O–Si), 800 (SiMe$_2$). ^{1}H NMR spectrum, δ, ppm: 2.06 s (6H, MeCO), 0.27 s (48H, SiMe$_2$). ^{29}Si NMR spectrum: δ_{Si} –21.5 ppm. Mass spectrum, m/z (I_{rel}, %): 43 (8) [MeCO]$^+$, 59 (8) [MeCOO]$^+$, 73 (100) [Me$_2$SiO]$^+$, 267 (51) [MeCOOSiMe$_2$OSiMe$_2$OSiMeO]$^+$, 355 (67) [M – Me$_2$SiOSiMe$_2$OSiMe$_2$OSiMe$_2$COOMeO]$^+$. Found, %: C 34.47; H 7.91; Si 35.79. C$_{20}$H$_{54}$O$_{11}$Si$_8$. Calculated, %: C 35.40; H 7.96; Si 33.00.

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