A Synthetic Pathway to 1,3-Bis(trisilylmethyl)disiloxane $[(H_3Si)_3CSiH_2]_2O$ – the Octasila Analogue of Dineopentyl Ether $[(H_3C)_3CCH_2]_2O$

Sebastian Bommers and Hubert Schmidbaur*

Anorganisch-Chemisches Institut der Technischen Universität München,
Lichtenbergstraße 4, D-85747 Garching, Germany

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A synthetic route to partly silylated tetra(silyl)methanes $(ArH_2Si)_nC(SiH_3)_{4-n}$, to their precursors $(TfH_2Si)_nC(SiH_2Ar)_{4-n}$ (Ar = *p*-tolyl, phenyl; Tf = CF_3SO_3; $n = 3, 2, 1$), and to 1,3-bis(trisilylmethyl)disiloxane $[(H_3Si)_3CSiH_2]_2O$ (**9**) is reported. Starting from symmetrical tetrakis(arylsilyl)methanes (aryl = *p*-tolyl, phenyl), we have obtained the selectively dearylated (arylsilyl)silylmethanes $(ArH_2Si)_nC(SiH_3)_{4-n}$ (Ar = *p*-tolyl, phenyl; $n = 3, 2, 1$) by Si–Ar cleavage with equivalent quantities of trifluoromethanesulfonic acid (triflic acid) and hydrogenation of the corresponding silyl triflates $(TfH_2Si)_nC(SiH_2Ar)_{4-n}$ ($n = 3, 2, 1$) with $LiAlH_4$. The synthesis of **9** has been accomplished by treating trisilyl(*p*-tolylsilyl)methane *p*-$TolH_2SiC(SiH_3)_3$ (**7**) with stoichiometric amounts of triflic acid to give (trisilylmethyl)silyl triflate $TfH_2SiC(SiH_3)_3$ (**8**) and hydrolysing the latter with water.

Small hydrogen-rich silane molecules are of considerable interest for chemical vapour deposition (CVD) of silicon alloys with various elements[1,2,3]. Tetrasilylmethane $C(SiH_3)_4$ is an example of the inclusion of *single* carbon atoms not bound to hydrogen into the framework of amorphous silicon a-Si,C:H[4]. Similar effects are observed with simple silylamines, -hydrazines, or -hydroxylamines as single-source precursors of a-Si,N:H and a-Si,N,O:H[5]. Following the same train of thoughts, we now report on the synthesis of 1,3-bis(trisilylmethyl)disiloxane $[H_3Si)_3CSiH_2]_2O$ (**9**), a tailor-made ternary Si/C/O system where, in addition to the CSi_4 unit, the OSi_2 fragment is provided in the molecule for the generation of silicon networks containing both carbon and oxygen.

Small volatile compounds with carbon in the environment of four silicon atoms are still not very common. Virtually all of these are homoleptic, the interstitial carbon bearing only one type of substituent. For the series of tetrakis(silyl)methanes $(RH_2Si)_4C$ the examples with R = Ph, *p*-Tol, H, Br. Cl have been reported[6,7]. The general synthetic route to these compounds is shown by Eqs. (1)–(3), which is also a summary of the continuing efforts in this laboratory to find an effective pathway to various poly(silyl)alkanes.

For maximum yields of the intermediates, it proved most convenient to use chloro(*p*-tolyl)silane as a starting material. For the cleavage of the aryl groups from the crucial intermediate tetrakis(*p*-tolylsilyl)methane (**1**), treatment with an excess of anhydrous liquid hydrogen bromide has been successfully employed. The use of trifluoromethanesulfonic acid (triflic acid) as a protodesilylating[8–11] agent instead of HBr proved to be of no advantage, owing to the very rapid decomposition of the resulting tetra(triflate) $(TfH_2Si)_4C$ (CF_3SO_3 = Tf). Published results have indicated that the stability of poly[(trifluoromethylsulfonyloxy)silyl]methanes $(TfHSi)_nCH_{4-n}$ (n = 4, 3, 2) decreases with increasing number of SiH_2Tf groups at the central carbon atom ($n = 4 \ll 3 < 2$)[12]. The experiments provided evidence that the products of partial dearylation are thermally much more stable, which suggested a route to intermediates with certain substitution patterns amenable to the synthesis of products with a novel combination of functionalities. The title compound with both Si_4C and SiOSi building blocks has therefore been chosen as a synthetic target.

$$\mathrm{ArH_2SiCl\ (Ar = Ph,\ p\text{-}Tol)} \xrightarrow{\mathrm{CBr_4/Mg}} \underset{\mathbf{1}}{\mathrm{(ArH_2Si)_4C}} \qquad (1)$$

$$\mathbf{1} \xrightarrow[\mathrm{-\ 4\,ArH}]{\mathrm{HX\ (X = Br,\ Cl)}} \mathrm{(XH_2Si)_4C} \qquad (2)$$

$$\mathrm{(XH_2Si)_4C} \xrightarrow{\mathrm{LiAlH_4}} \mathrm{(H_3Si)_4C} \qquad (3)$$

Results

Based on the foregoing the selective partial Si–Aryl cleavage in $(ArH_2Si)_4C$ molecules by triflic acid has been investigated. Up to three of the equivalent *p*-tolyl groups can be converted into the corresponding silyl triflate functions in clearly distinguished steps [Eqs. (4)–(6)] by starting from tetrakis(*p*-tolylsilyl)methane (**1**).

The electrophilic attack at a given *p*-tolyl ring appears to be strongly influenced by the presence of other triflate units

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in the molecule. Thus, under carefully controlled reaction conditions (−60°C), no random mixture of all kinds of cleavage products is observed, but the reactions rather show a surprisingly high selectivity. It is only under more forcing conditions that a scrambling of triflate and aryl groups is finally taking place. Compound **1** can be converted into the silyltriflates **2**, **3**, **4** by slowly adding the appropriate amount of triflic acid.

$$\underset{\mathbf{1}}{(p\text{-TolH}_2\text{Si})_4\text{C}} \xrightarrow{1\ \text{CF}_3\text{SO}_3\text{H}} \underset{\mathbf{2}}{(p\text{-TolH}_2\text{Si})_3\text{CSiH}_2\text{Tf}} \quad (4)$$

$$\underset{\mathbf{1}}{(p\text{-TolH}_2\text{Si})_4\text{C}} \xrightarrow{2\ \text{CF}_3\text{SO}_3\text{H}} \underset{\mathbf{3}}{(p\text{-TolH}_2\text{Si})_2\text{C}(\text{SiH}_2\text{Tf})_2} \quad (5)$$

$$\underset{\mathbf{1}}{(p\text{-TolH}_2\text{Si})_4\text{C}} \xrightarrow{3\ \text{CF}_3\text{SO}_3\text{H}} \underset{\mathbf{4}}{p\text{-TolH}_2\text{SiC}(\text{SiH}_2\text{Tf})_3} \quad (6)$$

The stability of the products decreases as the number of SiH_2Tf groups is increased (**4** < **3** < **2**). The silyl triflates slowly undergo isomerisation at ambient temperature, and workup of the crude products under standard conditions is not recommended. As derived from ^{1}H-NMR and ^{29}Si-NMR spectroscopic data [chemical shifts, ^1J(SiH) coupling constants] the decomposition ensues from inter- and intramolecular H/Tf exchange and leads to derivatives with $SiHTf_2$ and SiH_3 moieties. This isomerisation does not affect the stoichiometry of the following reduction step. Treatment of the freshly prepared silyl triflates **2**, **3**, **4** with a suspension of $LiAlH_4$ in diethyl ether affords the corresponding silanes **5**, **6**, **7** in good to moderate yields [Eqs. (7)−(9)].

$$\underset{\mathbf{2}}{(p\text{-TolH}_2\text{Si})_3\text{CSiH}_2\text{Tf}} \xrightarrow{\text{LiAlH}_4} \underset{\mathbf{5}}{(p\text{-TolH}_2\text{Si})_3\text{CSiH}_3} \quad (7)$$

$$\underset{\mathbf{3}}{(p\text{-TolH}_2\text{Si})_2\text{C}(\text{SiH}_2\text{Tf})_2} \xrightarrow{\text{LiAlH}_4} \underset{\mathbf{6}}{(p\text{-TolH}_2\text{Si})_2\text{C}(\text{SiH}_3)_2} \quad (8)$$

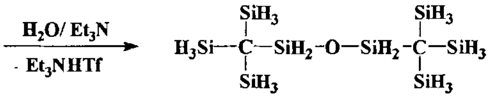
$$\underset{\mathbf{4}}{p\text{-TolH}_2\text{SiC}(\text{SiH}_2\text{Tf})_3} \xrightarrow{\text{LiAlH}_4} \underset{\mathbf{7}}{p\text{-TolH}_2\text{SiC}(\text{SiH}_3)_3} \quad (9)$$

The compounds **5**—**7** are airstable, colourless liquids with an intensive odour, which can be distilled in vacuo without decomposition (see Experimental).

All attempts to replace tetrakis(*p*-tolylsilyl)methane, an expensive starting material owing to the high price of the *p*-tolyl reagents, by the comparatively cheap tetrakis(phenylsilyl)methane analogue have met with only limited success, since tetrakis(phenylsilyl)methane is difficult to separate from the byproducts of its synthesis, mainly bis- and tris(phenylsilyl)methane. Treatment of the crude product mixture with what are inevitably somewhat unstoichiometric amounts of triflic acid, followed by hydride/triflate substitution, is associated with an unacceptable loss of product. Bis(phenylsilyl)disilylmethane $(PhH_2Si)_2C(SiH_3)_2$ and (phenylsilyl)trisilylmethane $(PhH_2Si)C(SiH_3)_3$ have been isolated, however, and characterized by their spectroscopic data (see Experimental).

Both trisilyl(*p*-tolylsilyl)methane and trisilyl(phenylsilyl)methane can be used as synthons for derivatives with (trisilylmethyl)silyl substituents $-SiH_2C(SiH_3)_3$. Protodesilylation with triflic acid affords the (trisilylmethyl)silyl triflate $(H_3Si)_3CSiH_2Tf$ (**8**) in quantitative yield. In this compound the Tf/H isomerisation is much slower than for compounds **2**−**4**. This key intermediate has been employed for the next reaction step without separating it from toluene or benzene produced in the preparation. The reaction of **8** with stoichiometric amounts of water, dissolved in a mixture of diethyl ether and triethylamine to trap the triflic acid released in the process, affords 1,3-bis(trisilylmethyl)disiloxane (**9**) [Eqs. (10), (11)].

$$\underset{\mathbf{7}}{p\text{-TolH}_2\text{SiC}(\text{SiH}_3)_3} \xrightarrow{\text{CF}_3\text{SO}_3\text{H}} \underset{\mathbf{8}}{\text{TfH}_2\text{SiC}(\text{SiH}_3)_3} \quad (10)$$



$$\xrightarrow[-\ \text{Et}_3\text{NHTf}]{\text{H}_2\text{O}/\ \text{Et}_3\text{N}} \underset{\mathbf{9}}{\text{H}_3\text{Si}-\underset{\text{SiH}_3}{\overset{\text{SiH}_3}{\text{C}}}-\text{SiH}_2-\text{O}-\text{SiH}_2-\underset{\text{SiH}_3}{\overset{\text{SiH}_3}{\text{C}}}-\text{SiH}_3} \quad (11)$$

1,3-Bis(trisilylmethyl)disiloxane (**9**) is a colourless liquid (b.p. 55−60/2 Torr). Solutions in C_6D_6 show two ^{1}H-NMR signals with silicon satellites, δ = 4.77 [$^1J(^1H^{29}Si)$ = 227 Hz, 4 H, SiH_2O] and 3.81 [$^1J(^1H^{29}Si)$ = 207 Hz, 18 H, SiH_3]. The ^{17}O resonance appears as a singlet at δ = 7.7, the ^{13}C resonance at δ = −29 as a pseudodecet [$^2J(^{13}CSi^1H)$ = 5 Hz]. The ^{29}Si-NMR spectrum shows a signal split into a triplet of decets of triplets at δ = −18.8 [$^1J(^{29}Si^1H)$ = 227, $^3J(^{29}SiCSi^1H)$ = 5, $^3J(^{29}SiOSi^1H)$ = 1.22 Hz] for the SiH_2 groups and a quartet signal of unresolved multiplets at δ = −54.2 [$^1J(^{29}Si^1H)$ = 207 Hz] for the SiH_3 groups. Electron impact mass spectra (m/z = 286, M^+) and IR spectra further confirm the proposed formula (see Experimental).

In summary, selective Si−aryl cleavage in tetrakis(*p*-tolylsilyl)methane (**1**) with stoichiometric amounts of triflic acid opens up a synthetic route to the tetrasilylmethane derivatives $(p\text{-TolH}_2Si)_nC(SiH_3)_{4-n}$ (n = 3, 2, 1) with well-defined functionality. The preparation of the silyltriflate intermediates $(TfH_2Si)_nC(SiH_2Ar)_{4-n}$ (n = 3, 2, 1) as well as the following reduction step present no experimental difficulties and give acceptable yields and high product purity. The phenyl analogues can also be prepared, but the synthetic procedure is far less satisfactory. Starting from trisilyl(*p*-tolylsilyl)methane, we have thus easily obtained the dineopentyl ether analogue 1,3-bis(trisilylmethyl)disiloxane (**9**) by hydrolysis of (trisilylmethyl)silyl triflate (**8**).

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Experimental

All experiments were carried out under pure dry nitrogen. Glassware and solvents were purified, dried, and kept under nitrogen. – NMR: C_6D_6 and $CDCl_3$ as solvents, reference tetramethylsilane, Jeol GX 270, GX 400 and Bruker WT 100 SY spectrometers.

(p-Tolylsilyl)[(trifluoromethylsulfonyloxy)silyl]methanes $(p\text{-}TolH_2Si)_3CSiH_2Tf$ (**2**), $(p\text{-}TolH_2Si)_2C(SiH_2Tf)_2$ (**3**), $p\text{-}TolH_2SiC(SiH_2Tf)_3$ (**4**). – *General Procedure:* To a solution of tetrakis(*p*-tolylsilyl)methane (**1**) in toluene, stoichiometric amounts of trifluoromethanesulfonic acid were added dropwise at temperatures around −60°C. After 2 h the mixture was allowed to warm to room temp. and stirred for 1 h. The crude products were used in the following reduction step without any further purification. The yields were virtually quantitative.

2: ^1H NMR (C_6D_6): δ = 7.3 (d, ArH), 6.9 (d, ArH), ^3J(HCCH) = 7.8 Hz; 4.8 (s, SiH_2Tf), ^1J(HSi) = 250 Hz; 4.75 (s, SiH_2Ar), ^1J(HSi) = ca. 200 Hz; 2.1 (s, CH_3). – $^{13}C\{^1H\}$ NMR (C_6D_6): δ = 141.3 (ArC-1); 136.7 (ArC-2/4); 129.3 (ArC-3/5); 125.1 (ArC-6); 118.9 (q, CF_3), $^1J(CF_3)$ = 319 Hz; 21.4 (CH_3); −15.3 (Si_4C). – ^{29}Si NMR (C_6D_6): δ = −7 (tsept, SiH_2Tf), ^1J(SiH) = 250, ^3J(SiCSiH) = 4.8 Hz; −32.5 (tm, SiH_2Ar), ^1J(SiH) = 207 Hz.

3: ^1H NMR (C_6D_6): δ = 7.3 (m, ArH), 6.9 (m, ArH); 4.8 (s, SiH_2Tf); 4.7 (s, SiH_2Ar). – ^{29}Si NMR (C_6D_6): δ = −8.8 (tm, SiH_2Tf), ^1J(SiH) = 244 Hz; −32.6 (tm, SiH_2Ar), ^1J(SiH) = 200 Hz.

4: Due to rapid H/Tf scrambling, no reliable spectroscopic data available.

Silyl(p-tolylsilyl)methanes $(p\text{-}TolH_2Si)_3CSiH_3$ (**5**), $(p\text{-}TolH_2Si)_2C(SiH_3)_2$ (**6**), $p\text{-}TolH_2SiC(SiH_3)_3$ (**7**). – *General Procedure:* To a suspension of $LiAlH_4$ in diethyl ether the freshly prepared silyl triflate was added dropwise at room temp. The resulting mixture was stirred under reflux conditions for 1 h. Aqueous workup and distillation of the organic phase yielded the particular silylmethane in high purity.

5: Yield 78%, b.p. 180°C/10^{-3} Torr. – ^1H NMR (C_6D_6): δ = 7.4 (d, ArH), 6.9 (d, ArH), ^3J(HCCH) = 7.8 Hz; 4.8 (s, SiH_2), ^1J(HSi) = 201 Hz; 3.9 (s, SiH_3), ^1J(HSi) = 204 Hz; 2.1 (s, CH_3). – $^{13}C\{^1H\}$ NMR (C_6D_6):; δ = 140.2 (ArC-1); 136.7 (ArC-2/6); 129 (ArC-3/5); 128 (ArC-4); 21.6 (CH_3); −24.3 (Si_4C). – ^{29}Si NMR (C_6D_6): δ = −28.4 (tm, SiH_2), ^1J(SiH) = 204 Hz; −56.4 (qsept, SiH_3), ^1J(SiH) = 204, ^3J(SiCSiH) = 4.6 Hz. – MS (EI, 70 eV), *m/z*: 406 [$C_{22}Si_4H_{30}$], 313–311 [$C_{15}Si_4H_x$], 281–279 [$C_{15}Si_3H_x$], 224–222 [$C_8Si_4H_x$], 194–191 [$C_8Si_3H_x$]. – $C_{22}H_{30}Si_4$ (406.8): calcd. C 64.7, H 7.5, Si 27.7; found C 64.8, H 7.4, Si 27.5.

6: Yield 66%, b.p. 135°C/10^{-3} Torr. – ^1H NMR (C_6D_6): δ = 7.4 (d, ArH), 6.9 (d, ArH), ^3J(HCCH) = 8 Hz; 4.7 (s, SiH_2), ^1J(SiH) = 200 Hz; 3.9 (s, SiH_3), ^1J(SiH) = 205 Hz; 2.0 (s, CH_3). – $^{13}C\{^1H\}$ NMR (C_6D_6): δ = 140 (ArC-1); 136.1 (ArC-2/6); 128.7 (ArC-3/5); 126.6 (ArC-4); 21.3 (CH_3); −29.8 (Si_4C). – ^{29}Si NMR (C_6D_6): δ = −26.5 (tm, SiH_2), $^1J(SiH_2)$ = 200 Hz; −54 (qm, SiH_3), ^1J(SiH) = 204 Hz. – MS (EI, 70 eV), *m/z*: 316 [$C_{15}Si_4H_{24}$], 227–222 [$C_8Si_4H_x$], 195–191 [$C_8Si_3H_x$], 165–161 [$C_8Si_2H_x$], 121–119 [C_7SiH_x]. – $C_{15}H_{24}Si_4$ (316.7): calcd. C 56.9, H 7.6, Si 35.5; found C 54.7, H 7.7, Si 36.1.

7: Yield 52%, b.p. 80°C/10^{-3} Torr. – ^1H NMR (C_6D_6): δ = 7.4 (d, ArH); 6.9 (d, ArH), ^3J(HCCH) = 8 Hz; 4.6 (s, SiH_2); 3.8 (s, SiH_3), ^1J(HSi) = 205 Hz; 2.0 (s, CH_3). – $^{13}C\{^1H\}$ NMR (C_6D_6): δ = 140 (ArC-1); 136.1 (ArC-2/6); 128.7 (ArC-3/5); 126.5 (ArC-4); 21.3 (CH_3); −20.8 (Si_4C). – ^{29}Si NMR (C_6D_6): δ = −24.5 (tm, SiH_2), ^1J(SiH) = 207 Hz; −51.4 (qm, SiH_3), ^1J(SiH) = 200 Hz. – MS (EI, 70 eV), *m/z*: 226 [$C_8Si_4H_{18}$], 136–134 [CSi_4H_x]. – $C_8H_{18}Si_4$ (406.8): calcd. C 42.4, H 7.9, Si 49.6; found C 43.2, H 7.8, Si 48.9.

$(PhH_2Si)_2C(SiH_3)_2$, $PhH_2SiC(SiH_3)_3$: To a product mixture of tetrakis(phenylsilyl)methane [containing about 70% of tetrakis(phenylsilyl)methane, together with other (phenylsilyl)methanes], dissolved in toluene, was added trifluoromethanesulfonic acid in appropriate stoichiometric quantities. The subsequent reduction with $LiAlH_4$ and distillation afforded both $(PhH_2Si)_2C(SiH_3)_2$ and $PhH_2SiC(SiH_3)_3$ in low yield.

$(PhH_2Si)_2C(SiH_3)_2$: B.p. 100°C/10^{-3} Torr. – ^1H NMR (C_6D_6): δ = 7.5 (m, ArH); 7.1 (m, ArH); 4.7 (s, SiH_2); 3.8 (s, SiH_3), ^1J(SiH) = 207 Hz. – $^{13}C\{^1H\}$ NMR (C_6D_6): δ = 136.4 (ArC-1); 135.6 (ArC-2/6); 130.6 (ArC-4); 128.2 (ArC-3/5); −29.8 (Si_4C). – ^{29}Si NMR (C_6D_6): δ = −26.2 (tm, SiH_2), ^1J(SiH) = 203 Hz; −53.8 (qoct, SiH_3), ^1J(SiH) = 205, ^3J(SiCSiH) = 4.6 Hz. – MS (EI, 70 eV), *m/z*: 283–279 [$C_{13}Si_4H_x$], 254–251 [$C_{13}Si_3H_x$], 210–208 [$C_7Si_4H_x$], 181–177 [$C_7Si_3H_x$].

$PhH_2SiC(SiH_3)_3$: B.p. 120°C/50 Torr. – ^1H NMR (C_6D_6): δ = 7.2 (m, ArH); 7.1 (m, ArH); 4.7 (s, SiH_2); 3.9 (s, SiH_3). – $^{13}C\{^1H\}$ NMR (C_6D_6): δ = 136.1 (ArC-2/6); 130.5 (ArC-1); 128.6 (ArC-4); 128.3 (ArC-3/5); −35.1 (Si_4C). – ^{29}Si NMR (C_6D_6): δ = −24.3 (tm, SiH_2), ^1J(SiH) = 207 Hz; −51.5 (qm, SiH_3), ^1J(SiH) = 205 Hz. – MS (EI, 70 eV), *m/z*: 212 [$C_7Si_4H_{16}$], 136–134 [CSi_4H_x], 107–104 [CSi_3H_x].

$TfH_2SiC(SiH_3)_3$ (**8**): To a solution of 2.3 g (10 mmol) of **7** in toluene 0.88 ml (10 mmol) of CF_3SO_3H was added at −60°C. The mixture was allowed to warm to ambient temp. and then used in the following reaction step without any further purification. The yield was almost quantitative. – ^1H NMR ($CDCl_3$): δ = 4.9 (s, SiH_2Tf), ^1J(HSi) = 250 Hz; 3.9 (s, SiH_3), ^1J(HSi) = 207 Hz. – ^{29}Si NMR (C_6D_6): δ = −3.1 (toct, SiH_2Tf), ^1J(SiH) = 246, ^3J(SiCSiH) = 5 Hz; −53.1 (qm, SiH_3), ^1J(SiH) = 212 Hz. – ^{13}C NMR (C_6D_6): δ = 118.4 (q, CF_3), ^1J(CF) = 318 Hz; −31 (CSi_4).

$[(H_3Si)_3CSiH_2]_2O$ (**9**): To a solution of freshly prepared **8** (above) a solution of stoichiometric amounts of H_2O and Et_3N in 20 ml of diethyl ether was added dropwise at room temp. After separation from the oily $Et_3NH^+OSO_2CF_3^-$ phase distillation of the organic layer yielded 0.80 g (56%) of **9**. – ^1H NMR (C_6D_6): δ = 4.8 (s, SiH_2O), ^1J(HSi) = 227 Hz; 3.8 (s, SiH_3), ^1J(HSi) = 207 Hz. – ^{17}O NMR (C_6D_6): δ = 7.7. – ^{19}Si NMR (C_6D_6): δ = −18.8 (toct, SiH_2O), ^1J(SiH) = 227, ^3J(SiCSiH) = 5, ^3J(SiOSiH) = 1.2 Hz; −54.2 (qm, SiH_3), ^1J(SiH) = 207 Hz. – ^{13}C NMR (C_6D_6): δ = −29 (Ψ-dec), ^1J(CSi) = 30, ^2J(CSiH) = 5 Hz. – MS (EI, 70 eV), *m/z*: 268–283 [$C_2Si_8OH_x$], 256–253 [$C_2Si_7OH_x$], 224–221 [$C_2Si_6OH_x$], 183–181 [CSi_5OH_x], 135 [CSi_4H_{11}]. – IR (Film) [cm^{-1}]: 2151 s ν(SiH), 1095 w ν(SiO), 867 s δ(SiH), 836 m ν(SiC).

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