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Facile Syntheses of New Incompletely Condensed Polyhedral Oligosilsesquioxanes: [(c-C_5H_9)$_7$Si$_7$O$_9$(OH)$_3$], [(c-C_7H_{13})$_7$Si$_7$O$_9$(OH)$_3$], and [(c-C_7H_{13})$_6$Si$_6$O$_7$(OH)$_4$]

Frank J. Feher,* Theodore A. Budzichowski,† Rusty L. Blanski, Keith J. Weller, and Joseph W. Ziller

Department of Chemistry, University of California, Irvine, California 92717

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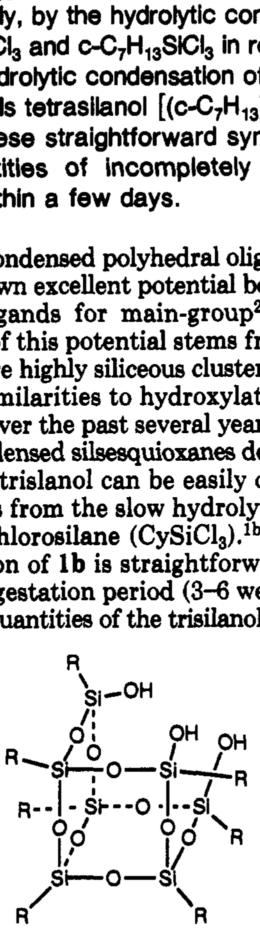
Summary: Facile syntheses of three new incompletely condensed polyhedral oligosilsesquioxanes are reported. Trisilanols [(c-C_5H_9)$_7$Si$_7$O$_9$(OH)$_3$ (**1a**) and [(c-C_7H_{13})$_7$Si$_7$O$_9$(OH)$_3$] (**1c**) are prepared in 29% and 26% yields, respectively, by the hydrolytic condensation reactions of c-C_5H_9SiCl$_3$ and c-C_7H_{13}SiCl$_3$ in refluxing aqueous acetone. The hydrolytic condensation of c-C_7H_{13}SiCl$_3$ at 25 °C also affords tetrasilanol [(c-C_7H_{13})$_6$Si$_6$O$_7$(OH)$_4$] (**2**) in 7% yield. These straightforward syntheses produce multigram quantities of incompletely condensed silsesquioxanes within a few days.

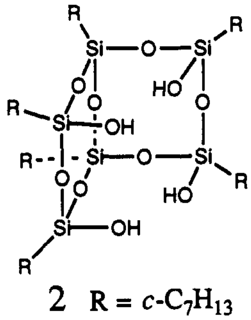
Incompletely condensed polyhedral oligosilsesquioxanes have recently shown excellent potential both as models for silica[1] and as ligands for main-group[2] and transition metals.[3] Much of this potential stems from the fact that silsesquioxanes are highly siliceous clusters with structural and electronic similarities to hydroxylated silica surface sites. Our work over the past several years has focused on incompletely condensed silsesquioxanes derived from **1b**.[1-3] This fascinating trisilanol can be easily obtained in multigram quantities from the slow hydrolytic condensation of cyclohexyltrichlorosilane (CySiCl$_3$).[1b,4]

The preparation of **1b** is straightforward but requires an inconvenient gestation period (3–6 weeks) before synthetically useful quantities of the trisilanol can be obtained.



1a R = c-C_5H_9
b c-C_6H_{11}
c c-C_7H_{13}

In this paper we report facile syntheses of two structurally similar trisilanols (**1a** and **1c**), which can be obtained from the hydrolytic condensation reactions of c-C_5H_9SiCl$_3$ and c-C_7H_{13}SiCl$_3$, respectively. We also report the synthesis of (c-C_7H_{13})$_6$Si$_6$O$_7$(OH)$_4$ (**2**), an incompletely condensed silsesquioxane, which is formally derived from the hydrolytic cleavage of two adjacent silicon atoms from a cube-octameric polyhedral oligosilsesquioxane. These syntheses, which produce multigram quantities of incompletely condensed silsesquioxanes within a few days, provide ready access to "T_7(OH)$_3$" and "T_6(OH)$_4$"[5] silsesquioxane frameworks.



2 R = c-C_7H_{13}

Results and Discussion

The hydrolytic condensation reactions of both c-C_5H_9SiCl$_3$ and c-C_7H_{13}SiCl$_3$, which are readily available from the hydrosilylation of cyclopentene and cycloheptene,[6] respectively, afford white microcrystalline precipitates. With the conditions described for the preparation of **1b** (H_2O/acetone),[1b] the hydrolytic condensation of c-C_5H_9SiCl$_3$ (23 g, 0.113 mol) afforded 1.1 g of crude precipitate after 7 days at 25 °C; an additional 1.3 g of precipitate was collected after an additional 7 days. ^{29}Si and ^{13}C NMR spectroscopy indicated that the crude product was almost entirely **1a**.[7] Analytically pure **1a** was obtained in 15% overall yield (based on starting c-C_5H_9SiCl$_3$) by using a procedure similar to that described for the purification of **1b**.[1b]

A similar reaction performed with c-C_7H_{13}SiCl$_3$ (40 g, 0.173 mol) afforded 4.75 g of precipitate after 6 weeks. NMR spectral data (^{29}Si, ^{13}C) indicated that the crude product contained trisilanol **1c** in ~60% yield, as well as a new compound in ~40% yield. This new compound, which was extracted from the crude product mixture with CH$_2$Cl$_2$ and recrystallized from CH$_2$Cl$_2$/acetone, exhibited two resonances with relative integrated intensities of 2:1 in both the ^{29}Si NMR spectrum and the methine region

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(1) (a) Feher, F. J.; Newman, D. A. *J. Am. Chem. Soc.* **1990**, *112*, 1931–6. (b) Feher, F. J.; Newman, D. A.; Walzer, J. F. *J. Am. Chem. Soc.* **1989**, *111*, 1741–8.

(2) (a) Feher, F. J.; Budzichowski, T. A.; Weller, K. J. *J. Am. Chem. Soc.* **1989**, *111*, 7288–9. (b) Feher, F. J.; Walzer, J. F. *Inorg. Chem.* **1990**, *29*, 1604–11. (c) Feher, F. J.; Gonzales, S. L.; Ziller, J. W. *Inorg. Chem.* **1988**, *27*, 3440–2. (d) Feher, F. J.; Weller, K. J. *Organometallics* **1990**, *9*, 2638–40. (e) Feher, F. J.; Budzichowski, T. A. *Organometallics* **1991**, *10*, 812–5.

(3) (a) Feher, F. J.; Blanski, R. L. *J. Chem. Soc., Chem. Commun.* **1990**, 1614–6. (b) Feher, F. J. *J. Am. Chem. Soc.* **1986**, *108*, 3850–2. (c) Feher, F. J.; Walzer, J. F. *Inorg. Chem.* **1991**, *30*, 1689–94. (d) Feher, F. J.; Walzer, J. F.; Blanski, R. L. *J. Am. Chem. Soc.* **1991**, *113*, 3618–9.

(4) Brown, J. F.; Vogt, L. H. *J. Am. Chem. Soc.* **1965**, *87*, 4313–7.

(5) In silsesquioxane chemistry the symbols M, D, T, and Q are used to represent framework silicon atoms which possess 3, 2, 1, and 0 organyl substituents, respectively. Subscripts are used to indicate the number of framework silicon atoms with the various designations. Trisilanols **1a–c** possess T_7(OH)$_3$ frameworks and tetrasilanol **2** possesses a T_6(OH)$_4$ framework.

(6) Selin, T. G.; West, R. *J. Am. Chem. Soc.* **1962**, *84*, 1863–8.

(7) Most (>95%) of the crude sample is soluble in chloroform. The remaining material was completely insoluble in all common organic solvents. A mass spectrum of this material exhibited peaks at *m/e* 968.8 (M$^+$, 1%) and 899.6 (M$^+$ − C_5H_9, 100%) consistent with a cube-octameric T_8 silsesquioxane (i.e., [(c-C_5H_9)$_8$Si$_8$O$_{12}$]).

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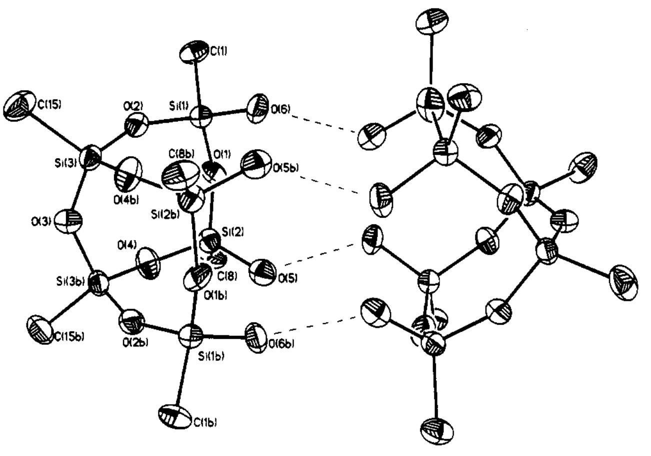


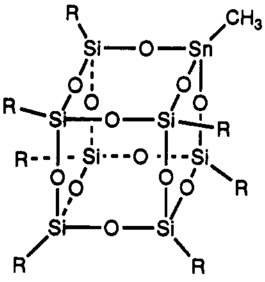
Figure 1. ORTEP plot from a preliminary X-ray crystal structure of **2** with thermal ellipsoids plotted at 50% probability. The molecules crystallize as hydrogen-bonded dimers in the space group $I\bar{4}$. There are two crystallographically independent "molecular halves" per asymmetric unit, each of which generates (by $\bar{4}$ and C_2) a complete dimeric unit. For clarity, only one dimeric unit is illustrated and only C's attached to Si are shown.

of the ^{13}C NMR spectrum (22–26 ppm). Our assignment of this new compound as **2** on the basis of spectroscopic data was confirmed by a single-crystal X-ray diffraction study.[8]

An ORTEP plot of **2** from a preliminary X-ray diffraction study is shown in Figure 1. The molecules cocrystallize from CH_2Cl_2/acetone as strongly hydrogen-bonded dimers in the tetragonal space group $I\bar{4}$. Although the marginal quality of the structure does not permit a meaningful discussion of metrical data (current R_F = ~10%), it is clear from Figure 1 that the molecules adopt an unstrained structure that is formally derived from the hydrolytic removal of two adjacent Si vertices from a completely condensed "T_8" silsesquioxane.

Unlike the hydrolytic condensation of c-$C_6H_{11}SiCl_3$,[1b] which has stubbornly resisted our attempts to accelerate trisilanol formation, the rates and courses of the reactions with c-$C_5H_9SiCl_3$ and c-$C_7H_{13}SiCl_3$ can be dramatically improved by refluxing the reaction mixture. For example, the hydrolytic condensation of c-$C_5H_9SiCl_3$ (65 g, 0.32 mol) in refluxing aqueous acetone afforded **1a** in 29% yield after 3 days. Similarly, the hydrolytic condensation of c-$C_7H_{13}SiCl_3$ (20 g, 0.086 mol) gave 3.45 g (26%) of precipitate, which contained >95% **1c** (by ^{13}C NMR spectroscopy). The reactions of c-$C_5H_9SiCl_3$ and c-$C_7H_{13}SiCl_3$ in refluxing aqueous acetone are clearly the best routes to **1a** and **1c**. Unfortunately, tetrasilanol **2** is not stable in refluxing aqueous acetone and it must still be obtained (along with **1c**) from the slow hydrolytic condensation of c-$C_7H_{13}SiCl_3$ at 25 °C.

Not surprisingly, trisilanols **1a–c** exhibit nearly identical reactivity patterns. The reactions of **1a–c** with Me_3SnCl, for example, all proceed readily to give the expected "corner-capped" products (i.e., **3a–c**). Somewhat unexpectedly, however, the solubility properties of these stoichiometrically similar molecules are quite different.[9] In contrast to **1b** (R = c-C_6H_{11}), which is soluble in a variety of common organic solvents (e.g., $CHCl_3$, Et_2O, THF, C_6H_6), the corresponding cyclopentyl- and cycloheptyl-substituted derivatives have substantially lower solubilities. In $CHCl_3$ (25 °C), for example, the solubilities of **1a**, **1b**, and **1c** are 4.0, 90, and 4.0 mg/mL, respectively.



3a R = c-C_5H_9
b c-C_6H_{11}
c c-C_7H_{13}

The results presented here represent an important advance in the development of silsesquioxanes as models for silica; it is now possible to prepare multigram quantities of incompletely condensed silsesquioxanes in a conveniently short period of time! This clearly overcomes many of the obstacles associated with studying silsesquioxanes, but there are other important consequences of this work. First, the synthesis of **2** finally provides a tetrahydroxylic silsesquioxane ligand that should be capable of accommodating two metal ions in a completely condensed silsesquioxane framework. This tetrasilanol should prove to be useful for developing chemistry relevant to bimetallic silica-supported catalysts (e.g., surface dichromates or dimolybdates). More importantly, the availability of three closely related families of incompletely condensed silsesquioxanes now provides a means by which mechanistically informative "crossover" experiments can be easily incorporated into reactivity studies. Since incompletely condensed silsesquioxanes are the best models developed to date for hydroxylated silica surface sites, these developments should further enhance the utility of these models.

Experimental Section

General experimental protocol and procedures for the preparation and purification of **1b** are described in ref 1b. Cyclopentyltrichlorosilane (c-$C_5H_9SiCl_3$) and cycloheptyltrichlorosilane (c-$C_7H_{13}SiCl_3$) were prepared by the hydrosilylation of cyclopentene and cycloheptene, respectively.[6]

NMR spectra were recorded on a General Electric GN-500 spectrometer (^1H, 500.10 MHz; ^{13}C, 125.76 MHz; ^{29}Si, 99.35 MHz). ^{29}Si NMR spectra were recorded with inverse-gated proton decoupling in order to minimize (negative) nuclear Overhauser enhancement effects. Electron impact mass spectral analyses were conducted on a VG 7070e high-resolution mass spectrometer. Melting points were measured in sealed capillaries under dry nitrogen with a Mel-Temp melting point apparatus and are uncorrected.

Hydrolytic Condensation of c-$C_5H_9SiCl_3$ (Room Temperature). c-$C_5H_9SiCl_3$ (23.0 g, 0.113 mol) was added cautiously and

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(8) Tetrasilanol **2** is extremely soluble in many common organic solvents, but well-formed crystals were eventually obtained by allowing acetone to slowly diffuse into a CH_2Cl_2 solution of **2**. Crystal data for [$C_{49}H_{82}O_{11}Si_8$·(CH_2Cl_2)·(acetone) (fw 1073.3)]: tetragonal $I\bar{4}$, a = b = 26.241 (2) Å, c = 15.706 (2) Å; V = 10816 (1) Å^3; D_{calc} = 1.32 g/cm^3 (Z = 8). A total of 4998 unique reflections with $4.0 \leq 2\theta \leq 50.0°$ were collected on a Siemens $P2_1$ diffractometer at −110 °C with the use of graphite-monochromated Mo Kα radiation. The structure was solved by direct methods (SHELXTL PLUS). Full-matrix least-squares refinement of positional and thermal parameters (anisotropic for Si, O, and isotropic for C) led to convergence with an R factor of 12.8% for 354 variables refined against 4343 data with $|F_o| > 4.0\sigma|F_o|$. Three of the cycloheptyl groups have disordered conformations that could not be successfully modeled. ^1H NMR spectroscopy clearly indicated the presence of both CH_2Cl_2 and acetone in the crystals (~1:1), but neither could be completely located nor refined. Two large peaks (3 e/Å^3) were located in a difference-Fourier synthesis and refined as chlorine atoms from CH_2Cl_2. This lowered the R factor to 8%, but it did not noticeably improve the quality of the structure.

(9) C.f.: Feher, F. J.; Budzichowski, T. A. *J. Organomet. Chem.* **1989**, *373*, 153–63.

with vigorous stirring to 575 mL of aqueous acetone (80% v/v) in a 1-L Wheaton glass bottle. The bottle was sealed with a screw cap and allowed to stand at room temperature. Filtration of the reaction mixture after 7 days afford 1.1 g of white precipitate; an additional 1.3 g was collected from the filtrate after another 7 days. The crude product was suspended in 20 times its weight of pyridine, and then it was filtered to remove particulates. The filtrate was neutralized by pouring over ice-cold aqueous HCl (1 mL of concentrated HCl/mL of pyridine). The precipitated product was collected on a Buchner funnel, washed until neutral with copious amounts of water, and then dried in air. Exhaustive extraction of the solid with diethyl ether (Soxhlet extraction) afforded analytically pure $(c\text{-}C_5H_9)_7Si_7O_9(OH)_3$ (**1a**) as a white microcrystalline powder (15%). ^{1}H NMR (500.1 MHz, $CDCl_3$, 25 °C): δ 6.00 (br s, 3 H), 1.79 (m, 20 H), 1.56 (m, 36 H), 0.97 (m, 7 H). $^{13}C\{^1H\}$ NMR (125.03 MHz, 25% v/v pyridine/$CDCl_3$, 25 °C): δ 26.72, 26.64, 26.56, 26.28, 26.26 (s, for CH_2); δ 23.30, 21.95, 21.57 (s, 3:3:1 for CH). $^{29}Si\{^1H\}$ NMR (99.35 MHz, 25% v/v pyridine/$CDCl_3$, 25 °C): δ −57.73, −65.96, −67.31 (s, 3:1:3). MS (70 eV, 200 °C) (assignment, relative intensity): *m/e* 955 ($M^+ - H_2O - C_5H_9$, 100%), 859 ($M^+ - H_2O - 2C_5H_9$, 3%). Anal. Calcd for $C_{35}H_{66}O_{12}Si_7$ (found): C, 48.00 (47.75); H, 7.60 (7.53).

Hydrolytic Condensation of $c\text{-}C_5H_9SiCl_3$ (Reflux). Distilled water (360 mL) was added cautiously and with vigorous stirring to a solution of $c\text{-}C_5H_9SiCl_3$ (65 g, 0.319 mol) and acetone (1.3 L) in a 3-L round-bottom flask, then the solution was refluxed for 65 h. The resulting white solid was collected on a Buchner funnel and washed with acetone. In order to remove any remaining resinous material, the filter cake was broken up and suspended in acetone (300 mL, 2 h with stirring). After filtration and drying (40–50 °C, 8 h), the crude product (19 g) was suspended in 20 times its weight of pyridine and stirred overnight. A small amount (0.13 g) of insolubles[7] was removed by filtration through a medium sintered-glass Buchner funnel; then the pyridine solution was carefully poured into an equal volume of concentrated HCl mixed with crushed ice. The precipitated product was collected on a Buchner funnel, washed until neutral with copious amounts of water, and then dried overnight in air at 40–50 °C. Exhaustive extraction of the solid with diethyl ether (Soxhlet extraction) afforded analytically pure **1a** as a white microcrystalline powder (11.5 g, 29%). Yields of **1a** obtained from several reactions performed on 2 or 3 times this scale have been as high as 45%.

Hydrolytic Condensation of $c\text{-}C_7H_{14}SiCl_3$ (Room Temperature). Distilled water (182 mL) was added cautiously and with vigorous stirring to a solution of $c\text{-}C_7H_{13}SiCl_3$ (40 g, 0.173 mol) in acetone (688 mL) in a 1-L Wheaton glass bottle. The bottle was sealed with a screw cap and allowed to stand at room temperature. A white precipitate began to form after 24 h and was accompanied by gel formation after 1 week. The solution was decanted after 6 weeks to leave a gelatinous white solid, which was triturated with acetone and filtered. The filter cake was washed with acetone (3 × 20 mL) and air-dried to afford 4.75 g of a fine white powder.

Tetrasilanol **2** was quantitatively extracted from the powder with CH_2Cl_2 (2 × 100 mL). Analytically pure $[(c\text{-}C_7H_{13})_6Si_6O_7(OH)_4]$ (**2**) (1.90 g, 7%) was obtained by evaporating the extracts to ~20 mL and then carefully layering the extracts with acetone (40 mL) to slowly crystallize the product. ^{1}H NMR (500.1 MHz, $CDCl_3$, 25 °C): δ 7.22 (br s, 4 H), 1.9–1.3 (br m, 72 H), 0.83 (m, 6 H). $^{13}C\{^1H\}$ NMR (125.03 MHz, $CDCl_3$, 25 °C): δ 29.65, 29.63, 29.30, 28.59, 28.32, 28.30, 27.96, 27.93, 27.91 (s, CH_2); δ 24.24, 23.75 (s, 2:1 for CH). $^{29}Si\{^1H\}$ NMR (99.35 MHz, $CDCl_3$, 25 °C): δ −58.61, −67.65 (s, 2:1). MS (70 eV, 200 °C) (assignment, relative intensity): *m/e* 895 ($M^+ - 2H_2O$, 40%), 815 ($M^+ - H_2O - C_7H_{13}$), 799 ($M^+ - 2H_2O - C_7H_{12}$, 55%). Anal. Calcd for $C_{42}H_{82}O_{11}Si_6$ (found) after desolvation at 60 °C (10^{-3} mTorr): C, 54.15 (54.36); H, 8.87 (9.03). Mp: 274–280 °C dec.

The CH_2Cl_2-insoluble solid was almost entirely **1c** (by ^{13}C NMR spectroscopy). Analytically pure $[(c\text{-}C_7H_{13})_7Si_7O_9(OH)_3]$ (**1c**) (2.50 g, 9.5%) was obtained by extracting the solid with hot pyridine/$CHCl_3$ (20%, v/v), evaporating the volatiles (25 °C, 10^{-3} Torr), and recrystallizing the residue from hot $CHCl_3$. ^{1}H NMR (500.1 MHz, $CDCl_3$, 25 °C): δ $^{13}C\{^1H\}$ NMR (125.03 MHz, 25% v/v pyridine/$CDCl_3$, 25 °C): δ 28.93, 28.77, 28.61, 27.97, 27.86, 27.75, 27.48, 27.40, 27.31 (s, 6:6:2:2:6:6:6:6:2 for CH_2); δ 23.97, 23.58, 23.19 (s, 3:3:1 for CH). $^{29}Si\{^1H\}$ NMR (99.35 MHz, 25% v/v pyridine/$CDCl_3$, 25 °C): δ −58.44, −66.73, −68.08 (s, 3:1:3). MS (70 eV, 200 °C) (assignment, relative intensity): *m/e* 955 ($M^+ - H_2O - C_7H_{13}$, 100%), 859 ($M^+ - H_2O - 2C_7H_{13}$, 3%). Anal. Calcd for $C_{49}H_{94}O_{12}Si_7$ (found): C, 54.91 (54.88); H, 8.84 (9.15). Mp: 328–340 °C dec.

Like the hydrolytic condensation of $c\text{-}C_6H_{11}SiCl_3$,[1b] the course of the reaction does not appear to be effected by periodically decanting the reaction mixture. After 4 weeks more at room temperature an additional 2.40 g of precipitate with the same composition of **1c** and **2** was isolated. The total yield of condensed products after 10 weeks was 7.15 g (~27%).

Hydrolytic Condensation of $c\text{-}C_7H_{14}SiCl_3$ (Reflux). Distilled water (92 mL) was added cautiously and with vigorous stirring to a solution of $c\text{-}C_7H_{13}SiCl_3$ (20 g, 0.0864 mol) in acetone (344 mL). The solution was refluxed for 3 days, and then it was cooled to afford a white gelantinous precipitate, which was triturated with acetone as described above to afford 3.45 g (26%) of fine white powder. The crude product contained >95% **1c** (by ^{13}C NMR spectroscopy), so extraction of the powder with CH_2Cl_2 yielded no **2**. Recrystallization from hot $CHCl_3$ (100 mL) afforded analytically pure **1c**.

Preparation of $(c\text{-}C_5H_9)_7Si_7O_{12}SnMe$ (3a). Via the procedure described for the preparation of **3b**,[1b] solid methyltrichlorostannane (82 mg, 0.342 mmol) was added to a solution of **1a** (300 mg, 0.343 mmol) and Et_3N (1 mL) in benzene (10 mL). The mixture for stirred for 2 h, and then the volatiles were removed in vacuo (25 °C, 10^{-4} Torr). Extraction of the residue with hexanes and solvent removal in vacuo (25 °C, 10^{-4} Torr) afforded **3a** as an amorphous white solid. Recrystallization by slow diffusion of acetonitrile into a concentrated benzene solution of **3a** afforded 120 mg (35%) of colorless crystals. ^{1}H NMR (500.1 MHz, C_6D_6, 25 °C): δ 2.00 (br m, 15 H), 1.79 (br m, 14 H), 1.70 (br m, 14 H), 1.52 (br m, 13 H); 1.25 (br m, 7 H), 0.286 (s, Me, J_{Sn-H} = 43, 100 Hz). $^{13}C\{^1H\}$ NMR (125.03 MHz, C_6D_6, 25 °C): δ 28.44, 27.98, 27.55, 27.52 (s, CH_2); δ 24.07, 23.15, 22.84 (s, 3:3:1 for CH); δ −4.32 (Sn–CH_3). $^{29}Si\{^1H\}$ NMR (99.35 MHz, C_6D_6, 25 °C): δ −62.45, −65.50, −67.00 (s, 3:1:3). MS (70 eV, 200 °C) (assignment, relative intensity): *m/e* 937 ($M^+ - C_5H_9$, 100%), 869 ($M^+ - 2C_5H_9$, 50%). Anal. Calcd for $C_{36}H_{66}O_{12}Si_7Sn$ (found): C, 42.97 (42.92); H, 6.61 (6.82).

Preparation of $(c\text{-}C_7H_{13})_7Si_7O_{12}SnMe$ (3c). Solid methyltrichlorostannane (45 mg, 0.187 mmol) was added to a solution of **1a** (200 mg, 0.186 mmol) and 5% (v/v) Et_3N/benzene (15 mL). The mixture for stirred for 12 h, then the volatiles were removed in vacuo. Extraction with benzene (2 × 10 mL), evaporation of the filtrate to ~2 mL and crystallization by acetonitrile diffusion afforded 165 mg (74%) of **3c** as colorless crystals. ^{1}H NMR (500.1 MHz, $CDCl_3$, 25 °C): δ 1.9–1.3 (br m, 84 H), 0.94 (s, 3 H), 0.83 (br m, 7 H). $^{13}C\{^1H\}$ NMR (125.03 MHz, $CDCl_3$, 25 °C): δ 29.55, 29.43, 29.40, 28.61, 28.54, 28.47, 28.01, 27.96 (s, CH_2); δ 25.01, 24.01, 23.90 (s, 3:3:1 for CH). $^{29}Si\{^1H\}$ NMR (99.35 MHz, $CDCl_3$, 25 °C): δ −62.80, −65.89, −67.30 (s, 3:3:1). MS (70 eV, 200 °C) (assignment, relative intensity: *m/e* 1188 ($M^+ - CH_3$, 2%), 1106 ($M^+ - C_7H_{13}$, 100%), 1092 ($M^+ - CH_3 - C_7H_{13}$, 8%), 1009 ($M^+ - C_7H_{13}, - C_7H_{12}$ 82%), 993 ($M^+ - CH_3 - 2C_7H_{13}$, 23%). Anal. Calcd for $C_{42}H_{82}O_{11}Si_6$ (found): C, 49.93 (49.97); H, 7.88 (7.89).

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Supplementary Material Available: X-ray crystal data for **2**, including a textual presentation of experimental procedures, tables of crystal data, atomic coordinates, thermal parameters, bond lengths, and bond angles, and figures of the molecule (19 pages); listings of calculated and observed structure factors for **2** (36 pages). Ordering information is given on any current masthead page.