Synthesis, Derivatization, and Structure of the Silanetriol [$C_5H_4(SiMe_3)$]$Si(OH)_3$: Unique Hydrogen Bonding in a Highly Symmetrical Tubular Assembly[†]

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*Summary: (5-(Trimethylsilyl)cyclopenta-1,3-dien-5-yl)silanetriol, [$C_5H_4(SiMe_3)$]$Si(OH)_3$ (**2**), has been synthesized by controlled hydrolysis of the silicon trichloride [$C_5H_4(SiMe_3)$]$SiCl_3$ (**1**). The crystal structure of compound **2** confirms a new structure type for silanetriols: intermolecular hydrogen bonding leads to a highly symmetrical tubelike arrangement. Stannylation of **2** utilizing $N(SnMe_3)_3$ yields [$C_5H_4(SiMe_3)$]$Si(OSnMe_3)_3$ (**3**) as a valuable synthon for the silanetriol.*

Introduction

In recent years the silanetriols have attracted a great deal of attention because they represent versatile building blocks for the construction of polyhedral silsesquioxanes or metallasilsesquioxanes.[1] With respect to the transformation of such oligomers into supramolecular structures special efforts have been directed toward the preparation of stable silanetriols bearing hydrolyzable functionalities as substituents. Our approach to this topic is the application of cyclopentadienyl (Cp) ligands for the synthesis of silanetriols. Because of the excellent leaving-group ability of the Cp moiety,[2] isolation of Cp-functionalized silanetriols might present a new perspective in this field. Moreover, Cp chemistry offers the advantage of fine-tuning the steric and electronic properties by modifying the substitution pattern of the Cp system.[2] In the context of silanol chemistry we have successfully pursued this strategy: previously, we described the synthesis and the crystal structure of the silanediol $Cp^*_2Si(OH)_2$[3] as well as the silanetriol $Cp^*Si(OH)_3$.[4]

In this paper we report on the synthesis and structural characterization of (5-(trimethylsilyl)cyclopenta-1,3-dien-5-yl)silanetriol, [$C_5H_4(SiMe_3)$]$Si(OH)_3$ (**2**). Furthermore, we demonstrate the complete substitution of the silanol protons in the reaction of **2** with $N(SnMe_3)_3$, resulting in the formation of [$C_5H_4(SiMe_3)$]$Si(OSnMe_3)_3$ (**3**).

Results and Discussion

The silicon trichloride [$C_5H_4(SiMe_3)$]$SiCl_3$ (**1**) serves as the starting material for the preparation of the silanetriol **2**. It can be obtained as an oily liquid in high yield by a two-step synthesis from $C_5H_4(SiMe_3)$. Controlled hydrolysis of **1**, applying a method first described by Takiguchi,[5] leads to the quantitative formation of the silanetriol **2**. The sequence for the synthesis of **2** is depicted in Scheme 1. Compound **2** is obtained as a colorless microcrystalline solid and is readily soluble in polar solvents such as diethyl ether, THF, acetone, dimethyl sulfoxide, acetonitrile, and ethanol and nearly insoluble in aliphatic and aromatic solvents. Compound **2** can be heated as a solid or as an ethereal solution for a prolonged time without self-condensation taking place; exposure to air leads to slow decomposition.

A complete substitution of the three Si−OH protons of **2** can be easily performed. Thus, by a literature procedure[6] the silanetriol **2** reacts with 1 equiv of $N(SnMe_3)_3$ to give in good yield the stannylated derivative [$C_5H_4(SiMe_3)$]$Si(OSnMe_3)_3$ (**3**) as a viscous oil (see Scheme 1). Compounds of the type $RSi(OSnMe_3)_3$ represent useful synthons for silanetriols due to the pronounced reactivity of the Sn−O bond toward electrophiles and nucleophiles.[7]

Compounds **1**−**3** were characterized by elemental analysis, mass spectrometry, and IR and multinuclear NMR spectroscopy; in the case of **2** an X-ray analysis was also performed.

Selected spectroscopic properties of the silanetriol **2** will be explained in more detail. The IR spectrum of **2** shows only one strong, broad absorption band at 3334 cm^{-1} in the characteristic region for OH-stretching frequencies, indicating that all three OH groups are involved in hydrogen bonding.[8] Concerning the ^{29}Si NMR spectrum of **2** a resonance at −72.3 ppm (DMSO-d_6) for the SiO_3 unit is observed, which is shifted considerably upfield in comparison with other C-bonded silanetriols.[9] This strong shielding implies electron donation by the $C_5H_4(SiMe_3)$ fragment. The remarkable stability of the silanetriol **2** may thus be considered as a consequence of the reduced electrophilicity of the silicon atom in addition to steric effects of the organic ligand.

Crystallographic data for the silanetriol **2** are collected in Table 1; selected structural parameters are given in Table 2. Important features of the crystal structure of **2** are depicted in Figures 1−4. The structure of **2** can be described as a polymeric arrangement of molecular subunits based on hydrogen bonding. This interaction leads to a highly symmetrical network in the shape of a tubular structure. The resulting tubes

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[†] Dedicated to Professor Manfred Weidenbruch on the occasion of his 60th birthday.

[⊗] Abstract published in *Advance ACS Abstracts,* November 1, 1997.

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Scheme 1

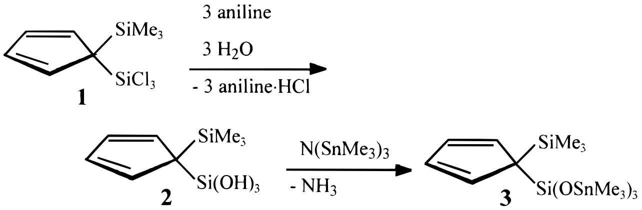

Table 1. Crystallographic Data for Diffraction Studies of 2

empirical formula	$C_8H_{16}O_3Si$
fw	216.39
cryst color, habit	colorless, plates
cryst size	1.00 × 0.30 × 0.20 mm^3
cryst syst	monoclinic
space group	*C*2/*c*
unit cell dimens	$a = 28.429(9)$ Å $b = 6.489(2)$ Å $c = 26.799(7)$ Å $\beta = 109.14(2)°$
V	4669(2) $Å^3$
Z	16
density (calcd)	1.231 Mg/m^3
abs coeff	0.281 mm^{-1}
F(000)	1856
wavelength	0.710 73 Å (graphite monochromator)
θ range for data collection	1.50−22.50°
index ranges	$0 \leq h \leq 30$, $0 \leq k \leq 6$, $-28 \leq l \leq 27$
no. of rflns collected	3118
no. of indep rflns	3044 ($R_{int} = 0.0722$)
abs corr	none
no. of data/restraints/params	3041/0/247
goodness of fit on F^2	1.106
final *R* indices ($I > 2\sigma(I)$)[a]	R1 = 0.0816 for 2078 rflns, wR2 = 0.2022
R indices (all data)	R1 = 0.1215, wR2 = 0.2414
largest and mean Δ/σ	0.000 and 0.000
largest diff peak and hole	+0.5 and −0.7 e/$Å^3$

[a] $R1 = \sum||F_o| - |F_c||/\sum|F_o|$, $wR2 = [\sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2]]^{1/2}$; $w(calcd) = 1/[\sigma^2(F_o^2) + (0.1189P)^2 + 30.6622P]$, where $P = (F_o^2 + 2F_c^2)/3$.

Table 2. Selected Structural Parameters for 2

	Bond Lengths (Å)		
Si(1)−O(1)	1.625(5)	Si(1′)−O(1′)	1.645(5)
Si(1)−O(2)	1.634(5)	Si(1′)−O(2′)	1.611(5)
Si(1)−O(3)	1.625(5)	Si(1′)−O(3′)	1.627(5)
Si(1)−C(1)	1.832(7)	Si(1′)−C(1′)	1.868(7)
Si(2)−C(1)	1.913(7)	Si(2′)−C(1′)	1.912(7)
C(1)−C(2)	1.495(10)	C(1′)−C(2′)	1.476(11)
C(2)−C(3)	1.346(12)	C(2′)−C(3′)	1.353(11)
C(3)−C(4)	1.424(12)	C(3′)−C(4′)	1.414(14)
C(4)−C(5)	1.350(11)	C(4′)−C(5′)	1.311(13)
C(1)−C(5)	1.503(11)	C(1′)−C(5′)	1.465(11)
	Bond Angles (deg)		
O(1)−Si(1)−O(2)	111.7(3)	O(1′)−Si(1′)−O(2′)	104.0(3)
O(2)−Si(1)−O(3)	109.6(3)	O(2′)−Si(1′)−O(3′)	109.8(3)
O(1)−Si(1)−O(3)	104.1(3)	O(1′)−Si(1′)−O(3′)	111.1(3)
C(2)−C(1)−C(5)	101.4(6)	C(2′)−C(1′)−C(5′)	103.3(7)
C(1)−C(2)−C(3)	110.2(8)	C(1′)−C(2′)−C(3′)	108.3(8)
C(2)−C(3)−C(4)	109.3(7)	C(2′)−C(3′)−C(4′)	108.4(8)
C(3)−C(4)−C(5)	109.5(8)	C(3′)−C(4′)−C(5′)	110.7(8)
C(4)−C(5)−C(1)	109.6(7)	C(4′)−C(5′)−C(1′)	109.2(8)
Si(1)−C(1)−Si(2)	116.9(4)	Si(1′)−C(1′)−Si(2′)	115.5(4)

may be considered as hydrophilic channels formed by the $Si(OH)_3$ units which are covered by a lipophilic wrapping consisting of the $C_5H_4(SiMe_3)$ moieties.

Some aspects of the molecular subunit will be described first. There are two molecules in the asymmetric unit. In view of their nearly identical structural parameters the following discussion refers only to one of these molecules, shown in Figure 1.

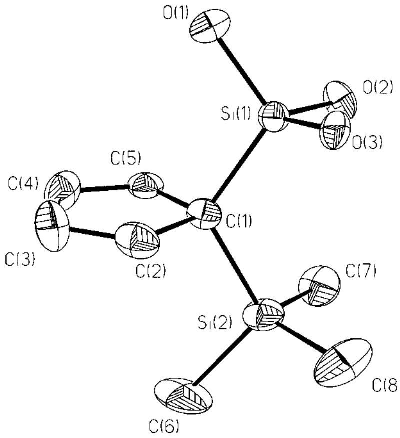

Figure 1. Molecular structure of **2**, showing one of the two crystallographically independent molecules.

The Cp moiety of **2** reveals a distorted-pseudotetrahedral geometry at C(1) (the Si−C−Si angle is increased to 116.9(4)° and the C−C−C angle decreased to 101.4(6)°), which is in agreement with similiar structures bearing two silyl fragments at the allylic carbon atom.[10] A significant difference in length between the two Si−C(Cp) bonds is found, with the $Si(OH)_3$−C(Cp) distance being considerably shorter (Si(1)−C(1) = 1.832(7) Å) compared with the $SiMe_3$−C(Cp) bond (Si(2)−C(1) = 1.913(7) Å). This observation is satisfactorily explained by the concept that shortening of a chemical bond occurs as a consequence of decreased p-orbital participation caused by electron-attracting substituents.[11] The Si−O bond lengths in the range of 1.611(5)−1.645(5) Å and the O−Si−O bond angles at 104.0(3)-111.7(3)° are comparable with those observed for other silanetriols.[12] The bond distances within the Cp ring system show the expected variation between double and single bonds.

The most interesting feature of the crystal structure of **2** concerns the type of polymer which is formed by hydrogen bonding between the molecular units. This interaction leads to a highly symmetrical structure in the shape of a one-dimensional tubular network. Each tube is created from four columns, which are formed by stacking the $[C_5H_4(SiMe_3)]Si(OH)_3$ units on top of each other. The polymeric arrangement can be easily understood by noting the horizontal projection of the tubular network depicted in Figure 2. Further details concerning the structure of **2** can be deduced from Figure 3, which shows a side view of the tubular network (the $C_5H_4(SiMe_3)$ moieties are omitted for clarity).

All molecules within a certain column are oriented exactly in the same direction, with one Si−OH bond directed along the column and the other two Si−OH bonds across the column. Opposite and neighboring columns differ from each other in two aspects. First, concerning the $Si(OH)_3$ fragment, Si−OH bonds in neighboring columns point in different directions, whereas in opposite columns they show strictly the same orientation. The second point is the displacement of the

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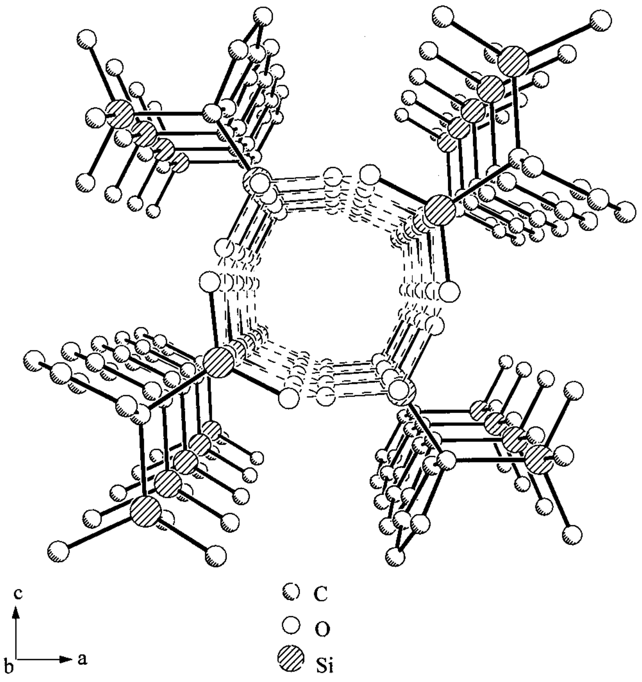

Figure 2. Top view of a tube composed of four molecular columns.

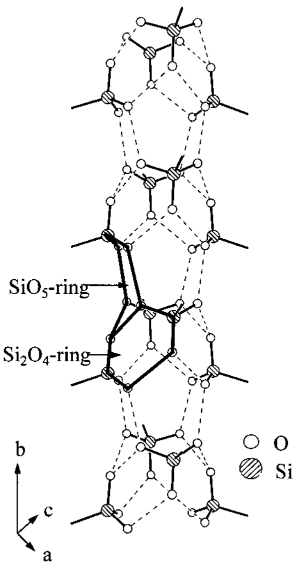

Figure 3. Side view of the hydrogen-bonded tubular network of **2**. The $C_5H_4(SiMe_3)$ moieties are omitted for clarity.

molecular units belonging to different columns. Molecules of opposite columns are shifted about the same amount with respect to those of neighboring columns. The high symmetry of the polymeric structure is manifested in a C_2 axis along the center of the tube and represents the unique feature of this structure. It marks the crucial difference from the only other known tubular structure, where a lower symmetry and incomplete hydrogen bonding are observed.[13b]

Every oxygen and every hydrogen atom of the $[C_5H_4(SiMe_3)]Si(OH)_3$ unit is involved in a single intermolecular hydrogen bond. The intermolecular O−O distances are in the range 2.693−2.787 Å, which are comparable with those for other silanetriols,[13] implying strong hydrogen bonding. The hydrogen-bonded network is composed of six-membered SiO_5 and Si_2O_4 rings in boat conformation (hydrogen atoms are ignored), which are connected in a very regular manner, as is emphasized in Figure 3.

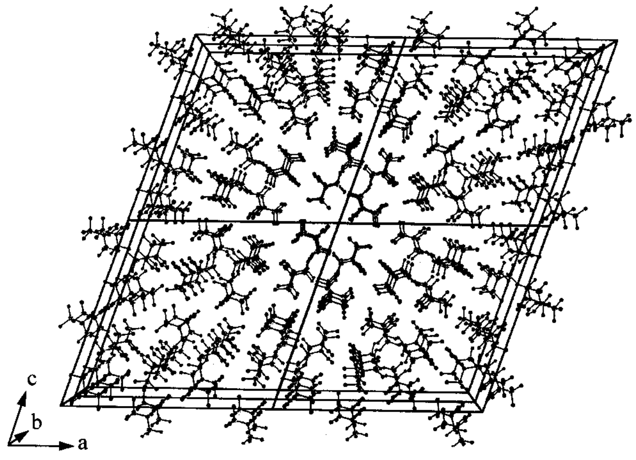

Figure 4. Arrangement of the tubes in the crystal structure of **2**.

The overall three-dimensional structure of **2** shows the specific arrangement of the individual tubes relative to each other. It can be seen from a horizontal projection, given in Figure 4, that all tubes are organized parallel to each other. On the basis of this observation crystals of **2** should reveal a remarkable anisotropy in their physical properties.

We are currently investigating condensation reactions of compound **2**. Due to the excellent leaving-group ability of Cp systems compound **2** represents an important building block for the design of new materials with a Si−O backbone.

Experimental Section

General Comments. All experiments were carried out under an atmosphere of purified and dry argon using standard Schlenk techniques. Solvents were dried and distilled prior to use by standard methods. Melting point determinations were conducted by using a Büchi 510 melting point apparatus. The elemental analyses were performed by the Microanalytical Laboratory of the chemistry faculty at the University of Bielefeld. NMR spectra were recorded on a Bruker Avance DRX 500 spectrometer (^1H, 500.1 MHz; $^{13}C\{^1H\}$, 125.8 MHz; $^{29}Si\{^1H\}$, 99.4 MHz; $^{119}Sn\{^1H\}$, 186.5 MHz). Chemical shifts are reported with reference to the respective solvent as internal standard (^1H, ^{13}C) or to an external standard (^{29}Si, $SiMe_4$; ^{119}Sn, $SnMe_4$). IR spectra were obtained on a Bruker Vector 22 spectrometer (only strong absorption values are given). Mass spectrometry (EI, 70 eV) was performed using a VG Autospec spectrometer. Only characteristic fragments and isotopes of highest abundance are mentioned.

Materials. $C_5H_4(SiMe_3)$ was prepared following the reported procedure.[14] *n*-Butyllithium was used as a 1.5 M solution in *n*-hexane (Aldrich). Silicon tetrachloride (Aldrich) was purified by distillation and stored over magnesium filings. Aniline (Aldrich) was purified by distillation. Tris(trimethylstannyl)amine, $N(SnMe_3)_3$, was prepared according to the literature.[15]

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$[C_5H_4(SiMe_3)]SiCl_3$ (1). A stirred solution of 9.69 g (70.1 mmol) of $C_5H_4(SiMe_3)$ in 500 mL of *n*-hexane was treated dropwise at 0 °C with 47 mL (70.1 mmol) of *n*-BuLi. After complete addition the reaction mixture was warmed to room temperature while stirring was continued for a further 12 h. The resulting suspension was slowly dropped into a cooled solution (−78 °C) of 36.0 g (210 mmol) of $SiCl_4$ in 50 mL of *n*-hexane. Afterward the cooling bath was removed and the reaction mixture was stirred for another 6 h. The precipitated LiCl was removed by filtration and volatiles stripped off applying reduced vacuum (20 Torr). The remaining viscous oil was distilled under vacuum to afford $[C_5H_4(SiMe_3)]SiCl_3$ as a colorless oil in 79% yield. Bp: 50 °C/0.8 Torr. ^1H NMR ($CDCl_3$; δ): 5,5-isomer, 0.09 (s, 9H, *SiMe$_3$*), 6.58, 6.92 (d, 2H, *vinyl-H*), additional signals due to other isomers, 0.03 (s, 9H, *SiMe$_3$*), 3.33 (s, 1H, *allyl-H*) ppm. ^{13}C NMR ($CDCl_3$; δ): 1.17 (*SiMe$_3$*), 60.0 (*allyl-C*), 132.3, 135.2 (*vinyl-C*) ppm. ^{29}Si NMR ($CDCl_3$; δ): 1.65 (*SiMe$_3$*), 2.25 (*SiCl$_3$*) ppm. IR (KBr; ν): 529 (vs), 567 (vs), 587 (vs), 738 (vs), 822 (vs), 843 (vs), 947 (m), 976 (s), 1023 (m), 1046 (m), 1084 (m), 1121 (s), 1254 (vs), 1410 (w), 1439 (w), 1463 (w), 2901 (m), 2960 (s), 3070 (w), 3092 (w), 3110 (w) cm^{-1}. MS (*m/z* (relative intensity, %) [fragment]): 270 (17, [M$^+$]), 255 (39, [M$^+$ − Me]), 201 (31, [M$^+$ − 2Cl]), 73 (100, [$SiMe_3{}^+$]). Anal. Calcd for $C_8H_{13}Cl_3Si_2$ (271.75): C, 35.36; H, 4.82. Found: C, 35.18; H, 4.95.

$[C_5H_4(SiMe_3)]Si(OH)_3$ (2). A solution of 4.63 g (17.0 mmol) of $[C_5H_4(SiMe_3)]SiCl_3$ in 40 mL of Et_2O was very slowly dropped into a suspension of 4.76 g (57.1 mmol) of aniline and 0.92 g (57.1 mmol) of H_2O in 300 mL of Et_2O, cooled to 0 °C. After the addition was complete, stirring was continued for 3 h, keeping the temperature at 0 °C. Following the removal of anilinium hydrochloride by filtration, the solvent was distilled off in vacuo, leaving a colorless solid. Washing this residue with pentane affords 3.61 g (16.7 mmol) of $[C_5H_4(SiMe_3)]Si(OH)_3$. Yield: 98%. Mp: 118 °C dec. ^1H NMR (DMSO-d_6; δ): 5,5-isomer, 0.00 (s, 9H, *SiMe$_3$*), 5.76 (s, 3H, *OH*), 5.78, 6.10 (m, 2H, *vinyl-H*); additional signals due to other isomers, −0.08 (s, 9H, *SiMe$_3$*), 2.97, 3.01 (s, 1H, *allyl-H*), 5.29 (s, 3H, *OH*) ppm. ^{13}C NMR (DMSO-d_6; δ): 1.80 (*SiMe$_3$*), 84.9 (*allyl-C*), 109.1, 111.0 (*vinyl-C*) ppm. ^{29}Si NMR (DMSO-d_6; δ): −72.3 (*Si(OH)$_3$*), 10.8 (*SiMe$_3$*) ppm. IR (KBr; ν): 482 (s), 734 (s), 746 (vs), 796 (vs), 825 (s), 841 (s), 915 (vs), 982 (vs), 1025 (s), 1121 (s), 1254 (vs), 2963 (s), 3076 (s), 3334 (vs, br) cm^{-1}. MS (*m/z* (relative intensity %) [fragment]): 207 (2, [$M_2{}^{2+}$ − H_2O; condensation product]), 138 (16, [$SiMe_3(C_5H_5)^+$]), 123 (30, [$SiMe_2(C_5H_5)^+$]), 73 (100, [$SiMe_3{}^+$]). Anal. Calcd for $C_8H_{16}Si_2O_3$ (216.39): C, 44.40; H, 7.45. Found: C, 44.28; H, 7.45.

$[C_5H_4(SiMe_3)]Si(OSnMe_3)_3$ (3). The treatment of a suspension of 0.78 g (3.60 mmol) of $[C_5H_4(SiMe_3)]Si(OH)_3$ in 5 mL of hexane with a solution of 1.82 g (3.6 mmol) of $N(SnMe_3)_3$ in 5 mL of hexane at room temperature leads to strong gas production. For completion of the reaction stirring was continued for 18 h. Then the solvent was stripped off in vacuo, leaving 2.51 g (3.56 mmol) of a slightly yellow oil. No further purification of this material was conducted. Yield: 99%. ^1H NMR (C_6D_6; δ): 5,5-isomer, 0.18 (s, 9H, *SiMe$_3$*), 0.27 (s, 27H, $^2J(^{117}Sn-H) = 56$ Hz, $^2J(^{119}Sn-H) = 58$ Hz, *SnMe$_3$*), 3.55 (s, br, 1H, *allyl-H*), 6.75, 6.91 (m, br, 3H, *vinyl-H*); additional signals due to other isomers, 0.37 (s, 9H, *SiMe$_3$*) ppm. ^{13}C NMR (C_6D_6; δ): 5,5-isomer, −6.62 (*SiMe$_3$*), −2.41 (*SnMe$_3$*), 68.9 (*allyl-C*), 101.4 (*allyl-C*), 128.9, 137.4 (*vinyl-C*); additional signals due to other isomers, 0.21 (*SiMe$_3$*), 130.1, 133.2, 142.6 (*vinyl-C*) ppm. ^{29}Si NMR (C_6D_6; δ): 5,5-isomer, −10.1 (*SiMe$_3$*), −58.8 (*Si(OSnMe$_3$)$_3$*); additional signals due to other isomers, −10.7 (*SiMe$_3$*), −57.4 (*Si(OSnMe$_3$)$_3$*) ppm. ^{119}Sn NMR (C_6D_6; δ): 5,5-isomer, 95.1 (*SnMe$_3$*); additional signals due to other isomers, 96.2 (*SnMe$_3$*) ppm. IR (KBr; ν): 538 (vs), 730 (vs), 770 (vs), 835 (vs), 909 (s), 938 (vs), 973 (vs), 1018 (vs), 1098 (s), 1246 (s), 1261 (s), 1420 (m), 2916 (s), 2957 (s) cm^{-1}. MS (*m/z* (relative intensity %) [fragment]): 673 (6, [M$^+$ − 2Me − H]), 567 (23, [M$^+$ − CpSiMe$_3$]), 495 (23, [M$^+$ − 2Me − OSnMe$_3$]), 165 (100, [$OSnMe_3{}^+$]). Anal. Calcd for $C_{17}H_{40}O_3Si_2Sn_3$ (704.91): C, 28.96; H, 5.71. Found: C, 28.81; H, 5.84.

X-ray Structure Determination. A single crystal of **2** was obtained by slow diffusion of pentane into an Et_2O solution. It was coated with a layer of hydrocarbon oil, attached to a glass fiber, and cooled to to 173(2) K for data collection on a Siemens P2(1) diffractometer. Crystallographic programs used for structure solution were Siemens SHELXTL Plus and SHELXL-93. The structure was solved by using direct methods and was refined by using full-matrix least squares with anisotropic thermal parameters for all non-hydrogen atoms. All hydrogen atoms were fixed at the calculated positions using a riding model.

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Supporting Information Available: Tables giving measurement and program parameters, crystal data and structure refinement details, atomic coordinates and isotropic displacement parameters, bond lengths and angles, and anisotropic displacement parameters for **2** (6 pages). Ordering information is given on any current masthead page.

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