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Tris- and Tetrakis(lithiomethyl)silanes: An Easy Access to New Building Blocks for Organosilicon Compounds[†]

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Tris(lithiomethyl)silanes, $RSi(CH_2Li)_3$, and tetrakis(lithiomethyl)silane, $Si(CH_2Li)_4$, were prepared by the reductive C−S bond cleavage with lithium *p*,*p*′-di-*tert*-butylbiphenylide (LiDBB) and characterized by trapping with Bu_3SnCl. The yields of the isolated trapping products were 42−81% (>95% NMR yield of the crude product), indicating a high-yield synthesis of the corresponding poly(lithiomethyl)silane building blocks. Tetrakis(lithiomethyl)silane is the first compound containing four lithioalkyl groups without any stabilization of the metalated carbon atoms by π-systems. Tetrakis(lithiomethyl)silane was used for the synthesis of 2,2,3,3,7,7,8,8-octamethyl-2,3,5,7,8-pentasilaspiro[4,6]nonane (**10**), a new spirocyclic disilane. The single-crystal X-ray diffraction study of **10** indicates two five-membered rings in envelope conformation with ecliptically arranged methyl groups connected to a spiro compound by a central silicon atom. DFT geometry optimizations [B3LYP/6-31G(d) level] and NMR calculations (GIAO method, HF/6-31+G(d,p) level) of **10** confirm the observed conformation of **10** as an energetic minimum and support the experimental results.

Introduction

Organic compounds containing two or more lithium atoms in one molecule are defined as polylithium organic compounds. Two recent reviews of polymetalated compounds have been published by Maercker.[1] The known triply, quadruply, or multiply metalated molecules are of the tetralithiomethane type or are polylithiated alkenes, arenes or π-systems. The polylithium compound **1** is the first known triply metalated organic compound, where the metalated carbon atoms are not stabilized by π-systems.[1−3]

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Despite the large number of dilithioalkyl compounds, only a few examples of 1,3-dilithioalkyl compounds have been described.[4,5] These have great synthetic potential as bifunctional building blocks, e.g., for the synthesis of cyclobutane derivatives,[4,5] but were not available, mainly due to the lack of appropriate synthetic routes. Also, their decomposition by β-elimination of LiH (e.g., 1,3-dilithiopropane decomposes at −60 °C to allyllithium[6]) prevented their preparation.[1]

As a part of our systematic studies[4,7,8] on the structural unit "$-\mathbf{CR_2}-\mathbf{M}-\mathbf{CR_2}-$" [M = element of groups 14−16, partly with substituents (R = H, alkyl, aryl)], we have investigated the synthesis of bis(lithiomethyl)silanes with the structural unit "$Li\mathbf{CH_2}-\mathbf{SiR_2}-\mathbf{CH_2}Li$" (M = SiR_2). The silicon atom stabilizes the lithio substituent in the α-position and prevents the β-elimination reaction.[9]

We succeeded in preparing the first non π-stabilized bis(lithiomethyl)silanes by reductive cleavage of C−S bonds with electron transfer reagents.[4,10] Bis(phenylthiomethyl)silanes, synthesized by reaction of dichlorosilanes with (phenylthiomethyl)lithium, were converted to the corresponding bis(lithiomethyl)silanes by reaction with lithium naphthalenide ($LiC_{10}H_8$) or lithium *p*,*p*′-di-*tert*-butylbiphenylide (LiDBB). These then were derivatized by reaction with chlorostannanes and chlorosilanes.

[†] Dedicated to Professor Helmut Werner on the occasion of his 66th birthday.

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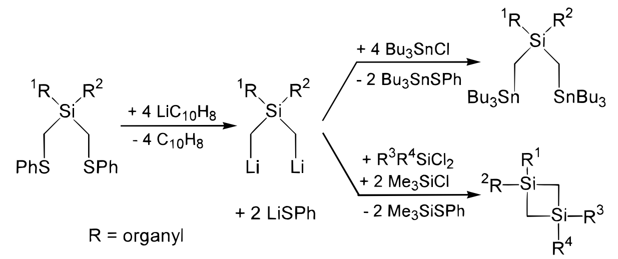
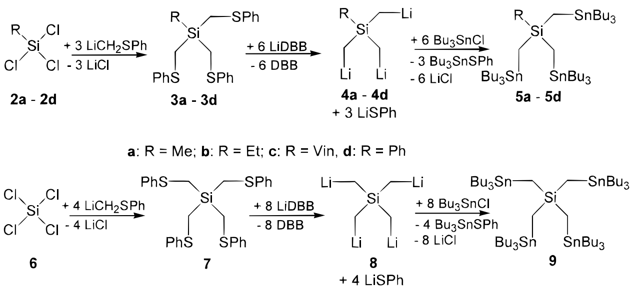
10.1021/om000596z CCC: $19.00

Scheme 1



Scheme 2



We report here the application of the reductive cleavage of C−S bonds with $LiC_{10}H_8$ and LiDBB to the first synthesis of tris(lithiomethyl)silanes and tetrakis(lithiomethyl)silane, as well as some of their synthetic conversions (Scheme 1).

Results and Discussion

Preparation of Tris(lithiomethyl)silane and Tetrakis(lithiomethyl)silane Reagents. The tris(phenylthiomethyl)silanes **3a**−**d** and tetrakis(phenylthiomethyl)silane (**7**) used for reductive cleavage were prepared by reaction of (phenylthiomethyl)lithium with the corresponding chlorosilanes **2a**−**d** and **6** (Scheme 2). (Phenylthiomethyl)lithium was synthesized by two methods: reaction of thioanisole, 1,4-diazabicyclo[2,2,2]octane (DABCO), and *n*-BuLi in THF[11] and reaction of thioanisole and *n*-BuLi in diethyl ether.[12] (Phenylthiomethyl)lithium prepared by the second procedure was easier to handle since the reagent obtained by the first procedure decomposes at 0 °C.

The tris(phenylthiomethyl)silanes **3a**−**d** were isolated by Kugelrohr distillation (**3a**−**c**) or by crystallization (**3a**, **3d**) in 53%−60% yield. Tetrakis(phenylthiomethyl)silane (**7**) was isolated by crystallization in 52% yield.

The reductive C−S bond cleavage is a special metalation reaction which occurs readily at low temperature.[10] The use of electron transfer reagents such as $LiC_{10}H_8$ or LiDBB results in a fast reaction in a homogeneous phase. LiDBB is preferred since it has a higher reduction potential compared with $LiC_{10}H_8$, resulting in a cleaner reaction. This reaction was effective in replacing the thiophenyl groups of tris(phenylthiomethyl)silanes **3a**−**d** with lithium to give the corresponding tris(lithiomethyl)silanes, **4a**−**d** (Scheme 2), in a procedure that proceeded readily in THF at −40 °C.

The $RSi(CH_2Li)_3$ thus formed were derivatized with Bu_3SnCl [>95% overall yield by NMR of the crude product; 42%−81% yield of isolated pure $RSi(CH_2SnBu_3)_3$ (Scheme 2)]. Use of $LiC_{10}H_8$ as electron transfer reagent resulted in the same products but required longer reaction times and gave lower yields. The use of $LiC_{10}H_8$ at higher temperature or longer reaction time resulted in partial deprotonation of the acidic $SiCH_2SPh$ groups by the formed (lithiomethyl)silanes, leading to undesired byproducts. Tetrakis(lithiomethyl)silane (**8**) was prepared analogously from $Si(CH_2SPh)_4$ using LiDBB and characterized by the reaction with Bu_3SnCl [>95% overall yield by NMR of the crude product; 42% yield of isolated pure $Si(CH_2SnBu_3)_4$ (**9**)].

The presence of the tris(lithiomethyl)silanes and tetrakis(lithiomethyl)silane could not be demonstrated by NMR studies of the reaction mixtures,[13] and it was uncertain whether the poly(lithiomethyl)silanes were present before the Bu_3SnCl was added or whether they were formed during the addition of Bu_3SnCl. However, two observations indicate that the poly(lithiomethyl)silanes were formed before the addition of the trapping reagent: (i) The green or blue-green color of LiDBB had disappeared on addition of the (phenylthiomethyl)silanes, indicating completion of reaction. (ii) Only partly metalated species were formed at lower temperature or after shorter reaction times and trapped with Bu_3SnCl. In earlier studies[4] on the synthesis of bis(lithiomethyl)silanes we also isolated trapping products of only partly metalated (e.g., monometalated) compounds with Bu_3SnCl. The reaction mixtures begin to decompose around 0 °C by proton abstraction caused by the lithium bases.

We tried to prepare the carbon analogues of **5a** and **9** (Si replaced by C) in an analogous manner. The phenylthiomethyl compounds 1-(phenylthio)-2,2-bis(phenylthiomethyl)propane and 1,3-bis(phenylthio)-2,2-bis(phenylthiomethyl)propane were prepared by the reaction of $CH_3C(CH_2Cl)_3$ and $C(CH_2Br)_4$ with sodium thiophenolate. On reaction of the $CH_3C(CH_2SPh)_3$ and $C(CH_2SPh)_4$ thus prepared with LiDBB, we were not able to obtain evidence for the presence of the carbon analogues of the poly(lithiomethyl)silanes $CH_3C(CH_2Li)_3$ or $C(CH_2Li)_4$. However, the CH_2S bonds cleaved and the other cleavage product, LiSPh, was trapped by Bu_3SnCl to give Bu_3SnSPh. In these reactions only half of the LiDBB in the reaction mixture was used. The other half reacted with Bu_3SnCl to form the corresponding distannanes. The product of the central organic part of $CH_3C(CH_2SPh)_3$ and $C(CH_2SPh)_4$ could not be identified in the reaction mixture. The characterized products indicate that half of the CH_2S groups were cleaved by reduction with LiDBB and the other half by LiSPh elimination, partly after transmetalation. Formation of cyclopropanes can be an explanation of these observations.[14] Similar behavior was observed during the attempted analogous synthesis of 1,3-dilithiopropanes.[15] Therefore the silicon atom plays a central role in the reactivity of the presented structural types.

Preparation of 2,2,3,3,7,7,8,8-Octamethyl-2,3,5,7,8-pentasilaspiro[4,6]nonane Starting from Tetrakis-

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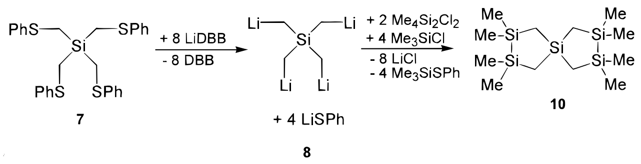
Scheme 3



(lithiomethyl)silane. Cyclic carbosilanes are of interest as starting materials for the preparation of carbosilane polymers[16] and/or Si−C ceramics on pyrolysis[17] of such polymers. Polymeric linear carbosilanes can be produced by ring-opening reactions of 1,3-disilacyclobutanes[16] or thermal rearrangement of polysilanes.[18] Spirocyclic, reactive carbodisilanes such as **10** would be interesting and potentially useful monomers but, to date, were unknown.

Tetrakis(lithiomethyl)silane (**8**) served well in the synthesis of **10** in a reaction with 1,2-dichloro-1,1,2,2-tetramethyldisilane at −50 °C. As the reaction mixture was allowed to warm, at 0 °C we added chlorotrimethylsilane to intercept the formed S-nucleophiles and unreacted C-nucleophiles before they could react with **10**. Kugelrohr distillation and recrystallization from ethanol gave spirosilane **10** (Scheme 3) as colorless crystals (20% yield), which were suitable for X-ray diffraction studies.

Spirosilane **10** crystallized from ethanol in the monoclinic crystal system, space group $C2/c$. The asymmetric unit contains half a molecule of **10**. The silicon center Si(3) of the spiro compound represents the inversion center of the molecule. The Si−C bond lengths are in the range 1.869(3)−1.891(3) Å as well as the Si−Si bond lengths of 2.354(1) Å, all in the normal range.[19] The two five-membered rings are in an envelope conformation with ecliptically arranged methyl groups [torsion angles C(4)−Si(1)−Si(2)−C(5) 2.5°, Si(3)−C(2)−Si(1)−Si(2) 23.9°, Si(3)−C(1)−Si(2)−Si(1) −28.0°]. The C(1)−Si(3)−C(2) angle of 104.3(2)° at the spirocenter is smaller than a tetrahedral angle. A further feature of **10** is the small C−Si−Si ring angles [both 99.3(1)°] at the disilane fragment, which indicates a strained ring system. The results of DFT calculations [B3LYP/6-31G(d) level] confirm an analogous conformation and geometry as an energy minimum for **10**.[20,21] NMR calculations (GIAO method[22] on the HF/6-31+G(d,p) level based on the calculated structure at the B3LYP/6-31G(d) level) results in ^{29}Si NMR shifts of −8.9/−11.3 ppm for the disilane and 20.6 ppm for the central silicon atoms. These calculated data support the experimental results.

Conclusions

Starting from chlorosilanes and thioanisole, poly(lithiomethyl)silane reagents were prepared by a two-step synthetic procedure: synthesis of the poly(phenylthiomethyl)silanes and reductive cleavage of C−S bonds of the latter with LiDBB. This easy access to tris- and tetrakis(lithiomethyl)silanes starting from inexpensive starting materials makes these new reagents available for further reactions. The synthetic potential was shown by the preparation of the spirosilane **10**. Further work is currently in progress to synthesize poly(lithiomethyl)element compounds of elements of groups 14−16.

Experimental Section

Melting points: Fa. Büchi, type 510. ^{1}H NMR [solvent CDCl$_3$; internal standard CHCl$_3$ ($\delta = 7.20$)]: Bruker AC-200P (200.13 MHz). ^{13}C NMR [solvent and internal standard CDCl$_3$ ($\delta = 77.05$)]: Bruker AC-200P (50.32 MHz). Assignment of the ^{13}C NMR data was supported by DEPT experiments and relative intensities of the resonance signals. ^{29}Si NMR (INEPT): [solvent CDCl$_3$; external standard TMS ($\delta = 0$)]: Bruker AC-200P (39.76 MHz). ^{119}Sn NMR: [solvent CDCl$_3$; external standard SnMe$_4$ ($\delta = 0$)]: Bruker AC-200P (74.63 MHz). EI-MS (70 eV): Finnigan-MAT 8430. The selected m/z values given refer to the isotopes ^{1}H, ^{12}C, ^{28}Si, ^{32}S, and ^{120}Sn. Microanalyses: Fa. Beller, Göttingen; Leco Elemental Analyser CHN 900, Institute of Inorganic Chemistry, Saarbrücken. Kugelrohr distillation: GKR-501, Fa. Büchi, the boiling points given are oven temperatures. All reactions were carried out under oxygen-free and dried under argon. The solvents were dried according to common procedures.

General Procedure for the Synthesis of 3a−d and 7. Method A: At −40 °C a cooled solution of 0.24 mol of phenylthiomethyllithium in 250 mL of THF, prepared from thioanisole and n-BuLi/DABCO (1,4-diazabicyclo[2.2.2]octane),[11] was added to a solution of 0.08 mol of the appropriate trichlorosilane or 0.06 mol of SiCl$_4$ in 30 mL of THF. The reaction mixture was warmed to room temperature, 200 mL of Et$_2$O and then 50 mL of water were added, and the mixture was extracted three times with Et$_2$O. The combined organic solutions were washed three times with 0.5 N HCl and dried over Na$_2$SO$_4$. The solvent was evaporated in vacuo, and the residue was purified by Kugelrohr distillation and/or crystallization to give the appropriate (phenylthiomethyl)silane.

Method B. At −40 °C a cooled solution of 0.24 mol of (phenylthiomethyl)lithium in 200 mL of diethyl ether/hexane, prepared from thioanisole and n-BuLi in diethyl ether,[12] was added to a solution of 0.08 mol of the appropriate trichlorosilane or 0.06 mol of SiCl$_4$ in 30 mL of diethyl ether. The reaction mixture was warmed to room temperature and filtered. The solvent was evaporated in vacuo, and the residue was purified by Kugelrohr distillation and/or crystallization.

Methyltris(phenylthiomethyl)silane (3a): 53%; bp 205 °C/10^{-3} mbar (oven temperature); mp 54−55 °C (ethanol). ^{1}H NMR (CDCl$_3$): δ 0.39 (s, 3H; SiCH_3), 2.43 (s, 6H; SiCH_2S),

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7.05−7.35 (m, 15H; SC$_6$H$_5$). ^{13}C{^{1}H} NMR (CDCl$_3$): δ −5.1 (Si*C*H$_3$), 15.5 (3C, Si*C*H$_2$S), 125.3 (3C, C-4 of SC$_6$H$_5$), 126.6 (6C), 128.8 (6C) (C-2, −6 and C-3, −5 of SC$_6$H$_5$), 138.9 (3C, C-1 of SC$_6$H$_5$). ^{29}Si NMR (CDCl$_3$): δ 2.3. MS (El): *m*/*z* 412 (44, M$^+$), 289 (53, M$^+$ − CH$_2$SC$_6$H$_5$), 179 (100, M$^+$ −2 SC$_6$H$_5$ - CH$_3$), 165 (50, M$^+$ − CH$_2$SC$_6$H$_5$ − SC$_6$H$_5$ − CH$_3$), 151 (52, M$^+$ − 2 CH$_2$SC$_6$H$_5$ − CH$_3$). Anal. Calcd for $C_{22}H_{24}S_3Si$: C, 64.03; H, 5.86. Found: C, 64.1; H, 6.0.

Ethyltris(phenylthiomethyl)silane (3b): 64%; bp 225 °C/10^{-3} mbar (oven temperature). ^{1}H NMR (CDCl$_3$): δ 0.85−1.20 (m, 5H; SiC*H*$_2$C*H*$_3$), 2.44 (s, 6H; SiC*H*$_2$S), 7.0−7.35 (m, 15H; SC$_6$H$_5$). ^{13}C{^{1}H} NMR (CDCl$_3$): δ 3.9 (Si*C*H$_2$CH$_3$), 7.2 (SiCH$_2$*C*H$_3$), 14.0 (3C, Si*C*H$_2$S), 125.2 (3C, C-4 of SC$_6$H$_5$), 126.7 (6C), 128.8 (6C) (C-2, −6 and C-3, −5 of SC$_6$H$_5$), 139.0 (3C, C-1 of SC$_6$H$_5$). ^{29}Si NMR (CDCl$_3$): δ 3.8. MS (El): *m*/*z* 426 (27, M$^+$), 303 (52, M$^+$ − CH$_2$SC$_6$H$_5$), 193 (100, M$^+$ − SC$_6$H$_5$ − CH$_2$SC$_6$H$_5$ − H), 165 (50, M$^+$ − 2 CH$_2$SC$_6$H$_5$ − CH$_3$). Anal. Calcd for $C_{23}H_{26}S_3Si$: C, 64.74; H, 6.14. Found: C, 63.6; H, 6.3.

Tris(phenylthiomethyl)vinylsilane (3c). 60%; bp 215 °C/10^{-3} mbar (oven temperature). ^{1}H NMR (CDCl$_3$): δ 2.56 (s, 6H; SiC*H*$_2$S), 6.05−6.31 (m, 3H; SiC*H*═C*H*$_2$), 7.10−7.40 (m, 15H; SC$_6$H$_5$). ^{13}C{^{1}H} NMR (CDCl$_3$): δ 14.4 (3C, Si*C*H$_2$S), 125.2 (3C, C-4 of SC$_6$H$_5$), 126.6 (6C), 128.7 (6C) (C-2, −6 and C-3, −5 of SC$_6$H$_5$), 130.7 (Si*C*H═CH$_2$), 133.5 (3C, C-1 of SC$_6$H$_5$), 137.1 (SiCH═*C*H$_2$). ^{29}Si NMR (CDCl$_3$): δ −8.4. MS (El): *m*/*z* 424 (8, M$^+$), 300 (92, M$^+$ − CH$_2$SC$_6$H$_5$ − H), 235 (100, M$^+$ − SC$_6$H$_5$ − C$_6$H$_5$ − 3 H), 177 (68, M$^+$ − 2 CH$_2$SC$_6$H$_5$ − H. Anal. Calcd for $C_{23}H_{24}S_3Si$: C, 65.04; H, 5.7. Found: C, 64.6; H 5.9.

Phenyltris(phenylthiomethyl)silane (3d): 57%; mp 62−63 °C (diethyl ether). ^{1}H NMR (CDCl$_3$): δ 2.70 (s, 6H; SiC*H*$_2$S), 7.0−7.5, 7.7−7.75 (m, 20H; SC$_6$H$_5$, SiC$_6$H$_5$). ^{13}C{^{1}H} NMR (CDCl$_3$): δ 14.7 (3C, SiCH$_2$S), 125.3 (3C, C-4 of SC$_6$H$_5$), 126.9 (6C, C-2, -6 and C-3, −5 of SC$_6$H$_5$), 128.3 (2C, C-2, -6 and C-3, -5 of SiC$_6$H$_5$), 128.8 (6C, C-2, -6 and C-3, -5 of SC$_6$H$_5$), 130.7 (C-4 of SiC$_6$H$_5$), 131.5 (C-1 of SiC$_6$H$_5$), 134.5 (2C, C-2, -6 and C-3, -5 of SiC$_6$H$_5$), 138.9 (3C, C-1 of SC$_6$H$_5$). ^{29}Si NMR (CDCl$_3$): δ −6.3. MS (El): *m*/*z* 474 (3, M$^+$), 273 (16, M$^+$ − CH$_2$SC$_6$H$_5$ − C$_6$H$_5$ − H), 124 (100, CH$_3$SC$_6$H$_5$$^+$), 109 (46, SC$_6H_5$$^+$). Anal. Calcd for $C_{27}H_{26}S_3Si$: C, 68.31; H, 5.52. Found: 68.5; H, 5.7.

Tetrakis(phenylthiomethyl)silane (7): 52%; mp 65−66 °C (ethanol). ^{1}H NMR (CDCl$_3$): δ 2.51 (s, 8H; SiC*H*$_2$S), 7.0−7.3 (m, 20H; SC$_6$H$_5$). ^{13}C{^{1}H} NMR (CDCl$_3$): δ 14.1 (4C, Si*C*H$_2$S), 125.6 (4C, C-4 of SC$_6$H$_5$), 127.2 (8C), 128.9 (8C) (C-2, -6 and C-3, -5 of SC$_6$H$_5$), 138.4 (4C, C-1 of SC$_6$H$_5$). ^{29}Si NMR (CDCl$_3$): δ 1.1. MS (El): *m*/*z* 520 (67, M$^+$), 397 (16, M$^+$ − CH$_2$SC$_6$H$_5$), 287 (100, − H$_2$CSC$_6$H$_5$ − SC$_6$H$_5$ − H), 273 (12, M$^+$ − 2 H$_2$CSC$_6$H$_5$ -H); Anal. Calcd for $C_{28}H_{28}S_4Si$: C, 64.57; H, 5.42. Found: C, 64.8; H, 5.7.

General Procedure for the Synthesis of 4a−d and 8 and Transformation to 5a−d and 9. At −60 °C 10 mmol of the appropriate tris(phenylthiomethyl)silane in 5 mL of THF was added to a freshly prepared solution of 64 mmol of LiDBB in 70 mL of THF and stirred for 6 h at −40 °C. Bu$_3$SnCl (20.8 g, 64 mmol) was added to the reaction mixture at −78 °C. After warming to room temperature the solvent was evaporated in vacuo and the residue was extracted with hexane and filtered. After evaporating the solvent the residue was purified by Kugelrohr distillation to give the appropriate tris(stannylmethyl)silane. DBB and Bu$_3$SnSPh were removed at 100−120 °C/10^{-3} mbar, and the high-boiling (stannylmethyl)silanes were isolated at the temperatures listed below. **9** was prepared analogously (10 mmol of **7**, 84 mmol of LiDBB, reaction time 8 h at −50 °C, 84 mmol Bu$_3$SnCl). The NMR yields of the appropiate (stannylmethyl)silanes were determined by ^{29}Si NMR (direct measurement). Only one ^{29}Si NMR signal was observed for the crude products before Kugelrohr distillation.

Methyltris[(tributylstannyl)methyl]silane (5a): 49%; bp. 225 °C/10^{-3} mbar (oven temperature). ^{1}H NMR (CDCl$_3$): δ −0.34 (s, ^{2}*J*(H,^{119,117} Sn) = 67.6/65.1 Hz, 6H; SiC*H*$_2$Sn), −0.06 (s, 3H; SiC*H*$_3$), 0.5−0.9, 1.15−1.55 (m, 81H; SnC*H*$_2$C, CC*H*$_2$C, CC*H*$_2$CH$_3$, CC*H*$_3$). ^{13}C{^{1}H} NMR (CDCl$_3$): δ −3.4 (3C, ^{1}*J*(C,^{119,117}Sn) = 202.1/193.2 Hz, ^{3}J(C,Sn) = 15.9 Hz; Si*C*H$_2$Sn), 4.3 (^{3}*J*(C,Sn) = 9.5 Hz; Si*C*H$_3$), 10.6 (9C, ^{1}*J*(C,^{119,117}Sn) = 318.3/307.8 Hz; Sn*C*H$_2$C), 13.7 (9C, CCH$_3$), 27.5 (9C, ^{3}*J*(C,^{119,117}Sn) = 58.0/55.3 Hz; C*C*H$_2$CH$_3$), 29.3 (9C, ^{2}*J*(C,Sn) = 19.3 Hz; SnCH$_2$*C*H$_2$C). ^{29}Si NMR (CDCl$_3$): δ 7.7 (^{2}*J*(Si,Sn) = 21.6 Hz). ^{119}Sn NMR (CDCl$_3$): δ −0.7. MS (El): *m*/*z* 551 (8, M$^+$ − ^{118}Sn(C$_4$H$_9$)$_3$ − 2 C$_4$H$_9$; M$^+$ = C$_{40}$H$_{90}$Si^{118}Sn$_2$120Sn), 495 (7, M$^+$ ^{118}Sn(C$_4$H$_9$)$_3$ − 3 C$_4$H$_9$ +H). Anal. Calcd for $C_{40}H_{90}SiSn_3$: C, 50.29; H, 9.50. Found: C, 50.6; H, 9.3.

Ethyltris[tributylstannyl)methyl]silane (5b): 57%; bp 225 °C/10^{-3} mbar (oven temperature). ^{1}H NMR (CDCl$_3$): δ −0.35 (s, ^{1}*J*(H,$^{119/117}$Sn) = 68.3/65.7 Hz, 6H; SiC*H*$_2$Sn), 0.35−0.9, 1.10−1.55 (m, 86H; SiC*H*$_2$C, SnC*H*$_2$C, CC*H*$_2$C, CC*H*$_3$). ^{13}C{^{1}H} NMR (CDCl$_3$): δ −5.8 (9C, ^{1}*J*(C,$^{119/117}$Sn) = 202.5/194.1 Hz, ^{3}*J*(C,Sn) = 15.2 Hz; Si*C*H$_2$Sn), 8.2 (SiCH$_2$*C*H$_3$), 10.5 (9C, ^{1}*J*(C,$^{119/117}$Sn) = 325.4/311.2 Hz; Sn*C*H$_2$C), 11.9 (^{3}*J*(C,Sn) = 10.3 Hz; Si*C*H$_2$CH$_3$), 13.7 (9C, CCH$_3$), 27.5 (9C, ^{3}*J*(C,$^{119/117}$Sn) = 58.1/55.7 Hz; C*C*H$_2$CH$_3$), 29.3 (9C, ^{2}*J*(C,Sn) = 19.3 Hz; SnCH$_2$*C*H$_2$C). ^{29}Si NMR (CDCl$_3$): δ 10.5 (^{2}*J*(Si,Sn) = 21.1 Hz). ^{119}Sn NMR (CDCl$_3$): δ −0.9. MS (El): *m*/*z* 665 (44, M$^+$ − CH$_2$120Sn(C$_4$H$_9$)$_3$; M$^+$ = C$_{41}$H$_{94}$Si^{118}Sn$_2$120Sn), 565 (58, M$^+$ ^{120}Sn(C$_4$H$_9$)$_3$ − 2 C$_4$H$_9$), 291 (100, Sn(C$_4$H$_9$)$_3$$^+$). Anal. Calcd for $C_{41}H_{94}SiSn_3$: C, 50.70; H, 9.75. Found: C, 51.7; H, 9.9.

Vinyl[tris[(tributylstannyl)methyl]silane (5c): 44%; bp 225 °C/10^{-3} mbar (oven temperature). ^{1}H NMR (CDCl$_3$): δ −0.29 (s, ^{1}*J*(H,$^{119/117}$Sn) = 66.5/62.9 Hz, 6H; SiC*H*$_2$Sn), 0.60−1.45 (m, 81H; SnC*H*$_2$C, CC*H*$_2$C, CC*H*$_3$), 5.58 (dd, ^{2}*J*(H,H) = 4.3 Hz, ^{3}*J*(H,H) = 19.8 Hz; SiCH═C*H*$_2$, 1H; SiCH═C*H*$_2$; H$_{\mathrm{cis}}$), 5.83 (dd, ^{2}*J*(H,H) = 4.2 Hz, ^{3}*J*(H,H) = 14.5 Hz, 1H; SiCH═C*H*$_2$; H$_{\mathrm{trans}}$), 6.11 (dd, ^{3}*J*(H,H$_{\mathrm{trans}}$) = 14.5 Hz, ^{3}*J*(H,H$_{\mathrm{cis}}$) = 19.8 Hz, 1H; SiC*H*═CH$_2$). ^{13}C{^{1}H} NMR (CDCl$_3$): δ −5.4 (3C, ^{1}*J*(C,$^{119/117}$Sn) = 200.1/195.2 Hz, ^{3}*J*(C,Sn) = 14.4 Hz; Si*C*H$_2$Sn), 10.7 (9C, ^{1}*J*(C,$^{119/117}$Sn) = 323.8/309.6 Hz; Sn*C*H$_2$C), 13.7 (9C, C*C*H$_3$), 27.5 (9C, ^{3}*J*(C,$^{119/117}$Sn) = 58.4/55.8 Hz; C*C*H$_2$CH$_3$), 29.3 (9C, ^{2}*J*(C,Sn) = 19.3 Hz; SnCH$_2$*C*H$_2$C), 130.0 (Si*C*H═CH$_2$), 143.8 (SiCH═*C*H$_2$). ^{29}Si NMR (CDCl$_3$): δ 0.3 (^{2}*J*(Si,Sn) = 21.2 Hz). ^{119}Sn NMR (CDCl$_3$): δ −1.4. MS (El): *m*/*z* 563 (100, M$^+$ − ^{118}Sn(C$_4$H$_9$)$_3$ − 2 C$_4$H$_9$ + 2 H; M$^+$ = C$_{41}$H$_{92}$Si^{118}Sn$_2$120Sn), 291 (38,^{120}Sn(C$_4$H$_9$)$_3$$^+$). Anal. Calcd for $C_{41}H_{92}SiSn_3$: C, 50.80; H, 9.57. Found: C, 51.1; H, 9.7.

Phenyltris[(tributylstannyl)methyl]silane (5d): 47%; bp 230 °C/10^{-3} mbar (oven temperature). ^{1}H NMR (CDCl$_3$): δ −0.07 (s, ^{2}*J*(H,Sn) = 65.5 Hz, 6H; SiC*H*$_2$Sn), 0.5−0.9, 1.1−1.55 (m, 81H; SnC*H*$_2$C, CC*H*$_2$C, CC*H*$_2$CH$_3$, CC*H*$_3$), 7.2−7.3, 7.35−7.45 (m, 5H; SiC$_6$*H*$_5$). ^{13}C{^{1}H} NMR (CDCl$_3$): δ −4.8 (3C, ^{1}*J*(C,$^{119/117}$Sn) = 197.3/188.6 Hz, ^{3}*J*(C,Sn) = 12.6 Hz; Si*C*H$_2$Sn), 10.6 (9C, ^{1}*J*(C,$^{119/117}$Sn) = 324.1/309.7 Hz; Sn*C*H$_2$C), 13.7 (9C, C*C*H$_3$), 27.5 (9C, ^{3}*J*(C,$^{119/117}$Sn) = 59.4/57.0 Hz; C*C*H$_2$CH$_3$), 29.3 (9C, ^{2}*J*(C,Sn) = 19.3 Hz; SnCH$_2$*C*H$_2$C), 127.5 (2C), 133.2 (2C) (C-2/C-6 and C-3/C-5 of SiC$_6$H$_5$), 128.3 (C-4 of SiC$_6$H$_5$), 143.4 (^{3}*J*(C,Sn) = 10.4 Hz; C-4 of SiC$_6$H$_5$). ^{29}Si NMR (CDCl$_3$): δ 3.1 (^{2}*J*(Si,Sn) = 21.0 Hz). ^{119}Sn NMR (CDCl$_3$): δ −2.9. MS (El): *m*/*z* 847 (0.3, M$^+$ − C$_4$H$_9$ + 2 H; M$^+$ = C$_{45}$H$_{92}$Si^{118}Sn$_2$120Sn), 791 (0.3, M$^+$ − 4 C$_4$H$_9$ + 3 H), 713 (36, −H$_2$C^{118}Sn(C$_4$H$_9$)$_3$), 671 (13, M$^+$ − ^{118}Sn(C$_4$H$_9$)$_3$ − C$_4$H$_9$), 613 (22, M$^+$ − ^{118}Sn(C$_4$H$_9$)$_3$ − 2 C$_4$H$_9$), 343 (97), 229 (70), 128 (100); Anal. Calcd for $C_{45}H_{92}SiSn_3$: C, 53.13; H, 9.11. Found: C, 53.2; H, 9.0.

Tetrakis[(tributylstannyl)methyl]silane (9): 42%; bp 250 °C/10^{-3} mbar (oven temperature). ^{1}H NMR (CDCl$_3$): δ −0.32 (s, 8H; ^{2}*J*(H,$^{119/117}$Sn) = 68.1/66.2 Hz; SiC*H*$_2$Sn), 0.6−0.95, 1.15−1.55 (m, 108H; SnC*H*$_2$C, CC*H*$_2$C, CC*H*$_2$CH$_3$, CC*H*$_3$). ^{13}C{^{1}H} NMR (CDCl$_3$): δ −1.8 (4C, ^{1}*J*(C,$^{119/117}$Sn) = 204.5/195.6 Hz, ^{3}*J*(C,Sn) = 14.3 Hz; Si*C*H$_2$Sn), 10.7 (12C, ^{1}*J*(C,$^{119/117}$Sn) = 321.2/306.8 Hz; Sn*C*H$_2$C), 13.7 (12C, C*C*H$_3$), 27.6 (12C, ^{3}*J*(C,$^{119/117}$Sn) = 58.3/55.9 Hz; C*C*H$_2$CH$_3$), 29.4 (12C, ^{2}*J*(C,Sn) = 19.2 Hz; SnCH$_2$*C*H$_2$C). ^{29}Si NMR (CDCl$_3$): δ 10.0 (^{2}*J*(Si,Sn) = 20.7 Hz). ^{119}Sn NMR (CDCl$_3$): δ −2.9. MS (El): *m*/*z* 897 (86, M$^+$ − ^{118}Sn(C$_4$H$_9$)$_3$ − C$_4$H$_9$ + H; M$^+$ = C$_{52}$H$_{116}$Si^{118}Sn$_2$120Sn$_2$), 839 (100, M$^+$ − ^{118}Sn(C$_4$H$_9$)$_3$ − 2 C$_4$H$_9$), 783 (22, M$^+$

$-$ $^{118}Sn(C_4H_9)_3$ $-$ 3 C_4H_9 + H), 291 (78, $Sn(C_4H_9)_3^+$). Anal. Calcd for $C_{52}H_{116}SiSn_4$: C, 50.19; H, 9.40. Found: C, 51.3; H, 9.5.

1-(Phenylthio)-2,2-bis(phenylthiomethyl)propane. To 3.9 g of sodium (171 mmol) and 200 mL of ethanol was added thiophenole (18.8 g, 171 mmol) at room temperature. After addition of 10 g of 1,3-dichloro-2-(chloromethyl)-2-methylpropane (57 mmol), the reaction mixture was heated at reflux for 1 week. After hydrolysis and drying of the organic phase over sodium sulfate the solvent was removed in vacuo and the residue was distilled (Kugelrohr) (47% yield). 1-(Phenylthio)-2-(chloromethyl)-2-(phenylthiomethyl)propane was isolated as a second product. Bp: 215 °C/10^{-3} mbar (oven temperature). ^{1}H NMR ($CDCl_3$): δ 1.23 (s, 3H; CCH_3), 3.22 (s, 6H; CCH_2S), 7.10$-$7.45 (m, 15H; SC_6H_5). ^{13}C NMR ($CDCl_3$): δ 23.8 (CCH_3), 41.4 (C_2CC_2), 43.4 (3C) (CCH_2S), 126.0 (3C) (C-4 of C_6H_5), 128.8 (6C), 129.5 (6C) (C-2/C-6 and C-3/C-5 of SC_6H_5), 136.9 (3C) (C-1 of SC_6H_5). MS (El): *m*/*z* (%) 396 (49, M^+), 177 (45, $M^+ - 2\ SC_6H_5 - H$), 123 (100, $CH_2SC_6H_5^+$), 109 (12, $^+SC_6H_5$). Anal. Calcd for $C_{23}H_{24}S_3$: C, 69.65; H, 6.09. Found: C, 69.2; H, 6.1.

1,3-Bis(phenylthio)-2,2-bis(phenylthiomethyl)propane.[23] The preparation was analogous to that of 1-(phenylthio)-2,2-bis(phenylthiomethyl)propane by reaction with pentaerithrityl tetrabromide (reaction time 60 h reflux) (87% yield); mp 70$-$71 °C (ethanol). ^{1}H NMR ($CDCl_3$): δ 3.23 (s, 8H; CCH_2S), 7.05$-$7.35 (m, 20H; C_6H_5). ^{13}C NMR ($CDCl_3$): δ 41.0 (4C) (CCH_2S), 45.7 (C_2CC_2), 126.4 (4C) (C-4 of SC_6H_5), 128.9 (8C), 130.1 (8C) (C-2/C-6 and C-3/C-5 of SC_6H_5), 136.3 (4C) (C-1 of SC_6H_5). MS (El): *m*/*z* (%) 504 (10, M^+), 218 (100, $H_5C_6SSC_6H_5$), 109 (62, $^+SC_6H_5$). Anal. Calcd for $C_{29}H_{28}S_4$: C, 69.00; H, 5.59. Found: C, 68.3; H, 5.7.

2,2,3,3,7,7,8,8-Octamethyl-2,3,5,7,8-pentasilaspiro[4,6]-nonane (10). Tetrakis(phenylthiomethyl)silane (**7**) (1.04 g, 2 mmol) was added to a freshly prepared solution of 16 mmol of LiDBB in THF at $-$50 °C and stirred for 8 h. Four millimoles of 1,2-dichloro-1,1,2,2-tetramethyldisilane was added to the mixture at $-$50 °C. After warming to 0 °C, 8 mmol of $ClSiMe_3$ was added. Hydrolysis and extraction with Et_2O, drying of the organic phase over sodium sulfate, and evaporation of the solution yielded a residue. The product can be separated from the higher boiling DBB by distillation by Kugelrohr. Recrystallization from ethanol gave **10** as colorless crystals (20%): bp 68 °C/10^{-3} mbar; mp 65$-$67 °C. ^{1}H NMR ($CDCl_3$): δ $-$0.21 (s, 8H; $SiCH_2Si$), 0.13 (s, 24H; $SiCH_3$). $^{13}C\ \{^1H\}$ NMR ($CDCl_3$): δ $-$1.3 (8C, $SiCH_3$), 4.8 (4C, $SiCH_2Si$). ^{29}Si NMR ($CDCl_3$): δ $-$13.6 (4 Si, $SiCH_3$), 17.4 (1Si, $Si(CH_2)_4$). MS (EI): *m*/*z* (%) 316 (26, M^+), 301 (14, $M^+ - CH_3$), 257 (6, $M^+ - Si(CH_3)_2\ H$), 243 (53, $C_9H_{23}Si_4$), 227 (75, $C_8H_{19}Si_4^+$), 73 (100, $(CH_3)_3Si^+$). Anal. Calcd for $C_{12}H_{32}Si_5$: C, 45.49; H, 10.18. Found: C, 45.2; H, 10.2.

X-ray Structure Determination of 10 (colorless crystals from ethanol). Crystallographic data: $C_{12}H_{32}Si_5$, $M = 316.83$, monoclinic, space group $C2/c$, $a = 11.378(2)$, $b = 11.544(2)$, $c = 15.573(3)$ Å, $\beta = 104.99(3)°$, $V = 1975.8(7)$ Å^3, $Z = 4$, $D_c = 1.065$ Mg/m^3, type of radiation Mo Kα, $\lambda = 0.71073$ Å, $\mu = 0.115$ mm^{-1}. Measurements: Stoe IPDS diffractometer, 173 K. The structure was solved using direct and Fourier methods; 8126 reflections measured with θ in the range 2.62$-$27°, 2082 unique reflections; 1564 with $I > 2\sigma(I)$; refinement by full-matrix least-squares methods (based on F_o^2, SHELXL-93[24]); anisotropic thermal parameters for all non-H atoms in the final cycles; the H atoms were refined isotropically; $R = 0.0528$ [$I > 2\sigma(I)$], $wR_2 = 0.1388$ (all data). SHELXS-86 and SHELXL-93 computer programs were used.[24]

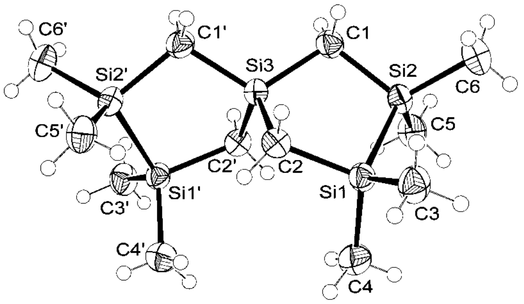

Figure 1. Selected bond length [Å] and angles [deg] of **10**: Si(1)$-$Si(2) 2.3540(10), Si(1)$-$C(2) 1.886(3), Si(2)$-$C(1) 1.891(3), Si(3)$-$C(1) 1.876(3), Si(3)$-$C(2) 1.880(3); C(2)$-$Si(1)$-$Si(2) 99.33(9), C(1)$-$Si(2)$-$Si(1) 99.26(9), C(1)$-$Si(3)$-$C(2) 104.27(12), C(1′)$-$Si(3)$-$C(2) 111.19(12), C(2)$-$Si(3)$-$C(2′) 110.4(2), Si(3)$-$C(1)$-$Si(2) 108.12(14), Si(3)$-$C(2)$-$Si(1) 109.03(13) (ORTEP; displacement ellipsoids at 50% probability level).

Computational Methods. The structure of **10** was calculated with the GAUSSIAN 98W[20] program, fully minimized, and gives a total energy of $-$1609.58158941 hartrees without symmetry by using the 6-31G(d) basis set implemented in the program and the B3LYP-DFT method.[21] Stationary points were identified by frequency analysis. For NMR calculations the GIAO method[22] on the HF/6-31+G(d,p) level—based on the calculated structure on the B3LYP/6-31G(d) level—was used.

B3LYP/6-31G(d) Geometry Optimized 10. The inversion center of **10** was not defined for the calculation, but a pseudo inversion center was observed in the minimized molecule. The two silicon atoms of one disilane unit are not correlated by symmetry. The scheme of labeling is comparable to labels in Figure 1. Selected bond length [Å] and angles [deg]: Si(1)$-$Si(2) 2.377, Si(1)$-$C(2) 1.916, Si(2)$-$C(1) 1.913, Si(3)$-$C(1) 1.904, Si(3)$-$C(2) 1.906; C(2)$-$Si(1)$-$Si(2) 99.9, C(1)$-$Si(2)$-$Si(1) 99.4, C(1)$-$Si(3)$-$C(2) 104.7, C(1′)$-$Si(3)$-$C(2) 112.0, C(2)$-$Si(3)$-$C(2′) 110.3, Si(3)$-$C(1)$-$Si(2) 110.1, Si(3)$-$C(2)$-$Si(1) 110.1.

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Supporting Information Available: Tables of crystal data, final atomic coordinates, tables of hydrogen atom coordinates, thermal parameters for the non-hydrogen atoms, and complete lists of bond distances and angles of **10**. This material is available free of charge via the Internet at http://pubs.acs.org.

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