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Ladder Structure of a Lithium Organyl: Synthesis and Crystal Structure of a Mixed Aggregate of *n*-BuLi and an (Aminomethyl)(lithiomethyl)silane

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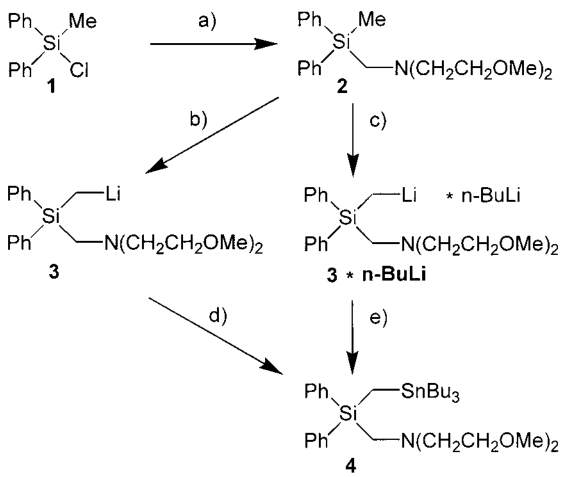
Summary: A mixture of n-BuLi and the (aminomethyl)(lithiomethyl)silane ***3*** *shows a higher reactivity than the separate compounds. A single-crystal X-ray structure of the mixed aggregate between n-BuLi and the (lithiomethyl)silane ([***3·n-BuLi***]$_2$) establishes a ladder structure of the lithiumorganyl in the solid state.*

Introduction

Organolithium compounds are important reagents in organic and organometallic synthesis, but the complex mechanisms of their reactions are only poorly understood.[1] Mixtures of organolithium compounds and alkali metal alkoxides are known to be more reactive than the separate reagents.[2] Such "superbase" reagents have been studied for more than 20 years, but the nature of the actual reactive species as well as their structure in solution remains controversial.[2] Up to now, mixtures of two different lithium alkyls have not been used to increase the reactivity of organolithium compounds, and only one compound of this type has been characterized in the solid state.[3]

We present here two new features in organolithium chemistry: (i) the enhanced reactivity of a mixture of *n*-BuLi and a (lithiomethyl)silane; (ii) the first lithium alkyl that has a ladder structure in the solid state. Ladder structures have been described for Li−N, Li−O, Li−P, and Li−S systems, but not for lithium alkyls.[1] To our knowledge, the only known example of a ladder structure of lithium organyls is that formed by a mixture of a lithium alkenyl and a lithium alkyl compound.[1c] The present crystal structure bridges the gap in the structural chemistry of lithium compounds.[4]

Scheme 1[a]



[a] (a) + $LiCH_2N(CH_2CH_2OMe)_2$/−LiCl; (b) + *t*-BuLi, pentane/−*t*-BuH; (c) + 2 *n*-BuLi, hexane, toluene/−*n*-BuH; (d) + Bu_3SnCl/−LiCl; (e) + 2 Bu_3SnCl/−2 LiCl, − $SnBu_4$.

Results and Discussion

Starting from [bis(2-methoxyethyl)](phenylthiomethyl)amine and chloromethyldiphenylsilane (**1**) the (aminomethyl)silane **2** was obtained by a reported procedure in 70% yield.[5] Metalation of **2** with *t*-BuLi in pentane in a 1:1 ratio at −78 °C gave **3**. During the metalation of **2** with *n*-BuLi in toluene at −78 °C we observed that 2 equiv of *n*-BuLi were required for complete metalation of the $SiCH_3$ group to give **3·*n*-BuLi**. Trapping experiments with Bu_3SnCl gave the same silane product **4** in both metalation reactions, but in the case of the latter reaction 1 equiv of $SnBu_4$, the trapping product of *n*-BuLi, was formed as well. On cooling a solution of **3·*n*-BuLi** in toluene or in hexane, orange crystals of the mixed aggregate [**3·*n*-BuLi**]$_2$ were formed.

[**3·*n*-BuLi**]$_2$ crystallizes from toluene in the space group $P2_1/c$ as a dimer. The central four-membered ring is formed by the two Li(2) atoms and the two α-carbon atoms of *n*-BuLi [C(21)]. The lithium atoms Li(2) and Li(2)′ have three carbon contacts [Li(2)−C(1)′ 2.215(5), Li(2)−C(21) 2.303(5), Li(2)−C(21)′ 2.251(5) Å]. A coordination of the oxygen of a (2-methoxyethyl)amino group completes the coordination sphere of Li(2) [Li(2)−O(2) 2.071(4) Å]. Similarly the carbon atom C(21) shows

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[1] (a) *Methoden Org. Chem.*, 4th ed.; Houben-Weyl: 1952; E19d, 1993. (b) *Lithium Chemistry;* Sapse, A.-M., Schleyer, P.v. R., Eds.; Wiley & Sons: New York, 1995. (c) Gardiner, M. G.; Raston, C. L.; Cloke, F. G. N.; Hitchcock, P. B. *Organometallics* **1995**, *14*, 4.1339.

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[3] (a) Wijkens, P.; van Koten, E. M.; Janssen, M. D.; Jastrzebski, J. T. B. H.; Spek, A. L.; van Koten, G. *Angew. Chem.* **1995**, *107*, 239; *Angew. Chem., Int. Ed. Engl.* **1995**, *34*, 219. (b) A mixed aggregate of *n*-BuLi and an alkenyllithium compound: Ruhlandt-Senge, K.; Ellison, J. J.; Wehmschulte, R. J.; Pauer, F.; Power, P. P. *J. Am. Chem. Soc.* **1993**, *115*, 11353.

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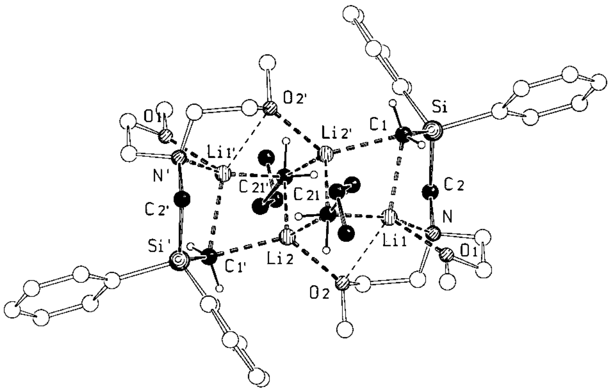

Figure 1. View of the molecular structure of [**3·*n*-BuLi**]$_2$. Selected bond distances (Å) and angles (deg): Si−C(1) 1.783(5), O(1)−Li(1) 2.087(7), O(2)−Li(1) 2.494(7), N−Li(1) 2.197(8), Li(2)−C(1)′ 2.233(8), C(21)−Li(2)′ 2.231(8), O(2)−Li(2) 2.090(7), Li(1)−C(21) 2.260(8), Li(1)−Li(2)′ 2.479(9), Li(2)−Li(2)′ 2.560(12), Li(1)−C(1) 2.340(8), Si−C(2) 1.906(4); Li(1)−C(1)−Li(2)′ 65.6(3), C(1)−Li(1)−C(21) 99.4(3), Li(1)−C(21)−Li(2)′ 67.0(3), C(1)′−Li(2)−C(21)′ 103.6(3), Li(2)−C(21)−Li(2)′ 68.0(3), C(21)−Li(2)−C(21)′ 112.0(3).

three lithium contacts. Two four-membered rings, formed by the two lithium atoms Li(1) and Li(2) and two carbon atoms [C(1) and C(21)], are connected to the opposite sides of the central four-membered ring, forming a ladder structure. Li(1) has two carbon contacts [Li(1)−C(1) 2.339(6), Li(1)−C(21) 2.266(5) Å], and the coordination sphere is completed by a Li(1)−N [219.6(5) pm], a Li(1)−O(1) [2.043(5) Å], and a longer Li(1)−O(2) [2.387(5) Å] contact. The metalated carbon atom of the (lithiomethyl)silane shows contacts with two different lithium atoms [Li(1) and Li(2)].

The silicon−carbon bond length [1.794(3) Å] to the lithiated carbon atom is shorter than the "normal" silicon−carbon bond length of 1.86−1.89 Å, reported in the literature.[6] This effect is known for metalated carbon atoms bonded to silicon.[1a,7] We observed slightly longer Si−C bonds of the nonmetalated carbon atoms of [**3·*n*-BuLi**]$_2$ [Si−C(2) 1.909(3), Si−C(9) 1.910(3), Si−C(15) 1.897(3) Å]. No conformation indicating negative hyperconjugation was found.[8]

A ball-and-stick drawing of the molecular structure of [**3·*n*-BuLi**]$_2$ and selected bond distances and angles are given in Figure 1. A second view of this new structural type is given in Figure 2. The four lithium atoms are arranged in one plane forming equilateral triangles. The lithiated carbon atoms are located above and below the triangular planes, a structural type known, for example, for [*n*-BuLi]$_6$.[9] The lithiated carbon atoms of the (lithiomethyl)silane part are arranged on the sides of the triangles, slightly above the Li−Li ring plane. This structure type has been observed for [*t*-BuLi·THF]$_2$,[9] and these two different structural types are combined in the crystal structure of [**3·*n*-BuLi**]$_2$ to give the ladder structure.

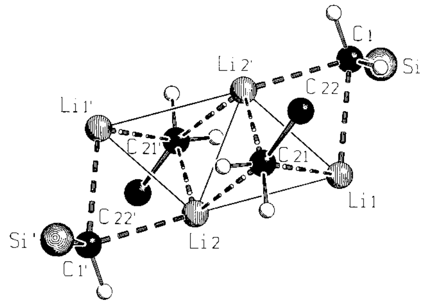

Figure 2. View of the central part of the molecular structure of [**3·*n*-BuLi**]$_2$.

A mixture of **3** and *n*-BuLi shows a higher reactivity than **3** or *n*-BuLi alone. Neither *n*-BuLi nor **3** lithiates benzene at room temperature, but a 1:1 mixture of **3** and ***n*-BuLi** metalated benzene at room temperature in a few hours.

We attempted to obtain information about the structure of **3·*n*-BuLi** in solution. A solution of [**3·*n*-BuLi**]$_2$ in benzene at 25 °C shows a broad resonance at −0.3 ppm in the ^{7}Li NMR. In the ^{13}C NMR spectrum the lithiated carbon atoms are observed at −14.4 and 13.9 ppm. The latter resonance is the same as the signal of *n*-BuLi in benzene (13.9 ppm for the lithiated carbon atom). Down to −70 °C no ^{13}C−^{6}Li spin−spin coupling was observed in the ^{13}C NMR spectrum of [^{6}Li]-labeled **3·*n*-Bu[^{6}Li]** in toluene.[10] A solution of **3** in benzene showed a resonance at −0.4 in the ^{7}Li NMR spectrum and a resonance at −14.4 for the lithiated carbon atom in the ^{13}C NMR spectrum. We found no significant difference between the sum of the NMR spectra of a solution of [**3·*n*-BuLi**]$_2$ and **3** or *n*-BuLi taken separately in benzene. In the ^{6}Li CP-MAS spectrum of {**[^{6}Li]3·*n*-Bu^{6}Li**}$_2$ a broad signal was observed at 2.0 ppm. The lithiated carbon atoms of the mixed aggregate show a significant shift in the ^{13}C CP-MAS spectrum (−16.7 ppm for Si*C*H$_2$Li and 5.6 ppm for Li*C*H$_2$C) when compared with the solution data. These results indicate the presence of a mixture of the aggregates of now interacting **3** and (*n*-BuLi)$_6$ in solution. The different reactivity of the mixture of **3** and *n*-BuLi then must be due to another species, present only in small amounts, which has a higher reactivity, probably [**3·*n*-BuLi**]$_2$.

Further related work on the synthesis, structure, and reactivity of (aminomethyl)(lithiomethyl)silanes is in progress in order to better understand the behavior of these systems in solution and the solid state.

Experimental Section

Melting points: Schmelzpunktsbestimmungsgerät, Fa. Büchi, Typ 510. ^{1}H NMR [solvent CDCl$_3$; internal standard CHCl$_3$ (δ 7.20)]: Bruker AC-200P (200.13 MHz). ^{13}C NMR [solvent and internal standard CDCl$_3$ (δ 77.05)]: Bruker AC-200P (50.32 MHz). Assignment of the ^{13}C NMR data was supported by DEPT experiments. ^{29}Si NMR (INEPT) (solvent CDCl$_3$; external standard TMS): Bruker AC-200P (39.76 MHz). ^{119}Sn NMR (solvent CDCl$_3$; external standard SnMe$_4$): Bruker AC-200P (74.63 MHz). ^{7}Li NMR (solvent C$_6$D$_6$; external

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(10) The NMR spectral data of **3·*n*-*BuLi*** could not be obtained at lower temperatures due to its crystallization.

standard LiCl): Bruker AC-200P (77.7 MHz). ^{13}C CP/MAS (external standard adamantane): Bruker MSL-200S (50.3 MHz). ^{6}Li CP/MAS (external standard LiCl): Bruker MSL-200S (29.4 MHz). EI-MS (70 eV): Finnigan-MAT 8430. The selected *m*/*z* values given refer to the isotopes ^{1}H, ^{12}C, ^{14}N,^{16}O, ^{28}Si, and ^{120}Sn. Microanalyses: Fa. Beller, Göttingen; Leco Elemental Analyser CHN 900, Institut 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, dry argon. The solvents were dried according to common procedures.

Methyl[bis(2-methoxyethyl)aminomethyl]diphenylsilane (**2**). The synthesis from methylchlorodiphenylsilane and [bis(2-methoxyethyl)aminomethyl]lithium was analogous to literature.[5] Yield: 92%; bp 145 °C (oventube temperature)/10^{-3} mbar. ^{1}H NMR ($\mathrm{CDCl_3}$): δ 0.60 (s, $^{2}J(\mathrm{H,Si}) = 6.5$ Hz, 3H; $\mathrm{SiC}H_3$), 2.58 (t, $^{3}J(\mathrm{H,H}) = 6.1$ Hz, 4H; $\mathrm{NC}H_2\mathrm{C}$), 2.59 (s, 2H; $\mathrm{SiC}H_2\mathrm{N}$), 3.19 (s, 6H; $\mathrm{OC}H_3$), 3.30 (t, $^{3}J(\mathrm{H,H}) = 6.1$ Hz, $\mathrm{OC}H_2\mathrm{C}$), 7.25−7.55 (m, 10H; Ph). ^{13}C NMR (50.3 MHz, $\mathrm{CDCl_3}$, 25 °C, $\mathrm{CDCl_3}$): δ −4.2 ($\mathrm{Si}C\mathrm{H_3}$), 45.3 ($\mathrm{Si}C\mathrm{H_2N}$), 57.2 (2C, $\mathrm{N}C\mathrm{H_2C}$), 58.7 (2C, $\mathrm{O}C\mathrm{H_3}$), 71.0 (2C, $\mathrm{O}C\mathrm{H_2C}$), 127.8 (2C, *m*-Ph), 129.2 (*p*-Ph), 134.7 (2C, *o*-Ph), 136.9 (*i*-Ph). ^{29}Si NMR (39.8 MHz, $\mathrm{CDCl_3}$, 25 °C, TMS): δ −12.3; EI-MS (70 eV): *m*/*z* 343 ($\mathrm{M^+}$, 3), 298 ($\mathrm{M^+ - CH_2OCH_3}$, 100), 266 ($\mathrm{M^+ - C_6H_5}$, 4), 197 ($\mathrm{(C_6H_5)_2SiCH_3{}^+}$, 15), 146 ($\mathrm{CH_2N(CH_2CH_2OCH_3)_2{}^+ - H}$, 95), 78 ($\mathrm{C_6H_5{}^+ + H}$, 18), 59 ($\mathrm{CH_2CH_2OCH_3{}^+}$, 76). Anal. Calcd for $\mathrm{C_{20}H_{29}NO_2Si}$ (343.5): C, 69.93; H, 8.51; N, 4.08. Found: C, 69.6; H, 8.7; N, 4.0.

(Lithiomethyl)[bis(2-methoxyethyl)aminomethyl]diphenylsilane (**3**). To a solution of 0.3 g (0.9 mmol) of silane **2** in 2.0 mL of pentane was added 0.6 mL (1.0 mmol) of 1.7 M *t*-BuLi in pentane at −78 °C. **3** was formed immediately as a red oil. At room temperature the pentane was separated, and **3** was washed with 2 mL of pentane and dried 1 h at room temperature in vacuo. Yield: 0.3 g (95%). ^{1}H NMR ($\mathrm{C_6D_6}$): δ −1.27 (s, 2H; $\mathrm{SiC}H_2\mathrm{Li}$), 2.40−2.50 (m, 4H; $\mathrm{NC}H_2\mathrm{C}$), 2.54 (s, 2H; $\mathrm{SiC}H_2\mathrm{N}$), 2.93 (s, 6H; $\mathrm{OC}H_3$), 3.00−3.10 (m, 4H; $\mathrm{OC}H_2\mathrm{C}$), 7.00−7.40 and 7.90−8.10 (m, 10H; Ph). ^{13}C NMR ($\mathrm{C_6D_6}$): δ −14.4 ($\mathrm{Si}C\mathrm{H_2Li}$), 47.5 ($\mathrm{Si}C\mathrm{H_2N}$), 56.0 (2C, $\mathrm{N}C\mathrm{H_2C}$), 58.7 (2C, $\mathrm{O}C\mathrm{H_3}$), 69.5 (2C, $\mathrm{O}C\mathrm{H_2C}$), 127.5 (2C, *m*-Ph), 127.8 (*p*-Ph), 135.3 (2C, *o*-Ph), 145.9 (*i*-Ph). ^{29}Si NMR ($\mathrm{C_6D_6}$): δ −4.5. ^{7}Li NMR ($\mathrm{C_6D_6}$): δ −0.4.

(Lithiomethyl)[bis(2-methoxyethyl)aminomethyl]diphenylsilane·*n*-BuLi (3·*n*-BuLi). To a solution of 0.3 g (0.9 mmol) of silane **2** in 2.0 mL of toluene was added 1.1 mL (1.8 mmol) of 1.6 M *n*-BuLi in hexane at −78 °C, and the reaction mixture was warmed to room temperature over a period of 30 min. **3·*n*-BuLi** crystallized at room temperature as orange rhombohedral plates. Yield: 0.31 g (83%). ^{1}H NMR ($\mathrm{C_6D_6}$): δ −1.19 (s, 2H; $\mathrm{SiC}H_2\mathrm{Li}$), 0.8−1.7 (m, 9H; $\mathrm{CC}H_2\mathrm{Li}$, $\mathrm{CC}H_2\mathrm{C}$, $\mathrm{CC}H_2\mathrm{CH_3}$, $\mathrm{CC}H_3$), 2.25−2.45 (m, 4H; $\mathrm{NC}H_2\mathrm{C}$), 2.51 (s, 2H; $\mathrm{SiC}H_2\mathrm{N}$), 2.90 (s, 6H; $\mathrm{OC}H_3$), 2.90−3.00 (m, 4H; $\mathrm{OC}H_2\mathrm{C}$), 7.00−7.40 u. 7.90−8.10 (m, 10H; Ph). ^{13}C NMR (50.3 MHz, $\mathrm{C_6D_6}$, 25 °C, $\mathrm{C_6D_6}$): δ −14.4 ($\mathrm{Si}C\mathrm{H_2Li}$), 13.9 ($\mathrm{C}C\mathrm{H_2Li}$), 14.2 ($\mathrm{H_3}C\mathrm{C}$), 25.1 ($\mathrm{C}C\mathrm{H_2C}$), 27.6 ($\mathrm{C}C\mathrm{H_2C}$), 47.4 ($\mathrm{Si}C\mathrm{H_2N}$), 55.8 (2C, $\mathrm{N}C\mathrm{H_2C}$), 58.7 (2C, $\mathrm{O}C\mathrm{H_3}$), 69.5 (2C, $\mathrm{O}C\mathrm{H_2C}$), 127.5 (2C, *m*-Ph), 127.9 (*p*-Ph), 135.3 (2C, *o*-Ph), 145.9 (*i*-Ph). ^{29}Si NMR ($\mathrm{C_6D_6}$): δ −4.6. ^{7}Li NMR ($\mathrm{C_6D_6}$): δ −0.3; [^{6}Li]**3**·*n*-Bu^{6}Li: ^{13}C CP/MAS: δ −16.7 ($\mathrm{Si}C\mathrm{H_2Li}$), 5.6 ($\mathrm{Li}C\mathrm{H_2C}$), 12.9 ($\mathrm{C}C\mathrm{H_3}$), 31.7 (2C, $\mathrm{C}C\mathrm{H_2C}$, $\mathrm{C}C\mathrm{H_2CH_3}$), 47.0 ($\mathrm{Si}C\mathrm{H_2N}$), 51.4, 55.0, 56.4, 57.5, 60.0 ($\mathrm{N}C\mathrm{H_2C}$, $\mathrm{O}C\mathrm{H_3}$), 67.1, 69.2 ($\mathrm{O}C\mathrm{H_2C}$), 128.0, 134.4, 145.1, 146.5, 151.4 (Ph). ^{6}Li CP/MAS: δ 2.0.

(Tributylstannylmethyl)[bis(2-methoxyethyl)aminomethyl](diphenyl)silane (**4**). (a) To a solution of 0.3 g (0.9 mmol) of silane **2** in 2.0 mL of pentane was added 0.6 mL (1.0 mmol) of 1.7 M *t*-BuLi in pentane at −78 °C. **3** was formed immediately as a red oil. At room temperature the pentane was separated, and the residue was dissolved in 2.0 mL toluene and reacted with 0.33 g (1 mmol) of $\mathrm{Bu_3SnCl}$ 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. Yield: 0.5 g (88%).

(b) A solution of 0.37 g (0.9 mmol) of **3·*n*-*BuLi*** in 2.0 mL of toluene was reacted with 0.59 g (1.8 mmol) of $\mathrm{Bu_3SnCl}$ 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 (stannylmethyl)silane **4** and $\mathrm{SnBu_4}$. Yield: 0.49 g (86%) of **4**.

Bp: 200 °C (ovenrohr temperature)/10^{-3} mbar. ^{1}H NMR ($\mathrm{CDCl_3}$): δ 0.22 (s, $^{2}J(\mathrm{H,{}^{119,117}Sn}) = 64.2/61.6$ Hz, 2H; $\mathrm{SiC}H_2\mathrm{Sn}$), 0.35−0.95, 1.00−1.45 (m, 27H; $\mathrm{SnC}H_2\mathrm{C}$, $\mathrm{CC}H_2\mathrm{C}$, $\mathrm{CC}H_2\mathrm{CH_3}$, $\mathrm{CC}H_3$), 2.55 (t, $^{3}J(\mathrm{H,H}) = 6.2$ Hz, 4H; $\mathrm{NC}H_2\mathrm{C}$), 2.56 (s, 2H; $\mathrm{SiC}H_2\mathrm{N}$), 3.15 (s, 6H; $\mathrm{OC}H_3$), 3.25 (t, $^{3}J(\mathrm{H,H}) = 6.2$ Hz; $\mathrm{OC}H_2\mathrm{C}$), 7.20−7.55 (m, 10H; Ph). ^{13}C NMR ($\mathrm{CDCl_3}$): δ −11.9 ($\mathrm{Si}C\mathrm{H_2Sn}$) ($^{1}J(\mathrm{C,{}^{119,117}Sn}) = 173.9/166.0$ Hz), 10.5 (3C, $\mathrm{Sn}C\mathrm{H_2C}$) ($^{1}J(\mathrm{C,{}^{119,117}Sn}) = 329.5/315.2$ Hz), 13.7 (3C, $\mathrm{C}C\mathrm{H_3}$), 27.4 (3C, $\mathrm{C}C\mathrm{H_2CH_3}$) ($^{3}J(\mathrm{C,{}^{119,117}Sn}) = 59.7/57.0$ Hz), 29.0 (3C, $\mathrm{C}C\mathrm{H_2C}$) ($^{2}J(\mathrm{C,Sn}) = 19.3$ Hz), 46.7 ($\mathrm{Si}C\mathrm{H_2N}$) ($^{3}J(\mathrm{C,Sn}) = 11.2$ Hz), 57.2 (2C, $\mathrm{N}C\mathrm{H_2C}$), 58.6 (2C; $\mathrm{O}C\mathrm{H_3}$), 71.0 (2C; $\mathrm{O}C\mathrm{H_2C}$), 127.7 (2C, *m*-Ph), 129.0 (*p*-Ph), 134.9 (2C, *o*-Ph), 138.2 (2C, *i*-Ph). ^{29}Si NMR ($\mathrm{CDCl_3}$): δ −9.8 ($^{2}J(\mathrm{Si,Sn}) = 21.3$ Hz). ^{119}Sn NMR ($\mathrm{CDCl_3}$): δ −1.4. EI-MS (70 eV): *m*/*z* 633 ($\mathrm{M^+}$, 10), 588 ($\mathrm{M^+ - H_3COCH_2}$, 72), 576 ($\mathrm{M^+ - CH_2CH_2CH_2CH_3}$, 60), 146 ($\mathrm{H_2CN(C_2H_4OCH_3)_2{}^+}$, 100). Anal. Calcd for $\mathrm{C_{32}H_{55}NO_2SiSn}$ (632.6): C, 60.76; H, 8.76; N, 2.21. Found: C, 61.0; H, 9.0; N, 2.1.

X-ray Structure Determination of [3·*n*-BuLi]$_2$ (orange crystals from toluene/hexane). Crystallographic data: $\mathrm{C_{24}H_{37}Li_2NO_2Si}$, $M = 413.52$, monoclinic, space group $P2_1/c$, $a = 10.867(4)$, $b = 11.244(2)$, $c = 20.027(7)$ Å, $\beta = 98.58(5)°$, $V = 2419.6(14)$ Å^3, $Z = 4$, $D_c = 1.135$ Mg/m^3, type of radiation Mo Kα, $\lambda = 0.71073$ Å, $\mu = 0.115$ mm^{-1}. Measurements: Stoe IPDS diffractometer, $T = 173$ K. The structure was solved using direct and Fourier methods; 20 760 reflections measured with θ in the range 2.62−25°, 4046 unique reflections; 2082 with $I > 2\sigma(I)$; refinement by full-matrix least-squares methods (based on $F_o{}^2$, SHELXL-93); anisotropic thermal parameters for all non-H atoms in the final cycles; the H atoms were refined on a riding model in their ideal geometric positions, except H(1a), H(1b), H(21a), and H(21b), which were refined isotropically; $R = 0.0516$ [$I > 2\sigma(I)$], $wR_2 = 0.1296$ (all data). SHELXS-86 and SHELXL-93 computer programs were used.

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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 together with an ORTEP plot of **3·*n*-BuLi**. This material is available free of charge via the Internet at http://pubs.acs.org.

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