

Synthesis of a Novel Binuclear Chlorotin(II) Alkyl and a Lithium Trialkylstannate Zwitterionic Cage Molecule: Crystal Structures of $[\text{Sn}(\text{Cl})\text{R}^{\text{N}}]_2$ and $[(\text{SnR}^{\text{N}}_3)\text{Li}(\mu^3\text{-Cl})\text{Li}(\text{tmeda})]_2$ [$\text{R}^{\text{N}} = \text{CH}(\text{SiBu}^t\text{Me}_2)\text{C}_5\text{H}_4\text{N-2}$]

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The reaction of SnCl_2 with $[\text{LiR}^{\text{N}}(\text{tmeda})]_2$ [$\text{R}^{\text{N}} = \text{CH}(\text{SiBu}^t\text{Me}_2)\text{C}_5\text{H}_4\text{N-2}$; $\text{tmeda} = N,N,N,N$ -tetramethylethylenediamine)] in various stoichiometric ratios afforded a binuclear chlorotin(II) alkyl $[\text{Sn}(\text{Cl})\text{R}^{\text{N}}]_2$ (**1**) and zwitterionic lithium trialkylstannate complexes $[(\text{SnR}^{\text{N}}_3)\text{Li}]\{\mu^3\text{-Cl}\}[\text{Li}(\text{tmeda})_2](\mu^2\text{-Cl})$ (**2**) and $[(\text{SnR}^{\text{N}}_3)\text{Li}](\mu^3\text{-Cl})\text{Li}(\text{tmeda})_2$ (**3**). X-ray structure analysis has shown that the anionic N-functionalized alkyl ligand $(\text{R}^{\text{N}})^-$ in **1** acts as a bridging ligand joining the two tin atoms to form an eight-membered ring in a "boat" conformation. Compounds **2** and **3** consist of one and two lithium trialkylstannate ion pairs $[\text{SnR}^{\text{N}}_3]^- \text{Li}^+$ bound to the chlorine atom of the $[\text{LiCl}(\text{tmeda})]_2$ fragment, respectively. The lithium ion in the ion pair is coordinated via the pyridyl nitrogens to form a cage.

Introduction

Organolithium compounds are widely used as alkylating agents for the synthesis of stannylenes SnR_2 . For example, $\text{Sn}\{\text{CH}(\text{SiMe}_3)_2\}_2$, $\text{Sn}\{\text{C}_6\text{H}_2(\text{CF}_3)_3\text{-2,4,6}\}_2$, $\text{Sn}\{\text{C}_6\text{H}_2\text{Pr}^i\text{-2,4,6}\}_2$, and $\text{Sn}\{\text{C}(\text{SiMe}_3)_2\text{C}_5\text{H}_4\text{N-2}\}_2$ have been synthesized from the appropriate lithium reagents.^{1–5} Likewise, monoalkylated compound such as $[\text{Sn}\{\text{C}(\text{SiMe}_3)_2\text{C}_5\text{H}_4\text{N-2}\}\text{Cl}]$ are accessible by the reaction of $[\text{Li}\{\text{C}(\text{SiMe}_3)_2\text{C}_5\text{H}_4\text{N-2}\}_2]$ with SnCl_2 in 1:1 stoichiometric ratio or by the redistribution reaction of $\text{Sn}\{\text{C}(\text{SiMe}_3)_2\text{C}_5\text{H}_4\text{N-2}\}$ with SnCl_2 .⁵

Tin(II) compounds are known to possess both acid and base properties; the metal center can either react with electrophiles or act as a Lewis acid and thus be susceptible to nucleophilic attack. It has been shown that nucleophilic addition of organolithium reagents to organotin(II) compounds formed triorganostannate compounds. For example, $[(\text{Sn}(\text{furyl})_3\text{Li}(\text{furyl})_3\text{Sn})^-]$, $[\text{HC}\{\text{SiMe}_2\text{N}(4\text{-CH}_3\text{C}_6\text{H}_4)\}_3\text{SnLi}(\text{THF})_3]$, and $[\text{Bu}^n\text{Sn}(\text{NC}_5\text{H}_4\text{-C,N})_3\text{LiBr}(0.5\text{thf})]$ have been isolated and structurally characterized.^{6–8} Recently, we have shown that the tin(II) dialkyl $[\text{SnR}'_2]$ [$\text{R}' = \text{CH}(\text{SiMe}_3)\text{C}_9\text{H}_8\text{N-8}$] behaves as a Lewis base and reacts with SnX_2 to form donor–

acceptor compounds $\text{R}'_2\text{Sn}\rightarrow\text{SnX}_2$ ($\text{X} = \text{Cl}, \text{Br}, \text{I}$).^{9,10} The Lewis acidity of the tin(II) alkyl $\text{Sn}\{\text{CH}(\text{SiMe}_3)_2\}_2$ was found to be low, as noted by the lack of thermal stability of the 1:1 base–adduct formed. The isolation of the product of the addition of LiR to SnR_2 to form lithium trialkylstannate LiSnR_3 [$\text{R} = \text{CH}(\text{SiMe}_3)_2$] was unsuccessful.¹⁰

We have recently reported the synthesis and structures of some group 14 metal alkyls MR'_2 [$\text{M} = \text{Ge}, \text{Sn}, \text{Pb}$; $\text{R}' = \text{CPh}(\text{SiMe}_3)\text{C}_5\text{H}_4\text{N-2}$ or $\text{CH}(\text{SiMe}_3)\text{C}_9\text{H}_8\text{N-8}$].¹¹ We describe here the synthesis and X-ray structures of a series of products isolated from the alkylation reaction of SnCl_2 with $[\text{LiR}^{\text{N}}(\text{tmeda})]_2$ [$\text{R}^{\text{N}} = \text{CH}(\text{SiBu}^t\text{Me}_2)\text{C}_5\text{H}_4\text{N-2}$] in different stoichiometric ratios.

Results and Discussion

The reactions of $[\text{LiR}^{\text{N}}(\text{tmeda})]_2$ with SnCl_2 in various stoichiometric ratios afforded tin(II) alkyl compounds $[\text{Sn}(\text{Cl})\text{R}^{\text{N}}]_2$ (**1**) and zwitterionic lithium trialkylstannate complexes $[(\text{SnR}^{\text{N}}_3)\text{Li}]\{\mu^3\text{-Cl}\}[\text{Li}(\text{tmeda})_2](\mu^2\text{-Cl})$ (**2**) and $[(\text{SnR}^{\text{N}}_3)\text{Li}](\mu^3\text{-Cl})\text{Li}(\text{tmeda})_2$ (**3**) (Scheme 1). Alkylation using 1 equiv of $[\text{LiR}^{\text{N}}(\text{tmeda})]_2$ with SnCl_2 afforded the binuclear chlorotin(II) alkyl $[\text{R}^{\text{N}}\text{SnCl}]_2$ (**1**) in good yield (75%). Similar reactions employing 3 and 3.5 equiv of $[\text{LiR}^{\text{N}}(\text{tmeda})]_2$ yielded $[(\text{SnR}^{\text{N}}_3)\text{Li}]\{\mu^3\text{-Cl}\}[\text{Li}(\text{tmeda})_2](\mu^2\text{-Cl})$ (**2**) and $[(\text{SnR}^{\text{N}}_3)\text{Li}](\mu^3\text{-Cl})\text{Li}(\text{tmeda})_2$ (**3**), respectively. Compounds **1–3** have been characterized by NMR spectroscopy and X-ray structure analysis. Binuclear tin(II) compounds similar to **1** such as chlorotin(II) amides $[\text{Sn}(\mu\text{-Cl})(\text{NR}_2)]_2$ [$\text{NR}_2 =$

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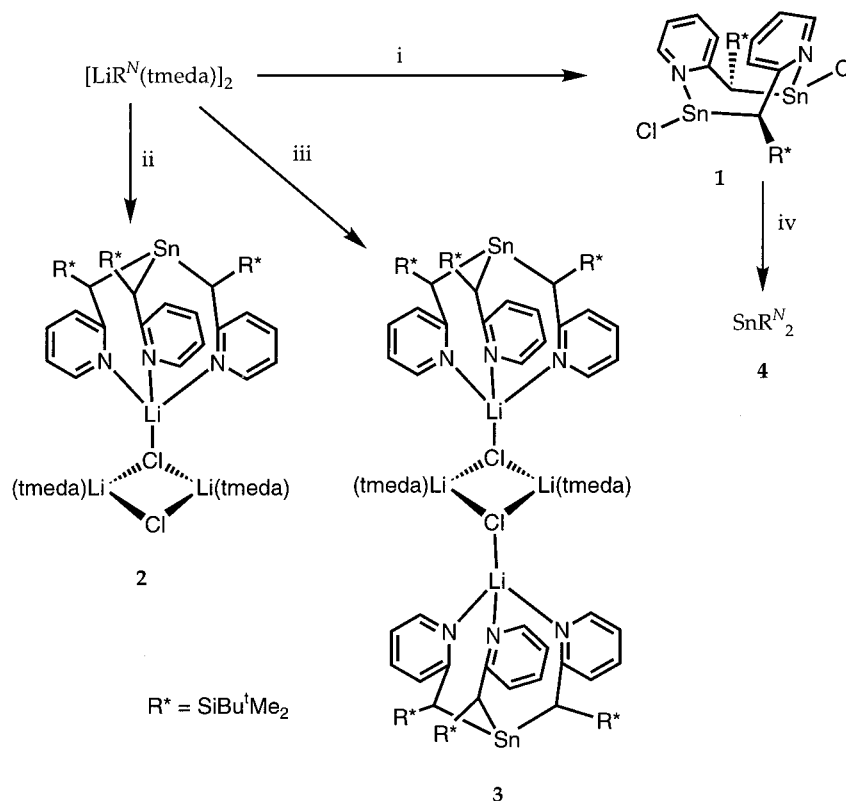
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Scheme 1. Preparation of Tin(II) Alkyl Compounds Containing the 2-Pyridyl(*tert*-butyldimethylsilyl)methyl Ligand [CH(SiBu^tMe₂)C₅H₄N-2] (R^N)^a



^a Conditions and reagents: (i) 1.5SnCl₂, Et₂O; (ii) SnCl₂, Et₂O; (iii) 1.8SnCl₂, Et₂O; (iv) [LiR^N(tmeda)]₂.

CCMe₂(CH₂)₃CMe₂ or N(SiMe₃)₂] having chloro-bridges have been prepared by the redistribution reaction of Sn(NR₂)₂ with SnCl₂.¹² In contrast, attempts to prepare ClSn{CH(SiMe₃)₂} by similar methods were unsuccessful. It was reported that the reaction of Sn{CH(SiMe₃)₂}₂ with SnCl₂ afforded [Sn{CH(SiMe₃)₂}₂Cl₂] and tin metal.¹⁰

The X-ray structure of **1** as shown in Figure 1 is a binuclear molecule with (R^N)⁻ acting in a bidentate C,N-bridging mode between two tin(II) atoms forming an eight-membered ring in a "boat" conformation. The closely related and more bulky alkyl ligand [C(SiMe₃)₂C₅H₄N-2] (R'^N)⁻ in the monomeric compound [R'^NSnCl] has been shown to function as a bidentate C,N-chelate ligand.⁵ In previous studies, we have found that the less bulky ligand R^N [R^N = CH(SiBu^tMe₂)C₅H₄N-2] can behave both as a C,N-chelate and in the bridging bonding mode, as demonstrated in the structures of binuclear compounds (MR^N)₂ (M = Fe and Co).¹³ In **1**, the geometry at the tin centers (sum of bond angles Σ = 277.4° and 276.9°) is consistent with the presence of a stereoactive lone pair. The Sn–C and Sn–N distances of 2.308(av) and 2.277(av) Å are in good agreement with the corresponding distances of 2.32(2) and 2.27(2) Å, respectively, in [C(SiMe₃)₂C₅H₄N-2]. The tin–tin distance of 4.47 Å in **1** is too long to be considered as a bonding interaction.

The reaction of SnCl₂ with 3 equiv of [LiR^N(tmeda)]₂ gave the self-assembled lithium trialkylstannate cage

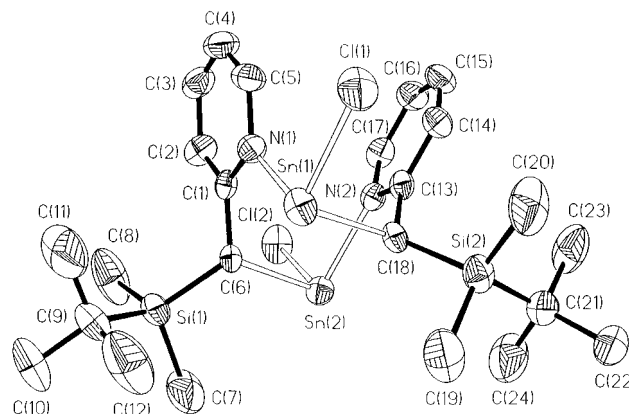


Figure 1. Perspective drawing of **1** with the atomic numbering scheme. Thermal ellipsoids are shown at the 35% probability levels. Selected bond distances [Å] and angles [deg]: Sn(1)–Cl(1) 2.438(3), Sn(1)–N(1) 2.287(7), Sn(1)–C(18) 2.309(6), Sn(2)–Cl(2) 2.454(3), Sn(2)–C(6) 2.306(6), Sn(2)–N(2) 2.267(5); Cl(1)–Sn(1)–N(1) 92.0(2), Cl(1)–Sn(1)–C(18) 95.8(2), N(1)–Sn(1)–C(18) 89.6(2), Cl(2)–Sn(2)–C(6) 94.3(2), Cl(2)–Sn(2)–N(2) 92.8(2), C(6)–Sn(2)–N(2) 89.8(2).

molecule [(SnR^N₃)Li]{(μ³-Cl){Li(tmeda)₂}(μ²-Cl)} (**2**). The alkylation reaction with an excess (3.5 equiv) of [LiR^N(tmeda)]₂ led to the formation of the double cage molecule [(SnR^N₃)Li]{(μ³-Cl)Li(tmeda)]₂ (**3**). The trialkylstannate [SnR^N₃]⁻ is believed to result from the nucleophilic addition of SnR^N₂ with [R^N]⁻. Wright and co-workers recently have reported the structures of [Buⁿ–Sn(NC₅H₄–C,N)₃MBr(0.5thf)] (M = Li, Cu) and [Pb(2-Pyridyl)₃Li(0.5thf)] isolated from the reaction of lithium pyridyl Li(C₅H₄N) with MCp₂ (M = Sn and Pb).⁸ Similar

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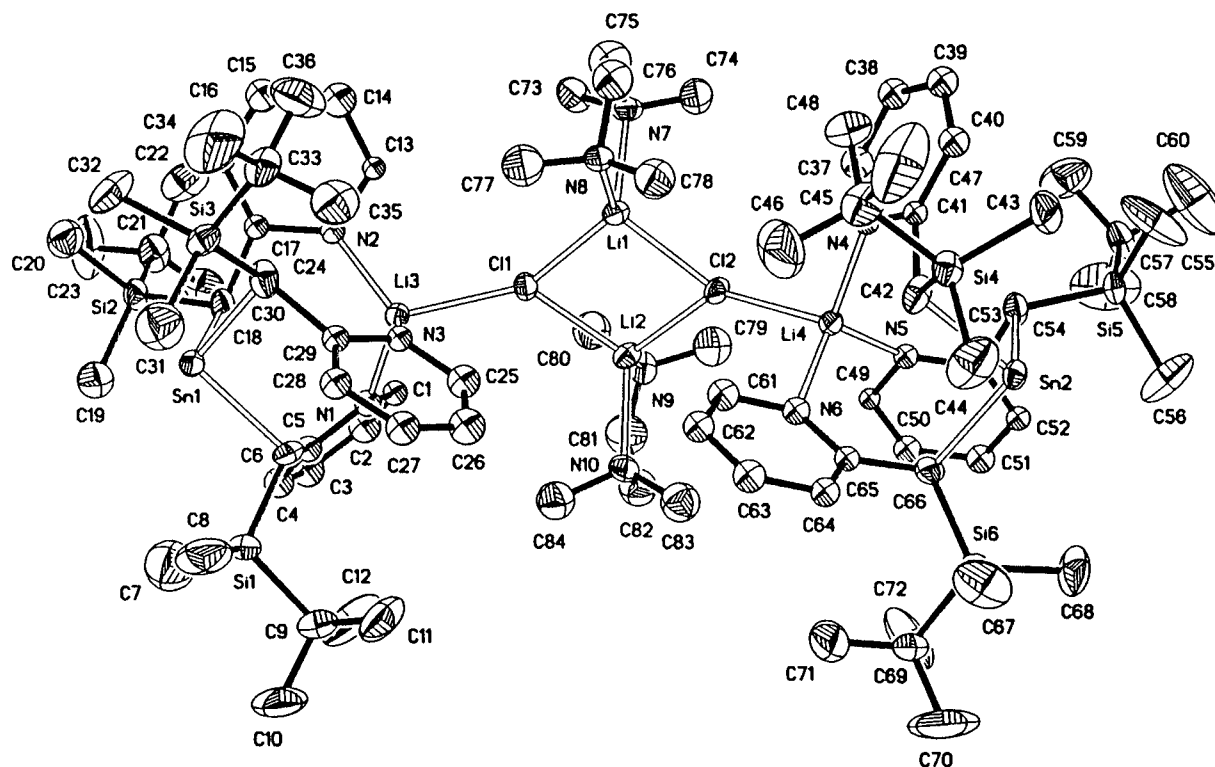


Figure 2. Perspective drawing of **3** with the atomic numbering scheme. Thermal ellipsoids are shown at the 35% probability levels. Selected bond distances [Å] and angles [deg]: Sn(1)–C(18) 2.351(9), Sn(1)–C(30) 2.330(11), Sn(1)–C(6) 2.357(9), Li(3)–N(1) 2.08 (2), Li(3)–N(2) 2.09(2), Li(3)–N(3) 2.00(2), Li(3)–Cl(1) 2.504(14), Li(2)–Cl(1) 2.331(2), Li(2)–Cl(2) 2.33(2), Li(1)–Cl(1) 2.365(14), Li(1)–Cl(2) 2.347(13); C(6)–Sn(1)–C(18) 94.1(4), C(6)–Sn(1)–C(30) 95.4(4), C(18)–Sn(1)–C(30) 94.5(4), N(1)–Li(3)–N(2) 111.4(3), N(1)–Li(3)–N(3) 117.3(2), N(2)–Li(3)–N(3) 120.3(8), Li(2)–Cl(1)–Li(1) 80.0(5), Li(2)–Cl(2)–Li(1) 80.4(5), Cl(1)–Li(2)–Cl(2) 100.6(5), Cl(1)–Li(1)–Cl(2) 98.8(5).

structural features in $\text{Li}[\text{M}(\text{C}_5\text{H}_4\text{N})_3]$ with $\text{M}(\text{C}_5\text{H}_4\text{N})_3$ acting as a tridentate ligand coordinating to lithium have been found.⁸ Moreover, sodium tri(pyrazol-1-yl)-germanate $[(\text{THF})_3\text{Na}\{\text{(pz)}_3\text{Ge}\}]$ and -stannate $[(\text{THF})_2\text{(pzH)}\{\text{(pz)}_3\text{Sn}\}]_2$ (pz = pyrazol-1-yl) with similar structural features have been reported by Stalke and co-workers.¹⁴

The reaction of SnCl_2 with 2 equiv of $[\text{LiR}^{\text{N}}(\text{tmeda})]_2$ gave compound **2** in moderate yield; the expected dialkyl compound was not isolated. However, stepwise reaction of compound **1** with a further equivalent of $[\text{LiR}^{\text{N}}(\text{tmeda})]_2$ afforded the tin(II) dialkyl SnR^{N}_2 , which has been characterized by its ^{119}Sn and ^1H NMR spectra. Attempts to isolate good-quality crystals of **4** for X-ray structure determination have been unsuccessful.

The structure of **3** as shown in Figure 2 comprises two $(\text{SnR}^{\text{N}}_3)^-\text{Li}^+$ ion pairs, with the lithium ion bound to the chlorine atoms of the four-membered chloro-bridged Li_2Cl_2 unit. The $[\text{SnR}^{\text{N}}_3]^-$ moiety acts as a tridentate ligand coordinated to lithium via the pyridyl nitrogen atoms to form a cage-like structure. The structure of the $[\text{LiCl}(\text{tmeda})]_2$ unit resembles that of the 1:1 adducts of lithium(I) halides, $[\text{LiX}(\text{tmeda})]$ (X = Cl, Br, I), reported by Raston and co-workers.¹⁵ The geometry at the tin center (sum of bond angles $\Sigma = 283.2^\circ$) is trigonal pyramidal, with the tin atom located 1.25 Å above the plane of the three α -carbons. The two trian-

gular planes defined respectively by the three α -carbons and the pyridyl nitrogens are staggered with a twist angle of about 58.5° . The average Sn–C distance of 2.34(1) Å is consistent with covalent bonding as compared to the corresponding values of 2.32 and 2.36(av) Å in $[\text{R}^{\text{N}}\text{SnCl}]$ and $[\text{SnR}^{\text{N}}_2]$, respectively.⁵ The structure of **2** is similar to that of **3** except that only one $\text{SnR}^{\text{N}}_3\text{Li}$ ion pair is connected to the chlorine atom of the Li_2Cl_2 ring.

Experimental Section

General Procedures. All manipulations were carried out under an inert atmosphere of argon gas by standard Schlenk techniques or in a dinitrogen glovebox. Solvents were dried over and distilled from CaH_2 (hexane) and/or Na (Et_2O). SnCl_2 was purchased from Aldrich and used without further purification. $[\text{Li}\{\text{CH}(\text{SiBu}^t\text{Me}_2)\text{C}_5\text{H}_4\text{N}-2\}(\text{tmeda})]$ was prepared according to the literature.¹⁶ The ^1H and ^{119}Sn NMR were recorded at 250 and 186.5 MHz, respectively, using a Bruker WM-250 or ARX-500 instrument. All spectra were recorded in benzene- d_6 , and the chemical shifts δ are relative to SiMe_4 and SnMe_4 for ^1H and ^{119}Sn NMR, respectively.

Preparation of $[\text{Sn}(\text{Cl})\text{R}^{\text{N}}]_2$ [$\text{R}^{\text{N}} = \text{CH}(\text{SiBu}^t\text{Me}_2)\text{C}_5\text{H}_4\text{N}-2$] (1**).** To a slurry of SnCl_2 (0.276 g, 1.46 mmol) in hexane (30 mL) was added dropwise a solution of $[\text{Li}\{\text{CH}(\text{SiBu}^t\text{Me}_2)\text{C}_5\text{H}_4\text{N}-2\}(\text{tmeda})]_2$ (0.472 g, 0.73 mmol) in ether (25 mL). After stirring for 4 h, the white solid formed was separated from the pale yellow mixture by filtration. The pale yellow filtrate was concentrated to ca. 10 mL and kept at -20°C for 18 h to yield a white, crystalline solid, which was collected by filtration, washed with cooled pentane, and dried under vacuum to

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yield 0.39 g (75%) of **1**. Single crystals suitable for X-ray structure determination were recrystallized from a solvent mixture of ether and hexane. Anal. Calcd for C₂₄H₄₀N₂Cl₂Si₂-Sn₂: C, 39.98; H, 5.59; N, 3.89. Found: C, 39.95; H, 5.64; N, 3.94. ¹H NMR (250 MHz, C₆D₆, 25 °C, SiMe₄): δ = 0.33 (s, 6H, SiMe₂), 0.86 (s, 9H, Bu⁴), 2.49 (s, ²J(H, ¹¹⁹Sn); C₅H₄N, 5.75 (t, 1H), 6.78 (t, 1H), 7.19 (m, 1H), 8.06 (d, 1H). ¹¹⁹Sn NMR (186.5 MHz, C₆D₆, 25 °C, SnMe₄): δ = 75.9 (s).

Preparation of [(SnR^N₃)Li]₂{LiCl(tmeda)}₂ (2**).** To a slurry of SnCl₂ (0.15 g, 0.79 mmol) in hexane (30 mL) was added dropwise a solution of [Li{CH(SiBu⁴Me₂)C₅H₄N-2}-(tmeda)]₂ (0.50 g, 0.79 mmol) in ether (30 mL). After stirring for 5 h, the reaction mixture was filtered and the filtrate was concentrated to ca. 10 mL and kept at -20 °C for 18 h to yield a yellow crystalline solid, which was collected by filtration, washed with cooled pentane, and dried under vacuum to yield 0.39 g (64%) of **2**. Anal. Calcd for C₄₈H₉₂N₇Cl₂Li₃Si₃Sn: C, 53.48; H, 8.60; N, 9.10. Found: C, 53.12; H, 8.55; N, 8.63. ¹H NMR (250 MHz, C₆D₆, 25 °C, SiMe₄): δ = 0.27 (s, 9H), 0.71 (s, 9H), 0.79 (s, 27H), 1.69 (s, 3H), 2.01 (s, 8H), 2.15 (s, 24H), 6.32 (t, 3H), 6.97 (t, 3H), 7.10 (d, 3H), 8.89 (d, 3H). ¹¹⁹Sn NMR (186.5 MHz, C₆D₆, 25 °C, SnMe₄): δ = 209 (s).

Preparation of [(SnR^N₃)Li]₂{LiCl(tmeda)}₂ (3**).** The procedure for the preparation of **3** was similar to **2** except that 3 equiv of [Li{CH(SiBu⁴Me₂)C₅H₄N-2}-(tmeda)]₂ was used (yield = 58%). Anal. Calcd for C₈₄H₁₅₂N₁₀Cl₂Li₄Si₆Sn₂: C, 54.38; H, 8.26; N, 7.39. Found: C, 55.84; H, 8.48; N, 7.75. ¹H NMR (250 MHz, C₆D₆, 25 °C, SiMe₄): δ = 0.24 (s, 18H), 0.78 (s, 18H), 0.89 (s, 54H), 1.63 (s, 6H), 2.10 (s, 8H), 2.14 (s, 24H), 6.29 (m, 6H), 6.99 (m, 6H), 7.11 (m, 6H), 8.58 (m, 6H). ¹¹⁹Sn NMR (186.5 MHz, C₆D₆, 25 °C, SnMe₄): δ = 201.5 (s).

Preparation of SnR^N₂ (4**).** To a solution of **1** (0.26 g, 0.36 mmol) in 20 mL of ether was added slowly a solution of [Li{CH(SiBu⁴Me₂)C₅H₄N-2}-(tmeda)]₂ (0.25 g, 0.38 mmol) in 15 mL of ether. The resulting mixture was stirred at room temperature for 6 h and then filtered. The pale yellow filtrate was concentrated (ca. 10 mL) and stored at -20 °C for 3 days to yield yellow solid of **4** (0.21 g, 55%). ¹H NMR (250 MHz, C₆D₆, 25 °C, SiMe₄): δ 0.20 (bs, 6H), 0.94 (s, 9H), 2.14 (s, 1H), 6.34 (t, 1H), 6.49 (d, 1H), 6.91 (m, 1H), 7.96 (d, 1H). ¹¹⁹Sn NMR (186.5 MHz, C₆D₆, 25 °C, SnMe₄): δ = 165 (s).

X-ray Crystallography. Crystal data (Mo K α radiation, 295 K): for (**1**), C₂₄H₄₀Cl₂N₂Si₂Sn₂, *M* = 721.0, triclinic, space group *P*1, *a* = 8.296(3) Å, *b* = 12.293(5) Å, *c* = 16.835(9) Å, *a*

= 76.640(0)°, β = 86.450(0)°, γ = 71.990(10)°, *V* = 1588.4(15) Å³, *Z* = 4, *F*(000) = 720, *D*_c = 1.508 mg m⁻³, μ = 1.830 mm⁻¹, $2\theta_{\max}$ = 45°, *N* = 4641, *N*_o = 3312, *R* = 0.040, *wR* = 0.050; for (**2**), (C₅₂H₉₈Cl₂Li₄N₇OSi₃Sn, *M* = 1132.05, monoclinic, space group *P*2₁/*c*, *a* = 17.157(3) Å, *b* = 19.911(3) Å, *c* = 23.724(9) Å, α = 90°, β = 100.93°, γ = 90°, *V* = 7957(4) Å³, *Z* = 4, *F*(000) = 2408, *D*_c = 0.945 g cm⁻³, μ = 0.464 mm⁻¹, $2\theta_{\max}$ = 26.73°, *N* = 11042, *N*_o = 9012, *R* = 0.0590, *wR* = 0.1415; for (**3**), (C₈₈H₁₆₂-Cl₂Li₄N₁₀OSi₆Sn₂, *M* = 1880.86, monoclinic, space group *P*2₁/*n*, *a* = 20.7230(10) Å, *b* = 25.8150(10) Å, *c* = 21.9910(10) Å, α = 90°, β = 93.650(10)°, γ = 90°, *V* = 11740.5(9) Å³, *Z* = 4, *F*(000) = 3992, *D*_c = 1.064 g cm⁻³, μ = 0.572 mm⁻¹, $2\theta_{\max}$ = 26.73°, *N* = 21178, *N*_o = 13567, *R* = 0.0775, *wR* = 0.2392.

Single crystals were sealed in 0.5 mm Lindemann glass capillaries under dinitrogen. X-ray data were collected on a R3m/V and Rigaku AFC7R diffractometers using graphite-monochromatized MoK α radiation (λ = 0.71073 Å) in the $\omega/2\theta$ scan mode. Unique reflections were measured, and "observed" reflections with $|F_o| \geq 3\sigma(|F_o|)$ were used in the structure solution and refinement. The weighing scheme used was $w = [\sigma^2 |F_o| + 0.0008 |F_o|^2]^{-1}$ for **1** and $w = [\sigma^2 |F_o| + 0.0005 |F_o|^2]^{-1}$ for **2** and **3**. The structures were solved by direct phase determination using the computer program SHELXTL-PC¹⁷ on a PC 486 and refined by full-matrix least-squares with anisotropic thermal parameters for the non-hydrogen atoms. Hydrogen atoms were introduced in their idealized positions and included in structure factor calculations with assigned isotropic temperature factors.

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Supporting Information Available: Tables of crystal data, bond distances and angles, atomic coordinates and equivalent isotropic temperature factors, anisotropic thermal parameters, and hydrogen atom coordinates and assigned isotropic temperature factors for **1–3**. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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