1482 *Organometallics* **1999,** *18,* 1482−1485

Synthesis of a Novel Binuclear Chlorotin(II) Alkyl and a Lithium Trialkylstannate Zwitterionic Cage Molecule: Crystal Structures of $[Sn(Cl)R^N]_2$ and $[(SnR^N_3)Li(\mu^3\text{-}Cl)Li(tmeda)]_2$ $[R^N = CH(SiBu^tMe_2)C_5H_4N\text{-}2]$

Wing-Por Leung,* Lin-Hong Weng, Wai-Him Kwok, Zhong-Yuan Zhou,
Ze-Ying Zhang, and Thomas C. W. Mak

*Department of Chemistry, The Chinese University of Hong Kong, Shatin,
New Territories, Hong Kong*

Received September 14, 1998

The reaction of $SnCl_2$ with $[LiR^N(tmeda)]_2$ $[R^N = CH(SiBu^tMe_2)C_5H_4N\text{-}2$; tmeda = *N*,*N*,*N*′,*N*′-tetramethylethylenediamine)] in various stoichiometric ratios afforded a binuclear chlorotin(II) alkyl $[Sn(Cl)R^N]_2$ (**1**) and zwitterionic lithium trialkylstannate complexes $[\{(SnR^N_3)Li\}\{(\mu^3\text{-}Cl)\{Li(tmeda)_2\}(\mu^2\text{-}Cl)]$ (**2**) and $[\{(SnR^N_3)Li\}(\mu^3\text{-}Cl)Li(tmeda)]_2$ (**3**). X-ray structure analysis has shown that the anionic N-functionalized alkyl ligand $(R^N)^-$ 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 $[SnR^N_3]^-Li^+$ bound to the chlorine atom of the $[LiCl(tmeda)]_2$ 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, $Sn\{CH(SiMe_3)_2\}_2$, $Sn\{C_6H_2(CF_3)_3\text{-}2,4,6\}_2$, $Sn\{C_6H_2Pr^i_3\text{-}2,4,6\}_2$, and $Sn\{C(SiMe_3)_2C_5H_4N\text{-}2\}_2$ have been synthesized from the appropriate lithium reagents.[1−5] Likewise, monoalkylated compound such as $[Sn\{C(SiMe_3)_2C_5H_4N\text{-}2\}Cl]$ are accessible by the reaction of $[Li\{C(SiMe_3)_2C_5H_4N\text{-}2]_2$ with $SnCl_2$ in 1:1 stoichiometric ratio or by the redistribution reaction of $Sn\{C(SiMe_3)_2C_5H_4N\text{-}2\}$ 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, $[(Sn(furyl)_3Li(furyl)_3Sn]^-$, $[HC\{SiMe_2N(4\text{-}CH_3C_6H_4)\}_3SnLi(THF)_3]$, and $[Bu^nSn(NC_5H_4\text{-}C,N)_3LiBr(0.5thf)]$ have been isolated and structurally characterized.[6−8] Recently, we have shown that the tin(II) dialkyl $[SnR'_2]_2$ $[R' = CH(SiMe_3)C_9H_8N\text{-}8]$ behaves as a Lewis base and reacts with SnX_2 to form donor−acceptor compounds $R'_2Sn{\rightarrow}SnX_2$ (X = Cl, Br, I).[9,10] The Lewis acidity of the tin(II) alkyl $Sn\{CH(SiMe_3)_2\}_2$ 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 $LiSnR_3$ $[R = CH(SiMe_3)_2]$ was unsuccessful.[10]

We have recently reported the synthesis and structures of some group 14 metal alkyls MR'_2 [M = Ge, Sn, Pb; $R' = CPh(SiMe_3)C_5H_4N\text{-}2$ or $CH(SiMe_3)C_9H_8N\text{-}8$].[11] We describe here the synthesis and X-ray structures of a series of products isolated from the alkylation reaction of $SnCl_2$ with $[LiR^N(tmeda)]_2$ $[R^N = CH(SiBu^tMe_2)\text{-}C_5H_4N\text{-}2]$ in different stoichiometric ratios.

Results and Discussion

The reactions of $[LiR^N(tmeda)]_2$ with $SnCl_2$ in various stoichiometric ratios afforded tin(II) alkyl compounds $[Sn(Cl)R^N]_2$ (**1**) and zwitterionic lithium trialkylstannate complexes $[\{(SnR^N_3)Li\}\{(\mu^3\text{-}Cl)\{Li(tmeda)_2\}(\mu^2\text{-}Cl)]$ (**2**) and $[\{(SnR^N_3)Li\}(\mu^3\text{-}Cl)Li(tmeda)]_2$ (**3**) (Scheme 1). Alkylation using 1 equiv of $[LiR^N(tmeda)]_2$ with $SnCl_2$ afforded the binuclear chlorotin(II) alkyl $[R^NSnCl]_2$ (**1**) in good yield (75%). Similar reactions employing 3 and 3.5 equiv of $[LiR^N(tmeda)]_2$ yielded $[\{(SnR^N_3)Li\}\{(\mu^3\text{-}Cl)\text{-}\{Li(tmeda)_2\}(\mu^2\text{-}Cl)]$ (**2**) and $[\{(SnR^N_3)Li\}(\mu^3\text{-}Cl)Li(tmeda)]_2$ (**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 $[Sn(\mu\text{-}Cl)(NR_2)]_2$ [NR_2 =

---

[1] Davidson, P. J.; Lappert, M. F. *J. Chem. Soc., Chem. Commun.* **1974**, 895.

[2] Lay, U.; Pritzkow, H.; Grützmacher, H. *J. Chem. Soc., Chem. Commun.* **1992**, 260.

[3] Masamune, S.; Sita, L. R. *J. Am. Chem. Soc.* **1985**, *107*, 6390.

[4] Schäfer, A.; Weidenbruch, M.; Saak, W.; Pohl, S.; Marsmann, H. *Angew. Chem., Int. Ed. Engl.* **1991**, *30*, 834.

[5] Engelhardt, L. M.; Jolly, B. S.; Lappert, M. F.; Raston, C. L.; White, A. H. *J. Chem. Soc., Chem. Commun.* **1988**, 336.

[6] Veith, M.; Ruloff, C.; Huch, V.; Töllner, F. *Angew. Chem., Int. Ed. Engl.* **1988**, *27*, 1381.

[7] Hellmann, K. W.; Gade, L. H.; Gevert, O.; Steinert, P. *Inorg. Chem.* **1995**, *34*, 4069.

[8] Beswick, M. A.; Belle, C. J.: Davies, M. K.; Halcrow, Raithby, A. S.; Wright, D. S. *J. Chem. Soc., Chem. Commun.* **1996**, 2619.

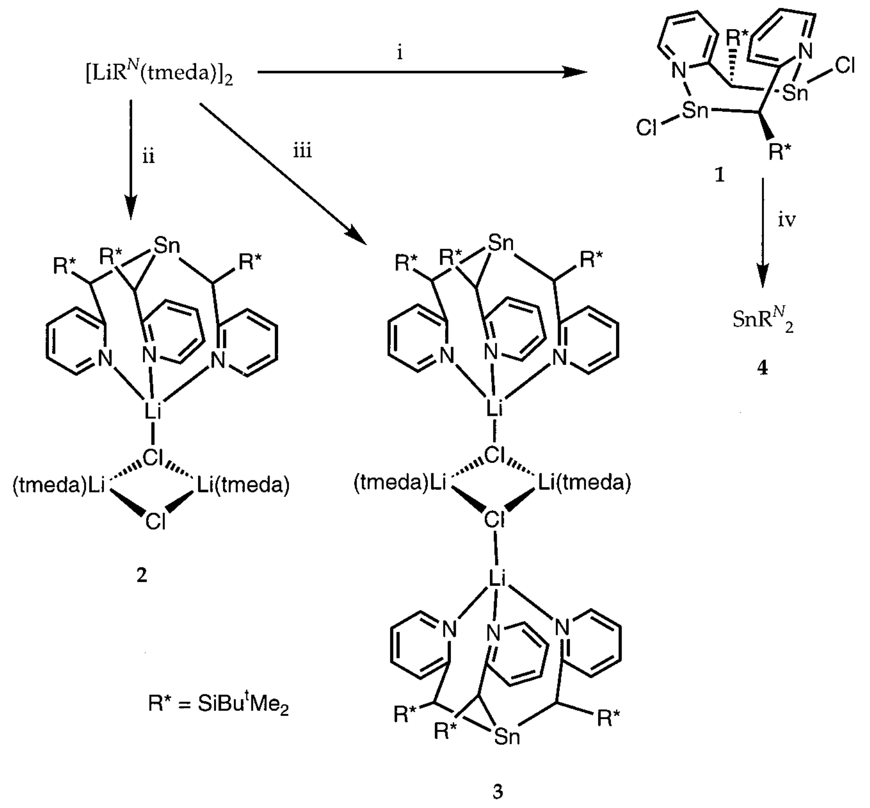
[9] Leung, W.-P.; Kwok, W.-H.; Xue, F.; Mak, T. C. W. *J. Am. Chem. Soc.* **1997**, *119*, 1145.

[10] Cotton, J. D.; Davidson, P. J.; Lappert, M. F. *J. Chem. Soc., Dalton Trans.* **1976**, 2275.

[11] Leung, W.-P.; Kwok, W.-H.; Weng, L.-H.; Law, L. T. C.; Zhou, Z.-Y.; Mak, T. C. W. *J. Chem. Soc., Dalton Trans.* **1997**, 4301.

10.1021/om9807699 CCC: $18.00
Publication on Web 03/19/1999

Scheme 1. Preparation of Tin(II) Alkyl Compounds Containing the 2-Pyridyl(*tert*-butyldimethylsilyl)methyl Ligand $[CH(SiBu^tMe_2)C_5H_4N\text{-}2]$ $(R^N)^a$



[a] Conditions and reagents: (i) $1.5SnCl_2$, Et_2O; (ii) $SnCl_2$, Et_2O; (iii) $1.8SnCl_2$, Et_2O; (iv) $[LiR^N(tmeda)]_2$.

$CCMe_2(CH_2)_3CMe_2$ or $N(SiMe_3)_2$] having chloro-bridges have been prepared by the redistribution reaction of $Sn(NR_2)_2$ with $SnCl_2$.[12] In contrast, attempts to prepare $ClSn\{CH(SiMe_3)_2\}$ by similar methods were unsuccessful. It was reported that the reaction of $Sn\{CH(SiMe_3)_2\}_2$ with $SnCl_2$ afforded $[Sn\{CH(SiMe_3)_2\}_2Cl_2]$ 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 $[C(SiMe_3)_2C_5H_4N\text{-}2]^-$ $(R'')^-$ in the monomeric compound $[R''SnCl]$ 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 [$R^N = CH(SiBu^tMe_2)C_5H_4N\text{-}2$] can behave both as a C,N-chelate and in the bridging bonding mode, as demonstrated in the structures of binuclear compounds $(MR^N_2)_2$ (M = Fe and Co).[13] In **1**, the geometry at the tin centers (sum of bond angles $\Sigma = 277.4°$ 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 $[C(SiMe_3)_2C_5H_4N\text{-}2]$. The tin−tin distance of 4.47 Å in **1** is too long to be considered as a bonding interaction.

The reaction of $SnCl_2$ with 3 equiv of $[LiR^N(tmeda)]_2$ gave the self-assembled lithium trialkylstannate cage molecule $[\{(SnR^N_3)Li\}\{(\mu^3\text{-}Cl)\{Li(tmeda)_2\}(\mu^2\text{-}Cl)]$ (**2**). The alkylation reaction with an excess (3.5 equiv) of $[LiR^N(tmeda)]_2$ led to the formation of the double cage molecule $[\{(SnR^N_3)Li\}(\mu^3\text{-}Cl)Li(tmeda)]_2$ (**3**). The trialkylstannate $[SnR^N_3]^-$ is believed to result from the nucleophilic addition of SnR^N_2 with $[R^N]^-$. Wright and co-workers recently have reported the structures of $[Bu^nSn(NC_5H_4\text{-}C,N)_3MBr(0.5thf)]$ (M = Li, Cu) and $[Pb(2\text{-}Pyridyl)_3Li(0.5thf)]$ isolated from the reaction of lithium pyridyl $Li(C_5H_4N)$ with MCp_2 (M = Sn and Pb).[8] Similar

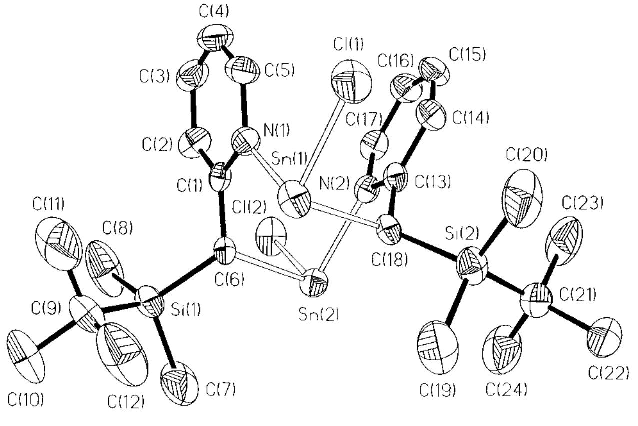


Figure 1. Perspective drawing of **1** with the atomic numbering scheme. Thermal ellipsoids are shown at the 35% probability levels. Selected bond distances [Å] and angles [deg]: Sn(1)−Cl(1) 2.438(3), Sn(1)−N(1) 2.287(7), Sn(1)−C(18) 2.309(6), Sn(2)−Cl(2) 2.454(3), Sn(2)−C(6) 2.306(6), Sn(2)−N(2) 2.267(5);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

---

(12) Chorley, R. W.; Hitchcock, P. B.; Jolly, B. S.; Lappert M. F.; Lawless, G. A. *J. Chem. Soc., Chem. Commun.* **1991**, 1302.

(13) Leung, W.-P.; Lee, H.-K.; Weng, L.-H.; Zhou, Z.-Y.; Mak, T. C. W. *J. Chem. Soc., Dalton Trans.* **1997**, 779.

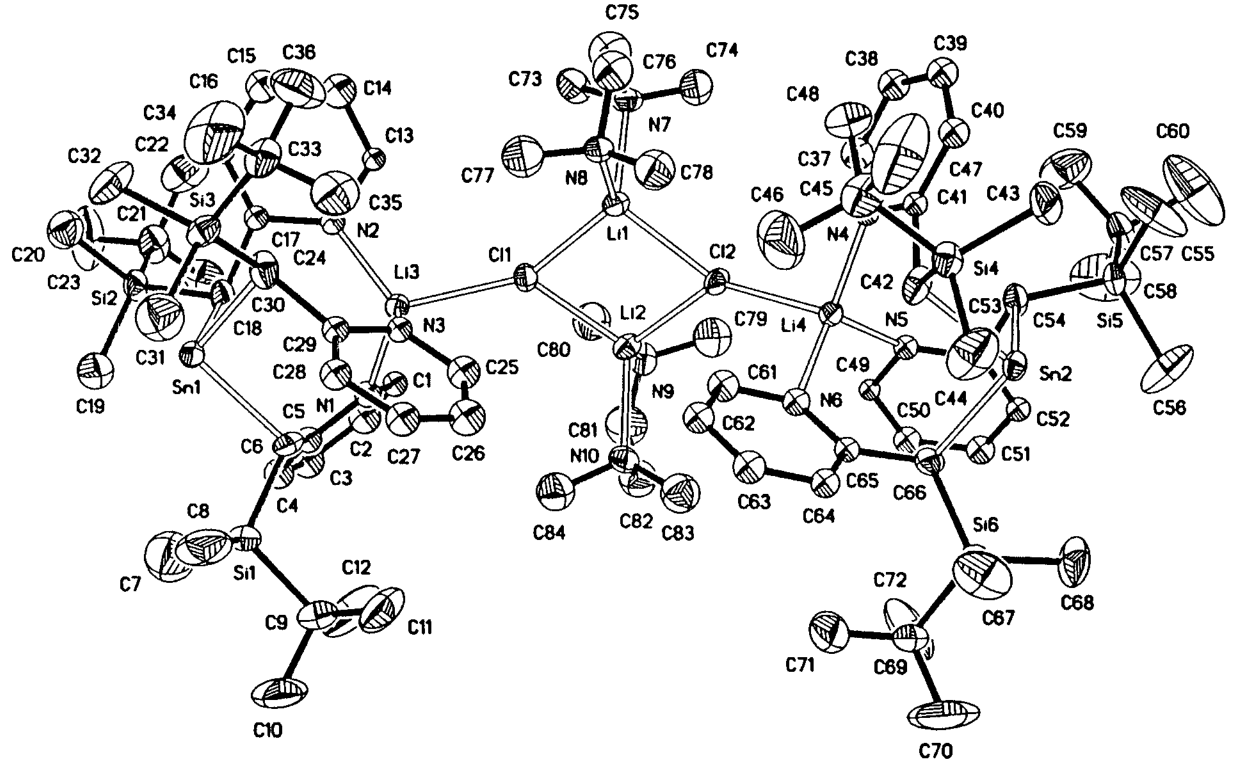


Figure 2. Perspective drawing of **3** with the atomic numbering scheme. Thermal ellipsoids are shown at the 35% probability levels. Selected bond distances [Å] and angles [deg]: Sn(1)–C(18) 2.351(9), Sn(1)–C(30) 2.330(11), Sn(1)–C(6) 2.357(9), Li(3)–N(1) 2.08 (2), Li(3)–N(2) 2.09(2), Li(3)–N(3) 2.00(2), 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 Li[M(C_5H_4N)$_3$] with M(C_5H_4N)$_3$ acting as a tridendate ligand coordinating to lithium have been found.[8] Moreover, sodium tri(pyrazol-1-yl)-germanate [(THF)$_3$Na{(pz)$_3$Ge}] and -stannate [(THF)$_2$-(pzH){(pz)$_3$Sn}]$_2$ (pz = pyrazol-1-yl) with similar structural features have been reported by Stalke and coworkers.[14]

The reaction of $SnCl_2$ with 2 equiv of [LiR^N(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 [LiR^N-(tmeda)]$_2$ afforded the tin(II) dialkyl SnR$^N_2$, which has been characterized by its ^{119}Sn and ^{1}H 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 (SnR$^N_3$)$^-$Li$^+$ ion pairs, with the lithium ion bound to the chlorine atoms of the four-membered chloro-bridged Li_2Cl_2 unit. The [SnR$^N_3$]$^-$ moiety acts as a tridentate ligand coordinated to lithium via the pyridyl nitrogen atoms to form a cagelike structure. The structure of the [LiCl(tmeda)]$_2$ unit resembles that of the 1:1 adducts of lithium(I) halides, [LiX(tmeda)] (X = Cl, Br, I), reported by Raston and co-workers.[15] The geometry at the tin center (sum of bond angles $\sum = 283.2°$) is trigonal pyramidal, with the tin atom located 1.25 Å above the plane of the three α-carbons. The two triangular 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 [R″SnCl] and [SnR″$_2$], respectively.[5] The structure of **2** is similar to that of **3** except that only one SnR$^N_3$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. [Li{CH(SiBu^{t}Me$_2$)C$_5$H$_4$N-2}(tmeda)] was prepared according to the literature.[16] The ^{1}H 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 ^{1}H and ^{119}Sn NMR, respectively.

Preparation of [Sn(Cl)R^N]$_2$ [R^N = CH(SiBu^{t}Me$_2$)C$_5$H$_4$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 [Li{CH(SiBu^{t}Me$_2$)-C$_5$H$_4$N-2}(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

---

[14] Steiner A.; Stalke, D. *J. Chem. Soc., Chem. Commun.* **1993**, 1702.

[15] Raston, C. L.; Skelton, B. W.; Whitaker, C. R.; White, A. H. *Aust. J. Chem.* **1988**, *41*, 1925.

[16] Leung, W.-P.; Weng, L.-H.; Wang, R.-J.; Mak, T. C. W. *Organometallics* **1995**, 4832.

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 $C_{24}H_{40}N_2Cl_2Si_2Sn_2$: C, 39.98; H, 5.59; N, 3.89. Found: C, 39.95; H, 5.64; N, 3.94. ^{1}H NMR (250 MHz, C_6D_6, 25 °C, $SiMe_4$): $\delta = 0.33$ (s, 6H, $SiMe_2$), 0.86 (s, 9H, Bu^t), 2.49 (s, ^2J(H,^{119}Sn); C_5H_4N, 5.75 (t, 1H), 6.78(t, 1H), 7.19(m, 1H), 8.06 (d, 1H). ^{119}Sn NMR (186.5 MHz, C_6D_6, 25 °C, $SnMe_4$): $\delta = 75.9$ (s).

Preparation of $[\{(SnR^N{}_3)Li\}\{LiCl(tmeda)\}_2]$ (2). To a slurry of $SnCl_2$ (0.15 g, 0.79 mmol) in hexane (30 mL) was added dropwise a solution of $[Li\{CH(SiBu^tMe_2)C_5H_4N\text{-}2\}(tmeda)]_2$ (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 $C_{48}H_{92}N_7Cl_2Li_3Si_3Sn$: C, 53.48; H, 8.60; N, 9.10. Found: C, 53.12; H, 8.55; N, 8.63. ^{1}H NMR (250 MHz, C_6D_6, 25 °C, $SiMe_4$): $\delta = 0.27$ (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). ^{119}Sn NMR (186.5 MHz, C_6D_6, 25 °C, $SnMe_4$): $\delta = 209$ (s).

Preparation of $[\{(SnR^N{}_3)Li\}_2\{LiCl(tmeda)\}_2]$ (3). The procedure for the preparation of **3** was similar to **2** except that 3 equiv of $[Li\{CH(SiBu^tMe_2)C_5H_4N\text{-}2\}(tmeda)]_2$ was used (yield = 58%). Anal. Calcd for $C_{84}H_{152}N_{10}Cl_2Li_4Si_6Sn_2$: C, 54.38; H, 8.26; N, 7.39. Found: C, 55.84; H, 8.48; N, 7.75. ^{1}H NMR (250 MHz, C_6D_6, 25 °C, $SiMe_4$): $\delta = 0.24$ (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). ^{119}Sn NMR (186.5 MHz, C_6D_6, 25 °C, $SnMe_4$): $\delta = 201.5$ (s).

Preparation of $SnR^N{}_2$ (4). To a solution of **1** (0.26 g, 0.36 mmol) in 20 mL of ether was added slowly a solution of $[Li\{CH(SiBu^tMe_2)C_5H_4N\text{-}2\}(tmeda)]_2$ (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%). ^{1}H NMR (250 MHz, C_6D_6, 25 °C, $SiMe_4$): 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). ^{119}Sn NMR (186.5 MHz, C_6D_6, 25 °C, $SnMe_4$): $\delta = 165$(s).

X-ray Crystallography. Crystal data (Mo Kα radiation, 295 K): for (**1**), $C_{24}H_{40}Cl_2N_2Si_2Sn_2$, $M = 721.0$, triclinic, space group $P1$, $a = 8.296(3)$ Å, $b = 12.293(5)$ Å, $c = 16.835(9)$ Å, $\alpha = 76.640(0)°$, $\beta = 86.450(0)°$, $\gamma = 71.990(10)°$, $V = 1588.4(15)$ Å^3, $Z = 4$, $F(000) = 720$, $D_c = 1.508$ mg m^{-3}, $\mu = 1.830$ mm^{-1}, $2\theta_{max} = 45$ °, $N = 4641$, $N_o = 3312$, $R = 0.040$, $wR = 0.050$; for (**2**), ($C_{52}H_{98}Cl_2Li_4N_7OSi_3Sn$, $M = 1132.05$, monoclinic, space group $P2_1/c$, $a = 17.157(3)$ Å, $b = 19.911(3)$ Å, $c = 23.724(9)$ Å, $\alpha = 90°$, $\beta = 100.93°$, $\gamma = 90°$, $V = 7957(4)$ Å^3, $Z = 4$, $F(000) = 2408$, $D_c = 0.945$ g cm^{-3}, $\mu = 0.464$ mm^{-1}, $2\theta_{max} = 26.73°$, $N = 11042$, $N_o = 9012$, $R = 0.0590$, $wR = 0.1415$; for (**3**), ($C_{88}H_{162}Cl_2Li_4N_{10}OSi_6Sn_2$, $M = 1880.86$, monoclinic, space group $P2_1/n$, $a = 20.7230(10)$ Å, $b = 25.8150(10)$ Å, $c = 21.9910(10)$ Å, $\alpha = 90°$, $\beta = 93.650(10)°$, $\gamma = 90°$, $V = 11740.5(9)$ Å^3, $Z = 4$, $F(000) = 3992$, $D_c = 1.064$ g cm^{-3}, $\mu = 0.572$ 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 MoKα radiation ($\lambda = 0.71073$ 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-PC[17] 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.

Acknowledgment. This work was supported by the Hong Kong Grants Council Earmarked Grant CUHK 306/94P.

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.

OM9807699

---

(17) Sheldrick G. M. In *Crystallographic Computing 3: Data Collection, Structure Determination, Proteins, and Databases*; Sheldrick, G. M., Kruger, C., Goddard, R., Eds.; Oxford University Press: New York, 1985; p 175.