Bulky Amidinate Complexes of Tin(IV). Synthesis and Structure of Sn(RNC(R′)NR)$_2$Cl$_2$ (R = Cyclohexyl, R′ = H, Me; R = SiMe$_3$, R′ = ^{t}Bu)

Yuanlin Zhou and Darrin S. Richeson*

Department of Chemistry, University of Ottawa, Ottawa, Ontario, Canada K1N 6N5

Received August 1, 1996⊗

Reaction of 2 equiv of lithium dicyclohexylformamidinate with SnCl$_4$ afforded the new species Sn(CyNC(H)NCy)$_2$Cl$_2$ (Cy = cyclohexyl) (**1**). Addition of MeLi and ^{t}BuLi to CyN═C═NCy and Me$_3$SiN═C═CSiMe$_3$, respectively, provided an entry to the high-yield syntheses of Sn(CyNC(Me)NCy)$_2$Cl$_2$ (**2**) and Sn(Me$_3$SiNC(CMe$_3$)NSiMe$_3$)$_2$Cl$_2$ (**3**). Spectroscopic and elemental analysis confirmed the formulas of these new species. Compounds **1** and **3** were further characterized by X-ray crystallography and shown to possess distorted octahedral coordination geometries of the metal center with the cis-dichloride groups. The structural determination of **3** represents the first structural confirmation of the new bulky amidinate ligand (Me$_3$SiNC(CMe$_3$)NSiMe$_3$)$^-$. Comparison of the solution structures of **1**−**3** was provided by NMR spectroscopy, which showed that while **1** was fluxional at room temperature, **2** and **3** exhibited restricted fluxionality. Differences in the solid state structures of **1** and **3** were attributed to the steric interactions within the ligands. Crystal data for **1**: orthorhombic, *Pnca, a* = 17.553(5) Å, *b* = 20.275(9) Å, *c* = 9.757(6) Å; empirical formula SnCl$_2$N$_4$C$_{26}$H$_{46}$(toluene), R_F 0.044, R_w 0.051. Crystal data for **3**: monoclinic *P*21/*n*, *a* = 13.515(8) Å, *b* = 16.179(8) Å, *c* = 15.974(4) Å, *β* = 97.69(6)°, empirical formula SnCl$_2$Si$_4$N$_4$C$_{22}$H$_{54}$, R_F 0.032, R_w 0.045.

Introduction

Motivated by an interest in the effects of ligand geometry on the coordination environments of posttransition elements in general and of tin specifically, we began a systematic study of the use of amidinates, formamidinates, and triazenates as supporting ligands for Sn(II/IV) complexes. This family of bidentate, three-atom bridging ligands with the general formula RNXNR$^-$ (X = CR′, CH, N) should present an ideal system for exploring the effects of changing the steric bulk and electronic properties of the supporting ligand on the product compounds. Through modification of the organic substituents on the nitrogen atoms and at the bridge position there is the potential for making rational modifications that can be tuned to stabilize desired structural and reactivity characteristics. For example, formamidinate ligands are known to favor unusual structural features in both transition metal and main group metal chemistry.[1−3]

Amidinato complexes of tin have been prepared with the metal in both the divalent and tetravalent oxidation states. However, these results have been, by and large, limited to use of *N,N′*-bis(trimethylsilyl)benzamidinate-based ligands, [Me$_3$SiNC(C$_6$H$_5$R)NSiMe$_3$].[4−11] Our initial attraction to the amidinate system was motivated by the possibility of overcoming this limitation and investigating the effects of changing the various substituents. We have relied on the generation of amidinate anions by the addition of alkyl anion equivalents to carbodiimides. For example, the employment of dicyclohexylformamidinate allowed the synthesis of a family of unusual binuclear indium complexes that are unique among the elements of group 13.[2] In addition, we have described the preparation of new bulky amidinate ligands and of several new complexes of these species with In(III).[12] Remarkable structural changes were observed in a unique family of tin−sulfido complexes in which changing the substituent on the bridging carbon atom of the amidinate allowed the isolation of both terminal and *μ*-sulfido species.[13]

Herein we report the synthesis and characterization of a novel family of monomeric six-coordinate Sn^{IV} compounds of the general formula Sn(RNC(R′)NR)$_2$Cl$_2$ (R = cyclohexyl, SiMe$_3$; R′ = H, Me, ^{t}Bu). We include the first structural characterization of a complex with the new amidinate (Me$_3$SiNC(^{t}Bu)NSiMe$_3$)$^-$ as a ligand. The crystallographic and spectroscopic characteristics of these complexes reveal some of the effects of varying the substituents in the amidinate ligands.

Results and Discussion

Complexes of the (CyNC(H)NCy) Anion. Reaction of the lithium salt of dicyclohexylformamidine[14] with SnCl$_4$ in a 2:1 stoichiometry afforded, after crystallization, the new species 1 (Scheme 1) in 74% yield. The ^{1}H and ^{13}C NMR spectra for 1 are consistent with a single environment for the formamidinate ligand as indicated by the single multiplet (3.07 ppm) for the cyclohexyl protons α to nitrogen and one resonance for the

---

⊗ Abstract published in *Advance ACS Abstracts*, January 1, 1997.

(1) For a review, see: Cotton, F. A.; Walton, R. A. *Multiple Bonds Between Metal Atoms*, 2nd ed.; J. Wiley, & Sons: New York, 1982.
(2) Zhou,Y.; Richeson, D. S. *Inorg.Chem.* **1996**, *35*, 1423.
(3) Berno, P.; Hao, S.; Minhas, R.; Gambarotta, S. *J. Am. Chem. Soc.* **1994**, *116*, 7417 and references therein.
(4) Dehnicke, K.; Ergezinger, C.; Hartmann, E.; Zinn, A.; Hösler, K. *J. Organomet. Chem.* **1988**, *352*, C1.
(5) Roesky, H. W.; Meller, B.; Noltemeyer, M.; Schmidt, H.-G.; Scholz, U.; Sheldrick, G. M. *Chem. Ber.* **1988**, *121*, 1403.
(6) Ergezinger, C.; Weller, F.; Dehnicke, K. *Z. Naturforsch.* **1988**, *43B*, 1621.
(7) Kidea, J. D.; Hiller, W.; Borgsen, B.; Dehnicke, K. *Z. Naturforsch.* **1989**, *44B*, 889.
(8) Appel, S.; Weller, F.; Dehnicke, K. *Z. Anorg. Allg. Chem.* **1990**, *583*, 7.
(9) Borgsen, B.; Dehnicke, K.; Fenske, D.; Baum, G. *Z. Anorg. Allg. Chem.* **1991**, *596*, 133.
(10) Kilimann, U.; Noltemeyer, M.; Edelmann, F. T. *J. Organomet. Chem.* **1993**, *443*, 35.
(11) Edelmann, F. T. *Coord. Chem. Rev.* **1994**, *137*, 403.
(12) Zhou, Y.; Richeson, D. S. *Inorg.Chem.* **1996**, *35*, 2448.
(13) Zhou, Y.; Richeson, D. S. *J. Am. Chem. Soc.*, in press.
(14) The lithium salt, LiCyNCHNCy (Cy = cyclohexyl), was prepared by the stoichiometric reaction of CyNHCHNCy with MeLi in Et$_2$O. CyNHCHNCy was prepared according to the published procedure: Saeed, A. H.; Selman, A. S. *J. Spectrosc.* **1982**, *27*, 123.

Scheme 1

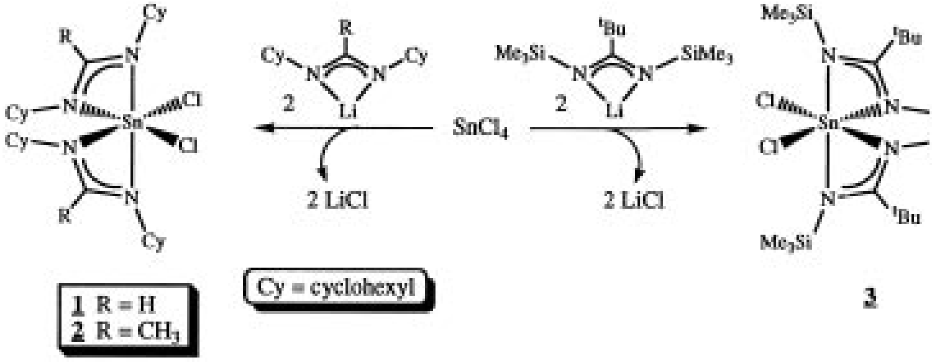


Table 1. Selected Atomic Bond Distances (Å)for **1**

Sn1−Cl1	2.388(3)	C3−C4	1.50(2)
Sn1−N1	2.160(7)	C4−C5	1.49(2)
Sn1−N2	2.202(7)	C5−C6	1.52(2)
N1−C1	1.45(1)	C7−C8	1.52(2)
N1−C13	1.32(1)	C7−C12	1.50(2)
N2−C7	1.46(1)	C8−C9	1.51(2)
N2−C13a	1.32(1)	C9−C10	1.49(2)
C1−C2	1.53(2)	C10−C11	1.53(2)
C1−C6	1.51(1)	C11−C12	1.53(2)
C2−C3	1.52(2)		

bridging carbon (156.8 ppm). The signal for the proton in the bridge position (NC*H*N) could not be located in the ^{1}H NMR spectrum and is perhaps obscured by accidental overlap with the residual protons of the NMR solvent. Elemental analysis confirmed the formula of this species to be $[Sn(CyNC(H)NCy)_2Cl_2]_n$ (Cy = cyclohexyl).

On the basis of the ready solubility of **1** in organic solvents, the ionic radius of Sn(IV) comparable to the transition metals, our previous isolation of the dimeric species $[In(CyNC(H)NCy)_2Cl]_2$, and the equivalence of the Cy groups in the NMR we anticipated that this complex may be dinuclear. In order to confirm the level of aggregation for this species, the structure of **1** was determined by single-crystal X-ray diffraction analysis, and the result is shown in Figure 1.

Complex **1** crystallized in the orthorhombic space group *Pnca* ($a = 17.553(5)$, $b = 20.275(9)$, $c = 9.757(6)$ Å) with four molecules of **1** and four molecules of solvent (toluene) in the unit cell. There were no anomalously short intermolecular contacts. The molecular geometry and atom-numbering scheme are shown in Figure 1.

In contrast to our previous report on the binuclear complex $[In(CyNC(H)NCy)_2Cl]_2$,[2] in which the indium centers were bridged by four formamidinate ligands, the structural analysis of **1** revealed a monomeric species with the metal center in a distorted octahedral environment. The coordination sphere of the Sn(IV) center included the four nitrogen atoms of the two chelating amidinate anions and was completed by two cis-chloride ligands. The Sn atom lies on a strict twofold axis which bisects the Cl−Sn−Cla angle resulting in a C_2 symmetric molecular geometry. In contrast to the solid state structural observation of two distinct Cy groups, the Cy groups appear to be equivalent in the ^{1}H and ^{13}C NMR spectra, which indicates that complex **1** is fluxional in solution. Tables 1 and 2 present a summary of selected bond distances and angles for **1**.

The two nitrogens and bridging carbon atoms for the amidinate ligand lie in a plane that includes the Sn atom (sum of the internal angles equals 360°). The termini of the amidinate ligand can be divided into two types, the cis-NCy groups (N2, N2a) and trans-NCy groups (N1, N1a). Both Sn−N bond lengths are slightly longer than the reported values for the five-coordinate benzamidinate complex $[Me_3SiNC(C_6H_5)NSiMe_3]SnCl_3$ (2.135, 2.152 Å).[6] In addition, the cis-SnN bond lengths (Sn−N2 2.202 Å) are slightly longer than the trans-SnN distances (Sn−N1 2.160 Å). This feature is likely due to the relative steric congestion experienced by the two groups. The Sn−Cl bond lengths of 2.388 Å correlate favorably with the reported benzamidinate complex (2.32−2.35 Å).[6]

Table 2. Selected Atomic Bond Angles (deg) for **1**

Cl1−Sn1−Cl1a	94.40(1)	N1−C1−C6	112.7(8)
Cl1−Sn1−N1	98.69(22)	C2−C1−C6	110.2(8)
Cl1−Sn1−N1a	98.59(22)	C1−C2−C3	110.6(9)
Cl1−Sn1−N2	91.35(21)	C2−C3−C4	111.5(9)
Cl1−Sn1−N2a	159.91(21)	C3−C4−C5	111.6(9)
N1−Sn1−N1a	154.5(3)	C4−C5−C6	111.2(10)
N1−Sn1−N2	99.5(3)	C1−C6−C5	112.1(9)
N1−Sn1−N2a	61.4(3)	N2−C7−C8	110.5(8)
N1a−Sn1−N2	61.4(3)	N2−C7−C12	110.9(8)
N2−Sn1−N2a	89.8(3)	C8−C7−C12	112.5(9)
Sn1−N1−C1	142.7(6)	C7−C8−C9	112.4(9)
Sn1−N1−C13	92.6(6)	C8−C9−C10	111.4(10)
C1−N1−C13	124.2(8)	C9−C10−C11	112.6(10)
Sn1−N2−C7	145.8(6)	C10−C11−C12	112.6(10)
Sn1−N2−C13a	90.9(5)	C7−C12−C11	112.0(10)
C7−N2−C13a	123.3(8)	N1−C13−N2a	115.1(8)
N1−C1−C2	111.1(8)		

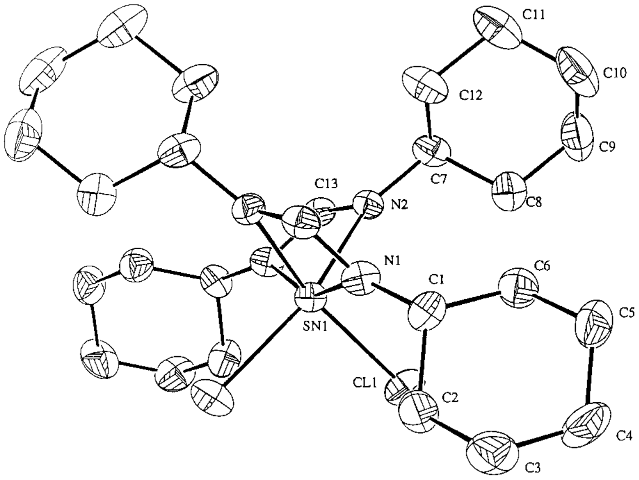


Figure 1. Molecular structure and atom numbering scheme for $Sn[CyNC(H)NCy]_2Cl_2$ (**1**) (Cy = cyclohexyl). Hydrogen atoms have been omitted for clarity.

Complexes of the (RNC(R′)NR) Anions (R = Cy, R′ = CH_3; R = $SiMe_3$, R′ = CMe_3). In an effort to extend this family of complexes as well as to enlarge the range of available amidinate ligands we have been investigating the introduction of variety of sterically demanding groups in the bridge position. Our synthetic methodology is based on the reaction of organolithium reagents with carbodiimide to generate the appropriate

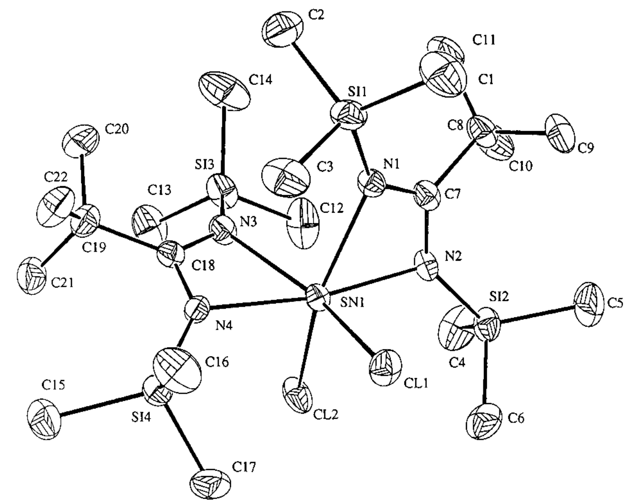


Figure 2. Molecular structure and atom numbering scheme for $Sn[Me_3SiNC(^tBu)NSiMe_3]_2Cl_2$ (**3**) (Cy = cyclohexyl). Hydrogen atoms have been omitted for clarity.

amidinate species. Complex preparation is achieved by addition of a stoichiometric amount of metal halide (e.g., $SnCl_4$) to this solution.

The reaction of MeLi with dicylohexylcarbodiimide proceeds smoothly in ether at room temperature. Subsequent addition of 0.5 equiv of $SnCl_4$ followed by recrystallization resulted in isolation of an 84% yield of $Sn(CyNC(Me)NCy)_2Cl_2$ (**2**) (Scheme 1). Spectroscopic characterization and microanalysis confirmed the formula of this complex.

Like complex **1**, the ^1H NMR gave only one multiplet for the cyclohexyl α-H at 3.03 ppm. However, the greater chemical shift dispersion in the ^{13}C NMR spectrum of complex **2** clearly indicated that there were, in fact, two different environments for the cyclohexyl groups in the molecule. In particular, two signals of equal intensity were observed for the α cyclohexyl carbons at 57.5 and 56.5 ppm. Although the mechanism for interconversion of the Cy groups in complex **1** is not yet clear, it appears that substitution of the methyne hydrogen in **1** with a methyl group substantially slows this process.

We have found that other amidinate ligands are also accessible through a similar reaction scheme. Thus, reaction of ^tBuLi with 1,3-bis(trimethylsilyl)carbodiimide in ether generated $Li[Me_3SiN(CMe_3)NSiMe_3]$. Further reaction of this species with $SnCl_4$ in a 2:1 ratio yielded the corresponding bis(amidinato)dichlorostannnate complex $[Me_3SiNC(CMe_3)NSiMe_3]_2SnCl_2$ (**3**) (Scheme 1). The formulation and proposed structure for **3** were based on spectroscopic evidence, microanalysis, and by analogy with **1** and **2**. In complex **3**, the $NSiMe_3$ groups were observed as a broad singlet at room temperature in both the ^1H NMR and ^{13}C NMR spectra. At −50 °C, the inequivalence of the trimethylsilyl groups was demonstrated by the appearance of two sharp resonances in the ^1H NMR for these substituents. A single-crystal X-ray diffraction analysis of **3** was undertaken in order to establish the monomeric nature, coordination geometry of the metal center, and connectivity of the ligand for this compound.

Complex **3** crystallized in the monoclinic space group $P2_1/n$ ($a = 13.515(8)$, $b = 16.179(8)$, $c = 15.974(4)$ Å; $\beta = 97.67(6)$) with four molecules in the unit cell. There were no anomalously short intermolecular contacts. The molecular geometry and atom-numbering scheme are shown in Figure 2.

The structural analysis revealed a molecular core with similarities to that of **1**: the metal center was in a distorted octahedral environment consisting of the four nitrogen atoms of the two amidinate anions with cis-chloride ligands completing the coordination sphere. Tables 3 and 4 present a summary of selected bond distances and angles for **3**. The bonding parameters within the two amidinate ligands are not dramatically different, and the two nitrogens and bridging carbon atoms for each of the individual amidinate ligands lie in a plane that includes the Sn atom (sum of the internal angles ~360°), resulting in a molecular geometry that is of approximately C_2 symmetry. Again, the termini of the amidinate ligand can be divided roughly into two types, the cis-$NSiMe_3$ groups (N1, N3) with an N1−Sn−N3 angle of 86.5° and trans-$NSiMe_3$ groups (N2, N4) with an N2−Sn−N4 angle of 170°. As with **1**, the two cis-SnN bond lengths are slightly longer (2.212(3), 2.230(3) Å) than the trans-SnN values (2.193(3), 2.202(3) Å) and all four bond lengths are longer than for the five-coordinate benzamidinate complex $[Me_3SiNC(C_6H_6)NSiMe_3]SnCl_3$.[6]

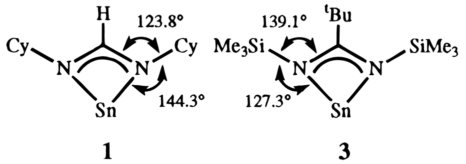
Table 3. Selected Atomic Bond Distances (Å) for **3**

Sn1−Cl1	2.3979(9)	Si2−N2	1.733(3)
Sn1−Cl1	2.4062(9)	Si3−N3	1.752(3)
Sn1−N1	2.212(3)	Si4−N4	1.785(3)
Sn1−N2	2.193(3)	N1−C7	1.324(5)
Sn1−N3	2.230(3)	N2−C7	1.343(4)
Sn1−N4	2.202(3)	N3−C18	1.342(5)
Si1−N1	1.763(3)	N4−C18	1.335(4)

Table 4. Selected Atomic Bond Angles (deg) for **3**

Cl1−Sn1−Cl2	102.46(4)	N3−Sn1−N4	61.29(11)
Cl1−Sn1−N1	92.06(7)	Sn1−N1−Si1	127.60(15)
Cl1−Sn1−N2	91.92(8)	Sn1−N1−C7	91.87(21)
Cl1−Sn1−N3	152.79(8)	Si1−N1−C7	139.1(3)
Cl1−Sn1−N4	94.24(8)	Sn1−N2−Si2	126.95(15)
Cl2−Sn1−N1	151.09(8)	Sn1−N2−C7	92.18(21)
Cl2−Sn1−N2	93.12(8)	Si2−N2−C7	139.0(3)
Cl2−Sn1−N3	91.36(8)	Sn1−N3−Si3	128.80(17)
Cl2−Sn1−N4	93.22(8)	Sn1−N3−C18	90.88(21)
N1−Sn1−N2	61.16(10)	Si3−N3−C18	138.2(3)
N1−Sn1−N3	86.48(11)	Sn1−N4−Si4	126.57(14)
N1−Sn1−N4	110.68(10)	Sn1−N4−C18	92.30(21)
N2−Sn1−N3	110.84(11)	Si4−N4−C18	139.66(25)
N2−Sn1−N4	170.01(10)		

Chart 1



1 **3**

While the gross structural features of **1** and **3** correlate well, closer inspection reveals some interesting differences between the two complexes. As might be anticipated, based on the fact that the $[Me_3SiNC(CMe_3)NSiMe_3]$ anion is likely sterically more demanding than $[CyNC(H)NCy]^-$, the average Sn−N and Sn−Cl bond lengths are slightly longer in **3** than in **1**. In addition, the angles around the N atoms show variations that correspond with the steric interactions within the ligand (Chart 1). In particular, the increase in the C_{bridge}−N−R (R = Cy, $SiMe_3$) angle and concomitant decrease in the Sn−N−R (R = Cy, $SiMe_3$) angle for **3** relative to **1** is presumably due principally to steric interactions of the $SiMe_3$ and ^tBu substituents versus the corresponding H and Cy groups. It may be these features which contribute to the observed decrease in fluxionality for **2** and **3** relative to **1**.

Conclusion

Amidinate ligands have proven to be useful in the preparation of a novel family of six-coordinate Sn complexes. A combination of X-ray crystallographic and spectroscopic studies confirm the C_2 symmetric structures of these compounds. The effects of substitution are manifest in the solution dynamics of these compounds, as exhibited through NMR spectroscopy, and in a comparison of the static structures of **1** and **3**. In addition, this report has extended the available amidinate ligands by demonstrating the synthesis and reactivity of a new bulky anionic ligand, $[Me_3SiN(CMe_3)NSiMe_3]^-$. Our continuing investigations are oriented at dissecting the steric and electronic features that influence the formation and reactivity of these and other main group and transition metal amidinate compounds.

Experimental Section

General Procedure. All reactions were carried out in either a nitrogen-filled drybox or under nitrogen using standard Schlenk-line techniques. Diethyl ether, hexane, and toluene were distilled under nitrogen from potassium. Deuterated benzene was dried by vacuum transfer from potassium. MeLi (1.4 M in diethyl ether), ^{t}BuLi (1.7 M in hexane), $SnCl_4$, dicyclohexylcarbodiimide and 1,3-bis(trimethylsilyl)carbodiimide were purchased from Aldrich and used without further purification. CyNHCHNCy and LiCyNCHNCy (Cy = cyclohexyl) were prepared according to the literature procedure.[14]

$Cl_2Sn[C_6H_{11}NCHNC_6H_{11}]_2$ (1). A Schlenk flask was charged with lithium *N,N′*-dicyclohexylformamidinate (1.58 g, 7.38 mmol), toluene (30 mL), and a stir bar. To this solution was added $SnCl_4$ (0.96 g, 3.69 mmol) dropwise. The reaction mixture was stirred overnight and then filtered. The solution was concentrated to ~10 mL and cooled to −30 °C in a freezer. The resulting white crystalline product was collected by filtration and dried in vacuo (1.90 g, 74%, 2.73 mmol), mp (sealed) 127−130 °C. Spectroscopic data: IR (Nujol, cm^{-1}) 1554 (s). ^1H NMR (C_6D_6, ppm) 3.07 (br, C_6H_{11}, 4H); 1.96−0.90 (m, C_6H_{11}, 40H). ^{13}C NMR (C_6D_6, ppm) 156.8 (s, N*C*HN); 57.5, 35.6, 25.9, 25.8 (4s, C_6H_{11}). Anal. Calcd for $C_{26}H_{46}Cl_2N_4Sn{\cdot}C_7H_8$: C 56.91; H 7.82; N 8.04. Found: C 56.52; H 7.90; N 8.10.

$Cl_2Sn[C_6H_{11}NC(Me)NC_6H_{11}]_2$ (2). A Schlenk flask was charged with dicyclohexylcarbodiimide (1.23 g, 5.96 mmol), diethyl ether (30 mL), and a stir bar. To this solution was added MeLi (4.3 mL, 1.4 M, 5.96 mmol), and the mixture was allowed to stir for an additional 30 min. Dropwise addition of $SnCl_4$ (0.78 g, 2.98 mmol) lead to precipitation of a white solid. The reaction mixture was stirred overnight and then filtered. The solvent was removed under oil pump vacuum, and the residue was dissolved in toluene (6 mL). Hexane (6 mL) was added to the toluene solution, and the mixture was cooled to −30 °C in a freezer. The resulting white crystalline product was collected by filtration and dried in vacuo (1.64 g, 84%, 2.50 mmol), mp (sealed) 231−234 °C. Spectroscopic data: IR (Nujol, cm^{-1}) 1524 (s). ^1H NMR (C_6D_6, ppm) 3.03 (br, C_6H_{11}, 4H); 2.42−1.18 (m, C_6H_{11}, 40H), 1.34 (s, Me). ^{13}C NMR (C_6D_6, ppm) 164.0 (s, N*C*MeN); 57.5, 56.5, 35.5, 34.8, 33.2, 26.7, 26.6, 26.1, 26.0, 25.6, 25.4 (11s, C_6H_{11}), 11.1 (Me). Anal. Calcd for $C_{28}H_{50}Cl_2N_4Sn{\cdot}{}^1/_4C_7H_8$: C 54.39; H 8.29; N 8.63. Found: C 54.52; H 8.00; N 8.55.

$Cl_2Sn[Me_3SiNC(CMe_3)NSiMe_3]_2$ (3). A Schlenk flask was charged with 1,3-bis(trimethylsilyl)carbodiimide (0.73 g, 3.92 mmol), diethyl ether (20 mL), and a stir bar. To this solution was added ^{t}BuLi (2.3 mL, 1.7 M, 3.92 mmol), and the mixture was allowed to stir for an additional 30 min. Dropwise addition of $SnCl_4$ (0.51 g, 1.96 mmol) lead to precipitation of a white solid. The reaction mixture was stirred overnight and then filtered. The solvent was removed under oil pump vacuum, the residue was dissolved in hexane (6 mL), and the solution was cooled to −30 °C in a freezer for three days. The resulting white crystals were collected by filtration and dried in vacuo (1.02 g, 77%, 1.51 mmol), mp (sealed) 114−116 °C. Spectroscopic data: ^1H NMR ($CDCl_3$, ppm) (at 25 °C) 1.28 (s, CMe_3, 18H); 0.34 (br, $SiMe_3$, 36H); (at −50 °C) 1.32 (s, CMe_3, 18H); 0.46, 0.30 (2s, $SiMe_3$, 36H). ^{13}C NMR (C_6D_6, ppm, 25 °C) 185.2 (s, N*C*HN); 39.6 ($C(CH_3)_3$), 30.7 (s, *C*Me_3), 6.1 (br, $SiMe_3$). Anal. Calcd for $C_{22}H_{54}Cl_2N_4Si_4Sn$: C 39.05; H 8.04; N 8.28. Found: C 39.01; H 8.19; N 8.20.

X-ray Crystallography. Data were collected on a Rigaku AFC6S diffractometer with graphite-monochromated Cu Kα radiation at −153 °C using the $\omega{-}2\theta$ scan technique to a maximum 2θ value of 50° for crystals mounted on glass fibers. Cell constants and orientation matrices were obtained from the least-squares refinement of 24 carefully centered high-angle reflections. Redundant reflections were removed from the data set. The intensities of three representative reflections were measured after every 150 reflections to monitor the crystal and instrument stability. In the case of **1**, absorption corrections (ψ scan) were made. For **3**, the data were corrected for Lorentz and polarization effects but not for absorption. Both structures was solved by direct methods. The non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were located in the difference Fourier maps and refined isotropically in the case of favorable observation/parameter ratio. The final cycle of full-matrix least-squares refinement was based on the number of observed reflections with $[I > 2.5\sigma(I)]$. Anomalous dispersion effects were included in the F_{calc}. All calculations were performed using the NRCVAX package. Details of the data collection, refinement and final atomic coordinates are reported in the Supporting Information.

Acknowledgment. This work was supported by the Natural Sciences and Engineering Research Council of Canada.

Supporting Information Available: Text providing a description of the structural solutions, ORTEP drawings, tables of atomic positions, thermal parameters, crystallographic data, and bond distances and angles for **1** and **3** (27 pages). Ordering information is given on any current masthead page.

IC960940D