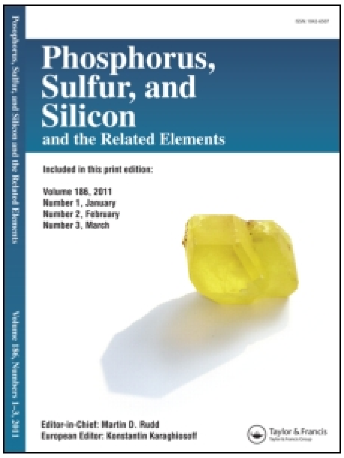


Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

A GENERAL ROUTE TO TIN-NITROGEN HETEROCUBANES

Jack F. Eichler[a]; Oliver Just[a]; William S. Rees[a]
[a] Georgia Institute of Technology, Atlanta, Georgia, USA

To cite this Article Eichler, Jack F. , Just, Oliver and Rees, William S.(2004) 'A GENERAL ROUTE TO TIN-NITROGEN HETEROCUBANES', Phosphorus, Sulfur, and Silicon and the Related Elements, 179: 4, 715 — 726
To link to this Article: DOI: 10.1080/10426500490426656
URL: http://dx.doi.org/10.1080/10426500490426656

Phosphorus, Sulfur, and Silicon, 179:715–726, 2004
Copyright © Taylor & Francis Inc.
ISSN: 1042-6507 print / 1563-5325 online
DOI: 10.1080/10426500490426656

A GENERAL ROUTE TO TIN-NITROGEN HETEROCUBANES

Jack F. Eichler, Oliver Just, and William S. Rees, Jr.
Georgia Institute of Technology, Atlanta, Georgia, USA

(Received August 18, 2003; accepted October 3, 2003)

*The reaction of the lithiated bis-stannylamine, $((CH_3)_3Sn)_2NLi\cdot THF$ (**1**), with $(C_5H_5)_2ZrCl_2$ yields the imido-bridged zirconium dimer, $[(C_5H_5)_2ZrN(Sn(CH_3)_3)]_2$ (**3**), which was characterized by single crystal x-ray diffractometry. The heteroleptic stannylamine, $[\{(CH_3)_3Sn\}\{(CH_3)_3Si\}]NLi\cdot Et_2O$ (**2**), prepared using (**1**) as a synthetic precursor, reacts with $SnCl_2$ to yield the tin-nitrogen heterocubane, $[Sn(\mu_3\text{-}NSi(CH_3)_3)]_4$ (**4**), which is fully characterized by single crystal x-ray diffractometry and standard spectroscopic techniques. The use of this class of stannylamine ligands allows access to heterometallic amido complexes and provides a general route to obtaining tin-nitrogen heterocubanes.*

The heavier congener of the ubiquitous $-SiMe_3$ unit, $-SnMe_3$, has enjoyed considerably less success as a substituent in metal amide compounds. Some examples of complexes that contain this tin(IV) moiety include the copper tetramer, $[Cu(\mu_2\text{-}N(SnMe_3)_2)]_4$,[1] the titanium-nitrogen cubane, $[CpTi(\mu_3\text{-}NSnMe_3)]_4$,[2] and the dimeric complexes of the group III metals, $[Me_2M\text{-}N(R)SnMe_3]_2$ (M = Al, Ga, In; R = ^{n}Pr, ^{i}Pr, ^{i}Bu).[3] Use of a *N*-lithiostannylamine as a starting material in the synthesis of metal amide complexes appears to be restricted to the aforementioned copper tetramer.[1] Hence, it is of interest to explore the coordination chemistry of these lithiated stannylamines. This report discusses the synthesis and characterization of the lithium amides, $(Me_3Sn)_2NLi\cdot THF$ (**1**) and $[(Me_3Sn)(Me_3Si)]NLi\cdot Et_2O$ (**2**), the imido-bridged zirconium dimer, $[(C_5H_5)_2Zr(\mu_2\text{-}NSnMe_3)]_2$ (**3**), and the

---

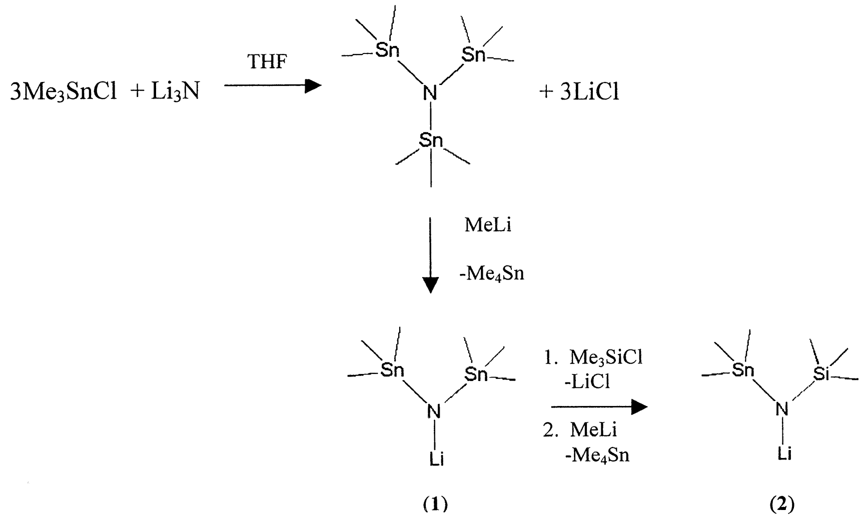
We gratefully acknowledge partial support of this project by the Georgia Institute of Technology Molecular Design Institute, under prime contract N00014-95-1-1116 from the Office of Naval Research.

Address correspondence to William S. Rees, Jr., Georgia Institute of Technology, Department of Chemistry, Atlanta, GA 30332-0400. E-mail: will.rees@chemistry.gatech.edu

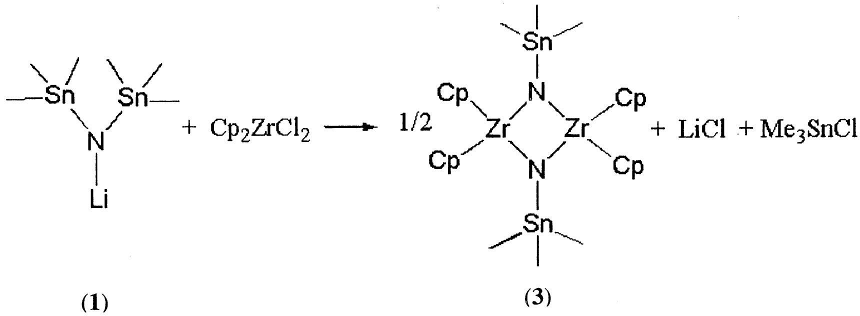
tin-nitrogen cubane previously reported by Veith et al.,[4] [Sn(μ_3-NSiMe$_3$)]$_4$ (**4**).

RESULTS

The synthesis of compound (**1**), prepared according to previous literature procedures,[1] is depicted in Scheme 1. The lithiated amine was recrystallized from THF and isolated as a colorless solid. The THF adduct of (**1**) was characterized by multinuclear NMR spectroscopy [^{1}H NMR: δ 0.31 (s, 18H, CH$_3$), 1.31 (m, 4H, CH$_2$), 3.62 (m, 4H, CH$_2$); ^{13}C NMR: δ 1.71 (CH$_3$), 25.22 (CH$_2$), 68.61 (CH$_2$); ^{119}Sn NMR: δ 63.3 (s); ^{7}Li NMR: δ 1.69 (s)] and elemental analyses [C$_{10}$H$_{26}$NOLiSn: C, 28.55; H, 6.23%. Found: C, 27.86; H, 6.24%]. Scheme 1 also illustrates the preparation of compound (**2**), which was synthesized via the reaction of (**1**) with Me$_3$SiCl, followed by the in situ reaction with MeLi. Compound (**2**) was isolated as a colorless solid after recrystallization from diethyl ether. The ether adduct of (**2**) was also characterized by multinuclear NMR spectroscopy [^{1}H NMR: δ 0.282 (s, 9H, CH$_3$Sn), 0.304 (s, 9H, CH$_3$Si), 0.985 (t, 6H, CH$_3$), 3.460 (q, 4H, CH$_3$); ^{13}C NMR: δ −1.29 (CH$_3$Sn), 5.89 (CH$_3$Si), 14.40 (CH$_3$), 63.83 (CH$_2$); ^{29}Si NMR: δ −12.68; ^{119}Sn NMR: δ 34.24; ^{7}Li NMR: δ 1.38] and elemental analyses [C$_{10}$H$_{28}$NOLiSiSn: C, 36.17; H, 8.50; N, 4.22%. Found: C, 35.92; H, 8.38; N, 4.28%].



SCHEME 1



SCHEME 2

The synthesis of (**3**) is shown in Scheme 2. The reaction of (**1**) with one equivalent of $(C_5H_5)_2ZrCl_2$ in THF resulted in the formation of (**3**), a zirconium dimer possessing bridging trimethylstannaimine units and terminal cyclopentyldienyl ligands. Compound (**3**) was recrystallized from diethyl ether as yellow prisms and characterized by single crystal x-ray diffractometry. The crystal structure of (**3**) is depicted in Figure 1.

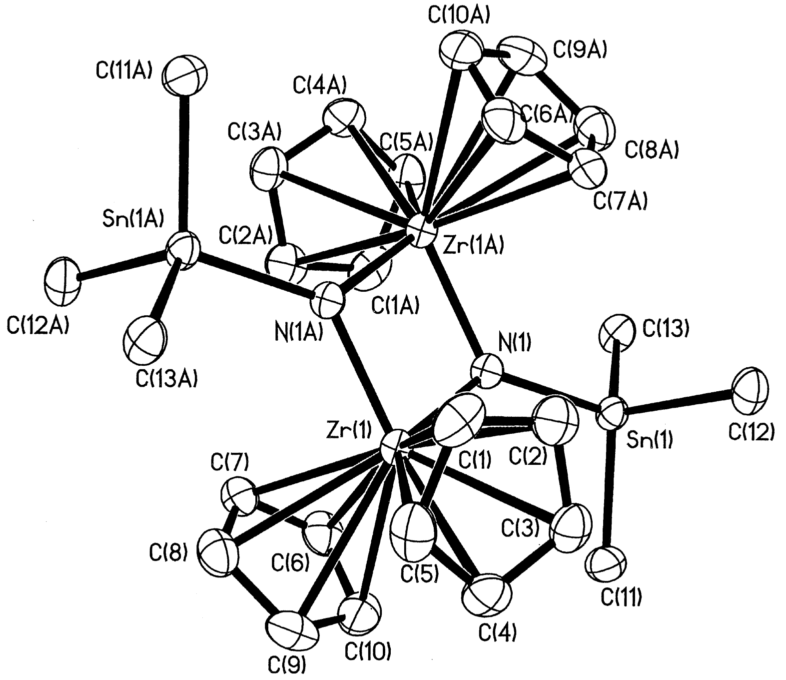
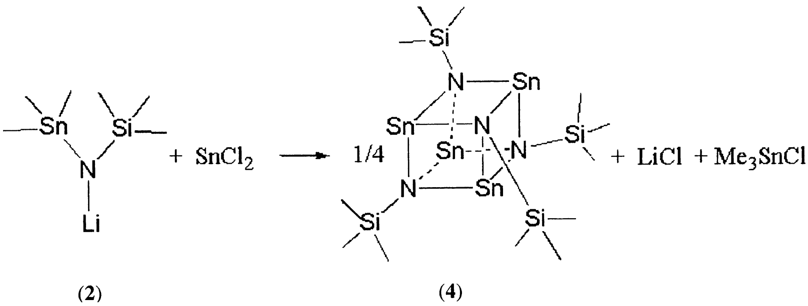


FIGURE 1 ORTEP representation (30% probability) of the structure of $[Cp_2Zr(\mu_2\text{-}NSnMe_3)]_2$ (**3**). Hydrogen atoms are omitted for clarity.



SCHEME 3

The preparation of (**4**) is illustrated in Scheme 3. The reaction of (**2**) with one equivalent of $SnCl_2$ in diethyl ether yielded the tin-nitrogen heterocubane (**4**). Recrystallization from hexane/toluene resulted in an orange, crystalline solid. Compound (**4**) was characterized by multinuclear NMR spectroscopy [^1H NMR: δ 0.26 (s, CH_3); ^{13}C NMR: δ −0.06 (CH_3); ^{119}Sn NMR: δ 782 (s)], UV/VIS spectroscopy [$\lambda_{max} = 330$ nm], elemental analyses [$C_{12}H_{36}N_4Si_4Sn_4$: C, 17.49; H, 4.41%. Found: C, 17.51; H, 4.42%], and single crystal x-ray diffractometry. The relevant crystallographic data are summarized in Tables I–V. The crystal structure of (**4**) is shown in Figure 2.

DISCUSSION

Even though the synthesis and structural characterization of lithiated stannylamines has been reported, their use for the preparation of metal amide complexes has apparently not been extensively investigated. Wrackmeyer and co-workers reported the structural characterization of *N*-lithiobis(trimethylstannyl)amine and *N*-lithiotrimethylsilyl(trimethylstannyl)amine, the structural analogues of **1** and **2**, as both the ^tBuOMe or *N,N′,N″*-pentamethyldiethylenetriamine (pmdta) adducts, which crystallize as a dimer and monomer, respectively. In addition to using different coordinating solvent, Wrackmeyer also reports a slightly modified synthetic procedure compared to that seen in Scheme 1.[5] Though there is no direct evidence of the structural nature of **1** or **2**, it is likely that they exist as dimers, with bridging amine and terminal solvent units, given the solid state structure of the ^tBuOMe adducts of the lithiated stannylamines reported by Wrackmeyer.

Compound **3** appears to be the initial report of a structurally characterized metal complex that possesses zirconium and tin metal centers bridged by nitrogen. The structural features of **3** are comparable

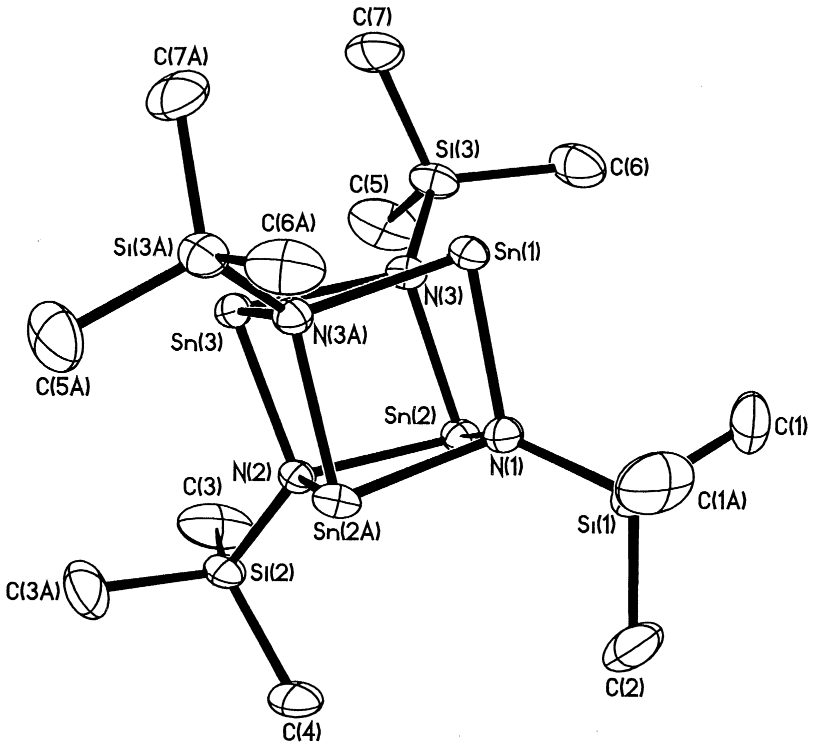


FIGURE 2 ORTEP representation (30% probability) of the structure of $[Sn(\mu_3\text{-}NSiMe_3)]_4$ (**4**). Hydrogen atoms are omitted for clarity.

to the zirconium dimer, $[(C_5H_5)_2ZrNC_6H_4C(CH_3)_3]_2$,[6] with **3** displaying slightly shorter Zr—N interatomic distances (2.058 Å vs. 2.096 Å) as well as slightly shorter Zr—Zr contacts (3.131 Å vs. 3.198 Å). Both structures contain planar Zr_2N_2 cores and each have similar angles about the zirconium and nitrogen atoms (**3**: N—Zr—N = 80.95°, Zr—N—Zr = 99.05°; $[(C_5H_5)_2ZrNC_6H_4C(CH_3)_3]_2$: N—Zr—N = 80.57°, Zr—N—Zr = 99.43°). The Sn—N interatomic distance in **3** falls within the range found in previous metal amide complexes [**3**: Sn—N = 2.047 Å; $\{(Me_3Sn)_2NLi(^tBuOMe)\}_2$: Sn—N = 2.032 Å;[5] $\{((CH_3)_5C_5)TiCl(NSn(CH_3)_3)\}_2$: Sn—N = 2.072 Å[7]].

Compound **4** was synthesized via the reaction of **2** with $SnCl_2$, as discussed above. This differs from the route employed by Veith et al.,[4] which involved the reaction of the cyclic diazastannylene, $Sn(N^tBu)_2SiMe_2$, with the primary amine, Me_2SiNH_2. The single crystal x-ray diffraction study of compound **4** produces a comparable result as reported by Veith et al.

TABLE I Crystal Data and Structure Refinement for **3**

Identification code	jfe11
Empirical formula	C26 H38 N2 Sn2 Zr2
Formula weight	798.40
Temperature	198(2) K
Wavelength	0.71073 A
Crystal system, space group	monoclinic, P2(1)/n
Unit cell dimensions	a = 8.2519(5) A alpha = 90 deg. b = 16.2209(11) A beta = 98.5650(10) deg. c = 10.3432(7) A gamma = 90 deg.
Volume	1369.03(16) A ^ 3
Z, Calculated density	2, 1.937 Mg/m ^ 3
Absorption coefficient	2.560 mm ^ −1
F(000)	776
Crystal size	0.34 × 0.20 × 0.07 mm
Theta range for data collection	2.35 to 28.80 deg.
Limiting indices	$-11 \leq h \leq 9$, $-20 \leq k \leq 19$, $-12 \leq l \leq 13$
Reflections collected/unique	8762/3265 [R(int) = 0.0475]
Completeness to theta = 28.80	91.5%
Max. and min. transmission	0.8452 and 0.4765
Refinement method	Full-matrix least-squares on F ^ 2
Data/restraints/parameters	3265/0/161
Goodness-of-fit on F ^ 2	0.964
Final R indices [I > 2 sigma(I)]	R1 = 0.0406, wR2 = 0.0935
R indices (all data)	R1 = 0.0727, wR2 = 0.1043
Largest diff. peak and hole	0.969 and −0.913 e.A ^ −3

In conclusion, this report describes the synthesis and characterization of the *N*-lithiostannylamines, **1** and **2**, isolated via recrystallization as analytically pure solids. Compounds **1** and **2** were used to synthesize the structurally characterized metal complex containing nitrogen-bridged zirconium and tin metal centers (**3**) and the tin-nitrogen heterocubane (**4**). *N*-lithiostannylamines should act as useful synthons to other metal amide complexes and based on the results of this work, may provide a general route to tin-nitrogen heterocubanes.

EXPERIMENTAL

All manipulations were carried out using standard Schlenk techniques or in a dry atmosphere glovebox. All solvents were dried over Na° and freshly distilled prior to use. MeLi, Me_3SiCl, $(C_5H_5)_2ZrCl_2$, and $SnCI_2$ were purchased and used as received with no additional purification. Compound **1** was prepared according to literature procedures[1] and recrystallized from THF.

TABLE II Atomic Coordinates (× 10^ 4) and Equivalent Isotropic Displacement Parameters (A^ 2 × 10^ 3) for jfe11

	x	y	z	U(eq)
Sn(1)	6936(1)	1621(1)	3866(1)	36(1)
Zr(1)	5741(1)	227(1)	6439(1)	33(1)
C(1)	3279(8)	844(4)	7334(7)	58(2)
C(2)	3742(8)	1449(4)	6562(7)	58(2)
C(3)	5278(9)	1755(4)	7134(6)	53(2)
C(4)	5714(8)	1339(4)	8278(6)	53(2)
C(5)	4485(9)	766(4)	8422(7)	62(2)
C(6)	8610(7)	−357(4)	6481(8)	56(2)
C(7)	7727(7)	−1012(4)	6832(6)	50(2)
C(8)	7328(8)	−878(4)	8051(7)	58(2)
C(9)	8005(9)	−139(5)	8497(8)	64(2)
C(10)	8769(8)	203(4)	7499(8)	64(2)
C(11)	9133(7)	1918(4)	5148(6)	54(2)
C(12)	5360(8)	2679(4)	3854(6)	54(2)
C(13)	7428(7)	1366(4)	1925(5)	46(1)
N(1)	5873(5)	604(2)	4559(4)	34(1)

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

(**2**): Isolated crystals of the THF adduct of **1** (5.14 g, 12.0 mmol) were dissolved in Et_2O (30 ml) and added drop-wise to a Et_2O (20 ml) solution of Me_3SiCl (1.33 g, 12.0 mmol) at 0°C. After the addition, the solution was warmed to room temperature and stirred overnight under argon. The next day, the solution was filtered to remove LiCI and the clear, colorless solution was reacted in situ with MeLi (7.64 ml of 1.6 M solution in hexanes, 12.0 mmol) at −30°C. After the addition, the solution was warmed to room temperature and stirred overnight. The next day, the Et_2O was reduced to about 20 ml under vacuum and colorless crystals of **2** were grown at −40°C. Multinuclear NMR and elemental analyses confirmed the identity of **2** as the ether adduct of the lithiated (trimethylstannyl)(trimethylsilyl)amine.

(**3**): Isolated crystals of the THF adduct of **1** (1.0 g, 2.45 mmol) were dissolved in 30 ml of THF and added drop-wise to a THF (20 ml) solution of $(C_5H_5)_2ZrCl_2$ (0.716 g, 2.45 mmol) at 0°C. The reaction mixture was warmed to room temperature and stirred overnight under argon. The next day, the THF was removed in vacuo, the solid residue extracted with 30 ml of hexane, and the solution filtered to remove any precipitate. The hexane was removed in vacuo and the yellow solid extracted with a minimum amount of diethyl ether. The clear, yellow solution produced a yellow crystalline solid at −40°C over a 24 h period.

(**4**): Isolated crystals of the Et_2O adduct of **2** (0.93 g, 2.21 mmol) were dissolved in 30 ml diethyl ether and added drop-wise to a Et_2O

TABLE III Bond Lengths [A] and Angles [deg] for jfe11

Sn(1)—N(1)	2.047(4)
Sn(1)—C(11)	2.136(6)
Sn(1)—C(13)	2.146(6)
Sn(1)—C(12)	2.152(6)
Zr(1)—N(1)	2.057(4)
Zr(1)—N(1)#1	2.060(4)
Zr(1)—C(6)	2.545(6)
Zr(1)—C(1)	2.559(6)
Zr(1)—C(10)	2.576(6)
Zr(1)—C(5)	2.584(7)
Zr(1)—C(7)	2.586(6)
Zr(1)—C(2)	2.595(6)
Zr(1)—C(4)	2.625(6)
Zr(1)—C(3)	2.626(6)
Zr(1)—C(8)	2.657(6)
Zr(1)—C(9)	2.682(7)
C(1)—C(2)	1.356(9)
C(1)—C(5)	1.392(9)
C(2)—C(3)	1.408(9)
C(3)—C(4)	1.363(9)
C(4)—C(5)	1.400(9)
C(6)—C(7)	1.367(9)
C(6)—C(10)	1.382(10)
C(7)—C(8)	1.367(9)
C(8)—C(9)	1.373(9)
C(9)—C(10)	1.402(10)
N(1)—Zr(1)#1	2.060(4)
N(1)—Sn(1)—C(11)	109.4(2)
N(1)—Sn(1)—C(13)	108.85(19)
C(11)—Sn(1)—C(13)	111.3(2)
N(1)—Sn(1)—C(12)	110.8(2)
C(11)—Sn(1)—C(12)	106.3(2)
C(13)—Sn(1)—C(12)	110.3(2)
N(1)—Zr(1)—N(1)#1	80.95(17)
N(1)—Zr(1)—C(6)	87.0(2)
N(1)#1—Zr(1)—C(6)	107.20(19)
N(1)—Zr(1)—C(1)	112.5(2)
N(1)#1—Zr(1)—C(1)	87.06(19)
C(6)—Zr(1)—C(1)	157.8(2)
N(1)—Zr(1)—C(10)	102.8(2)
N(1)#1—Zr(1)—C(10)	135.8(2)
C(6)—Zr(1)—C(10)	31.3(2)
C(1)—Zr(1)—C(10)	128.6(3)
N(1)—Zr(1)—C(5)	137.17(19)
N(1)#1—Zr(1)—C(5)	108.3(2)
C(6)—Zr(1)—C(5)	126.5(2)
C(1)—Zr(1)—C(5)	31.4(2)
C(10)—Zr(1)—C(5)	98.4(3)

(Continued on next page)

TABLE III Bond Lengths [A] and Angles [deg] for jfe11 *(Continued)*

N(1)—Zr(1)—C(7)	104.98(19)
N(1)#1—Zr(1)—C(7)	85.30(18)
C(6)—Zr(1)—C(7)	30.9(2)
C(1)—Zr(1)—C(7)	140.0(2)
C(10)—Zr(1)—C(7)	50.9(2)
C(5)—Zr(1)—C(7)	117.2(2)
N(1)—Zr(1)—C(2)	86.7(2)
N(1)#1—Zr(1)—C(2)	99.00(19)
C(6)—Zr(1)—C(2)	151.7(2)
C(1)—Zr(1)—C(2)	30.5(2)
C(10)—Zr(1)—C(2)	125.0(2)
C(5)—Zr(1)—C(2)	50.9(2)
C(7)—Zr(1)—C(2)	168.1(2)
N(1)—Zr(1)—C(4)	119.20(19)
N(1)#1—Zr(1)—C(4)	137.57(19)
C(6)—Zr(1)—C(4)	110.6(2)
C(1)—Zr(1)—C(4)	51.4(2)
C(10)—Zr(1)—C(4)	79.3(2)
C(5)—Zr(1)—C(4)	31.2(2)
C(7)—Zr(1)—C(4)	119.6(2)
C(2)—Zr(1)—C(4)	50.6(2)
N(1)—Zr(1)—C(3)	90.47(19)
N(1)#1—Zr(1)—C(3)	130.21(19)
C(6)—Zr(1)—C(3)	121.3(2)
C(1)—Zr(1)—C(3)	51.4(2)
C(10)—Zr(1)—C(3)	93.9(2)
C(5)—Zr(1)—C(3)	51.0(2)
C(7)—Zr(1)—C(3)	143.6(2)
C(2)—Zr(1)—C(3)	31.3(2)
C(4)—Zr(1)—C(3)	30.09(19)
N(1)—Zr(1)—C(8)	134.7(2)
N(1)#1—Zr(1)—C(8)	95.42(19)
C(6)—Zr(1)—C(8)	50.8(2)
C(1)—Zr(1)—C(8)	112.4(2)
C(10)—Zr(1)—C(8)	50.7(2)
C(5)—Zr(1)—C(8)	87.1(2)
C(7)—Zr(1)—C(8)	30.2(2)
C(2)—Zr(1)—C(8)	137.9(2)
C(4)—Zr(1)—C(8)	93.8(2)
C(3)—Zr(1)—C(8)	122.9(2)
N(1)—Zr(1)—C(9)	133.4(2)
N(1)#1—Zr(1)—C(9)	125.1(2)
C(6)—Zr(1)—C(9)	50.7(2)
C(1)—Zr(1)—C(9)	107.2(2)
C(10)—Zr(1)—C(9)	30.8(2)
C(5)—Zr(1)—C(9)	76.1(2)
C(7)—Zr(1)—C(9)	49.7(2)
C(2)—Zr(1)—C(9)	120.2(2)

(Continued on next page)

TABLE III Bond Lengths [A] and Angles [deg] for jfe11 *(Continued)*

C(4)—Zr(1)—C(9)	69.9(2)
C(3)—Zr(1)—C(9)	96.0(2)
C(8)—Zr(1)—C(9)	29.8(2)
C(2)—C(1)—C(5)	108.2(6)
C(2)—C(1)—Zr(1)	76.2(4)
C(5)—C(1)—Zr(1)	75.3(4)
C(1)—C(2)—C(3)	108.8(6)
C(1)—C(2)—Zr(1)	73.3(4)
C(3)—C(2)—Zr(1)	75.6(4)
C(4)—C(3)—C(2)	107.1(6)
C(4)—C(3)—Zr(1)	74.9(4)
C(2)—C(3)—Zr(1)	73.1(3)
C(3)—C(4)—C(5)	108.6(6)
C(3)—C(4)—Zr(1)	75.0(4)
C(5)—C(4)—Zr(1)	72.8(4)
C(1)—C(5)—C(4)	107.2(6)
C(1)—C(5)—Zr(1)	73.3(4)
C(4)—C(5)—Zr(1)	76.0(4)
C(7)—C(6)—C(10)	107.6(7)
C(7)—C(6)—Zr(1)	76.2(4)
C(10)—C(6)—Zr(1)	75.6(4)
C(8)—C(7)—C(6)	109.5(6)
C(8)—C(7)—Zr(1)	77.8(4)
C(6)—C(7)—Zr(1)	72.9(3)
C(7)—C(8)—C(9)	107.8(6)
C(7)—C(8)—Zr(1)	72.0(4)
C(9)—C(8)—Zr(1)	76.1(4)
C(8)—C(9)—C(10)	107.7(7)
C(8)—C(9)—Zr(1)	74.1(4)
C(10)—C(9)—Zr(1)	70.4(4)
C(6)—C(10)—C(9)	107.4(7)
C(6)—C(10)—Zr(1)	73.1(4)
C(9)—C(10)—Zr(1)	78.8(4)
Sn(1)—N(1)—Zr(1)	131.0(2)
Sn(1)—N(1)—Zr(1)#1	129.1(2)
Zr(1)—N(1)—Zr(1)#1	99.05(17)

Symmetry transformations used to generate equivalent atoms: #1 −x + 1, −y, −z + 1.

(20 ml) slurry of $SnCl_2$ at 0°C. The reaction mixture was warmed to room temperature and stirred overnight under argon. Subsequently, the Et_2O was removed in vacuo and the orange, solid residue extracted with 30 ml of a hexane/toluene solution. The solution was filtered to remove any precipitate and the clear, orange solution was reduced to about 20 ml under vacuum. The solution produced an orange crystalline solid at −40°C over a 24 h period.

TABLE IV Anisotropic Displacement Parameters (A^ 2 × 10^ 3) for jfe11

	U11	U22	U33	U23	U13	U12
Sn(1)	43(1)	34(1)	34(1)	2(1)	9(1)	−5(1)
Zr(1)	35(1)	31(1)	34(1)	−1(1)	11(1)	0(1)
C(1)	50(4)	50(4)	80(5)	−24(4)	26(4)	1(3)
C(2)	59(4)	50(4)	64(5)	−14(3)	9(4)	18(3)
C(3)	67(4)	41(3)	52(4)	−8(3)	18(3)	3(3)
C(4)	63(4)	56(4)	42(4)	−13(3)	8(3)	3(3)
C(5)	90(5)	54(4)	49(4)	4(3)	31(4)	4(4)
C(6)	39(3)	58(4)	71(5)	5(3)	8(3)	14(3)
C(7)	50(4)	44(3)	53(4)	2(3)	−2(3)	6(3)
C(8)	52(4)	60(4)	62(5)	16(3)	10(3)	0(3)
C(9)	59(4)	81(5)	48(4)	−4(4)	−7(3)	10(4)
C(10)	41(4)	53(4)	94(6)	0(4)	−6(4)	2(3)
C(11)	56(4)	54(4)	49(4)	1(3)	1(3)	−12(3)
C(12)	73(4)	41(3)	51(4)	6(3)	16(3)	−2(3)
C(13)	58(4)	46(3)	38(3)	0(3)	19(3)	−9(3)
N(1)	33(2)	34(2)	35(2)	1(2)	9(2)	−1(2)

The anisotropic displacement factor exponent takes the form: −2 pi^ 2 [h^ 2 a*^ 2 U11 + ⋯ + 2 h k a* b* U12].

TABLE V Hydrogen Coordinates (× 10^ 4) and Isotropic Displacement Parameters (A^ 2 × 10^ 3) for jfe11

	x	y	z	U(eq)
H(1)	2291	525	7161	56(18)
H(2)	3123	1638	5757	110(30)
H(3)	5902	2178	6785	90(30)
H(4)	6699	1425	8884	60(20)
H(5)	4476	389	9136	80(20)
H(6)	9042	−298	5674	130(40)
H(7)	7432	−1491	6304	80(20)
H(8)	6688	−1237	8513	90(30)
H(9)	7963	100	9340	220(60)
H(10)	9304	729	7519	270(70)
H(11A)	8859	2168	5929	80(13)
H(11B)	9775	2295	4720	80(13)
H(11C)	9752	1424	5372	80(13)
H(12A)	4479	2635	3139	118(18)
H(12B)	5978	3170	3756	118(18)
H(12C)	4921	2703	4662	118(18)
H(13A)	8522	1155	1968	121(18)
H(13B)	7323	1865	1419	121(18)
H(13C)	6660	965	1521	121(18)

REFERENCES

[1] P. Reiss and D. Fenske, *Z. Anorg. Allg. Chem.*, **626,** 1317 (2000).
[2] A. Decker, D. Fenske, and K. Maczek, *Angew. Chem. Int. Ed.*, **35,** 2863 (1996).
[3] K. Schmid, H. D. Hausen, K. W. Klinkhammer, and J. Weidlein, *Z. Anorg. Allg. Chem.*, **625,** 945 (1999).
[4] M. Veith, M. Opsolder, M. Zimmer, and V. Huch, *Eur. J. Inorg. Chem.*, **6,** 1143 (2000).
[5] C. Neumann, T. Seirfert, W. Storch, N. Vosteen, and B. Wrackmeyer, *Angew. Chem. Int. Ed.*, **40,** 3405 (2001).
[6] P. J. Walsh, E. J. Hollander, and R. G. Bergman, *J. Am. Chem. Soc.*, **110,** 8729 (1988).
[7] Y. Bai, H. W. Roesky, H. G. Schmidt, and M. Noltemeyer, *Z. Naturforsch, Teil B.*, **47,** 603 (1992).