DOI: 10.1002/ejic.200800142

Nickel Coordination Compounds of Stanna-*closo*-dodecaborate

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Dedicated to Prof. Dr. Dr. h. c. Bernt Krebs on the occasion of his 70th birthday

Keywords: Boranes / Nickel / Tin / ^{119}Sn NMR spectroscopy / N-heterocyclic carbenes

The reaction of the nickel complexes [(dppm)NiBr$_2$] [dppm = 1,1-bis(diphenylphosphanyl)methane] and [(dppe)NiBr$_2$] [dppe = 1,2-bis(diphenylphosphanyl)ethane] with stanna-*closo*-dodecaborate [Bu$_3$NH]$_2$[SnB$_{11}$H$_{11}$] (**1**) yielded the trigonal-bipyramidal coordination compounds [Bu$_3$NH]$_4$[(dppm)Ni(SnB$_{11}$H$_{11}$)$_3$] (**2**) and [Bu$_3$NH]$_4$[(dppe)Ni(SnB$_{11}$H$_{11}$)$_3$] (**3**). The square-planar compound [Bu$_3$NH]$_2$[(Im*i*Pr$_2$Me$_2$)$_2$-Ni(SnB$_{11}$H$_{11}$)$_2$] (**4**) (Im*i*Pr$_2$Me$_2$ = 1,3-diisopropyl-4,5-dimethyl-imidazoliumylidene) could be obtained by the reaction of the N-heterocyclic carbene complex [(Im*i*Pr$_2$Me$_2$)$_2$-NiBr$_2$] with [Bu$_3$NH]$_2$[SnB$_{11}$H$_{11}$] (**1**). The new coordination compounds were characterized by heteronuclear NMR spectroscopy and single-crystal X-ray structure analysis.

Introduction

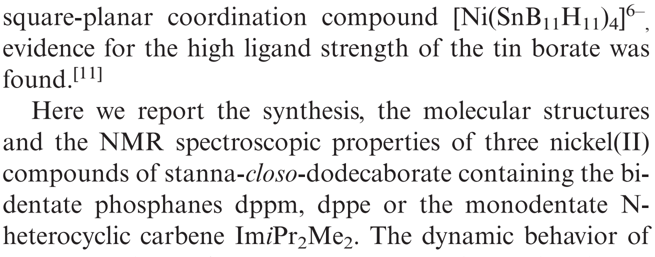
The formation of nickel–tin bonds is well established and has been studied for more than 30 years.[1,2] In general, nickel complexes with the metal atom in the formal oxidation states zero or two were treated with various tin ligands. Stannylenes for example react readily with the nickel(0) compounds [Ni(C$_2$H$_4$)$_3$], [Ni(CO)$_4$] or bis(toluene)nickel to give the respective (stannylene)nickel complexes.[3–5] Other systems, which feature nickel–tin bonds, are the recently described mixed-metal clusters [Sn$_9$Ni$_2$(CO)]$^{3-}$ and [Ni$_3$(μ-dppm)$_3$(μ$_3$-Cl)(μ$_3$-SnCl$_3$)].[6,7] Furthermore, the homoleptic diaminostannylene compounds [M{Sn(N*t*Bu)$_2$SiMe$_2$}$_4$Cl$_2$] (M = Ni, Pd and Pt), which exhibit a square-planar coordination at the metal centers, were reported by Veith and co-workers.[8] The nickel complexes [Ni(SnPh$_3$)(L)][BPh$_4$] [L = tris(2-diphenylphosphanylethyl)phosphane or tris(2-diphenylphosphanylethyl)amine] were found by the group of Sacconi, establishing the trigonal-bipyramidal coordination of nickel with polydentate phosphorus ligands and [SnPh$_3$]$^-$.[9] As one of the first coordination compounds of stanna-*closo*-dodecaborate, the nickel complex [Cp(PPh$_3$)Ni(SnB$_{11}$H$_{11}$)] was published in 1999.[10] Recently, we have reported a complete series of octahedrally coordinated complexes [M(SnB$_{11}$H$_{11}$)$_6$]$^{8-}$ of the nickel triade with the transition metals in the high formal oxidation state four.[11] Furthermore, with the square-planar coordination compound [Ni(SnB$_{11}$H$_{11}$)$_4$]$^{6-}$, evidence for the high ligand strength of the tin borate was found.[11]

Here we report the synthesis, the molecular structures and the NMR spectroscopic properties of three nickel(II) compounds of stanna-*closo*-dodecaborate containing the bidentate phosphanes dppm, dppe or the monodentate N-heterocyclic carbene Im*i*Pr$_2$Me$_2$. The dynamic behavior of [Bu$_3$NH]$_4$[(dppe)Ni(SnB$_{11}$H$_{11}$)$_3$] (**3**) was also explored.

Results and Discussion

Syntheses

The starting materials [(dppm)NiBr$_2$] and [(dppe)NiBr$_2$] were treated with 3 equiv. of [Bu$_3$NH]$_2$[SnB$_{11}$H$_{11}$] (**1**) in dichloromethane with formation of deep violet solutions (Scheme 1).



Scheme 1. Synthesis of [Bu$_3$NH]$_4$[(dppm)Ni(SnB$_{11}$H$_{11}$)$_3$] (**2**) and [Bu$_3$NH]$_4$[(dppe)Ni(SnB$_{11}$H$_{11}$)$_3$] (**3**).

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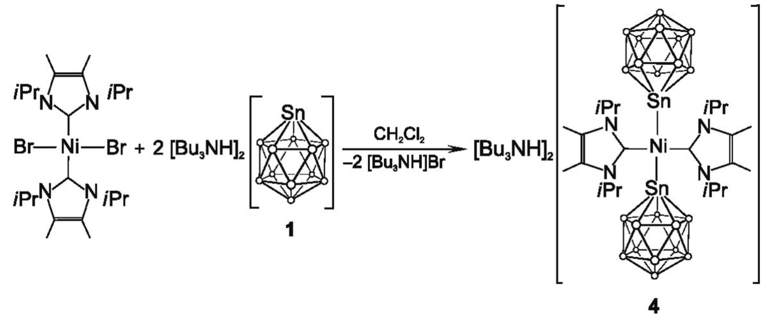

Scheme 2. Synthesis of $[Bu_3NH]_2[(ImiPr_2Me_2)_2Ni(SnB_{11}H_{11})_2]$ (**4**).

The completeness of the reaction was monitored by $^{11}B\{^1H\}$ NMR spectroscopy. The uncoordinated heteroborate shows resonances at δ = –6, –11 and –13 ppm with an intensity ratio of 1:5:5, whereas the coordination of stanna-*closo*-dodecaborate at a transition metal gives rise to one peak at $\delta \approx -15$ ppm.[10] After washing with water, the products **2** and **3** were obtained as dark violet solids in 64% and 70% yield, respectively. Compounds **2** and **3** are inert towards air and moisture and do not react with further equivalents of the heteroborate. Interestingly, the reaction of 2 equiv. of the tin nucleophile with the nickel electrophiles also gave the pentacoordinate compounds **2** and **3**, respectively. No other products could be identified in the ^{31}P NMR spectrum. The square-planar coordinated complex $[Bu_3NH]_2[(ImiPr_2Me_2)_2Ni(SnB_{11}H_{11})_2]$ (**4**) was obtained as a yellow solid from the reaction of 2 equiv. of stanna-*closo*-dodecaborate (**1**) with $[(ImiPr_2Me_2)_2NiBr_2]$ (Scheme 2), which was prepared in situ by the reaction of $[(thf)_2NiBr_2]$ with the free carbene ($ImiPr_2Me_2$). In contrast to the immediate formation of compounds **2** and **3**, compound **4** was obtained in 60% yield after a reaction time of 72 h.

Solid-State Structures

Slow diffusion of Et_2O into a tetrahydrofuran solution of the salt **2** gave large dark violet crystals which were suitable for single-crystal X-ray diffraction analysis. The molecular structure of the anion is shown in Figure 1 with selected bond lengths and angles, and the crystal data are listed in Table 1. The salt **2** crystallizes in the monoclinic space group $P2_1/n$ with the inclusion of one spatially disordered tetrahydrofuran molecule. The anion of **2** shows a pentagonal-bipyramidal structure with the phosphane coordinating in axial and equatorial positions. Due to the small bite angle of dppm, the trigonal-bipyramidal coordination is strongly distorted with an Sn1–Ni–P2 angle of 167.28° between the two apical atoms. The interatomic separations Ni–Sn1 (2.4644 Å), Ni–Sn2 (2.5435 Å) and Ni–Sn3 (2.4850 Å) are in the range of hitherto reported values for Ni–Sn bonds.[1–11] Dark violet crystals of compound **3** were obtained by slow diffusion of hexane into a dichloromethane solution of **3**. The salt crystallizes in the space group $P\bar{1}$ with inclusion of six partly disordered dichloromethane molecules. The structure of the transition metal complex is depicted in Figure 2, and the crystal data are listed in Table 1. In contrast to compound **2**, the trigonal bipyramid

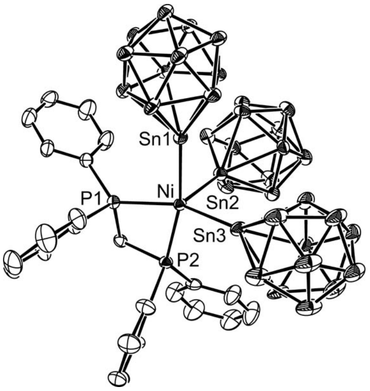

Figure 1. Molecular structure of the anion of $[Bu_3NH]_4[(dppm)Ni(SnB_{11}H_{11})_3]$ (**2**), H atoms and cations have been omitted for clarity, ellipsoids with 30%. Interatomic distances [Å] and angles [°]: Ni–Sn1 2.4644(8), Ni–Sn2 2.5435(8), Ni–Sn3 2.4850(8), Ni–P1 2.2122(17), Ni–P2 2.1920(17); P1–Ni–P2 74.82(6), Sn1–Ni–Sn2 92.50(3), Sn1–Ni–Sn3 87.12(3), Sn2–Ni–Sn3 100.01(3), Sn1–Ni–P1 94.16(5), Sn1–Ni–P2 167.28(5), Sn2–Ni–P1 112.05(5), Sn2–Ni–P2 97.49(5), Sn3–Ni–P1 147.81(5), Sn3–Ni–P2 98.80(5).

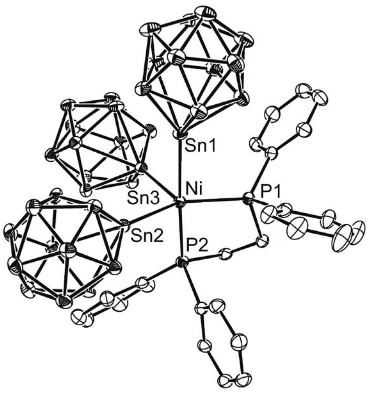

Figure 2. Molecular structure of the anion of $[Bu_3NH]_4[(dppe)Ni(SnB_{11}H_{11})_3]$ (**3**), H atoms and cations have been omitted for clarity, ellipsoids at 30% probability. Interatomic distances [Å] and angles [°]: Ni–Sn1 2.4939(9), Ni–Sn2 2.4815(8), Ni–Sn3 2.5236(9), Ni–P1 2.2027(17), Ni–P2 2.1880(17); P1–Ni–P2 87.93(6), Sn1–Ni–Sn2 84.34(3), Sn1–Ni–Sn3 91.93(3), Sn2–Ni–Sn3 105.51(3), Sn1–Ni–P1 89.93(5), Sn1–Ni–P2 175.12(6), Sn2–Ni–P1 139.68(6), Sn2–Ni–P2 94.46(5), Sn3–Ni–P1 114.57(5), Sn3–Ni–P2 92.95(5).

is undistorted, and the interatomic distances in the anion of **3** are of normal magnitude. Yellow single crystals of **4** were grown by slow diffusion of hexane into a dichloromethane solution of the (carbene)nickel complex. The structure refinement in space group $P2_1/n$ revealed that the asymmetric unit consists of two half molecules of the anion. The nickel atoms lie on a center of symmetry which generates the other half of the molecule. One of the *i*Pr groups of each half is disordered. The molecular structure is shown in Figure 3, and the crystal data are listed in Table 1. The Ni–Sn separation of 2.4720 Å is similar to the separation in the compounds **2** and **3** and the Ni–C bond length of 1.914 Å is comparable to the corresponding interatomic distance in the nickel complex [(1,3-dicyclohexylIm)$_2$NiI$_2$].[12] The square-planar coordination of the nickel center is almost ideal with an interatomic angle Sn1–Ni–C1 of 91.42°.

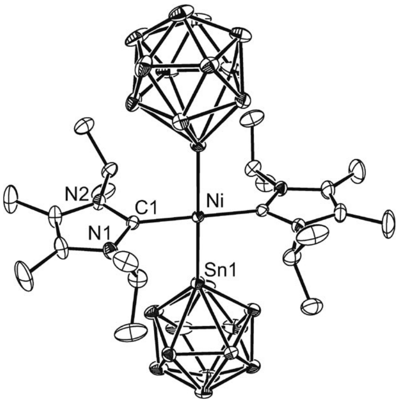

Figure 3. Structure of one anion of [(Im*i*Pr$_2$Me$_2$)$_2$Ni(SnB$_{11}$H$_{11}$)$_2$] (**4**) in the asymmetric unit, H atoms and cations have been omitted for clarity, ellipsoids with 30%; the dianion lies on a center of symmetry. Interatomic distances [Å] and angles [°]: Ni–Sn1 2.4720(2), Ni–C1 1.914(3); Sn1–Ni –C1 91.42(10).

NMR Spectroscopy in Solution

Compounds **2**, **3** and **4** exhibit one broad resonance in the ^{11}B{^{1}H} NMR spectrum at $\delta \approx -15$ ppm, which indicates Sn-coordination of the heteroborate through a metal–tin bond and can be assigned to B2–B12. The ^{31}P{^{1}H} NMR spectra of compound **2** and **3** at room temperature do not show the expected AB pattern but exhibit a broad singlet at $\delta = -11$ ppm for compound **2** and at $\delta = 64.7$ ppm for compound **3**. The corresponding tin satellites are not resolved into ^{117}Sn and ^{119}Sn satellites and show coupling constants of ^2J(Sn–P) = 360 Hz for compound **2** and ^2J(Sn–P) = 368 Hz as well as ^2J(Sn–P) = 1208 Hz for compound **3**. The remarkable difference of the ^{31}P chemical shifts of **2** and **3** of about 75 ppm can be ascribed to the well-known ring contribution ΔR to the coordination chemical shift of these complexes.[13,14]

Due to the fact that both ^{31}P{^{1}H} NMR spectra are broadened and not in agreement with the solid-state structures, ^{31}P{^{1}H} NMR spectra were measured at variable temperatures in order to obtain more structural information about these complexes in solution. Upon cooling of compound **2** to –60 °C, a second set of satellites appeared with ^2J(Sn–P) = 1340 Hz. Further cooling towards –80 °C resulted in no additional information, and the slow-exchange limit was not reached. In the ^{31}P{^{1}H} NMR spectra of compound **3** (Figure 4) at different temperatures, a new set of tin satellites could be detected in the 0 °C spectrum with ^2J(Sn–P) = 124 Hz. Further cooling led to a continuous broadening of the signal until coalescence was observed at –90 °C. Upon further cooling to –102 °C (CH$_2$Cl$_2$ solvent), two signals could be distinguished at $\delta = 78$ and 51 ppm, which can be ascribed to the equatorial and the apical phosphorus atoms. The slow-exchange limit was also not reached.

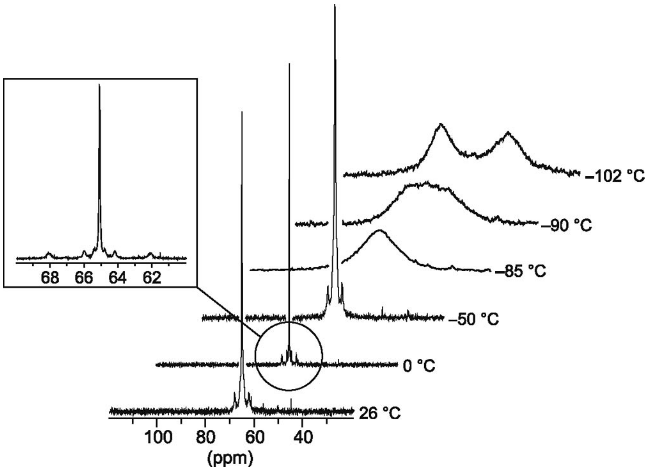

Figure 4. Variable-temperature ^{31}P{^{1}H} NMR spectra of **3**.

In the ^{119}Sn{^{1}H} NMR spectrum of compound **2** at room temperature, one broad signal with a linewidth of 3000 Hz can be detected at $\delta = -350$ ppm. This is in contrast to the ^{119}Sn{^{1}H} NMR spectrum (Figure 5) of compound **3**, which consists of two signals, a singlet at $\delta = -302$ ppm and a doublet of doublets at $\delta = -372$ ppm with an integration ratio of 1:2. The couplings of the signal at $\delta = -372$ ppm reflect the already observed coupling constants in the ^{31}P{^{1}H} NMR spectrum of **3** with ^2J(Sn–P) = 368 Hz and ^2J(Sn–P) = 1208 Hz. After cooling the sample, only a temperature dependence of the chemical shift towards lower frequencies was observed in the ^{119}Sn{^{1}H} NMR spectrum combined with a continuous loss of the coupling pattern. Compound **4** exhibits one broad singlet

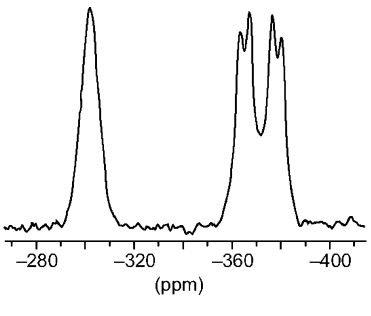

Figure 5. ^{119}Sn{^{1}H} NMR spectrum of **3** at room temperature.

in the ^{119}Sn{^{1}H} NMR spectrum at δ = –475 ppm. In comparison to other transition metal coordination compounds with the stanna-*closo*-dodecaborate, the ^{119}Sn NMR chemical shift of **4** shows a remarkable low-frequency shift.[10]

Finally, the nickel-coordinated carbon atom of the carbene gives rise to one resonance in the ^{13}C{^{1}H} NMR spectrum at δ = 160 ppm, flanked by tin satellites with a coupling constant of ^{2}J(Sn–C) = 246 Hz.

Conclusions

Stanna-*closo*-dodecaborate coordinates readily to the electrophiles [(dppm)NiBr$_2$] and [(dppe)NiBr$_2$] with the formation of trigonal-bipyramidal complexes, which show dynamic behavior in solution. With [(Im*i*Pr$_2$Me$_2$)$_2$Ni(SnB$_{11}$H$_{11}$)$_2$]$^{2-}$, a square-planar coordination at the nickel center was found.

Experimental Section

General: All manipulations were carried out under argon in Schlenk glassware. Solvents were dried and purified by standard methods and stored under argon. NMR spectra were recorded with a Bruker DRX-250 NMR spectrometer equipped with a 5 mm ATM probe head and operating at 250.13 (^{1}H), 80.25 (^{11}B), 62.90 (^{13}C), 101.25 (^{31}P), and 93.25 MHz (^{119}Sn) and a Bruker AVII+ 500 NMR spectrometer equipped with a 5 mm broad-band probe head, operating at 500.13 (^{1}H), 125.76 (^{13}C), and 202.46 MHz (^{31}P). Chemical shifts are reported in δ values in ppm relative to external TMS (^{1}H, ^{13}C), BF$_3$·Et$_2$O (^{11}B), 85% aqueous H$_3$PO$_4$ (^{31}P), or SnMe$_4$ (^{119}Sn) using the chemical shift of the solvent ^{2}H resonance frequency. Elemental analyses were performed at the Institut für Anorganische Chemie University of Tübingen using a Vario EL analyzer. The starting materials [(dppm)NiBr$_2$], [(dppe)NiBr$_2$][17] and Im*i*Pr$_2$Me$_2$[18] were prepared according to literature procedures. The compound [(Im*i*Pr$_2$Me$_2$)$_2$NiBr$_2$] was prepared in analogy to the synthesis of [(1,3-dicyclohexylIm)$_2$NiBr$_2$].[12] [Bu$_3$NH]$_2$[SnB$_{11}$H$_{11}$] (**1**) was synthesized by using a modified protocol of the original work of the group of Todd.[19]

Crystallography: X-ray data for compounds **2**–**4** were collected with a Stoe IPDS 2T diffractometer and corrected for Lorentz and polarization effects and absorption by air. The programs used in this work were Stoe's X-Area[20] and the WinGX suite of programs[21] including SHELXS[22] and SHELXL[23] for structure solution and refinement. Numerical absorption correction based on crystal-shape optimization was applied for **2** and **3** with Stoe's X-Red and X-Shape.[15,16] Crystal data are listed in Table 1. CCDC-680367, -680368, -680369 contain the supplementary crystallographic data for compounds **2**, **3**, **4**, respectively. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

[Bu$_3$NH]$_4$[(dppm)Ni(SnB$_{11}$H$_{11}$)$_3$] (2): A solution of [Bu$_3$NH]$_2$[SnB$_{11}$H$_{11}$] (932 mg,1.5 mmol) in dichloromethane (30 mL) was added to a solution of [(dppm)NiBr$_2$] (301 mg, 0.5 mmol) in dichloromethane (30 mL). The resulting dark violet solution was stirred for 1 h and extracted with water (3× 40 mL). The volume of the dichloromethane solution was reduced in vacuo to 20 mL and the product precipitated by the addition of hexane. A violet crystalline solid was obtained by filtration to give **2** (630 mg, 65% yield). Crystals suitable for single-crystal X-ray diffraction could be obtained by slow diffusion of diethyl ether into a tetrahydrofuran solution of **2**. C$_{73}$H$_{167}$B$_{33}$N$_4$NiP$_2$Sn$_3$ (1934.69): calcd. C 45.32, H 8.70, N 2.90; found C 45.74, H 8.10, N 2.71. ^{1}H NMR (CD$_2$Cl$_2$): δ = 7.07–7.89 (m, 20 H, P–C$_6$H$_5$), 2.96 (t, 24 H, –N–CH_2–CH$_2$–), 1.59 (m, 24 H, –CH$_2$–CH_2–CH$_2$–), 1.29 (m, 24 H, –CH$_2$–CH_2–CH$_3$), 0.87 (t, 36 H, –CH$_2$–CH$_2$–CH_3) ppm. ^{11}B{^{1}H} NMR (CD$_2$Cl$_2$): δ = –15.2 (B2–B12) ppm. ^{31}P{^{1}H} NMR (CD$_2$Cl$_2$): δ = –11.0 [s, ^{2}J(Sn-

Table 1. Crystal and structure refinement parameters for **2**, **3** and **4**.

	2	**3**	**4**
Empirical formula	C$_{77}$H$_{175}$B$_{33}$N$_4$NiOP$_2$Sn$_3$	C$_{80}$H$_{169}$B$_{33}$Cl$_{12}$N$_4$NiP$_2$Sn$_3$	C$_{47}$H$_{118}$B$_{22}$Cl$_2$N$_6$NiSn$_2$
M_r [g mol^{-1}]	2006.70	2446.04	1372.28
Wavelength [Å]	0.71073	0.71073	0.71073
Temperature [K]	173	173	173
Crystal system	monoclinic	triclinic	monoclinic
Space group	$P2_1/n$	$P\bar{1}$	$C2_1/c$
Z	4	2	8
a [Å]	15.5960(4)	14.7324(5)	52.3975(19)
b [Å]	28.4948(9)	17.3936(6)	10.6944(4)
c [Å]	25.6467(6)	24.7579(8)	31.2612(14)
α [°]	90	96.423(3)	90
β [°]	92.847(2)	90.556(3)	123.157(3)
γ [°]	90	92.633(3)	90
Volume [Å^3]	11383.5(5)	6297.0(4)	14665.2(10)
$\rho_{\text{calcd.}}$ [g/cm^{-3}]	1.213	1.290	1.243
μ [mm^{-1}]	0.883	1.054	1.038
R_{int}	0.0554	0.0680	0.0759
Reflections collected	59909	62258	67986
Unique reflections	18185	24682	14841
Observed reflections	13524	19331	12456
Parameters/restrains	1046/314	1158/14	834/237
Absorption correction	numerical[15,16]	numerical[15,16]	none
Max./min. transmission	0.7928/0.9348	0.7519/0.8764	–/–
R_1 (all/obsd. data)	0.0967/0.0657	0.1006/0.0764	0.0637/0.051
wR_2 (all/obsd. data)	0.1458/0.1333	0.1943/0.1779	0.1235/0.117
GOF (all/obsd. data)	1.095/1.095	1.102/1.097	1.052/1.046

P) = 368 Hz] ppm. ^{119}Sn{^{1}H} NMR (CD_2Cl_2): δ = –350 (br.) ppm.

$[Bu_3NH]_4[(dppe)Ni(SnB_{11}H_{11})_3]$ (3): To a solution of $[(dppe)NiBr_2]$ (378 mg, 0.6 mmol) in dichloromethane (120 mL) was added $[Bu_3NH]_2[SnB_{11}H_{11}]$ (1143 mg, 1.84 mmol). The resulting dark violet solution was stirred for 1 h and extracted with water (3× 40 mL). The volume of the dichloromethane solution was reduced in vacuo to 20 mL and the product precipitated by the addition of hexane. A violet crystalline solid was obtained by filtration to give **3** (830 mg, 70.5% yield). Crystals suitable for single-crystal X-ray diffraction could be obtained by slow diffusion of hexane into a dichloromethane solution of **3**. $C_{74}H_{169}B_{33}N_4NiP_2Sn_3$ (1948.72): calcd. C 45.61, H 8.74, N 2.88; found C 45.29, H 7.42, N 2.84. ^{1}H NMR (CD_2Cl_2): δ = 7.33–7.96 (m, 20 H, P–C_6H_5), 3.07 (t, 24 H, –N–CH_2–CH_2–), 1.67 (m, 24 H, –CH_2–CH_2–CH_2–), 1.29 (m, 24 H, –CH_2–CH_2–CH_3), 2.50 (br., 4 H, P–CH_2–CH_2–P), 0.97 (t, 36 H, –CH_2–CH_2–CH_3) ppm. ^{11}B{^{1}H} NMR (CD_2Cl_2): δ = –14.8 (B2–B12) ppm. ^{13}C{^{1}H} NMR (CD_2Cl_2): δ = 134.4 (br., P–C_6H_5), 129.7 (br., P–C_6H_5), 127.6 (P–C_6H_5), 51.5 (N–CH_2–CH_2–), 34.0 (br., P–CH_2–CH_2–P), 25.7 (N–CH_2–CH_2–), 20.4 (–CH_2–CH_2–CH_3), 13.8 (–CH_2–CH_2–CH_3) ppm. ^{31}P{^{1}H} NMR (CD_2Cl_2): δ = 64.7 [br., ^2J(P-Sn) = 1186, ^2J(P-Sn) = 368 Hz] ppm. ^{119}Sn{^{1}H} NMR (CD_2Cl_2): δ = –302 (br., 1 Sn), –372 [dd, 2 Sn, ^2J(Sn–P) = 1186, ^2J(Sn–P) = 368] ppm.

$[Bu_3NH]_2[(ImiPr_2Me_2)_2Ni(SnB_{11}H_{11})_2]$ (4): In a glove box Im-*i*Pr_2Me_2 (180 mg, 1 mmol) was added to a solution of $[(thf)_2NiBr_2]$ (181 mg, 0.5 mmol) in dichloromethane (20 mL). After stirring for 10 min, $[Bu_3NH]_2[SnB_{11}H_{11}]$ (621 mg, 1 mmol) was added, and stirring was continued for 72 h. The resulting yellow solution was extracted with water (3× 40 mL). The solvent was removed in vacuo to give 790 mg (61% yield) of a yellow solid. Slow diffusion of hexane into a dichloromethane solution of **4** gave yellow crystals suitable for single-crystal X-ray diffraction. $C_{46}H_{118}B_{22}N_6NiSn_2$ (1289.44): calcd. C 42.85, H 9.22, N 6.52; found C 42.08, H 8.05, N 6.70. ^{1}H NMR (CD_2Cl_2): δ = 6.78 [m, 4 H, –N–CH–$(CH_3)_2$], 2.95 (t, 12 H, –N–CH_2–CH_2–), 2.05 [s, 12 H, –C–(CH_3)], 1.64 (m, 12 H, –CH_2–CH_2–CH_2–), 1.57 [d, 24 H, –N–CH–$(CH_3)_2$], 1.29 (m, 12 H, –CH_2–CH_2–CH_3), 0.88 (t, 18 H, –CH_2–CH_2–CH_3) ppm. ^{11}B{^{1}H} NMR (CD_2Cl_2): δ = –15.4 (B2–B12) ppm. ^{13}C{^{1}H} NMR (CD_2Cl_2): δ = 160.3 [^2J(Sn-C) = 246 Hz, N–*C*–N], 129 (*C*–CH_3), 55.6 [N–*C*H–$(CH_3)_2$], 51.5 (N–CH_2–CH_2–), 25.7 (N–CH_2–CH_2–), 22.7 [N–CH–$(CH_3)_2$], 20.4 (–CH_2–CH_2–CH_3), 13.8 (–CH_2–CH_2–CH_3), 10.9 (CCH_3) ppm. ^{119}Sn{^{1}H} NMR (CD_2Cl_2): δ = –475 (s) ppm.

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Received: February 6, 2008