Oxidation of Germa- and Stanna-closo-dodecaborate

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Complete List of Authors: Wesemann, Lars; University of Tuebingen, Institut fur Anorganische Chemie
Gädt, Torben; University of Tübingen,
Dimmer, Lörg-Alexander; University of Tübingen,
Fleischhauer, Sebastian; University of Tübingen,
Frank, Anita; University of Tübingen,
Nickl, Claudia; University of Tübingen,
Wütz, Tobias; University of Tübingen,
Eichele, Klaus; University of Tübingen,
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Torben Gädt, a Jörg-Alexander Dimmer, a Sebastian Fleischhauer, a Anita Frank, a Claudia Nickl, a Tobias Wütz, a Klaus Eichele, a and Lars Wesemann a *

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Oxidation of group 14 heteroborates [GeB_{11}H_{11}]^{2–} and [SnB_{11}H_{11}]^{2–} yields a symmetrical dimer exhibiting a Ge–Ge bond or an unsymmetrical dimer bearing an Sn–B bond.
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The oxidation of closo-heteroborates \([\text{GeB}_{11}\text{H}_{11}]^{2-}\) and \([\text{SnB}_{11}\text{H}_{11}]^{2-}\) is presented. Upon oxidation germa-closo-dodecaborate yields a symmetrical dimer exhibiting a Ge–Ge bond between two clusters. This dimer shows sulphur insertion into the Ge–Ge bond at room temperature. In contrast, oxidation of the homologous tin cluster results in an unsymmetrical dimer bearing an Sn–B bond between two clusters. The Sn–B dimer is also the product of the hydride abstraction reaction. In the presence of the donor ligand 2,2'-bipyridine, the oxidation of closo-cluster \([\text{SnB}_{11}\text{H}_{11}]^{2-}\) leads to the Sn(IV)-half sandwich coordination compound \([\text{bipyO}\text{SnB}_{11}\text{H}_{11}]\) which dissolves in DMSO to give the Sn(IV)-adduct \([\text{bipy(DMSO)}\text{O}\text{SnB}_{11}\text{H}_{11}]\).

Introduction

Heteroborane cluster chemistry is an active field of research and many interesting studies have been published in recent years.\(^1\)\(^-\)\(^10\) In heteroborane clusters a BH vertex of a borane cluster skeleton is replaced by a main group element or a transition metal. Following the isolobal principle a BH unit can for example be substituted by a Ge atom or an anionic BH unit by an N atom respectively.\(^11\)\(^-\)\(^13\) In these cases the resulting heteroboranes do not have a substituent but a lone pair at the vertex of the heteroelement and therefore can potentially act as a Lewis base. For more than ten years we have been interested in the chemistry of group 14 heteroborane clusters carrying an unsubstituted group 14 element like germanium or tin.\(^14\) The monoheteroborane and diheteroborane clusters are the focus of our investigations with respect to synthesis, ligand properties in coordination chemistry and reactions with chalcogenes.\(^13\)\(^-\)\(^22\) Besides their chemistry the NMR spectroscopical properties in solution and in the solid state were also explored. Furthermore the Mössbauer spectroscopy results of the tin clusters and their derivatives were published.\(^23\) Due to the low oxidation state II of the group 14 elements in the clusters \([\text{GeB}_{11}\text{H}_{11}]^{2+}\) and \([\text{SnB}_{11}\text{H}_{11}]^{2+}\) we started to study oxidation reactions of these closo-heteroborates. The oxidation of the stanna-closo-dodecaborate \([\text{SnB}_{11}\text{H}_{11}]^{2-}\) with typical oxidation agents like hydrogen peroxide and iodine was investigated several years ago.\(^23\)\(^,\)\(^24\) The dianionic tin cluster \([\text{SnB}_{11}\text{H}_{11}]^{2-}\) reacts with hydrogen peroxide to give a tetrameric oxide \(\text{K}_{8}[\text{OSnB}_{11}\text{H}_{11}]\) (Chart 1).\(^23\)

![Chart 1](image)

**Chart 1** Two oxidation products of stanna-closo-dodecaborate (Sn= [SnB11H11]).

In this heterocubane type structure three oxygen atoms coordinate at each tin vertex and the tin atom exhibits a very small isomeric \(^{119}\)Sn Mössbauer shift indicating the oxidation state IV. In the case of the reaction of the cluster \([\text{SnB}_{11}\text{H}_{11}]^{2-}\) with iodine at \(-78^\circ\)C we found oxidative addition of the halogen at the tin vertex of the cluster.\(^24\) In this manuscript we present oxidation chemistry of germa- and stanna-closo-dodecaborate leading to unexpected Sn–B bond formation in the case of the tin cluster.

Results and discussion

In order to investigate the oxidation of germa-closo-dodecaborate \([\text{GeB}_{11}\text{H}_{11}]^{2-}\) we reacted a sample of the salt 1 with various oxidizing agents like hydrogen peroxide, iodine and ferrocenium hexafluorophosphate (Scheme 1).

![Scheme 1](image)

**Scheme 1** Reactions of germa-closo-dodecaborate with hydrogen peroxide, iodine and ferrocenium hexafluorophosphate.

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Oxidation of germa-closo-dodecaborate, a: I$_2$, b: 2 [Cp$_2$Fe][PF$_6$].

We were not able to characterize the product of the reaction of closo-cluster 1 with hydrogen peroxide. The $^{11}$B NMR spectrum shows a new substance but due to the lack of crystalline material we cannot identify the composition of the product. However, in the case of the reaction with iodine or ferrocinium hexafluorophosphate the dimeric oxidation product 2 was formed quantitatively and isolated as colourless crystals (Scheme 1). The molecular structure together with selected interatomic distances is presented in Figure 1.

The observed Ge–S separations of 2.178(1) Å are slightly longer than the Ge–S bond characterized in the sulphur adduct [H$_4$B$_3$Ge–S] of 2.102(1) Å. The found Ge–S–Ge structural motive is known in the literature from a variety of cyclic Ge-S structures but should only be compared with molecules showing a related sulphur bridge between two germanium moieties. These examples show longer Ge–S bond lengths and a comparable Ge–S–Ge angle: [S(Ge{CH$_2$})) 2.235(21) Å, 111.0°; [S–(GePh$_3$)$_2$] 2.212(1), 2.261(1), 2.227(2), 2.240(2), 111.2(1), 110.7(1)°; [S(Ge(SiMe$_3$)C(Ph)(C(SiMe$_3$)C(H$_2$N$_2$)_2)] 2.222(3), 2.226(3) Å, 101.4(1)°; [S(ge(ButCN)$_2$)$_2$, Ar. 2.6-4] 2.252(14), 2.2626(14) Å, 101.37(6)°; [S(ge(NSiMe$_3$)Ar)$_2$, (Ar = C$_6$H$_4$(CH$_2$)$_2$Me-2,6,4)] 2.2854(8), 2.2869(8) Å, 98.21(3)°.

In analogy to the oxidation of the germanium cluster 1 the reaction of the homologous tin cluster [SnB$_3$H$_{11}$]$_2$ with the ferrocium salt [Cp$_2$Fe][PF$_6$] was also investigated. To our surprise we did not isolate a homologous dimer showing a Sn-Sn bond from this reaction. Instead a dimer exhibiting a Sn-B bond was formed (Scheme 3). Dimer 5 was characterized by elemental analysis, mass spectrometry and heteronuclear NMR spectroscopy. Although many different counterions were applied in the crystallization, crystals yielding suitable X-ray data for publication were not obtained. In the solution of the crystal structure determination a severe disorder of the trianion [SnB$_3$H$_{11}$-(SnB$_3$H$_{11}$)]$^-$ prevents a discussion of interatomic distances.
The \(^{119}\text{Sn}^\text{1H}\) NMR spectrum of 5 shows two characteristic signals at \(-267\) [q] and \(-558\) [s] ppm respectively. The non-binomial quartet at \(-267\) ppm shows coupling to a \(^{11}\text{B}\) atom with a \(^1\text{J}^\text{(119\text{Sn}-11\text{B})}\) coupling constant of 850 Hz which is indicative of a direct Sn–B bond.\(^{23}\) The singlet at \(-558\) ppm is in a range comparable to the starting material 4 which shows a signal at \(-546\) ppm.\(^{15,16,36,37}\) Due to the low symmetry of the trianion 5, the \(^{11}\text{B}^\text{1H}\) NMR spectrum exhibits eight signals with an intensity ratio of 1:1:2:2:2:2:11. Assignment of the signals was carried out on the basis of \(^{11}\text{B}^\text{1H}\), \(^{31}\text{P}^\text{1H}\) NMR and \(^{11}\text{B}^\text{1H}\) COSY spectra. The oxidation product 5 was also formed in reaction with air or phenyl iodonium diacetate.

Since the formation of the trianion 5 can also be regarded as a hydride abstraction followed by nucleophilic attack of a \([\text{SnB}_3\text{H}_4]^\text{2-}\) cluster we studied the reaction of stanna-closo-dodecaborate with the Lewis acids [\(\text{B}^\text{C}_6\text{F}_3\text{H}_3\)] and [\(\text{CP}_3\text{Sn}^\text{2-}\text{Cl}_2\)]. Interestingly, with both reagents the Sn–B dimer 5 can be synthesized quantitatively.

In order to get further information about the oxidation pathways of the tin cluster the oxidation was also investigated in presence of donor ligands. In this reaction an acetonitrile solution of two equivalents of the oxidizing reagent were added to an acetonitrile solution of \(2,2^\prime\)-bipyridine and \([\text{Bu}_3\text{MeN}]_2[\text{SnB}_3\text{H}_4]\) (Scheme 4). Immediately a light brown precipitate was formed which was isolated by filtration. The solid is insoluble in common solvents except DMSO (vide infra). Elemental analysis confirms the formation of the bipy adduct 6 \([\text{bipySnB}_3\text{H}_4]\). The \(^{119}\text{Sn}^\text{MAS} NMR spectrum which shows a resonance at \(-146\) ppm (Figure 3) corroborates this finding since the comparable Sn(IV) cluster \([\text{MeSnB}_3\text{H}_4]\) shows a resonance at \(-197\) ppm whereas the chemical shift of \([\text{SnB}_3\text{H}_4]^\text{2-}\) is \(-546\) ppm.\(^{15,16,37}\)

Scheme 4 Oxidation of \([\text{SnB}_3\text{H}_4]^\text{2-}\) and reaction with 2,2\(^\prime\)-bipy.

Upon dissolving the oxidation product 6 in DMSO at room temperature the solution turns red and the DMSO adduct 7 is formed (Scheme 5). After one day orange red crystals of 7 were isolated and characterized by single crystal diffraction, elemental analysis and NMR spectroscopy. The molecular structure of 7 is depicted in Figure 4 together with selected interatomic distances and angles. The tin vertex is centered above the pentagonal B5-face with Sn–B distances of 2.305(4) – 2.328(4) Å. These interatomic Sn–B distances are shorter than the distances found in \([\text{OSnB}_3\text{H}_4]_4\)\(^-\) [Sn–B range: 2.337(5) – 2.390(5)Å] and also shorter than the Sn–B values found in the starting material.\(^{23,37}\) The tin vertex is coordinated with two exo-polyhedral ligands, viz. bipyridine and a DMSO molecule that is bonded to the tin vertex via its oxygen atom. The Sn–N separations are 2.241(3) and 2.233(3) Å respectively. Compared to the Sn(II)–N distances in 1-\((2,2^\prime\)-bipy\()-2,3,\text{Me}_2-1\text{Sn}-2,3,\text{C}_2\text{B}_3\text{H}_4\) [2.412(14), 2.374(15) Å] and 1–Sn(bipy)-2,3-(\text{SiMe}_3)_2-2,3-C_2B_3H_4 [2.49(1), 2.54(1)] the Sn–N distances of 7 are remarkably short.\(^{38,39}\) Further references from known bipyridine adducts of Sn(IV) species like \([\text{Me(Ph)SnCl}_4(\text{bipy})]\) \([\text{Sn–N} 2.389, 2.401 \text{Å}\) and \([\text{MeCl}_2(\text{bipy})\text{Sn(CH}_3)_2\text{SnMe}_3]\) \([\text{Sn–N} 2.270, 2.372 \text{Å}\) corroborate the finding that adduct 7 exhibits rather short Sn–N distances.\(^{40,41}\) The tin oxygen separation amounts to 2.266(2) Å which is a reasonable value compared to literature known distances between an Sn(IV) center and a DMSO ligand.\(^{42,44}\)
Compound 7 can also be viewed as a rare example of an η⁵-coordinated Sn(IV) atom which has not been documented for many ligands. Hosmane presented a rare example for a bent-sandwich complex of Sn(IV) with a carborane ligand.\(^{35,36}\) With the DMSO adduct 7 we have isolated the second example for a half-sandwich complex of type [L₂Sn(B₁₂H₁₁)]\(^{3-}\). The tetrameric oxide [(OSnB₂H₁₁)₄]\(^{3-}\) (Chart 1) is another example for this type of coordination. Formally these molecules can be separated into a L₂Sn(IV) fragment coordinated at a [B₁₂H₁₁]⁴⁻ ligand which is isomorph with the carboalide [C₂B₆H₁₁]⁴⁻ and cyclopentadiene [C₅] ligand.\(^{47}\) We would also like to discuss the presented heteroborates 6 and 7 with respect to Wade’s cluster rules.\(^{48}\) According to these rules the donor ligands contribute two electrons each to the electron count of the tin vertex and thus the tin vertex contributes in the case of 6 six and in the case of 7 eight electrons to the cluster count. In addition to the eleven BH-fragments which contribute 22 electrons both clusters exhibit an electron count higher than the half sandwich complex of type [L₂Sn(B₁₂H₁₁)]\(^{3-}\) necessary for a twelve vertex cluster and thus the tin vertex contributes in the case of 7 eight electrons to the cluster count. In

### Experimental

**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 operating at 250.13 (\(^{1}H\), 80.25 (\(^{11}B\), 62.9 (\(^{13}C\), 101.25 (\(^{31}P\) and 93.25 MHz (\(^{119}Sn\) and a Bruker AvanceII+500 NMR-spectrometer (\(^{1}H\), 500.13 MHz; \(^{13}C\), 125.76 MHz; \(^{119}Sn\), 186.50 MHz). Chemical shifts are reported in δ values in ppm relative to external TMS (\(^{1}H\), \(^{13}C\) BF₄⁻Et₂O (\(^{11}B\) or SnMe₄ (\(^{119}Sn\)) using the chemical shift of the solvent \(^{2}H\) resonance frequency. The solid-state \(^{119}Sn\) CP/MAS spectra were obtained at 74.63 MHz using a Bruker DSX-200 spectrometer (\(B_0 = 4.7\) T). Powdered samples of 6 were packed into 4 mm zirconia rotors. After cross-polarization (CP) the spectra were acquired under high-power proton decoupling and were referenced with respect to external SnMe₄ via external SnCy₄ at –97.4 ppm. Elemental analyses were performed at the Institut für Anorganische Chemie, University of Tübingen using a Vario EL analyzer.

**Crystallography:** X-Ray data for compounds 3 and 7 were collected on a Stoe IPDS 2T diffractometer and were corrected for Lorentz and polarization effects and absorption by air. The programs used for 3 and 7 are Stoe’s X-Area and the WinGX suite of programs including SHELXS and SHELXL for structure solution and refinement.\(^{50,51,52}\) Numerical absorption correction based on crystal shape optimization was applied for all compound with Stoe’s X-Red and X-Shape.\(^{53,54}\) X-Ray data for 2 were collected with a Bruker APEX-II CCD diffractometer equipped with a sealed tube source with molybdenum anode and graphite monochromator. For data reduction and absorption correction Bruker’s APEX2\(^{55}\) including the programs SAINT,\(^{56,57}\) SADABS\(^{58}\) and XPREP was used.\(^{59}\)

**Synthesis of [Bu₃MeN]₂[SnB₁₂H₁₁] (2):** To a solution of [Bu₃MeN]₂[GeB₁₁H₁₁] (100 mg, 0.160 mmol) in acetonitrile (10 mL), I₂ (21 mg, 0.08 mmol) was added as a solid. The colour of the reaction mixture vanished rapidly. After stirring for one hour, all volatile components were removed under reduced pressure. The residue was washed with water to remove [Bu₃MeN] and the solid was dried overnight. Colourless crystals of 2 (39 mg, 60 % yield) were obtained by layering a filtered solution of the residue in dichloromethane with n-hexane. \(^{1}H\) NMR (250.13 MHz, CDCl₃) δ: 0.94 [t, \(^{3}J(\text{H},\text{H}) = 7.3\) Hz, 1H, CH₂CH₂], 1.36 (m, 12H, CH₂CH₂), 1.59 (m, 12H, CH₂CH₂CH₂), 2.92 (s, 6H, NC₃H₃), 3.08 ppm (m, 12H, NC₃H₃CH₂). \(^{11}B\) [\(^{1}H\)] NMR (80.25 MHz, CDCl₃) δ: –7.0 to –14.5 ppm (s, 20B, B₂11). Elemental analysis (% calcd for C₁₂H₁₂B₂₂Ge₅N₂: C, 38.74; H, 10.25; N, 3.48; C₂₀H₁₆B₄₂Ge₅N₄S: C, 72.65; H, 11.05; N, 3.48). Compound 7 can also be viewed as a rare example of an η⁵-coordinated Sn(IV) atom which has not been documented for many ligands. Hosmane presented a rare example for a bent-sandwich complex of Sn(IV) with a carborane ligand.\(^{35,36}\) With the DMSO adduct 7 we have isolated the second example for a half-sandwich complex of type [L₂Sn(B₁₂H₁₁)]\(^{3-}\). The tetrameric oxide [(OSnB₂H₁₁)₄]\(^{3-}\) (Chart 1) is another example for this type of coordination. Formally these molecules can be separated into a L₂Sn(IV) fragment coordinated at a [B₁₂H₁₁]⁴⁻ ligand which is isomorph with the carboalide [C₂B₆H₁₁]⁴⁻ and cyclopentadiene [C₅] ligand.\(^{47}\) We would also like to discuss the presented heteroborates 6 and 7 with respect to Wade’s cluster rules.\(^{48}\) According to these rules the donor ligands contribute two electrons each to the electron count of the tin vertex and thus the tin vertex contributes in the case of 6 six and in the case of 7 eight electrons to the cluster count. In addition to the eleven BH-fragments which contribute 22 electrons both clusters exhibit an electron count higher than the half sandwich complex of type [L₂Sn(B₁₂H₁₁)]\(^{3-}\) necessary for a twelve vertex cluster and thus the tin vertex contributes in the case of 7 eight electrons to the cluster count. In addition to the eleven BH-fragments which contribute 22 electrons both clusters exhibit an electron count higher than the 26 electrons necessary for a twelve vertex \(\text{closo}\) cluster. However both molecules 6 and 7 show an icosahedral twelve vertex \(\text{closo}\) geometry.\(^{49}\) Obviously, the canonical Wade counting scheme fails to predict the correct cluster structures in the case of oxidized stannia-\(\text{closo}\)-dodecaborate species with main group element ligands.

**Synthesis of [Bu₃MeN]₂[SnB₁₂H₁₁] (3):** [Bu₃MeN]₂[GeB₁₁H₁₁] (2) (67 mg, 0.08 mmol) was dissolved in dichloromethane (12 mL). Excess sulphur was added as a solid and the reaction mixture was stirred for three days. The mixture was filtered and carefully layered with n-hexane. After several days, colourless crystals of 3 (35 mg, 50 % yield) were obtained. \(^{11}B\) [\(^{1}H\)] NMR (80.25 MHz, CDCl₃) δ: –13.7 (s, 2B, B₂12), –16.3 ppm (s, 20B, B₂12). Elemental analysis (%) calcd for C₁₂H₁₂B₄₂Ge₅N₂: C, 37.26; H, 9.86; N, 3.34; S, 3.83. Found: C, 37.25; H, 9.17; N, 3.39; S, 3.74.

**Synthesis of \([\text{Et₃HN}]_2[\text{SnB₁₂H₄p-} \text{SnB₁₂H₁₁}]\) (5):** 43.5 mg (0.100 mmol) [Et₃HN]₂[SnB₁₂H₁₁] were added and the colour of the solution turns to bright yellow immediately. After stirring for two hours the colour had disappeared and all volatiles were removed in vacuum. The remaining residue was washed three times each with water and diethyl ether. The resulting colourless solid was dried in vacuum. Crystals were obtained by slow diffusion of diethyl ether into an acetoniitile solution of 5. (31.7 mg, 79% yield) Alternatively 43.5 mg (0.100 mmol) [Et₃HN]₂[SnB₁₂H₁₁] were dissolved in 20 mL acetoniitile, two equivalents of...
[CP₂Fe][PF₆] dissolved in 10 mL acetonitrile were added and the colour of the solution instantly turned to bright yellow. Volatiles were removed in vacuum and the remaining residue was washed with water and diethyl ether. The resulting colourless solid was dried in vacuum and recrystallized from acetonitrile by slow diffusion of diethyl ether. (25.0 mg, 62% yield). ¹H¹¹B NMR (250.13 MHz, CD₂Cl₂) δ: 1.27 [t, ⁴JC(H-H) = 7.3 Hz, 2H, CH₃], 3.13 [q, ⁴JC(H-H) = 7.3 Hz, 18H, N-CH₂₂], ¹¹B¹¹B NMR (160.46 MHz, CD₂Cl₂) δ: -3.6 (B12), -8.0 (B9), -8.6 (B4/5), -10.0 (B3/6), -11.4 (B7/11, 8/10), -13.6 (B2), -15.8 (SnB₂), ¹³Sn¹³H NMR (186.43 MHz, CH₂Cl₂) δ: -267 [q, ⁴JC(Sn-Sn-B) = 850 Hz], -558 (s, br). Elemental analysis calcld (%) for ([bipy]SnB₂[H]₂): C, 29.39; H, 5.33; N, 5.34; S, 6.11.

Synthesis of [[bipy]-SnB₂[H]₂] (6): 260 mg (0.400 mmol) [Bu₄NMeN]₂[SnB₂[H]₂] and 62.5 mg (0.400 mmol) 2,2′-bipyridine were dissolved in 10 mL acetonitrile. To the stirred solution was added dropwise a solution of 265 mg (0.800 mmol) [FeCp₂][PF₆]. The colour of the reaction mixture changed to orange-brown and a light brown precipitate was formed. The solid was filtered off, washed three times with 10 mL CH₂Cl₂ and dried overnight. (85.8 mg, 53.0% yield). IR (ATR) 2496 (s, ν(B-H)), 1013 (m, δ(B-H)), 767 (m, δ(C-H)), 720 (m, cage breathing), 288 (w, ν(CH₃Sn-B-N)). Elemental analysis calcld (%) for C₁₀H₁₉B₂N₂Sn (404.91 g/mol): C, 29.32; H, 4.73; N, 6.92. found: C, 30.03; H, 4.61; N, 6.28.

Conclusions
The homologous closo-clusters germa-closo-dodecarbaborate and stanna-closo-dodecarbaborate show different reaction products upon oxidation. Whereas germa-closo-dodecarbaborate forms a symmetrical dimer exhibiting a Ge–Ge bond as the product of oxidation the tin cluster reacts to an unsymmetrical dimer bearing an Sn–B bond. The Sn–B dimer is also the product of the hydride abstraction reaction of [SnB₃[H]₂]²⁻. Furthermore in the presence of donor ligands the oxidation of the closo-cluster [SnB₁₃[H]₂]²⁻ leads to Sn(IV) half sandwich coordination compounds of the type [L₂SnB₁₃[H]₂] (x = 2–3).

Acknowledgements
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Notes and references
25. Details about the crystal structure solution are given in the Supporting Information.
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59. Details about the crystal structure solution are given in the Supporting Information.
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Oxidation of group 14 heteroborates [GeB_{11}H_{11}]^{2–} and [SnB_{11}H_{11}]^{2–} yields a symmetrical dimer exhibiting a Ge–Ge bond or an unsymmetrical dimer bearing an Sn–B bond.