Two Isomers of a Tris(dimethylamino)bis(trimethylstannyl) Triborane(5) and the Reaction of $(Me_3Sn)_2B_2(NMe_2)_2$ with $(Ph_3P)_2Pt(\eta^2\text{-}C_2H_4)$[‡]

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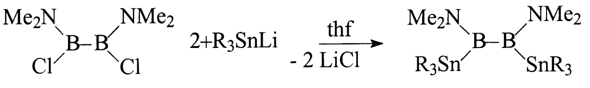
Reactions of $LiSnR_3$ (R = Me, Ph) with $Cl(Me_2N)$-B–$B(NMe_2)Cl$ lead to the corresponding 1,2-(triorganylstannyl)diboranes(4) **1a,b**. Similarly, when the triborane(5) $Cl(Me_2N)B$–$BNMe_2$–$B(NMe_2)Cl$ is reacted with Me_3SnLi at low temperature the bis(trimethylstannyl)triborane **2a** is formed. In contrast, when the dibromide $Br(Me_2N)B$–$BNMe_2$–$B(NMe_2)Br$ is allowed to react with Me_3SnLi only small amounts of **2a** are formed; the main product being the cyclic isomer **2b**. Compounds **1a** and **2b** were characterized by X-ray crystal structure analysis. Compound **2b** has quite short B–Sn bonds. Oxidative addition of **1a** with ethylenebis(triphenylphosphane)platinum(0) yields *cis*-bis(trimethylstannyl)bis(triphenylphosphane)platinum(II) (**3**) besides trimethylstannyl[trimethylstannylbis(dimethylamino)diboranyl(4)]bis(triphenylphosphane)platinum **4**. These compounds were characterized by NMR spectroscopy.

Transition metal-boryl compounds have come into focus in recent years as they have been proposed as intermediates in a number of catalytic systems. Many well-characterized transition metal-boryl compounds are known today, and the state of the art has been reviewed several times.[2] Many of these complexes were obtained by oxidative addition of diborane(4) compounds to various metal-complex fragments. The oxidative addition of boron−halogen bonds[3] or boron−tin bonds[4] was also successful. So far, no reactivity scale has been developed, although it has recently been shown that the B−B bond of diborane(4) halides is more reactive than the boron−halogen bond. For example the reaction of $B_2Cl_2(NMe_2)_2$ or B_2F_4 with $(C_2H_4)Pt(PPh_3)_2$ leads to $[Pt(PPh_3)_2\{BCl(NMe_2)\}_2]$[5] and $[Pt(PPh_3)_2(BF_2)_2]$,[6] respectively. However, the formation of diborane(4)-1-yl derivatives is also known.[7] In our studies on silyl-, germyl- and stannylboranes, as well as polyboranes,[4,8−10] we became interested in the competition of B−Sn bonds vs. B−B bonds in the oxidative addition of stannyldiboranes(4) and stannyltriboranes(5). In preparing suitable starting materials we detected an interesting pair of isomers derived from a triborane(5) derivative, which we report here together with a new stannyldiborane(4).

Some time ago we described the preparation of $B_2(SnMe_3)_2(NMe_2)_2$ (**1a**) by treatment of $B_2Cl_2(NMe_2)_2$ with Me_3SnLi [Equation (1)].[8a] In analogy to **1a** we have now also obtained $B_2(SnPh_3)_2(NMe_2)_2$ (**1b**).

The ^{11}B NMR spectrum of **1b** shows a broad signal within the expected range at $\delta^{11}B = 61$ ($h_{1/2} = \approx 1500$ Hz). Two different signal sets for the NMe_2 groups were found in the ^1H and ^{13}C NMR spectra consistent with a rotation barrier about the B−N bonds resulting from strong B−N π-bonding. In contrast to **1a**, which is a liquid, **1b** could be obtained as single crystals suitable for an X-ray structure determination (Figure 1). The B−N bond lengths support the view that strong π-bonding is present in **1b** as indicated by an average B−N bond length of 1.384(3) Å. The planes made up of the atoms bonded to the N atoms are almost coplanar with the planes at the boron atoms to which the nitrogen atoms are attached: [B(1)−N(1) = 5.3°,



(1)

1a R = Me
1b R = Ph

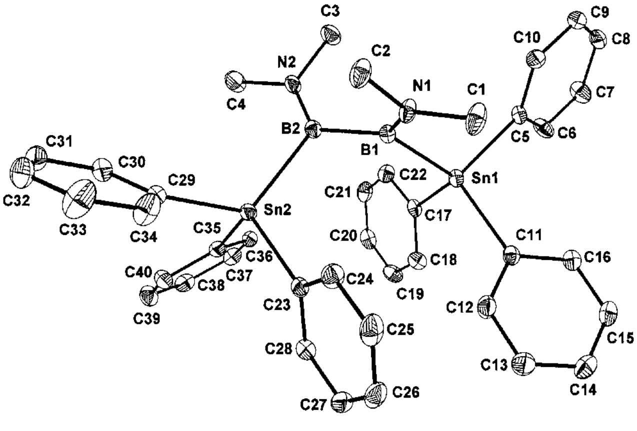


Figure 1. Molecular structure of **1b** in the crystal; hydrogen atoms omitted for clarity; thermal ellipsoids are drawn at a 25% probability level; selected bond lengths [Å] and bond angles [°]: Sn1−B1 2.276(3), Sn2−B2 2.282(3), N1−B1 1.383(3), N2−B2 1.384(3), B1−B2 1.677(4); N1−B1−B2 122.7(2), N1−B1−Sn(1) 118.9(2), B2−B1−Sn(1) 118.4(2), N2−B2−B1 125.7(2), N2−B2−Sn2 119.8(2), B1−B2−Sn2 114.4(2)

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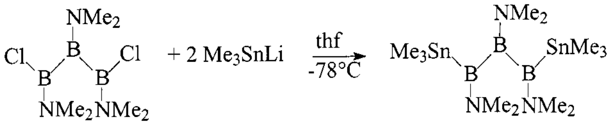
Table 1. NMR spectroscopic data for the isomers **2a** and **2b**

	2a	**2b**
$\delta^{11}B$	61.5 (2B) 54.7 (1B)	49.1 (2B) -51.1 (d, $^{1}J_{BSn} = 415$ Hz, 1B)
$\delta^{1}H$	0.30 (d, $^{2}J_{SnH} = 40.0$ Hz, 18 H) Sn*Me* 2.66 (s, 6 H) central N*Me*$_2$ 2.78 (s, 6 H) N*Me*$_2$ 2.88 (s, 6 H) N*Me*$_2$	0.42 (d, $^{2}J_{SnH} = 39.9$ Hz, 18 H) Sn*Me* 1.89 (s, 6 H) N*Me*$_2$ 2.12 (d, $^{5}J_{SnH} = 7.7$ Hz, 6 H) B$_2$N*Me*$_2$ 2.76 (s, 6 H) N*Me*$_2$
$\delta^{13}C$	-9.4 ($^{1}J_{SnC} = 196, 205$ Hz) Sn*Me* 45.6 central N*Me*$_2$ 48.3 ($^{3}J_{SnC} = 102$ Hz) N*Me*$_2$ 48.9 ($^{3}J_{SnC} = 85$ Hz) N*Me*$_2$	-5.3 ($^{1}J_{SnC} = 205$ Hz, $^{3}J_{SnC} = 19$ Hz) Sn*Me* 36.4 ($^{4}J_{SnC} = 30$ Hz) N*Me*$_2$ 37.2 ($^{4}J_{SnC} = 102$ Hz) N*Me*$_2$ 42.3 (broad) central N*Me*$_2$
$\delta^{119}Sn$	-152.0	-8.5 ($^{1}J_{BSn} = 415$ Hz)

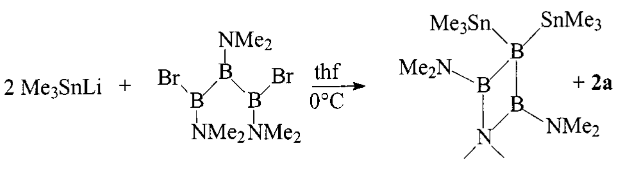
B(2)−N(2) = 2.9°]. The B−B−N−Sn planes of the two boron atoms are twisted relative to each other by 85.6°. Therefore, there is no electron delocalization across the B−B bond in this molecule although the molecule is isoelectronic and isolobal with a 1,3-butadiene. Rather, the space requirements of the substituents are taken care of by minimizing steric interactions, which results in the observed conformation. The B−B bond is a single bond although the B−B bond length [1.677(4) Å] is on the short side amongst the bond lengths known for diborane(4) molecules.[11] For the B−Sn bonds, atom distances of 2.282(3) Å and 2.276(3) Å were determined. These lengths are consistent with B−Sn bonds in molecules with minor steric hindrance.[1,10a]

The preparation of the corresponding triorganylstannyl triborane(5) compounds was much more complicated. The reaction of $Cl_2B_3(NMe_2)_3$ with two equivalents of Me_3SnLi affords compound **2a** in high yield [Equation (2)]. However, minor changes in the reaction conditions lead to a different product **2b** [Equation (3)] which proved to be an isomer of **2a**. Isomer **2b** is predominantly formed in the reaction of $Br_2B_3(NMe_2)_3$ with Me_3SnLi, whereas compound **2a** was formed only to an extent of about 30%. Compounds **2a** and **2b** can be separated and **2b** purified by fractional crystallization: This allowed an unequivocal assignment of their NMR spectroscopic data (listed in Table 1).



(2)

2a



(3)

2b

The acyclic triborane **2a** shows NMR spectra which are typical for symmetric, terminally substituted tris(dimethylamino)triborane(5) compounds. In the ^{11}B NMR spectrum two broad signals in the region for tricoordinate boron atoms are found.[12] The ^{1}H and ^{13}C NMR signals for the dimethylamino groups (three signal sets each) are consistent with a hindered rotation. The central amino group is represented by a single signal for symmetry reasons. These data point to the presence of strong B−N π-bonding. The ^{119}Sn NMR spectrum shows the expected[12] broad signal at $\delta^{119}Sn = -152$. In contrast, the NMR spectroscopic data of **2b** seemed, at first sight, not to be compatible with a triborane(5) framework, as a signal for a tetracoordinate boron atom was unexpected, and, as only one signal with double intensity for a tricoordinate boron atom was found, this NMR spectrum could only be explained by the presence of a tetracoordinate boron atom in the center of a chain of three boron atoms. The observed coupling constants: $^{1}J_{BSn} = 415$ Hz, $^{1}J_{CSn} = 205$ Hz and $^{3}J_{CSn} = 19$ Hz confirm that the tetracoordinate boron atom bears the $SnMe_3$ groups. The chemical shift of the central boron atom in **2b** is also unusual with $\delta^{11}{}_{B} = -51.1$. As the ^{1}H and ^{13}C NMR spectra indicate a high symmetry for the molecule the structure depicted for **2b** was deduced. However, the high-field shift of the tetracoordinate boron atom has so far no parallel as no boron atom carrying two boron and two tin atoms is known to date. The closest analogues for high-field shielding are $LiB(SiMe_3)_4$ [13] with $\delta^{11}B = -53.4$, $LiBH_3SnMe_3$ with $\delta^{11}B = -43.7$[8a] and $LiB(SnMe_3)_3$-NMePh with $\delta^{11}B = -13.1$.[1] This indicates that the central boron atom bears a significant negative charge. This assumption is confirmed by a signal at $\delta\delta^{119}Sn = -8.5$ as this Sn atom is deshielded by about 45 ppm relative to the tin atoms in the two stannyl-borates, and much more so than the tin atoms in isomer **2a.** The low-field singlet in the ^{1}H and ^{13}C NMR spectra of **2b** confirms the presence of a dimethylammonium group in a planar B_3N four-membered ring. Moreover, the remaining two signals for the dimethylamino groups fit with the suggested structure if they are coplanar with the ring. In this case the two methyl groups at each Me_2N group are nonequivalent, and, moreover, there is hindered rotation about these B−N bonds.

The structure derived from NMR spectroscopic data was fully confirmed by X-ray structure analysis: The molecular structure of **2b** is shown in Figure 2.

The striking key feature of the structure of **2b** is the 1-azonia-2,4-dibora-3-boratacyclobutane ring. The closest

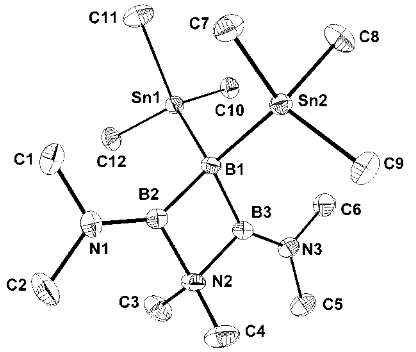


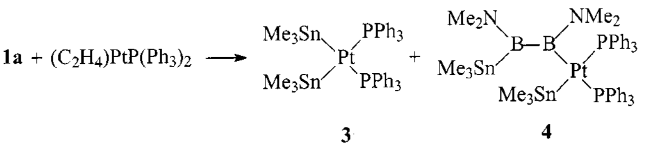
Figure 2. Molecular structure of **2b** in the crystal; hydrogen atoms are not depicted; thermal ellipsoids are drawn at a 25% probability level; selected bond lengths [Å] and bond angles [°]: Sn1−B1 2.223(3), Sn2−B1 2.225(3), N1−B2 1.382(4), N2−B2 1.605(4), N2−B3 1.603(4), N3−B3 1.379(4), B1−B2 1.660(4), B1−B3 1.662(5), N2−C3 1.483(4), N2−C4 1.498(4); Sn1−B1−Sn2 107.7(1), Sn1−B1−B2 116.6(2), Sn1−B1−B3 120.6(2), Sn2−B1−B2 116.7(2), Sn2−B1−B3 112.5(2), B2−B1−B3 81.3(2), B1−B3−N2 96.9(2), B1−B2−N2 96.9(2), B2−N2−B3 84.9(2)

other system for comparison would be an azapolyborane such as hypho-2,3-μ-(tBuNH)B_5H_{10} [14] which also has an alkylammonium fragment, although this compound is actually not best suited for a comparison with **2b**. The B_3N four-membered ring is exactly planar (angle sum = 360°). The intra-ring angles at the tetracoordinate atoms B1 and N1 are 81.3(2)° and 84.9(2)°, respectively. The compression at B1 is 12.6° (from tetrahedral symmetry) and for B2 and B3 38.7° (from trigonal-planar symmetry). Therefore, the four-membered ring is highly strained. In spite of this, the B−B bond lengths [av.: 1.661 Å] are almost unaffected as they are the same as in compound **1b**. They are actually only slightly longer than the B−N distances to the coordinated N atom [av.: 1.603 Å]. Thus, the expected increase in the B−B bond lengths that is normally observed upon changing to higher coordination is not found in this case. Therefore, the B−B distances in **2b** can be regarded as rather short. The sums of the bond angles at the nitrogen atoms N1 and N3 are 360° for both atoms and their bonding planes are coplanar with the B_3N ring. The two tricoordinate boron atoms are electronically stabilized by B−N double bonds as confirmed by the short B−N bonds. The two B−N bonds to the tetracoordinate N atom are single bonds. The B−Sn bonds are very short; they are the shortest found to date. Similar results were recently observed for B−Si[8d] and B−Ge[1] compounds. This indicates that for compounds of boron with the higher group 14 elements, a bond shortening with an increasing coordination number at the B atom seems to be a general rule.

Compound **2b** is, to the best of our knowledge, the first example of a tris(amino)triborane(5) in which the central B atom and not just the terminal boron atoms are doubly substituted. The mechanism by which **2b** is formed remains unclear. No equilibrium between the two isomers could be observed by NMR spectroscopy when a pure sample of either **2a** or **2b** was dissolved. The initial formation of **2a**, followed by a subsequent rearrangement to **2b**, can therefore be excluded. With regard to mechanistic speculations it is important to note that two conditions are in any case necessary for the formation of **2b**: The use of $Br_2B_3(NMe_2)_3$ as starting material, and reaction conditions which provide an initial excess of Me_3SnLi. This might indicate that the first step of the formation of **2b** could be a double substitution at one of the terminal boron atoms followed by a rearrangement of the substituents. The final formation of an azonia-bridged cyclic structure is also very likely. Anderson et al.[15] recently reported the favored formation of various 1-azonia-2-borata-5-borol compounds also bearing tri- and tetracoordinate boron and nitrogen atoms.

As we intended to explore whether the B−B or the B−Sn bond is more reactive towards transition metal centers, compound **1a** was reacted with $(C_2H_4)Pt(PPh_3)_2$. However, the product formed was not either of the expected B−B or B−Sn oxidative addition products but rather $(Me_3Sn)_2Pt(PPh_3)_2$ (**3**).[16]

The formation of **3** also requires the formation of a compound like $[B(NMe_2)]_n$, but the only other boron-containing compound which could be isolated was a small quantity of the diborane(4)-1-yl platinum compound **4** [Equation (4)].



(4)

The structure of **4** was derived from its NMR spectroscopic data. The ^{11}B NMR spectrum of compound **4** shows two signals at $\delta^{11}B = 59.8$ ($h_{1/2} = 480$ Hz) and $\delta^{11}B = 47.0$ ($h_{1/2} = 1840$ Hz). While the latter signal is almost the same as for the starting material, the signal of the boron atom bonded to the platinum atom is found in the range of platinum-boryl complexes.[9b] Apart from this, the B−Pt bond is confirmed by a very large half-width of its ^{11}B NMR signal of 1840 Hz. In the ^{31}P NMR spectrum two signal sets at $\delta^{31}P = 32.9$ [$^{1}J(^{31}P^{195}Pt) = 2895$ Hz; $^{2}J_{PP} = 12$ Hz; $^{2}J(^{31}P^{119}Sn) = 1364$ Hz, $^{2}J(^{31}P^{117}Sn) = 1304$ Hz] and $\delta^{31}P = 31.2$ [$^{1}J(^{31}P^{195}Pt) = 1656$ Hz; $^{2}J(^{31}P^{117}Sn) = 142$ Hz] are found proving the *cis* position of the phosphane groups in **4**. As this compound was obtained only in poor yield, attempts to grow single crystals failed. Nevertheless, the formation of **4** indicates that the B−Sn bonds in **1a** are more reactive than the B−B bonds.

Experimental Section

Experimental conditions, spectrometers and diffractometer are as described in previous contributions.[1]

1,2-Bis(triphenylstannyl)-1,2-bis(dimethylamino)diborane(4) (1b): Ph_3SnLi (2.9 mmol) was added to 50 mL of THF.[17] The solution was cooled to −78 °C and slowly treated with a solution of 1,2-dichloro-1,2-bis(dimethylamino)diborane(4)[18] (0.26 g, 1.44 mmol) in 25 mL of hexane. After stirring for 30 min. at ambient temperature all volatile components were removed in vacuo. The solid residue was treated with 50 mL of toluene and the resulting suspension filtered. The volume of the filtrate was reduced in vacuo to 30 mL and then layered with 30 mL of hexane. This afforded, after some days standing at room temp., 0.81 g (71%) of **1b** as colorless crystals, m.p.: 119 °C. − ^{1}H NMR (400 MHz, C_6D_6): δ = 2.65 (s, 6 H, C*H*$_3$), 2.80 (s, 6 H, C*H*$_3$), 7.17−7.22 (m, 18 H, *m*-Ph-*H* and *p*-Ph-*H*), 7.58 (ddd, $^3J_{HH}$ = 5.9 Hz, $^4J_{HH}$ = 2.5 Hz, $^3J_{HSn}$ = 39.4 Hz, 12 H, *o*-Ph-*H*). − ^{11}B NMR (64 MHz, C_6D_6): δ = 60.8 ($h_{1/2}$ = 1500 Hz). − ^{13}C NMR (100 MHz, C_6D_6): δ = 47.6 (N*C*H$_3$), 49.3 (N*C*H$_3$), 128.5 (t, J_{CSn} = 39.1 Hz, *C*-arom.), 128.9 (t, J_{CSn} = 30.2 Hz, *C*-arom.), 137.6 (t, J_{CSn} = 39.1 Hz, *C*-arom.), 137.8 (t, J_{CSn} = 36.9 Hz, *C*-arom.), 139.2 (*C*-arom.), 142.4 (*C*-arom.). − ^{119}Sn NMR (149 MHz, C_6D_6): no signal observed. − $C_{40}H_{42}B_2N_2Sn_2$ (809.7): calcd. C 59.32, H 5.23, N 3.46; found C 58.71, H 5.23, N 3.32.

1,2,3-Tris(dimethylamino)-1,2-bis(trimethylstannyl)triborane(5) (2a): 1,3-Dichloro-1,2,3-tris(dimethylamino)triborane(5)[12] (2.31 g, 9.8 mmol) was dissolved in 30 mL of hexane. The solution was then cooled to −78 °C and treated dropwise with a THF solution of Me_3SnLi (19.6 mmol).[19] After stirring for 12 h at ambient temperature all volatile components were removed in vacuo. The solid residue was then treated with 30 mL of toluene. After filtration the solution was reduced in volume to 20 mL and then cooled at −78 °C. This afforded 3.18 g (66%) of **2a** as a colorless, polycrystalline solid, m.p.: 51−53 °C. For spectroscopic data see Table 1. − $C_{12}H_{36}B_3N_3Sn_2$ (492.3): calcd. C 29.28, H 7.37, N 8.54; found C 29.64, H 7.55, N 8.75.

1-Azonia-2,4-dibora-3-borata-2,4-bis(dimethylamino)-3-bis(trimethylstannyl)-1-dimethylcyclobutane (2b): A stirred solution of Me_3SnLi[19] (5.2 mmol) in THF was cooled to 0 °C and treated with 1,3-dibromo-1,2,3-tris(dimethylamino)triborane(5)[12] (0.86 g, 2.6 mmol) in 30 mL of toluene. After stirring for 5 h at ambient temperature a suspension had been formed, and the solution showed an ^{11}B NMR spectrum with signals at $δ^{11}$B = 66.5, 56.9, 50.4, 46.5, 36.2, −33.2 ($h_{1/2}$ = 300 Hz), and −49.8 (d, $^1J_{BSn}$ = 415 Hz). Those at δ = 66.5 and 36.2 are assigned to compound **2a**, and those at δ = 46.5 and −49.8 to **2b**. The signals at δ = 56.9, 50.4 and −33.2 could not be assigned. As the signals were broad and not well separated, integration was not possible, but the ratio of **2a** and **2b** can be roughly estimated as 6:3. All volatile components were removed from the suspension in vacuo. The residue was treated with 50 mL of hexane. Cooling the filtrate to −78 °C afforded 0.8. g (62%) of **2b** as colorless prisms. m.p.: 110 °C. − Spectroscopic data see Table 1. − $C_{12}H_{36}B_3N_3Sn_2$ (492.3): calcd. C 29.28, H 7.37, N 8.54; found C 28.52, H 7.09, N 8.07.

Reaction of 1a with ($η^2$-Ethene)bis(triphenylphosphane)platinum(0): Compound **1a** (0.41 g, 1.0 mmol) was dissolved in about 40 mL of toluene and treated with ($η^2$-ethene)bis(triphenylphosphane)platinum(0).[20] After stirring for 12 h at ambient temperature, a brownish precipitate had been formed. The precipitate was filtered, washed with hexane and crystallized from $CHCl_3$/hexane. This afforded colorless crystals, which were identified as **3**.[16] The filtrate was reduced in volume to 20 mL and layered with 20 mL of hexane. Within a few days about 0.03 g of a brown solid separated which was identified as **4** by NMR spectroscopy. Yield: 0.96 g of **3** (92%).
3: ^{31}P (109 MHz, −60 °C, C_6D_6): δ = 32.5 (dd, $^1J_{PPt}$ = 3774 Hz, $^2J_{SnP}$ = 646 Hz). − ^{119}Sn NMR (100 MHz, $CDCl_3$): δ = −13.2 (d, $^2J_{PSn}$ = 646 Hz).
4: ^{1}H (270 MHz, C_6D_6): δ = −0.51 [3 H, Sn(C*H*$_3$)$_3$], 0.29 [3 H, Sn(C*H*$_3$)$_3$], 0.39 [Sn(C*H*$_3$)$_3$], 0.67 [3 H, Sn(C*H*$_3$)$_3$], 0.89 [6 H, Sn(C*H*$_3$)$_3$], 2.56 (s, 3 H, NC*H*$_3$), 2.72 (s, 3 H, NC*H*$_3$), 2.76 (s, 3 H, NC*H*$_3$), 2.86 (s, 3 H, NC*H*$_3$), 6.93 (br. m, 18 H, Ph-*H*), 7.50 (br. m, 12 H, Ph-*H*); − ^{31}P (109 MHz, C_6D_6): δ = 32.9 ($^1J_{PPt}$ = 2895 Hz, $^2J_{PP}$ = 12 Hz, $^2J_{P^{119}Sn}$ = 1364 Hz, $^2J_{P^{117}Sn}$ = 1304 Hz, $P_{cis\mathrm{B}}$), 31.2 (broad, $^1J_{PPt}$ = 1656 Hz, $^2J_{PSn}$ = 142 Hz, $P_{trans\mathrm{B}}$); − ^{11}B (64 MHz, C_6D_6): δ = 47.0 ($h_{1/2}$ = 1840 Hz, *B*Pt), 59.8 ($h_{1/2}$ = 480 Hz, *B*Sn).

Crystal Structure Determination: A suitable single crystal was covered with perfluoroether oil, mounted on the end of a glass fiber and placed on a goniometer head. The crystal was cooled to −80 °C with a cold stream of dinitrogen using an LT2 device. The unit cell was determined from the reflections of five series of 15 frames each taken at different angles and sweeping φ by 0.3°. A Siemens P4 diffractometer equipped with an area detector and graphite-monochromated Mo-$K_α$ radiation was used. Data collection was performed with the hemisphere mode of the SMART programs.[21] Data were reduced by SAINT[22] and an absorption correction applied with the program SADABS.[23] Structure solution and refinement was carried out with the SHELX-97 program suite. Non-hydrogen atoms were refined anisotropically, and hydrogen atoms were placed in calculated positions and included in the refinement as riding on the respective C atoms. Crystallographic and structure solution data are summarized in Table 2.
Crystallographic data (excluding structure factors) for the structures included in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-157308 (**1b**) and -157309 (**2b**). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road,

Table 2. Crystallographic data for **1b** and **2b**

	1b	**2b**
Chem. formula	$C_{40}H_{42}B_2N_2Sn_2$	$C_{12}H_{36}B_3N_3Sn_2$
Form. wt.	809.76	492.25
Cryst. size [mm]	0.10 × 0.20 × 0.20	0.10 × 0.10 × 0.40
Cryst. system	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/n$
a, [Å]	11.7751(9)	9.9514(8)
b, [Å]	22.525(2)	14.074(1)
c, [Å]	14.736(1)	15.665(1)
α, [°]	90	90
β, [°]	104.085(1)	90.797(1)
γ, [°]	90	90
V, [Å^3]	3790.8(5)	2193.7(3)
Z	4	4
ρ (calcd.), [Mg/m^3]	1.419	1.490
μ [mm^{-1}]	1.346	2.272
F(000)	1624	976
Index range	$-14 \le h \le 16$ $-28 \le k \le 27$ $-19 \le l \le 18$	$-12 \le h \le 12$ $-18 \le k \le 17$ $-19 \le l \le 18$
2 θ [°]	58.26	58.16
Temp, [K]	183(2)	193(2)
Refl. collected	21768	12423
Refl. unique	7694	4396
Refl. observed (4σ)	6305	3803
R (int.)	0.0240	0.0266
No. variables	419	193
Weighting scheme[a] x/y	0.033/0.237	0.043/0.155
GOOF	1.035	1.055
Final *R* (4s)	0.0242	0.0271
Final *wR*2	0.0561	0.0676
Larg. res. peak [e/Å^3]	0.249	0.492

[a] $w^{-1} = s^2F_o^2 + (xP)^2 + yP$; $P = (F_o^2 + 2F_c^2)/3$.

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