Reactions of *cis*-Stannyl-boryl-alkenes with Sulfur Bis(trimethylsilylimide) and *N*-Sulfinyl(trimethylsilyl)amine – X-ray Analysis of an 1-Amino-1λ⁴-thia-2-azonia-3-borata-1,4-cyclopentadiene

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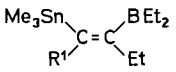
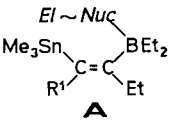
New heterocyclic systems (1-amino-1λ⁴-thia-2-azonia-3-borata-1,4-cyclopentadienes **4a** and **4b**, a 1λ⁴-thia-2-aza-3-bora-1-cyclopentanone **7** and a 1-trimethylstannyloxy-1λ⁴-thia-2-azonia-3-borata-1,4-cyclopentadiene **8**) are formed by reaction of (*E*)-2-diethylboryl-1-trimethylstannyl-1-butene (**1a**) and (*E*)-3-diethylboryl-2-trimethylstannyl-2-pentene (**1b**) with sulfur bis(trimethylsilylimide) (**2**) and *N*-sulfinyl(trimethylsilyl)amine (**3**). There is a marked influence of other substituents at the C=C bond on the product distribution. It appears that all reactions proceed via an intermediate borane adduct **A**. In **A** the proximity of the electrophilic site of **2** or **3** to various reactive sites of the alkene derivative opens the way to the final products. The proposed structures are supported by consistent ^{1}H-, ^{11}B-, ^{13}C-, ^{29}Si- and ^{119}Sn-NMR data. In the case of compound **4a** an X-ray analysis has been carried out.

Organometallic-substituted alkenes offer numerous reactive sites for useful transformations. Organometallic substituents in *cis* position at the C=C bond may exert a combined influence on the reactivity of the alkene. Alkene derivatives of type **1** with *cis* arrangement are readily accesible by organoboration of alkynyltin compounds[1]. The combined effect of substituents such as a boryl and a stannyl group is particularly attractive for studying the reactivity of **1** towards reagents which contain at least one nucleophilic and one electrophilic center (Nuc~El).

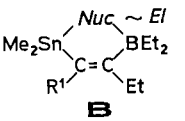


1	**a**	**b**
R^1	H	Me

In the weak borane adduct **A** the respective reactive centers can be in close contact. The result of the following intramolecular processes then depends mainly on R^1, the nature of the reagent used and, possibly, on the reaction conditions, e.g. on the solvent[2]. We have already shown that lithium amides[3], tin amides[2,3], tin alkoxides[2] or methanol[2] are useful reagents in this respect. Similarly, new heterocyclic systems are obtained when the reagent Nuc~El is linked first to the tin atom by nucleophilic displacement of Cl^- (**B**) as shown in the reaction of sulfur diimide anions with (*E*)-2-chloro(dimethyl)stannyl-3-diethylboryl-2-pentene[4]. In this paper, we report on the reaction of **1** with sulfur bis(trimethylsilylimide) (**2**) and *N*-sulfinyl(trimethylsilyl)amine (**3**). In these compounds[5] the nitrogen atoms, or possibly also the oxygen atom, are the nucleophilic centers, and the sulfur atom represents the electrophilic center in Nuc~El.



A



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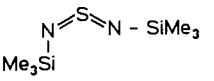
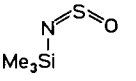


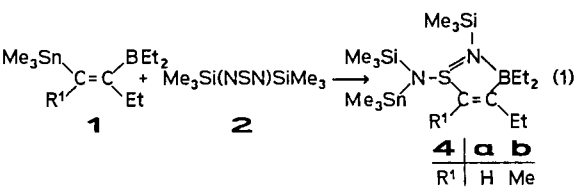
2



3

Results and Discussion

A smooth reaction takes place between **1a** and **2** in hexane when the reaction mixture is warmed from −78°C to ambient temperature. This process affords the 1-amino-1λ⁴-thia-2-azonia-3-borata-1,4-cyclopentadiene **4a** in almost quantitative yield (Eq. 1). Under the same conditions **4b** is also formed, but in a much lower yield (30%) together with numerous unidentified products. The structures of **4a** and **4b** are based on a consistent set of NMR data (see Table 1), and in the case of **4a** suitable crystals for X-ray analysis (vide infra) have been obtained.

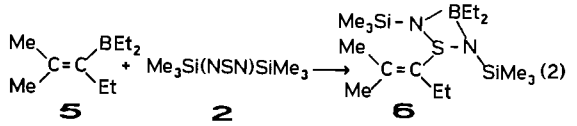


(1)

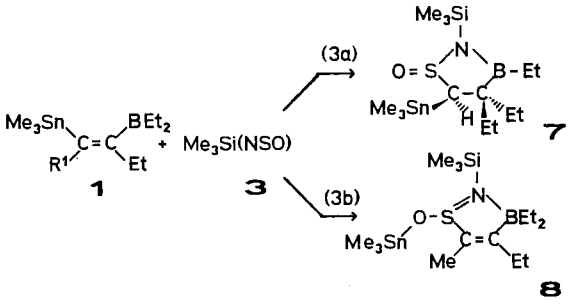
4	**a**	**b**
R^1	H	Me

  0009–2940/93/0303–0595 $ 10.00+.25/0

In the absence of the Me_3Sn group, the reaction takes a different course as shown for the alkenylborane **5** (Eq. 2). Again, one can assume the formation of a weak borane adduct analogous to A, followed by cleavage of the B–C= bond to give **6**.



The behavior of the *N*-sulfinylamine **3** in its reaction with **1a** is rather different from that of **2**. Under the same conditions, compound **3** reacts readily with **1a** to give a $1\lambda^4$-thia-2-aza-3-bora-1-cyclopentanone derivative **7** (Eq. 3a). However, the reaction of **3** with **1b** affords a 1-trimethylstannyloxy-$1\lambda^4$-thia-2-azonia-3-borata-1,4-cyclopentadiene **8**, analogous to **4b** (Eq. 3b). Starting from **A**, the attack at the C=C bond is accompanied by a 1,2-shift of an ethyl group from the boron to the neighbored olefinic carbon atom, leading to **7**. This type of reaction has also been observed in the treatment of **1a** with Me_3SnOMe or Me_3SnNMe_2 (in hexane)[2], and there are indications that this is the kinetically controlled process. The fast electrophilic attack at the C=C bond is hampered if R^1 = Me. Then the intermediate formation of the N–borane adduct is followed by electrophilic cleavage of the Sn–C= bond (as in the case of **4a**, **b**) to give the heterocycle **8**.



It appears that differing kinetic effects are important and that the sulfur atom in **3** is more electrophilic than in **2**. Thus, there is no indication of a reaction between **1a** and **2** according to Eq. 3b. This points to the influence of kinetic effects which can be expected in view of the difference between an S=O and an S=N–$SiMe_3$ fragment. Since the reaction between **1b** and **2** leads to numerous products (possibly including one analogous to **6**), these side reactions compete efficiently with the electrophilic attack at the Sn–C= bond. In contrast, steric hindrance to the clean reaction of **1b** with **3** is weaker than with **2**, and the electrophilic character of the sulfur atom in **3** is sufficiently high for preferred cleavage of the Sn–C= bond. There are reactions of organotin compounds (mostly with functional groups linked to tin) with sulfur diimides or *N*-sulfinylamines described in the literature[6]. In some of these the Sn–C bond is cleaved, and an organyl group migrates to the sulfur atom, comparable to the reaction in Eq. 1. However, the dependence of the reactions on the substituents R^1 (e.g. formationof **7** or **8**) indicates that the combined action of the boryl and stannyl groups is responsible for the reaction pathways reported here.

The compounds **4a**, **b**, **7** and **8** are colorless, crystalline solids which can be purified by repeated recrystallization. Compound **6** is a yellowish liquid (purity >90%), and so far we have failed to purify it by distillation, chromatography (decomposition) or crystallization at low temperature (–78°C). All products are extremely sensitive to traces of moisture which have caused severe problems with the application of other analytical methods, e.g. solid-state ^{13}C-, ^{29}Si- or ^{119}Sn-CP/MAS NMR spectroscopy of **4a**, **b**, **7** and **8**.

NMR Spectra

NMR data (Table 1) are fully in accord with the proposed structures of **4a**, **b**, **6** and **8**. In these compounds the tetracoordinate boron atom is evident from the typical[7,8] $\delta^{11}B$ values ($\delta^{11}B$ = 7–11). The ^1H- and ^{13}C-NMR spectra are readily assigned. Figure 1 shows the ^{13}C-NMR spectrum of compound **7** as a typical example with broadened ^{13}C resonance signals for boron-bound carbon atoms[8] and $^{117/119}Sn$ satellites arising from $J(Sn^{13}C)$. The $\delta^{29}Si$ values are in agreement with the presence of Me_3SiN groups[9], and the differing ^{29}Si environments in **4a**, **b** are assigned according to the geminal coupling constant $^2J(SnN^{29}Si)$[10]. In the case of **4a** and **4b**, the $\delta^{119}Sn$ values ($\delta^{119}Sn$ = 72.9, 73.6) are typical of

Table 1. ^{11}B-, ^{13}C-, ^{29}Si- and ^{119}Sn-NMR data[a] of the heterocycles **4a**, **4b**, **7** and **8**

	$\delta^{13}C$								
	C-4	C-5	N(SiMe3)(SnMe3) O-SnMe3	BNSiMe3	BEt	$\delta^{11}B$	$\delta^{29}Si$	$\delta^{119}Sn$
4a	188.2 [br] 25.5 12.3	119.2 [< 2]	3.6, 0.5 [373.0]	3.1	19.3,12.3 [br] 19.2,10.7[b] [br]	+7.1	+6.7 +11.9 [8.8]	+72.9
4b	177.0 [br] 24.2 12.4[c]	125.7 [10.2] 12.8[c]	3.8, -0.5 [371.9]	3.4	18.8,11.3 [br] 14.8,10.8[b] [br]	+8.7	+6.6 +12.3 [9.3]	+73.6
7	52.1 [br] 34.4[d] [35.0] 31.3[d] [34.5]	66.2 [288.2] -6.5 [344.7]	- -	1.8	10.3, 9.1 [br]	+56.5	+10.4	-20.2
8	178.3 [br] 23.0 12.0	129.6 [< 2] 12.6	-1.4 [390.0]	2.5	16.7,12.0 [br] 15.9, 10.7[b] [br]	+8.7	6.9	+165.6

[a] Measured in 5-mm (o.d.) tubes at 25°C in C_6D_6 (ca. 10%); coupling constants $J(^{119}Sn^{13}C)$, $^2J(^{119}Sn^{29}Si)$ in Hz are given in square brackets; [br] denotes the broad ^{13}C resonance signal of a boron-bound carbon atom. – [b] The assignment of the $^{13}C(CH_3)$ resonance signals may be reversed. – [c] The assignment of the $^{13}C(=CCH_3)$ and $^{13}C(CH_2CH_3)$ resonance signals may be reversed. – [d] $\delta^{13}C(CH_2CH_3)$ = 12.7, 11.2.

Me_3SnN groups, and in the case of **8**, the deshielding of ($\delta^{119}Sn = +165.6$) indicates the Me_3Sn-O linkage[11]. In contrast, substantial shielding of ^{119}Sn in **7** ($\delta^{119}Sn = -20.2$) is expected for a tin atom surrounded by four alkyl groups[11], in agreement with the observation of the coupling constants $^1J(Sn^{13}C)$ in the ^{13}C-NMR spectrum (Figure 1).

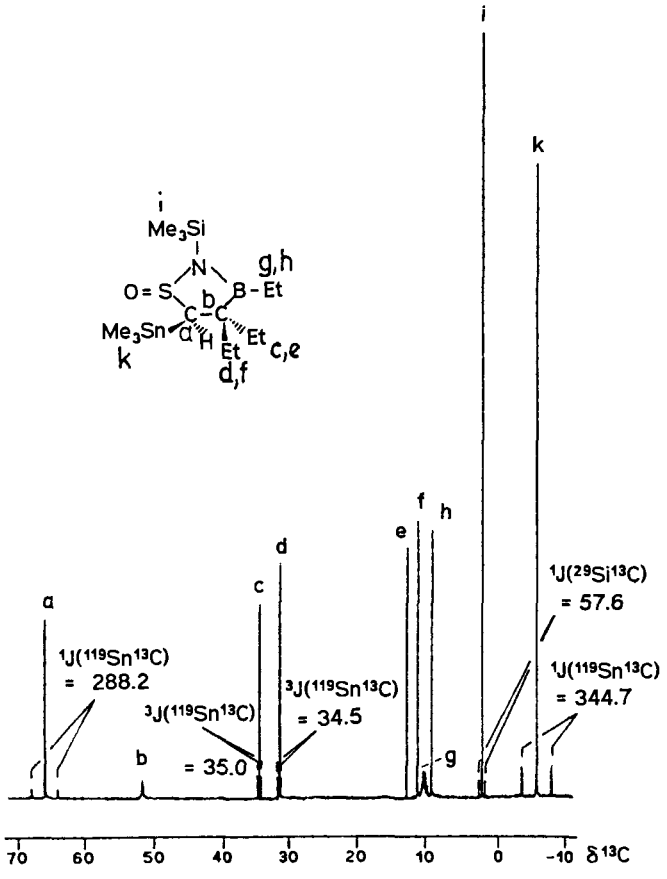
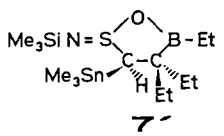
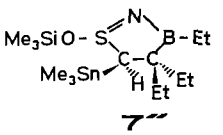


Figure 1. 75.5-MHz $^{13}C\{^1H\}$-NMR spectrum of **7** in C_6D_6 (coupling constants are given in Hz); note the $^{117/119}Sn$ satellites as indicated and the broad ^{13}C resonance signals for the boron-bound carbon atoms labeled b and g

The proposed structure of **7** does not follow unequivocally from the NMR data. Although the $\delta^{11}B$ value (56.8) clearly indicates the trigonal planar surrounding of the boron atom, the $\delta^{11}B$ value could, in principle, also be assigned to isomers of **7** (**7′** or **7″**).



7′



7″

The formation of **7′** would either require the O–borane adduct in the first step of the reaction or a rearrangement of **7**. Structure **7″** results from **7** by a 1,3-migration of the Me_3Si group. However, the deshielding of ^{13}C-5 ($\delta^{13}C$-5 = 66.2) and of 5-^1H ($\delta^1H = 2.80$) is in keeping with the sulfoxide moiety in **7**. If structure **7″** with a Me_3SiO group was correct, the ^{29}Si resonance signal should be located at significantly higher frequency.

X-ray Analysis of Compound 4a

Experimental data of the X-ray analysis are given in Table 2[12]. Selected bond distances and bond angles are given in Figure 2, and Table 3 contains atomic coordinates and equivalent isotropic displacement factors. The five-membered ring is almost planar (mean deviation 2.7 pm). The surrounding of N(2) is exactly trigonal planar, whereas for N(1) a pyramidal geometry is observed where the N(1)–Si(1) bond forms an angle of ca. 20° with the mean plane of the ring. The bond lengths are all in the expected range. There is little difference between the two values $d_{S-N(1)}$ [162.7(4) pm] and $d_{S-N(2)}$ [165.4(4) pm] which are both in between the ranges for S–N double and S–N single bonds. This indicates delocalization of the formal positive charge over the N–S–N moiety. In other molecular structures of comparable compounds[4,13], it has been observed that both *B*-ethyl groups are bent towards the ring. In the case of **4a**, the bulkiness of the $Me_3SnN(2)$ group appears to prevent this particular arrangement of one of the *B*-ethyl groups. Similarly, the arrangement of the $Me_3Si(Me_3Sn)N(2)$ group, where the Si(Sn)NS plane almost bisects the heterocycle, is enforced by steric constraints.

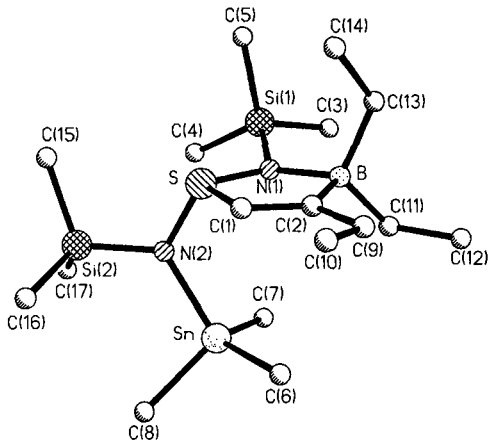


Figure 2. Molecular structure of **4a** (H atoms are not shown for simplicity); selected bond distances [pm] and angles [°] Sn–N(2) 210.3(4), Sn–C(7) 211.4(5), Si(1)–N(1) 175.5(4), Si(1)–C(4) 185.4(5), Si(2)–N(2) 180.2(4), Si(2)–C(16) 186.2(5), S–N(1) 162.7(4), S–C(1) 174.2(5), B–C(2) 162.3(6), B–C(13) 162.1(8), C(2)–C(9) 151.1(8), Sn–C(6) 211.3(5), S–N(2) 165.4(4), N(1)–B 163.8(7), B–C(11) 163.9(8), C(1)–C(2) 132.9(7), C(9)–C(10) 151.1(7), C(13)–C(14) 154.6(7); N(1)–S–N(2) 111.8(2), N(2)–S–C(1) 105.1(2), Si(1)–N(1)–B 125.0(3), Sn–N(2)–Si(2) 121.1(2), B–C(2)–C(1) 115.2(4), C(1)–C(2)–C(9) 122.3(4), B–C(11)–C(12) 117.7(4), N(1)–S–C(1) 96.1(2), Si(1)–N(1)–S 115.6(2), S–N(1)–B 114.2(3), Sn–N(2)–S 121.7(2), N(1)–B–C(2) 99.8(4), S–C(1)–C(2) 114.3(3), B–C(2)–C(9) 122.4(4), B–C(13)–C(14) 116.0(4)

Conclusions

Alkene derivatives of type **1** have again proved useful in reactions with reagents of the type Nuc~El. The structure of all the products obtained can be traced to the intermediacy of a weak borane adduct **A**, consistent with previous

studies of the reactivity of **1**[2,3]. The dependence of the product distribution on kinetic and electronic effects is fascinating since rather subtle changes induce a completely different course of the reaction (e.g., Eq. 3a and 3b). Considering the enormous diversity of systems with cumulated double bonds, similar to **2** and **3**, the reactions introduced here are promising for the synthesis of a large number of new heterocyclic systems.

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Experimental

All reactions and handling of samples for measurements were carried out under N_2 using carefully dried solvents and observing all precautions to exclude air and moisture. – Elemental analyses: Dornis and Kolbe, Mülheim and Pascher, Remagen. – EI MS: Varian MAT CH 7; 70 eV, direct inlet. – ^1H and ^{13}C NMR: Bruker AC 300 (300.13 and 75.5 MHz, respectively). – ^{11}B NMR: Jeol FX 90 Q (28.7 MHz) and Bruker AC 300 (96.3 MHz); $Et_2O \cdot BF_3$ (external). – ^{29}Si NMR [refocused INEPT pulse sequence based on $^2J(^{29}SiC^1H) = 7$ Hz]: Bruker AC 300 (59.7 MHz), Jeol FX 90 Q (17.8 MHz); Me_4Si (external). – ^{119}Sn NMR: Bruker AC 300 (111.9 MHz), Jeol FX 90 Q (33.3 MHz); Me_4Sn (external). – X-ray analysis: Nicolet R3m/V. – Starting materials: The alkene derivatives **1a** and **1b**[14] and the sulfur diimide **2**[15] were prepared according to literature procedures.

3,3,4-Triethyl-2-trimethylsilyl-1-[trimethylsilyl(trimethylstannyl)amino]-1λ⁴-thia-2-azonia-3-borata-1,4-cyclopentadiene (**4a**) *and 3,3,4-Triethyl-5-methyl-2-trimethylsilyl-1-[trimethylsilyl(trimethylstannyl)amino]-1λ⁴-thia-2-azonia-3-borata-1,4-cyclopentadiene* (**4b**): A solution of 1.03 g (5.00 mmol) of the sulfur diimide **2** in 50 ml of hexane was cooled to −78 °C. The alkene derivative **1a** (0.93 g, 5.00 mmol) was added in one portion, and the stirred mixture was warmed to room temp. and stirred for 5 h. After the solvent was removed, the residue was recrystallized from hexane or hexane/toluene (10:1) to give 2.00 g (81%) of compound **4a** as colorless crystals (m.p. 122 °C). – The same procedure was applied to the preparation of **4b**. However, the yield was lower (30%), and recrystallization had to be repeated several times in order to obtain colorless needles of **4b** (m.p. 125 °C).

4a: ^1H NMR (C_6D_6, 25 °C): δ [$J(^{119}Sn^1H)$] = 0.12 (s, 9H; $SiMe_3$); 0.26 [57.2] (s, 9H; $SnMe_3$); 0.27 (s, 9H, $SiMe_3$); 0.40–1.20 (m, 13H; BEt_2, CH_3); 2.26 (m, 2H; $=CCH_2$); 5.29 (s, 1H; =CH).

$C_{17}H_{43}BN_2SSi_2Sn$ (493.3)
Calcd. C 41.39 H 8.79 N 5.68 S 6.50 Si 11.39 Sn 24.06
Found C 41.22 H 8.63 N 5.82 S 6.55 Si 11.16 Sn 25.21

4b: ^1H NMR (C_6D_6, 25 °C): δ [$J(^{119}Sn^1H)$] = 0.15 (s, 9H; $SiMe_3$); 0.31 (s, 9H, $SiMe_3$); 0.32 [57.6] (s, 9H; $SnMe_3$); 0.65 (m, 4H), 1.08 (t, 6H) (BEt_2); 1.22 (t, 3H; CH_3); 1.66 (s, 3H; $=CCH_3$); 2.17 (m, 2H; $=CCH_2$).

$C_{18}H_{45}BN_2SSi_2Sn$ (507.3) Calcd. C 42.60 H 8.94 N 5.52
Found C 42.15 H 8.81 N 5.25

3,3-Diethyl-1-(1-ethyl-2-methyl-1-propenyl)-2,4-bis(trimethylsilyl)-1-thionia-2,4-diaza-3-boratacyclobutane (**6**): The same procedure was used as for the synthesis of **4a, b**. A yellowish oily liquid was left after the solvent had been removed. Attempts at purification of **6** by distillation or chromatography [Al_2O_3 (various grades), silica gel] led to decomposition, and the compound did not crystallize from pentane at −78 °C. – ^1H NMR (C_6D_6, 25 °C): δ = 0.06 (s, 18H; $SiMe_3$); 0.65 (m, 4H), 1.22 (t, 6H) (BEt_2); 1.15 (t, 3H; CH_3); 1.49 (s, 3H; $=CCH_3$), 1.82 (s, 3H; $=CCH_3$). – ^{13}C NMR: δ = 1.0 ($SiMe_3$); 22.7 [br], 10.7 (BEt_2); 22.4, 14.5 ($=CCH_3$); 15.9, 16.1 ($=CCH_2CH_3$); 145.1 (=CS); 146.1 [$(CH_3)_2C=$]. – ^{11}B NMR: δ = 10.8. – ^{29}Si NMR: δ = 0.7.

$C_{16}H_{39}BN_2SSi_2$ (360.6) Calcd. C 53.30 H 10.90 N 7.77
Found C 52.55 H 9.98 N 7.10

Table 2. Data of the crystal structure analysis of compound **4a**[12]

Temperature: 115 K; crystal size: 0.31 · 0.23 · 0.19 mm^3; space group: $P2_1/c$; Z = 4; a = 1420.3 (4), b = 1113.9 (3), c = 1653.2 (5), β(°) = 103.58 (2); V = 2542.0 (1) × 10^{-30} m^3; ρ (calcd.) = 1.289 g/cm^3; μ = 1.18 mm^{-1}; Mo-Kα radiation (graphite monochromator): λ = 71.069 pm; 4344 independent reflections, 3000 observed ($F_0 \geq 4\sigma(F)$); 2Θ range: $3 \leq 2\Theta \leq 50°$; no absorption correction; no extinction correction; number of parameters 278; R = 0.028, R_W = 0.028 [$w^{-1} = (\sigma^2(F_0) + 0.0005 \cdot F_0^2)$]; residual electron density: −0.490 e/Å³ (minimum) and 0.919 e/Å³ (maximum).

Table 3. Atomic coordinates (× 10^4) and equivalent isotropic displacement factors (Å² × 10^3)

	x	y	z	U_{eq}
Sn	3827(1)	2058(1)	5696(1)	19(1)*
Si(1)	2386(1)	-6(1)	7604(1)	16(1)*
Si(2)	1988(1)	3697(1)	6138(1)	8(1)*
S	1754(1)	1067(1)	5995(1)	13(1)*
N(1)	2360(3)	-19(3)	6538(2)	12(1)*
N(2)	2449(3)	2243(3)	5947(2)	13(1)*
B	2467(4)	-1220(5)	5995(3)	16(2)*
C(1)	1601(3)	362(4)	5032(3)	14(2)*
C(2)	1992(3)	-726(4)	5065(3)	16(2)*
C(3)	3318(4)	-1066(5)	8169(3)	29(2)*
C(4)	2717(4)	1513(5)	8036(3)	30(2)*
C(5)	1203(3)	-394(5)	7820(3)	26(2)*
C(6)	3793(4)	980(5)	4638(3)	26(2)*
C(7)	4771(4)	1442(5)	6803(3)	30(2)*
C(8)	4229(4)	3817(5)	5397(3)	27(2)*
C(9)	1933(4)	-1483(5)	4297(3)	24(2)*
C(10)	1273(4)	-1003(5)	3511(3)	35(2)*
C(11)	3612(4)	-1600(4)	6171(3)	20(2)*
C(12)	3868(4)	-2726(5)	5745(4)	40(2)*
C(13)	1799(4)	-2297(4)	6213(3)	20(2)*
C(14)	693(4)	-2088(5)	5955(3)	30(2)*
C(15)	817(4)	3517(5)	6470(3)	31(2)*
C(16)	1672(4)	4525(5)	5132(3)	31(2)*
C(17)	2879(4)	4483(5)	6946(3)	29(2)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

3,4,4-Triethyl-2-trimethylsilyl-5-trimethylstannyl-1λ⁴-thia-2-aza-3-bora-1-cyclopentanone (**7**): The procedure corresponded to that used for the preparation of **4a**, except for the stirring period at room temp. (1 h). After recrystallization from pentane, the product 7 (1.12 g, 83%) was obtained as colorless crystalline solid (m.p. 115°C). – MS: *m/z* (%) = 423 (0) [M^+], 394 (1) [$M^+ - C_2H_5$], 351 (10), 336 (73), 322 (95), 165 (100) [Me_3Sn^+], 73 (12). – ^{1}H NMR (C_6D_6, 25°C): δ [$J(^{119}Sn^1H)$] = 0.26 (s, 9H; $SnMe_3$); 0.29 (s, 9H; $SiMe_3$); 0.5–2.2 (m, 15H; Et, Et, BEt); 2.80 [53.3] (s, 1H; SCH).

$C_{14}H_{34}BNOSSiSn$ (422.1)
Calcd. C 39.85 H 8.13 N 3.32 B 2.56 S 7.60
Found C 38.17 H 7.57 N 3.10 B 2.47 S 7.21

3,3,4-Triethyl-5-methyl-2-trimethylsilyl-1-trimethylstannyloxy-1λ⁴-thia-2-azonia-3-borata-1,4-cyclopentadiene (**8**): Under the same conditions as applied to the synthesis of 7 compound **8** was isolated (1.63 g, 75%) as colorless crystalline solid (m.p. 116°C). – ^{1}H NMR (C_6D_6, 25°C): δ [$J(^{119}Sn^1H)$] = 0.23 (s, 9H; $SiMe_3$); 0.55 [60.5] (s, 9H; $SnMe_3$); 0.7–1.1 (m, 13H; CH_3, BEt_2); 1.74 (s, 3H; $=CCH_3$); 2.22 (m, 2H; $=CCH_2$).

$C_{15}H_{36}BNOSSiSn$ (436.1) Calcd. C 41.31 H 8.32 N 3.21
Found C 40.68 H 7.95 N 3.05

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