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arachno-Diazapentaboranes

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ABSTRACT

The reaction of appropriate amounts of BH_3 with diazadiboretidines $(RBNtBu)_2$ (**1a–e**; *R = Et, Pr, Bu, tBu, tBu(Me_3Si)N) transforms them into cyclic diazapentaboranes either of the type [—RB—(H_2)—BR—NtBu—BH—NtBu—]* (**2a–c**), *of the type [—HB—(H_2)—BR—NtBu—BH—NtBu—]* (**3a–d**), *or of the type [—HB—(H_2)—BH—NtBu—BH—NtBu—]* (**4**), *or into an acyclic product RBH—NtBu—BH—NtBu—BHR* (**2e**). *The structure of* **3c** *is characterized by a planar N_2B_3 ring skeleton with an unsymmetrical double-hydrogen bridge.*

RESULTS

The diazadiboretidines **1a–c** [1–4] readily react with the borane thf · BH_3 in a 1:1 ratio to give the five-membered ring products **2a–c** (Equation 1). The ^1H, ^{11}B, and ^{13}C NMR spectra of **2a–c** indicate two equivalent N*t*Bu and RB groups. The terminal and the two bridging hydrogens give IR absorptions in the ranges of 2450–2590 and 1730–1770 cm^{-1}, respectively. Typical BH-NMR coupling constants comprise the range 120–137 Hz. The 2,4-diaminodiazadiboretidine **1e** reacts in a similar way, but does not form a double hydrogen bridge in the product **2e** (Equation 2). The chain character of **2e** corresponds to a tendency of hydrodiaminoboranes $HB(NR_2)_2$ to avoid a dimerization via hydrogen bridges, the boron atom being electronically satisified by BN π bonds; the terminal BH bonds of **2a–c** document this tendency. The sterically overcrowded tetra-*tert*-butyldiazadiboretidine **1d** does not react with thf · BH_3 according to Equation 1. If a 1:1 ratio is employed, only half of the amount of **1d** will be consumed, giving the product **3d**, which will be quantitatively formed from **1d** by applying a 1:2 ratio, according to Equation 3. In contrast to **1c** [1], the ring skeleton of **1d** deviates distinctly from planarity [5], and by expanding the four-membered to a hypothetical five-membered ring **2d** the steric stress applied by four *t*Bu groups would even be worse. Thus, if **2d** was ever formed from **1d** and BH_3, the consecutive transformation of **2d** into **3d** by the action of excess BH_3 would be faster than the reaction of remaining **1d** with BH_3. The formation of **3a–c** can easily be achieved either by applying a 1:2 ratio of **1a–c** and thf · BH_3 (Equation 3) or also by the reaction of **2a–c** and thf · BH_3 in a 1:1 ratio.

Typical for the adopted structure of **3a–d** in solution are the NMR spectra that reveal two different N*t*Bu groups and three nonequivalent B atoms. Two of them show doublets indicating the presence of two BH groups. This structural picture is confirmed by a crystal structure determination of **3c**. The remaining BR fragment of **3a–d** will be replaced by BH, finally, by adding additional thf · BH_3. The product **4** can also be directly synthesized from **1a–d** by starting with a 1:3 ratio of the components (Equation 4). The N*t*Bu groups and two of the three BH groups of **4** are equivalent, according to the NMR spectra.

Iminoboranes RB≡N*t*Bu [3], which correspond to their cyclodimers **1a–c**, may be used instead of **1a–e** for synthesizing the products **2–4**. The more reactive of these iminoboranes (R = Et, Pr, Bu) cause unwelcome side products during their violent reaction with thf · BH_3 at −78°C. But the less reactive ones (R = N($SiMe_3$)*t*Bu, *t*Bu) give good yields of **2e** and **3d**, respectively, at −78°C, according to Equations 5 and 6. The product **4** may also be obtained from *t*BuB≡N*t*Bu directly (Equation 7). Since these iminoboranes are stable below 0°C, the diazadiboretidines **1d**, **e** are not intermedi-

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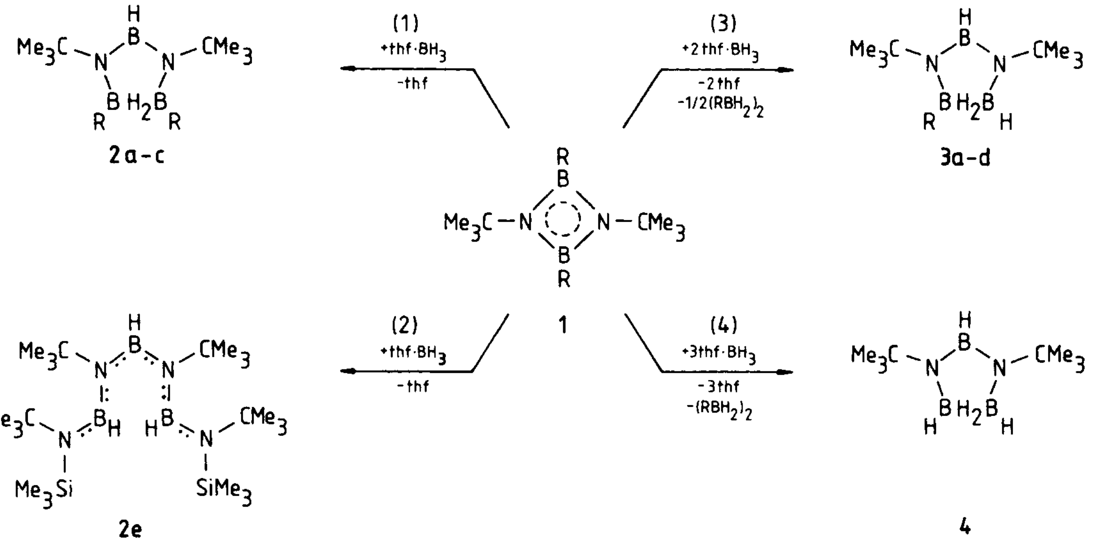

	R
a	Et
b	Pr
c	Bu
d	*t*Bu
e'	N(SiMe$_3$)*t*Bu

ates during reactions (5) and (6) at −78°C, though **1d**, **e** are the stabilization products of the corresponding iminoboranes at ambient temperature. Obviously, reaction (5) involves a hydroboration of the shorter BN bonds of two molecules of *t*Bu(Me$_3$Si)N$\stackrel{..}{-}$B$\stackrel{..}{=}$N*t*Bu by thf · BH$_3$. Initially, reaction (6) is likely to proceed in a similar way.

The products **2a–c**, **3a–d**, and **4** are thermally rather stable. The least stable one, **4**, decomposes at 140°C into diborane and the borazine (HBN*t*Bu)$_3$ (Equation 8). A protolytic cleavage of **4** can be achieved by *t*BuNH$_2$, according to Equation 9.

STRUCTURE OF 3c

The structure of the molecule **3c** in the crystal (Tables 1 and 2, Figure 1) is characterized by a planar five-membered ring with four of the neighboring atoms in the same and one (Hc) almost in the same

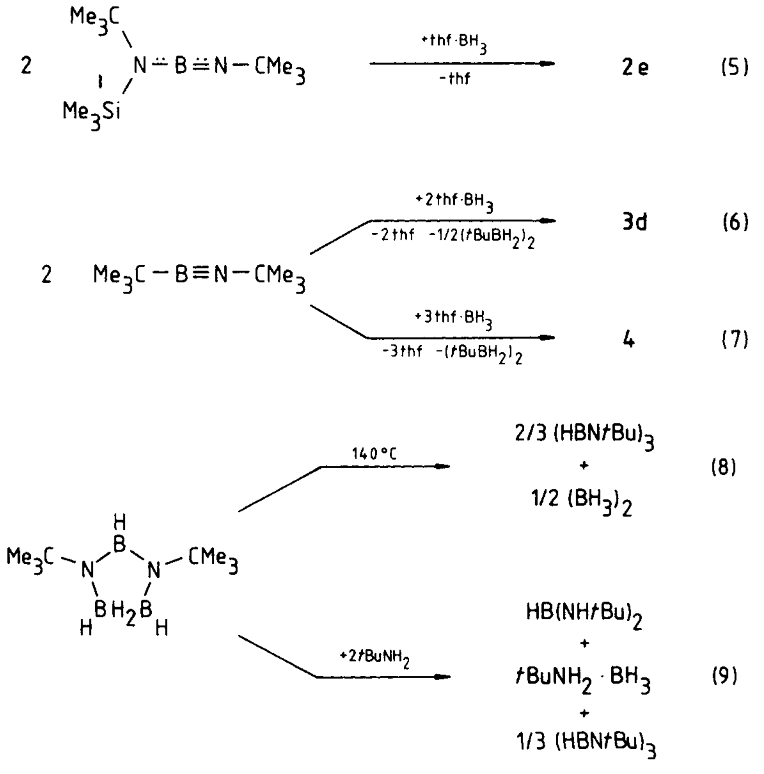

TABLE 1 Positional Parameters and their Estimated Standard Deviations of **3c**

Atom	*x*	*y*	*z*	*B(iso)*[a]
N1	0.9161(2)	0.8916(1)	0.661	3.86(4)
N2	0.7753(1)	0.9959(1)	0.6665(6)	3.84(4)
C1	1.1132(2)	0.9822(2)	0.661(1)	5.16(7)
C2	1.1473(2)	1.0719(2)	0.669(1)	6.25(8)
C3	1.2674(2)	1.0917(2)	0.665(1)	6.79(8)
C4	1.3002(3)	1.1781(3)	0.651(1)	9.3(1)
C11	0.9628(2)	0.8053(2)	0.6609(9)	4.37(6)
C12	1.0339(3)	0.7900(2)	0.5188(5)	6.00(9)
C13	0.8722(3)	0.7403(2)	0.660(1)	6.84(8)
C14	1.0341(3)	0.7948(2)	0.8144(6)	7.7(1)
C21	0.6639(2)	1.0349(2)	0.669(1)	4.26(5)
C22	0.6544(2)	1.0891(2)	0.5172(5)	5.50(8)
C23	0.6491(3)	1.0902(3)	0.8109(5)	7.3(1)
C24	0.5734(2)	0.9697(2)	0.667(1)	7.53(9)
B1	0.9837(2)	0.9662(2)	0.663(1)	3.81(6)
B2	0.8754(2)	1.0452(2)	0.666(1)	4.15(6)
B3	0.7995(2)	0.9096(2)	0.665(1)	4.04(6)
Ha	0.954(2)	1.023(1)	0.562(3)	0.1(5)
Hb	0.925(2)	1.011(2)	0.771(3)	1.0(6)
Hc	0.736(2)	0.863(1)	0.694(4)	1.2(5)
Hd	0.881(2)	1.114(1)	0.695(4)	1.4(6)

[a] Anisotropically refined atoms are given in the form of their isotropic equivalent displacement parameter, defined as: $B(iso) = (4/3)[a^2 \cdot B(1,1) + b^2 \cdot B(2,2) + c^2 \cdot B(3,3)]$.

plane. There are three larger ring angles at N1, N2, and B3 (111.5°–113.5°) and two smaller ones at B1 and B2 (100.1°, 102.4°). The four BN bond lengths, 1.421–1.453 Å, are slightly longer than normal BN double bonds but are comparable in size with the BN bonds in diazadiboretidines $(RBNR')_2$ and borazines $(RBNR')_3$ [6]; apparently, the hydrogen bridge does not prevent adjacent BN π bonds. The distances between boron and the terminal H atoms (B2–Hd: 1.14 Å, B3–Hc: 1.09 Å) are similar to those in B_2H_6 (1.09 Å) [7]. The geometry of the hydrogen bridge, however, is different from B_2H_6: Only one of the bridging H atoms is found in a plane nearly perpendicular to the ring plane (angle between the planes B1–Hb–B2 and B1–B2–B3: 88(2)°), but the second one is situated outside (interplanar angle B1–Ha–B2/B1–B2–B3: 115(1)°). BH bonds of rather different lengths (1.20–1.36 Å) constitute an unsymmetrical bridging situation (1.24 Å in B_2H_6) with a BB bond length (1.824 Å) within the broad range of cluster BB bonds [8].

DISCUSSION

The products **2–4** may be considered as derivatives of a cyclic diaminodiborane(6). In an acyclic diaminodiborane(6) the amino groups would be expected to enter the bridge position. 1,2:1,2-Doubly bridged diboranes are well known, putting up either or medium size have only recently been synthe- [10] with a common BH_2B-edge. According to the electron-counting rules for clusters, the products **2–4** may also be conceived as *arachno*-derivatives of a pentagonal bipyramid as the corresponding *closo*-cluster skeleton. Azaborane clusters of small or medium size have only recently been synthesized, including organic derivatives of *nido*-NB_3H_6 [11], *nido*-$N_2B_4H_6$ [12], and *arachno*-$NB_6H_{10}^{-}$ [13]. In all of them, and in **2–4** as well, the maximum coordination number of the N atoms remains three or four, and the nitrogen-containing fragment of such a cluster skeleton might be considered to be built up "classically," that is by two-center-two-electron bonds. The situation is different with larger azaborane cluster molecules like *nido*-$NC_2B_8H_{11}$ [14], *closo*-NB_9H_{10} [15], and *nido*-$NB_{10}H_{13}$ [16], which contain five-coordinated nitrogen.

TABLE 2 Selected Bond Distances X–Y (Å) and Bond Angles X–Y–Z (°) of **3c**[a]

B1–B2	1.824(3)	N1–C11	1.501(2)	N2–B2–Hb	100(1)
B2–N2	1.446(3)	C11–C12	1.496(7)	B1–Ha–B2	88(2)
N2–B3	1.421(2)	C11–C13	1.514(3)	B1–Hb–B2	91(2)
B3–N1	1.436(3)	C11–C14	1.564(7)	Ha–B1–Hb	85(1)
N1–B1	1.453(2)	N2–C21	1.482(2)	Ha–B2–Hb	90(1)
B1–Ha	1.30(2)	C21–C22	1.557(8)	N2–B2–Hd	125.8(9)
B1–Hb	1.36(3)	C21–C23	1.504(8)	Ha–B2–Hd	111(1)
B2–Ha	1.34(2)	C21–C24	1.514(3)	Hb–B2–Hd	104(2)
B2–Hb	1.20(3)	N1–B1–B2	100.1(1)	N1–B3–Hc	123.6(9)
B2–Hd	1.14(2)	B1–B2–N2	102.4(2)	N2–B3–Hc	121(1)
B3–Hc	1.09(2)	B2–N2–B3	111.5(2)	B2–B1–C1	126.4(2)
B1–C1	1.582(3)	N2–B3–N1	113.5(2)	N1–B1–C1	133.5(2)
C1–C2	1.506(3)	B3–N1–B1	112.5(1)	B2–N2–C21	121.5(1)
C2–C3	1.483(3)	N1–B1–Ha	114(1)	B3–N2–C21	127.0(2)
C3–C4	1.453(4)	N1–B1–Hb	99(1)	B1–N1–C11	123.8(1)
		N2–B2–Ha	116(1)	B3–N1–C11	123.7(2)

[a] Numbers in parentheses are estimated standard deviations in the least significant digit.

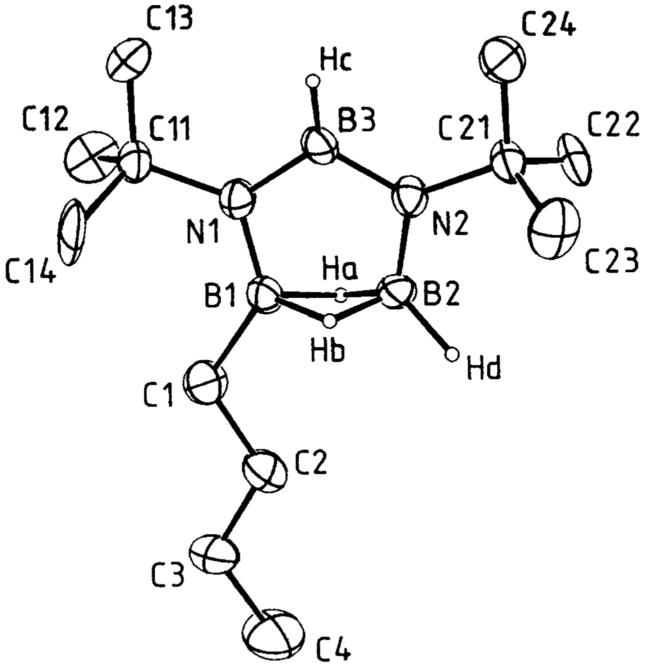

FIGURE 1 ORTEP drawing of **3c**. Vibrational ellipsoids are scaled to enclose 30% of the electron density. Carbon-bonded hydrogen atoms are omitted for clarity.

Apart from protic agents, the product **4** turns out to be of surprising stability. No reactions occurred with Lewis bases such as NEt_3 or PMe_3, oxidizing agents such as I_2, or $C_2(CN)_4$, reducing agents such as Li, Na, $NaBH_4$/diglyme, or thf · BH_3, or unsaturated organic molecules such as 2,3-dimethylbutadiene, 3-hexyne, or bis(trimethylsilyl)ethyne. We also did not succeed in capping the ring skeleton of **4** with one or two of any metal fragments that would not contribute further electrons to the cluster skeleton, thus furnishing a *nido*-pentagonal pyramid or *closo*-pentagonal bipyramid; the fragments $(OC)_3Cr$ and $(cod)Rh^+$ had been considered proper candidates.

EXPERIMENTAL

4,5-Dialkyl-1,3-di-tert-butyl-4,5-μ-dihydro-1,3-diazacyclopentaborane(7) (**2a–c**), *1,3,5,7-tetra-tert-butyl-1,7-bis(trimethylsilyl)-1,3,5,7-tetraza-2,4,6-triboraheptane* (**2e**), *4-alkyl-1,3-di-tert-butyl-4,5-μ-dihydro-1,3-diazacyclopentaorane(7)* (**3a–d**), *and 1,3-di-tert-butyl-4,5-μ-dihydro-1,3-diazacyclopentaborane(7)* (**4**)

As starting materials, the diazadiboretidines **1a–d** were synthesized according to known procedures from the aminoiminoborane $[Me_3Si(t\mathrm{Bu})N]BN(t\mathrm{Bu})$ and the appropriate alkylchloroborane $RBCl_2$ (**1a–c**) [2, 4], or simply by cyclodimerization of the corresponding iminoborane (**1d**) [5]. The compounds **1a–d** were dissolved in 10 mL pentane. BH_3 in a 0.95 molar solution in thf was added at −78°C in a stoichiometric amount with respect to Equations 1–4. The stirred solution was brought to ambient temperature during 30 min. Further stirring was applied for 2 h in the case of **3a–c**, 5 h in the case of **3d**, and 2 h in the case of **4**. The normal work-up was to remove all volatile materials in the high vacuo and to distill the product. The solid product **2e** was pure without sublimation or recrystallization. The crystalline product **3c** was recrystallized from ether. The liquid product **4** was purified after removing the solvents at 25°C/15 Torr by a condensation in vacuo at room temperature; the fraction between 1 and 10 Torr turned out to be spectroscopically clean. The amounts of starting material and the boiling points, yields, NMR spectra, and analytical data of the products are mentioned in Table 3. In order to gain **2e** from the corresponding iminoborane (Equation 5), the iminoborane was first prepared from 3.67 g (11.5 mmol) aminoborane $[Me_3Si(t\mathrm{Bu})N]_2BF$ [2]. Without removing Me_3SiF, the iminoborane was dissolved in 10 mL pentane, and 20.3 mL of a 0.85 molar solution of BH_3 in thf were added at −78°C. After stirring the cold solution for 10 min, all volatile products were removed in high vacuo. There remained 2.50 g (93%) of colorless solid **2e**. When the product **3d** was prepared from the iminoborane $t\mathrm{BuB{\equiv}N}t\mathrm{Bu}$ [5], 2.85 g (11.5 mmol) of a mixture of $t\mathrm{BuB{\equiv}N}t\mathrm{Bu}$ and the unremoved silane Me_3SiCl in 10 mL pentane were cooled down to −78°C, and 13.5 mL of the 0.85 molar solution of BH_3 in thf were added. After stirring the cold solution for 30 min, the solvent was removed in vacuo, and 1.18 g (87%) **3d** were distilled. Starting from 1.37 g (5.5 mmol) of a mixture of $t\mathrm{BuB{\equiv}N}t\mathrm{Bu}$ and Me_3SiCl and from 19.5 mL of a 0.85 molar solution of BH_3 in thf, the same procedure but with an additional stirring at room temperature for 2 days gave 0.35 g (70%) **4** after fractional condensation.

Thermolysis and Aminolysis of **4**

After heating 0.58 g (3.2 mmol) of **4** in a closed tube for 6 h at 140°C, the gaseous product B_2H_6 was trapped at −196°C. An amount of 0.49 g (92%) of 1,3,5-tri-*tert*-butylborazine was recovered from sublimation at 30°C/0.001 Torr and identified by its well-known properties [17].

A mixture of 0.38 g (2.15 mmol) of **4** and 0.315 g (4.3 mmol) *tert*-butylamine in 10 mL hexane was stirred at 25°C for 1 h. A precipitate of 0.16 g (86%) *tert*-butylamine-borane, $t\mathrm{BuNH_2} \cdot BH_3$, was gained by filtration and was characterized by its ^1H and ^{11}B NMR spectra [18]. After removing the solvent from the filtrate, a distillation at 50°C/2 Torr gave 0.235 g (70%) of bis(*tert*-butylamino)borane, $HB(NH t\mathrm{Bu})_2$. ^{1}H-NMR: $\delta = 1.10$ (s, 18 H, *t*Bu), 2.67

TABLE 3 Preparative, NMR,[a] IR,[b] and Analytical Data[c] of the Products **2–4**

	2a	**2b**	**2c**	**2e**	**3a**	**3b**	**3c**	**3d**	**4**
Preparative Data									
1a–e (g/mmol)	2.58/10.6	5.15/20.6	2.74/9.8	0.98/2.2	2.50/11.3	2.76/11.0	0.63/2.2	3.34/12.2	3.49/14.0
bp (°C/Torr)	65/0.001	70/0.001	100/0.001	[d]	45/5	51/0.02	[e]	70/0.001	
Yield (g/%)	1.99/79	5.24/97	1.87/65	0.86/86	1.65/71	1.47/59	0.46/90	2.07/72	2.38/95
NMR: δ [ppm]									
^{1}H tBu (s)	1.31	1.40	1.32	1.27[f]	1.29, 1.34	1.30, 1.37	1.30, 1.36	1.30, 1.34	1.31
R (broad)	1.08	0.8–1.7	0.6–1.6	0.18[f]	1.07	1.0–1.4	0.9–1.4	1.16	
H_t (q)	4.82	4.90	5.0	4.93[f]	4.0, 4.9	4.2, 4.8	4.2, 4.8	4.2, 4.9	4.5, 5.0[g]
H_b (broad)	2.1	2.1	2.1		2.0	2.2	2.15	2.1	2.0
^{11}B BR (broad)	11.1	10.6	10.5	37.1	15.6	14.1	15.1	17.2	
BH (d[h])	31.4(137)	31.8(120)	31.8(125)		31.3(129)	31.4(130)	31.7(100)	33.7(148)	32.1(129)[i]
BH (d[h])					−2.1(104)	−1.6(106)	−0.6(120)	−0.5(120)	2.2(132)[i]
^{13}C *t*Bu (q)	34.0	34.0	34.0	32.9, 33.4	33.3, 33.7	33.5, 33.9	33.2, 33.7	33.2, 35.0	33.3
*t*Bu (s)	53.5	53.5	53.5	54.5, 55.0	51.6, 53.6	51.8, 53.7	51.6, 53.4	51.7, 53.8	51.9
BC (broad)	8.6	19.3	15.4		9.1	20.1	25.6	20.8	
R (q)	13.3	17.8	14.0	6.3	11.9	17.4	14.0	32.2	
R (t)		22.9	26.4, 31.9			21.6	28.7, 30.4		
IR: $\bar{\nu}$ [cm^{-1}]	2578, 2486 1762	2589, 2460 1735	2589, 2459 1736	2447, 2361 2340	2579, 2481 1564	2580, 2485 1564	2581, 2477 1564	2580, 2476 1564	2574, 2358 1563
Analytical Data									
C (calcd./found)	61.1/61.1	63.7/63.9	65.8/66.1	56.7/56.0	57.8/56.2	59.6/59.5	61.1/61.5	61.1/60.7	53.5/54.0
H (calcd./found)	13.2/13.3	13.4/13.4	13.5/13.4	12.3/12.4	13.1/13.3	13.2/13.5	13.2/13.5	13.2/13.3	12.9/12.8
N (calcd./found)	11.9/11.9	10.6/10.6	9.6/9.6	12.0/12.0	13.5/13.3	12.6/12.2	11.9/11.8	11.9/12.1	15.6/15.5

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[a] Bruker WP80 (^{1}H, 80 MHz, int. TMS); Jeol JNM PS 100 (^{11}B, 32.08 MHz, ext. $BF_3 \cdot Et_2O$); Bruker WH 270 (^{13}C, 67.88 MHz, TMS via $CDCl_3$); solvent: $CDCl_3$.

[b] Perkin Elmer FT 1720: range: 1550–2600 cm^{-1}.

[c] Carlo Erba Elemental Analyzer 1106.

[d] mp 68°C.

[e] mp 136°C (from Et_2O).

[f] At −50°C; 3s, 12:6:1.

[g] 2 q, 2:1

[h] J_{BH} [Hz] in parentheses.

[i] 2 d, 1:2.

(broad, 2 H, NH); ^{11}B-NMR: δ = 26.1 (d, J = 129 Hz); MS: m/e = 156 (M^+, 16%), 141 ($M - Me$, 100%), and so on. The sublimation of the distillation residue at 30°C/0.001 Torr yields 0.16 g (91%) of 1,3,5-tri-*tert*-butylborazine [17].

Crystal and Molecular Structure of **3c**

A single crystal of **3c**, $C_{12}H_{31}B_3N_2$, of approximate dimensions $0.4 \times 0.7 \times 1.0$ mm^3 was used for both geometry and intensity data collection on an ENRAF Nonius CAD4 diffractometer. The compound crystallizes in the orthorhombic space group $Pna2_1$, No. 33, with the following cell parameters: a = 12.057(6), b = 16.120(78), c = 8.45(1) Å, V = 1641(3) Å^3, d_{calc} = 0.954 g/cm^3, Z = 4, μ = 0.494 cm^{-1}, $F(000)$ = 528. MoK$_\alpha$ radiation (0.7093 Å, graphite monochromator) was used to collect 2366 reflections with $3° < \theta < 30°$ in the omega-scan-mode: 1570 reflections showed $I > 2\sigma(I)$, and of these 1238 were symmetrically independent and were used for structure solution and subsequent refinement. The structure was solved by Direct Methods (MULTAN) [19] and a difference Fourier synthesis. In the final full-matrix refinement (SDP) [20] all nonhydrogen atoms were refined anisotropically and the four boron-bound hydrogen atoms isotropically; the rest of the hydrogen atoms were included according to a "riding" model (C—H = 0.98 Å, $B_H = 1.3 \cdot B_c$). A correction for secondary extinction was applied on F_c [21]. Refinement converged at R = 0.054, R_w = 0.065 for 170 parameters; $w^{-1} = \sigma^2(F)$. The highest residual electron density was 0.2 e/Å^3 [22].

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[22] A request for additional data may be directed to Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, D-7514 Eggenstein-Leopoldshafen with a citation of the registration number CSD 54816, the names of the authors, and the journal.