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N-Boranyl-*N*-(trimethylsilyl)pyrrolidine

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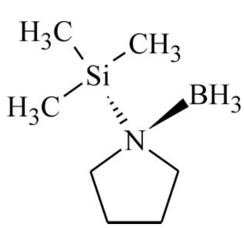
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.090; data-to-parameter ratio = 14.2.

The molecule of the title compound, $C_7H_{20}BNSi$, exhibits normal sp^3-hybridized B and N centres and the pyrrolidine ring has a slightly distorted envelope conformation. The B—N bond distance is longer than that found for related derivatives and possibly reflects the conformation of the ring and the effect of N-atom silylation.

Related literature

For related literature, see: Jaska *et al.* (2003); Mountford *et al.* (2003); Chitsaz *et al.* (2002).



Experimental

Crystal data

$C_7H_{20}BNSi$
M_r = 157.14
Monoclinic, $P2_1/n$
a = 8.613 (5) Å
b = 10.321 (5) Å
c = 11.753 (5) Å
β = 106.080 (5)°
V = 1003.9 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.17 mm^{-1}
T = 100 (2) K
0.4 × 0.3 × 0.2 mm

Data collection

Oxford Diffraction Xcalibur2 CCD diffractometer
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.934, T_{max} = 0.971
6834 measured reflections
2436 independent reflections
1970 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.090
S = 1.06
2436 reflections
171 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Selected torsion angles (°).

N—C1—C2—C3	20.37 (11)	C3—C4—N—C1	41.07 (11)
C1—C2—C3—C4	4.88 (11)	C2—C1—N—C4	−37.80 (11)
C2—C3—C4—N	−28.50 (11)		

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: CrysAlis RED; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *WinGX* (Farrugia, 1999); software used to prepare material for publication: *WinGX*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2228).

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supplementary materials

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N-Boranyl-*N*-(trimethylsilyl)pyrrolidine

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Comment

As part of a broader study aimed at using various borane derivatives as hydroborating agents for octenes, we have recently begun to explore the use of activated amine adducts of BH_3. Our goal in this work was to prepare and structurally characterize a relatively stable silylated secondary amine adduct of BH_3 as a potentially active hydroborating reagent for alkenes.

The molecular structure of (I), (Fig. 1), reflects sp^3 hybridized geometries for boron and nitrogen, consistent with the formation of a regular dative covalent bond to boron. Viewing (I) down the N–Si bond vector shows that the B–N bond roughly bisects the C5–Si–C7 angle to give a staggered conformation around the Si–N bond [C7–Si–N–B = 69 (1)°]. The BH_3 group therefore fits within the space generated by the closest two methyls of the $SiMe_3$ group.

The B–N bond of (I) (Table 1) is significantly longer than that of $B(C_6F_5)_3$.(pyrrolidine) [1.628 Å; Mountford *et al.*, 2003], $(pyrrolidine)_2(BH_2)_2$ [1.596 Å; Jaska *et al.*, 2003] and BH_3.(pyrrolidine) (1.591 Å; Chitsaz *et al.*, 2002). The origin of this effect is unclear, since the present structure exhibits no repulsive (short) B···Si steric interactions that might favour marked elongation of the B–N bond. Indeed the B–N–Si bond angle [108.8 (1)°] is close to the ideal sp^3 hybridized value of 109.5°.

It is possible that silylation of the pyrrolidine nitrogen in (I) reduces its σ-donor power, culminating in a weaker, longer N–B bond. Also relevant is the fact that the pyrrolidine ring of (I) adopts a slightly distorted envelope conformation, with N as the flap atom; N is displaced by 0.602 (3) Å from the mean plane of the other four atoms. The ring carbon atoms C2 and C3 tip towards the BH_3 unit leading to a short intramolecular contact between H3B and B [3.02 (1) Å]. It has also a pseudo mirror plane passing through atom N and the mid-point of C2—C3 bond, as evidenced by the torsion angles (Table 1). This conformation is not observed for the pyrrolidine adduct of BH_3 where the envelope conformation of the pyrrolidine ring is folded away from the BH_3 group, presumably due to the absence of the bulky $SiMe_3$ group.

There are no short intermolecular contacts between molecules of (I) in the crystal lattice and the 4-molecule unit cell (Fig. 2) shows the expected packing.

Experimental

B_2H_6 (b.p. 181 K) was generated by the reaction of $BF_3(OEt_2)$ (51.6 mmol, 6.53 ml) and $NaBH_4$ (1.46 g, 38.7 mmol) in bis(2-methoxyethyl) ether (20 ml) under nitrogen prior to being condensed at 166 K (liquid nitrogen/*iso*-octane slurrey). The liquid B_2H_6 was reacted with a solution of *N*-(trimethylsilyl)pyrrolidine (17.2 mmol, 3.0 ml) in hexane. Colorless crystals of (I) were obtained after 5 d.

Refinement

H atoms were located in difference syntheses and refined isotropically [C—H = 0.893 (15)-1.010 (12) Å, B—H = 1.104 (14)-1.121 (13) Å and U_{iso}(H) = 0.013 (3)-0.051 (5) $Å^2$].

Figures

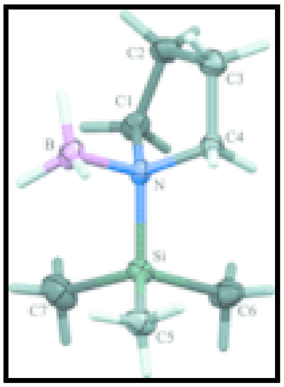

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 70% probability level.

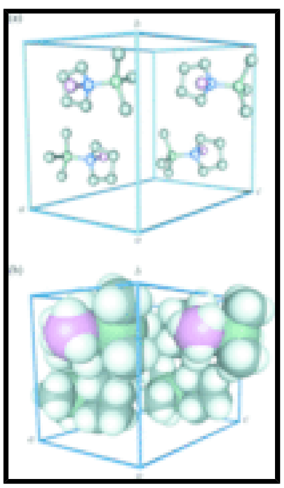

Fig. 2. (a) A packing diagram for (I). H atoms have been omitted for clarity. (b) CPK model of the unit cell contents of (I) including H atoms.

N-Boranyl-*N*-(trimethylsilyl)pyrrolidine

Crystal data

$C_7H_{20}BNSi$	F_{000} = 352
M_r = 157.14	D_x = 1.04 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 4177 reflections
a = 8.613 (5) Å	θ = 3.8–33.9º
b = 10.321 (5) Å	μ = 0.17 mm^{-1}
c = 11.753 (5) Å	T = 100 (2) K
β = 106.080 (5)º	Block, colorless

V = 1003.9 (9) Å^3	0.4 × 0.3 × 0.2 mm
Z = 4	

Data collection

Oxford Diffraction Xcalibur2 CCD diffractometer	2436 independent reflections
Radiation source: fine-focus sealed tube	1970 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.016
Detector resolution: 8.4190 pixels mm^{-1}	θ_{max} = 33.9º
ω scans	θ_{min} = 4.0º
Absorption correction: multi-scan (Blessing, 1995)	$h = -10 \rightarrow 13$
T_{min} = 0.934, T_{max} = 0.971	$k = -12 \rightarrow 13$
6834 measured reflections	$l = -12 \rightarrow 15$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0589P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
Least-squares matrix: full	$(\Delta/\sigma)_{max}$ = 0.001
$R[F^2 > 2\sigma(F^2)]$ = 0.031	$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$wR(F^2)$ = 0.090	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
S = 1.06	Extinction correction: none
2436 reflections	
171 parameters	
All H-atom parameters refined	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.85439 (13)	0.12797 (11)	0.34325 (10)	0.0169 (2)
C2	0.68293 (13)	0.15220 (11)	0.35366 (10)	0.0194 (3)
C3	0.66737 (13)	0.30030 (12)	0.36165 (10)	0.0194 (3)
C4	0.82484 (12)	0.35392 (11)	0.34436 (10)	0.0153 (2)
C5	1.24120 (13)	0.41217 (11)	0.40765 (11)	0.0198 (3)
C6	1.06149 (15)	0.29146 (14)	0.17331 (11)	0.0239 (3)
C7	1.24759 (15)	0.11565 (12)	0.36983 (13)	0.0257 (3)
B	1.00502 (16)	0.24707 (13)	0.53477 (12)	0.0185 (3)
N	0.94841 (11)	0.24928 (8)	0.38998 (8)	0.0124 (2)
Si	1.12767 (3)	0.26715 (3)	0.33653 (3)	0.01435 (12)

H1A	0.9113 (13)	0.0563 (12)	0.3895 (10)	0.021 (3)*
H1B	0.8533 (14)	0.1164 (13)	0.2613 (11)	0.023 (3)*
H2A	0.6659 (14)	0.1066 (13)	0.4225 (10)	0.024 (3)*
H2B	0.6058 (14)	0.1173 (12)	0.2842 (10)	0.021 (3)*
H3A	0.5724 (14)	0.3351 (12)	0.2980 (11)	0.022 (3)*
H3B	0.6545 (15)	0.3236 (13)	0.4371 (12)	0.028 (3)*
H4A	0.8639 (13)	0.4323 (12)	0.3881 (10)	0.017 (3)*
H4B	0.8130 (13)	0.3645 (11)	0.2611 (10)	0.013 (3)*
H5A	1.1743 (16)	0.4884 (14)	0.3951 (12)	0.037 (4)*
H5B	1.2849 (14)	0.3980 (12)	0.4911 (11)	0.023 (3)*
H5C	1.3241 (16)	0.4273 (13)	0.3727 (11)	0.033 (4)*
H6A	0.9977 (19)	0.2279 (14)	0.1352 (13)	0.042 (5)*
H6B	1.1574 (19)	0.2906 (14)	0.1439 (14)	0.041 (4)*
H6C	1.0107 (16)	0.3754 (17)	0.1528 (11)	0.046 (4)*
H7A	1.3255 (19)	0.1171 (15)	0.3272 (12)	0.051 (4)*
H7B	1.2976 (18)	0.1068 (15)	0.4542 (14)	0.051 (5)*
H7C	1.1859 (17)	0.0430 (16)	0.3421 (12)	0.047 (4)*
H8A	0.9040 (16)	0.2120 (13)	0.5674 (12)	0.033 (4)*
H8B	1.0372 (15)	0.3472 (13)	0.5678 (10)	0.030 (4)*
H8C	1.1110 (16)	0.1800 (14)	0.5637 (11)	0.040 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0208 (5)	0.0125 (5)	0.0170 (6)	−0.0037 (4)	0.0045 (4)	−0.0025 (5)
C2	0.0176 (5)	0.0210 (6)	0.0194 (6)	−0.0059 (4)	0.0047 (5)	−0.0015 (5)
C3	0.0166 (5)	0.0226 (6)	0.0197 (6)	0.0008 (4)	0.0061 (5)	−0.0010 (5)
C4	0.0167 (5)	0.0131 (5)	0.0161 (5)	0.0031 (4)	0.0046 (4)	0.0012 (4)
C5	0.0172 (5)	0.0173 (6)	0.0248 (6)	−0.0016 (4)	0.0057 (5)	−0.0027 (5)
C6	0.0239 (6)	0.0307 (7)	0.0183 (6)	−0.0053 (5)	0.0080 (5)	−0.0028 (5)
C7	0.0218 (6)	0.0198 (6)	0.0342 (7)	0.0042 (5)	0.0054 (6)	−0.0068 (6)
B	0.0217 (6)	0.0198 (6)	0.0128 (6)	−0.0014 (5)	0.0027 (5)	0.0012 (5)
N	0.0144 (4)	0.0098 (4)	0.0125 (4)	0.0001 (3)	0.0029 (4)	−0.0002 (3)
Si	0.01368 (16)	0.01410 (18)	0.01535 (19)	0.00015 (10)	0.00415 (12)	−0.00203 (12)

Geometric parameters (Å, °)

C1—N	1.5095 (14)	C5—H5B	0.959 (12)
C1—C2	1.5357 (17)	C5—H5C	0.931 (14)
C1—H1A	0.967 (12)	C6—Si	1.8606 (15)
C1—H1B	0.968 (12)	C6—H6A	0.893 (15)
C2—C3	1.5395 (18)	C6—H6B	0.980 (16)
C2—H2A	0.982 (12)	C6—H6C	0.971 (17)
C2—H2B	0.968 (12)	C7—Si	1.8551 (14)
C3—C4	1.5296 (16)	C7—H7A	0.943 (16)
C3—H3A	1.010 (12)	C7—H7B	0.969 (15)
C3—H3B	0.955 (13)	C7—H7C	0.925 (16)
C4—N	1.5068 (14)	B—N	1.6354 (17)
C4—H4A	0.967 (12)	B—H8A	1.104 (14)

C4—H4B	0.961 (11)	B—H8B	1.112 (13)
C5—Si	1.8553 (13)	B—H8C	1.121 (13)
C5—H5A	0.962 (14)	N—Si	1.8303 (13)

N—C1—C2	105.47 (9)	Si—C6—H6A	112.9 (10)
N—C1—H1A	107.0 (7)	Si—C6—H6B	108.4 (9)
C2—C1—H1A	115.4 (7)	H6A—C6—H6B	106.2 (12)
N—C1—H1B	108.7 (8)	Si—C6—H6C	111.4 (8)
C2—C1—H1B	110.8 (7)	H6A—C6—H6C	111.1 (12)
H1A—C1—H1B	109.2 (10)	H6B—C6—H6C	106.7 (11)
C1—C2—C3	105.57 (8)	Si—C7—H7A	108.1 (10)
C1—C2—H2A	110.7 (7)	Si—C7—H7B	111.0 (9)
C3—C2—H2A	113.0 (7)	H7A—C7—H7B	111.4 (13)
C1—C2—H2B	108.8 (7)	Si—C7—H7C	112.2 (9)
C3—C2—H2B	111.7 (8)	H7A—C7—H7C	104.5 (12)
H2A—C2—H2B	107.0 (10)	H7B—C7—H7C	109.5 (12)
C4—C3—C2	104.82 (9)	N—B—H8A	109.2 (7)
C4—C3—H3A	109.8 (7)	N—B—H8B	108.9 (6)
C2—C3—H3A	112.0 (7)	H8A—B—H8B	109.1 (9)
C4—C3—H3B	111.6 (8)	N—B—H8C	107.9 (7)
C2—C3—H3B	110.0 (8)	H8A—B—H8C	110.3 (10)
H3A—C3—H3B	108.7 (10)	H8B—B—H8C	111.4 (9)
N—C4—C3	104.97 (9)	C4—N—C1	102.14 (9)
N—C4—H4A	107.6 (7)	C4—N—B	110.97 (8)
C3—C4—H4A	115.0 (7)	C1—N—B	110.02 (8)
N—C4—H4B	108.0 (7)	C4—N—Si	112.67 (7)
C3—C4—H4B	109.2 (7)	C1—N—Si	112.16 (7)
H4A—C4—H4B	111.7 (9)	B—N—Si	108.77 (7)
Si—C5—H5A	111.9 (8)	N—Si—C7	108.42 (6)
Si—C5—H5B	110.3 (8)	N—Si—C5	108.43 (5)
H5A—C5—H5B	109.2 (11)	C7—Si—C5	113.07 (7)
Si—C5—H5C	108.0 (8)	N—Si—C6	108.71 (6)
H5A—C5—H5C	107.2 (11)	C7—Si—C6	108.96 (6)
H5B—C5—H5C	110.2 (11)	C5—Si—C6	109.17 (6)

N—C1—C2—C3	20.37 (11)	C4—N—Si—C7	−167.46 (7)
C1—C2—C3—C4	4.88 (11)	C1—N—Si—C7	−52.87 (9)
C2—C3—C4—N	−28.50 (11)	B—N—Si—C7	69.06 (8)
C3—C4—N—C1	41.07 (11)	C4—N—Si—C5	69.43 (9)
C3—C4—N—B	−76.15 (10)	C1—N—Si—C5	−175.98 (7)
C3—C4—N—Si	161.60 (7)	B—N—Si—C5	−54.05 (8)
C2—C1—N—C4	−37.80 (11)	C4—N—Si—C6	−49.15 (9)
C2—C1—N—B	80.10 (10)	C1—N—Si—C6	65.44 (8)
C2—C1—N—Si	−158.69 (7)	B—N—Si—C6	−172.63 (7)

Fig. 1

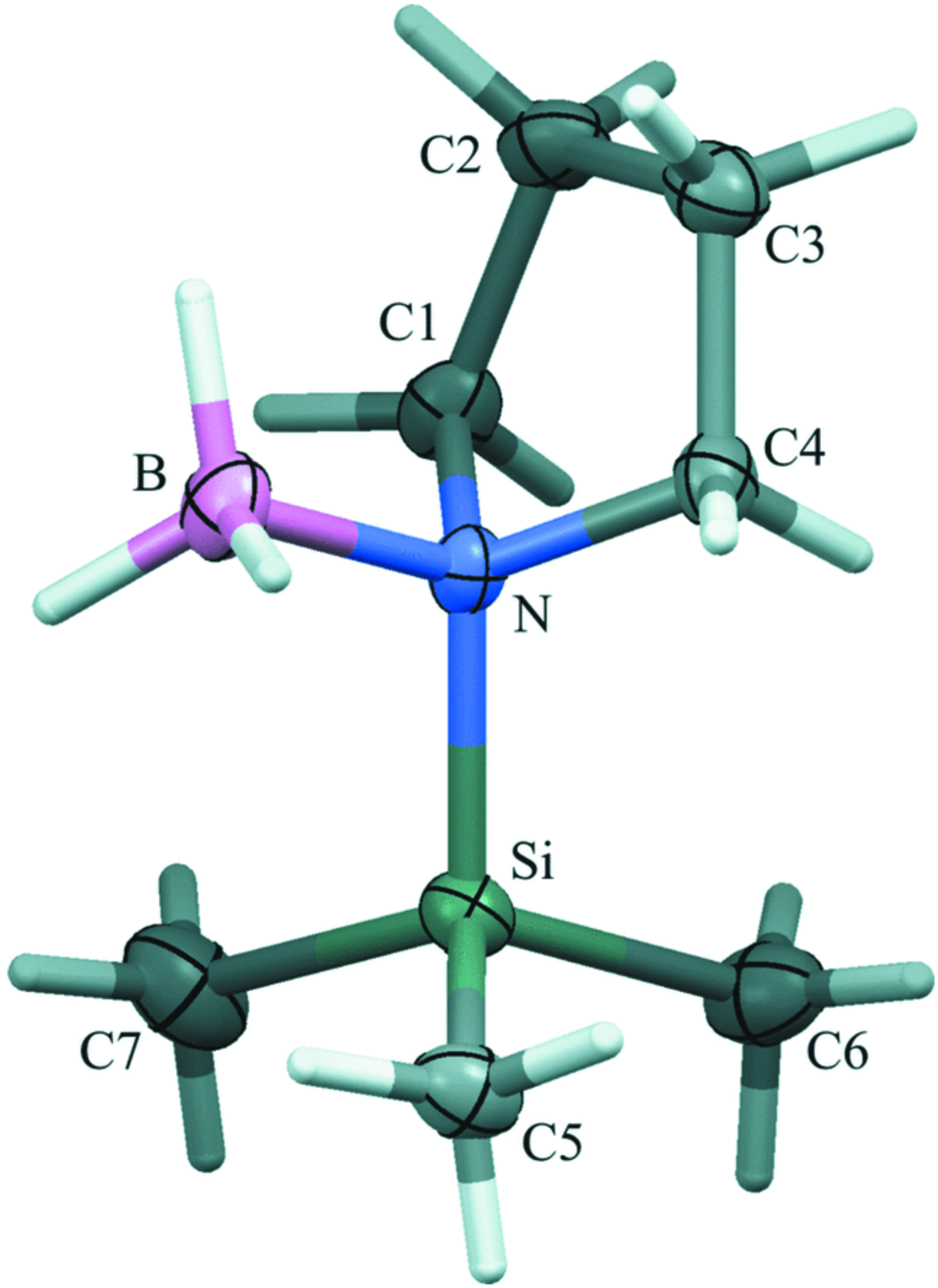

Fig. 2