2-Chloro-1,1-dimethyl-2-phenyl-2,3-dihydro-1H-1,2-benzazoniaborolate

Hong-Bo Tong, Xue-Hong Wei, Shu-Ping Huang, Jian-Feng Li and Dian-Sheng Liu*

School of Chemistry and Chemical Engineering, The Shanxi University, Shanxi, People's Republic of China

Correspondence e-mail: common@mail.sxu.edu.cn

Key indicators

Single-crystal X-ray study
$T = 180$ K
Mean σ(C–C) = 0.004 Å
R factor = 0.068
wR factor = 0.139
Data-to-parameter ratio = 14.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the crystal structure of the title compound, $C_{15}H_{17}BClN$, the coordination around both B and N is distorted tetrahedral. The five-membered heterocycle adopts a folded conformation.

Comment

Organoboron chemistry is intriguing from a number of points of view: structural (bornane clusters), bond theoretical (multicenter bonding) and practical (hydroboration and carbaboration). Boron is found in a large number of saturated and unsaturated heterocycles, in segments such as C—B—C, C—B—N, N—B—N, O—B—O, C—B—O, C—B—S, *etc*.

(I)
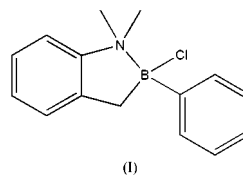
Recently, we have synthesized, in high yield, the title compound, (I), containing the C—B—N fragment. This compound has been characterized by single-crystal X-ray diffraction analysis.

The geometric parameters of the heterocyclic moiety of (I) are listed in Table 1 and the molecular structure is illustrated in Fig. 1. The five-membered heterocyclic ring adopts a folded conformation. The angle between the C1/B/N and C1/C2/C7/N planes is 29.1 (5)°. The torsion angles Cl—B—N—C7 [−84.4 (2) Å] and Cl—B—C1—C2 [82.2 (3) Å] indicate that the B—Cl bond is almost perpendicular to the Cl—B—N—C7 part of the heterocyclic ring.

Both the B and N atoms are sp^3 hybridized and coordination around each is distorted tetrahedral. The B—C bond length [1.608 (4) Å] agrees well with the value of 1.597 (22) Å for a four-coordinate B atom bonded to a Csp^3 atom (Allen *et al.*, 1987). The B—N bond is much longer [1.685 (4) Å]. A search of the Cambridge Structural Database (CSD; Allen, 2002) found four comparable compounds with a five-membered BNC$_3$ ring and four-coordinate B and N atoms. These entries and the values of the B—N bond lengths are: 1.720 (3) Å in KISZIV (Köster *et al.*, 1991), 1.716 (5) Å in RISHAC (Ashe *et al.*, 1997), 1.682 (4) and 1.684 (4) Å in WOPBEI (Schumann *et al.*, 2000) and 1.702 Å in YAXFUY (Köster *et al.*, 1993).
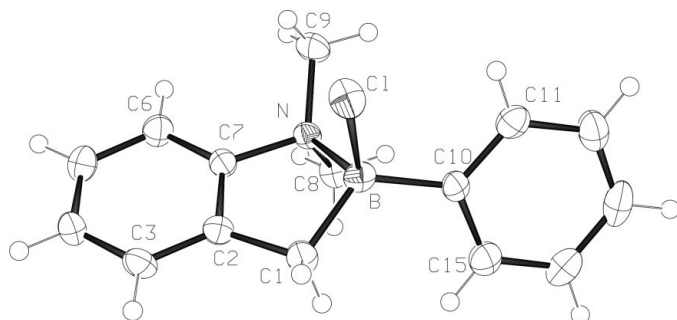
Experimental

n-Butyllithium (molar ratio 1:1) was added dropwise to a solution of *N,N*-dimethyl-*o*-toluidine in hexane at 273 K, and the temperature
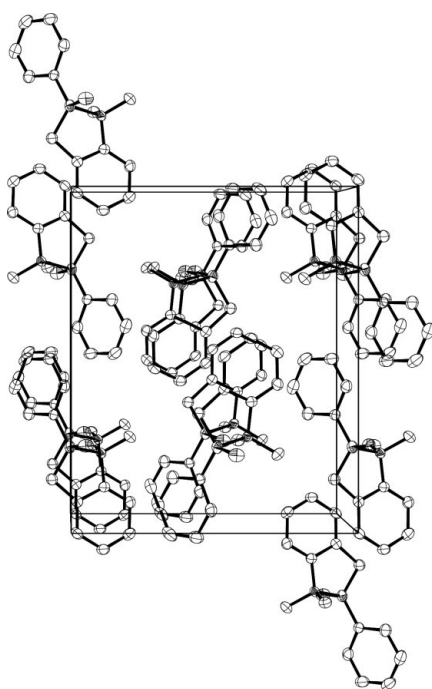
Figure 1
The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by small spheres of arbitrary radii.

Figure 2
A packing diagram of the title molecule, viewed down the *a* axis. For clarity, all H atoms have been omitted.

was allowed to rise to room temperature. The mixture was stirred for more than 5 h and then chlorotrimethylsilane (molar ratio 1:1) was added at 273 K. The resulting mixture was warmed slowly to room temperature and stirred for a further 12 h to yield a white precipitate (LiCl). The mixture was filtered and the title compound, (I), was isolated by distilling the filtrate as a colorless oil. A solution of (I) in toluene was cooled to 273 K and dichlorophenylborane added (ratio 1:1). The mixture was warmed slowly to room temperature and the solution refluxed for a further 12 h, eliminating chlorotrimethylsilane. A white solid was obtained, which was dissolved in CH_2Cl_2 (10 ml). The solution was concentrated carefully under vacuum, yielding a colorless crystal of the title compound. All reactions were performed under argon, using standard Schlenk techniques. The hexane was dried by distilling with a sodium–potassium alloy, CH_2Cl_2 was distilled from CaH_2 and toluene was distilled with sodium.

Crystal data

$C_{15}H_{17}BClN$	$D_x = 1.276$ Mg m^{-3}
$M_r = 257.56$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 1487
$a = 7.4639 (11)$ Å	reflections
$b = 14.798 (2)$ Å	$\theta = 2.8–26.3°$
$c = 12.2356 (18)$ Å	$\mu = 0.27$ mm^{-1}
$\beta = 97.180 (2)°$	$T = 180 (2)$ K
$V = 1340.8 (3)$ Å^3	Block, colorless
$Z = 4$	$0.40 \times 0.30 \times 0.30$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2355 independent reflections
	1905 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.037$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 25.0°$
	$h = -8 \rightarrow 8$
$T_{min} = 0.902$, $T_{max} = 0.925$	$k = -17 \rightarrow 14$
5435 measured reflections	$l = -14 \rightarrow 11$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.068$	$+ 0.4704P]$
$wR(F^2) = 0.139$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.16$	$(\Delta/\sigma)_{max} < 0.001$
2355 reflections	$\Delta\rho_{max} = 0.35$ e Å^{-3}
165 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Cl—B	1.905 (4)	N—C7	1.490 (4)
B—C1	1.608 (4)	N—C8	1.497 (4)
B—C10	1.611 (4)	C1—C2	1.495 (4)
B—N	1.685 (4)	C2—C7	1.392 (4)
N—C9	1.485 (4)		
C1—B—C10	121.2 (3)	C7—N—C8	107.3 (2)
C1—B—N	99.8 (2)	C9—N—B	116.1 (2)
C10—B—N	111.1 (2)	C7—N—B	103.0 (2)
C1—B—Cl	108.1 (2)	C8—N—B	110.0 (2)
C10—B—Cl	110.0 (2)	C2—C1—B	103.8 (2)
N—B—Cl	105.3 (2)	C7—C2—C1	113.2 (2)
C9—N—C7	112.4 (2)	C2—C7—N	111.2 (2)
C9—N—C8	107.8 (2)		
Cl—B—N—C7	−84.4 (2)	Cl—B—C1—C2	82.2 (3)

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry, with C—H distances of 0.98 Å and $U_{iso}(H) = 1.5U_{eq}(C)$, but each group was allowed to rotate freely about the C—N bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.95–1.00 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.

Ashe, A. J. III, Kampf, J. W. & Waas, J. R. (1997). *Organometallics*, **16**, 163–167.

Farrugia, L. J. (1997). *ORTEP*-3. University of Glasgow, Scotland.

Köster, R., Seidel, G. & Müller, G. (1991). *Chem. Ber.* **124**, 1017–1023.

Köster, R., Seidel, G. & Müller, G. (1993). *Chem. Ber.* **126**, 2211–2219.

Schumann, H., Wassermann, B. C., Schutte, S., Heymer, B., Nickel, S., Seuss, T. D., Wernik, S., Demchuk, J., Girgsdies, F. & Weimann, R. (2000). *Z. Anorg. Allg. Chem.* **626**, 2081–2095.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.