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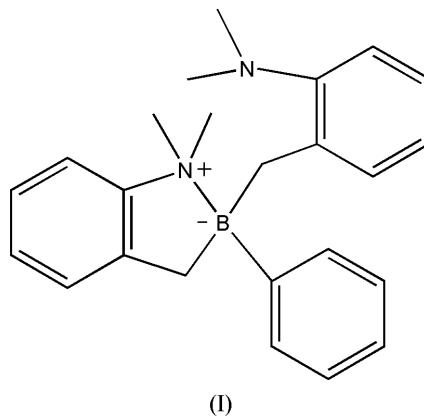
Key indicators

Single-crystal X-ray study
 $T = 190$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.087
 wR factor = 0.176
Data-to-parameter ratio = 14.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.[2-(Dimethylamino)benzyl][(N-B)-2-(dimethyl-
ammonio)benzyl]phenylborate

In the crystal structure of the title compound, $\text{C}_{24}\text{H}_{29}\text{BN}_2$, the coordination around both the B and N atoms is distorted tetrahedral. The five-membered ring adopts an envelope conformation, with the flap atom, B, displaced by 0.629 (6) Å from the plane of the other four atoms.

Comment

Among the group 3 elements B, Al, Ga, In and Tl, the organometallic chemistry of boron and aluminium clearly predominates. Organoboron chemistry is of interest from many perspectives, including electronic and structural, as well as its heterocyclic chemistry (Elschenbroich & Salzer, 1992). Only one other compound containing the fused five- and six-membered rings of the title compound, (I), has been characterized by X-ray crystallography. Its crystal structure, (II), was reported by our group some two years ago (Tong *et al.*, 2002). We have synthesized the title compound, (I), by reacting (II) with lithium *N,N*-dimethyl-*o*-toluidine, and report its structure here.



The molecular structure is illustrated in Fig. 1, and important geometric parameters are listed in Table 1. The five-membered ring adopts an envelope conformation, with the flap atom, B, displaced by 0.629 (6) Å from the plane of the other four atoms; the corresponding distance in (II) was 0.515 (1) Å. Denoting the ring C2–C7 as *R*1, the ring C11–C16 as *R*2 and the ring C19–C24 as *R*3, the dihedral angles *R*1/*R*2, *R*1/*R*3 and *R*2/*R*3 are 24.8 (5), 56.7 (5) and 81.0 (5)°, respectively. The coordination around both B and N is distorted tetrahedral.

For the title compound, the B–C1 bond length [1.621 (4) Å] agrees well with the value of 1.60 (2) Å for a four-coordinate B atom bonded to a *Csp*³ atom (Allen *et al.*, 1987); B–C10, however, is somewhat longer at 1.642 (4) Å.

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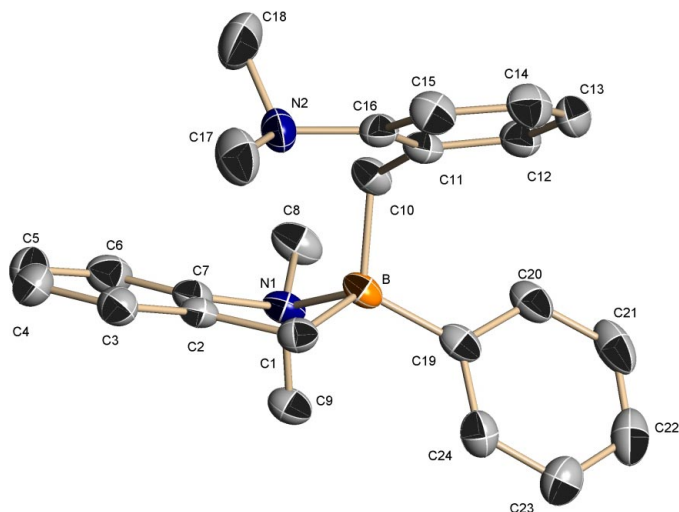


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted.

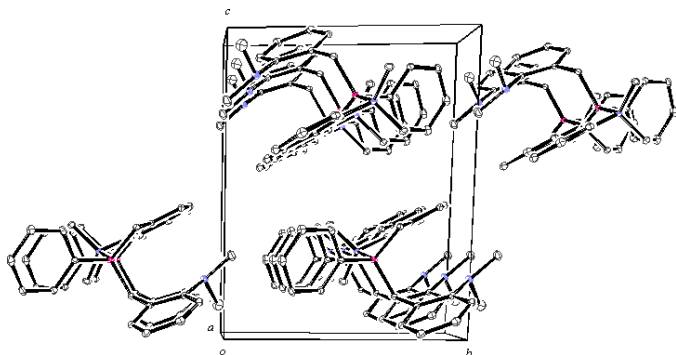


Figure 2
A packing diagram (ORTEP-3; Farrugia, 1997) of the title complex, viewed approximately down the *a* axis. H atoms have been omitted.

B—N1 [1.734 (4) Å] is much longer than in (II), where it is 1.685 (4) Å. B—N distances ranging from 1.682 (4) to 1.720 (3) Å have been reported in four other compounds containing the C₃BN ring (Schumann *et al.*, 2000; Ashe *et al.*, 1997; Köster *et al.*, 1991, 1993).

Experimental

All manipulations were carried out under argon or *in vacuo*, using standard Schlenk techniques. Solvents were pre-dried over sodium wire, distilled from sodium potassium alloy (hexane and toluene) and sodium benzophenone (diethyl ether), and stored over molecular sieves (4 Å). Deuterated solvents were likewise stored over such molecular sieves, and degassed prior to use. Chloro(trimethyl)silane, and *n*-butyllithium in hexanes (1.6 mol l⁻¹, FMC Corporation), were commercial samples, and were used without further purification.

n-Butyllithium in *n*-hexane (3.5 ml of a 1.6 mol l⁻¹ solution, 5.6 mmol) was added dropwise to a solution of *N,N*-dimethyl-*o*-toluidine (0.757 g, 5.6 mmol) and 1,2-bis(dimethylamino)ethane (0.650 g, 5.6 mmol) in hexane (20 ml) at *ca* 273 K. The mixture was stirred for *ca* 24 h at room temperature and the solvent extracted under vacuum. Diethyl ether (20 ml) was then added, followed by (II)

(1.44 g, 5.7 mmol) dropwise at 273 K. The resultant mixture was slowly warmed to room temperature and stirred for a further 12 h to give a white precipitate (LiCl). The mixture was filtered and the filtrate concentrated carefully under vacuum, yielding colourless crystals (1.63 g, 80%).

Crystal data

C₂₄H₂₉BN₂
M_r = 356.30
 Triclinic, *P* $\bar{1}$
a = 7.8909 (10) Å
b = 10.2693 (14) Å
c = 13.2831 (18) Å
 α = 86.490 (2)°
 β = 72.899 (2)°
 γ = 78.974 (3)°
V = 1009.8 (2) Å³

Z = 2
D_x = 1.172 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 4832 reflections
 θ = 3.1–26.5°
 μ = 0.07 mm⁻¹
T = 190 (2) K
 Block, colourless
 0.40 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.974, *T_{max}* = 0.987
 4212 measured reflections

3496 independent reflections
 2529 reflections with *I* > 2σ(*I*)
R_{int} = 0.026
 θ_{max} = 25.0°
h = -9 → 7
k = -12 → 11
l = -15 → 14

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.087
wR(*F*²) = 0.176
S = 1.16
 3496 reflections
 248 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0638P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.003$
 $\Delta\rho_{max} = 0.28 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.21 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

C1—B	1.621 (4)	C19—B	1.619 (5)
C10—B	1.642 (4)	B—N1	1.734 (4)
C19—B—C1	117.9 (3)	C7—N1—C8	113.1 (2)
C19—B—C10	114.5 (3)	C7—N1—C9	108.1 (2)
C1—B—C10	111.0 (3)	C8—N1—C9	107.2 (2)
C19—B—N1	108.7 (2)	C7—N1—B	102.4 (2)
C1—B—N1	96.8 (2)	C8—N1—B	114.8 (2)
C10—B—N1	105.7 (2)	C9—N1—B	111.1 (2)

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 0.96 Å and *U*_{iso}(H) = 1.5*U*_{eq}(C), but each group was allowed to rotate freely about its C—C 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 0.93–0.97 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C).

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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