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A BONBON 6-membered ring heterocycle

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Key indicators

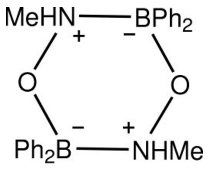
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.037
wR factor = 0.058
Data-to-parameter ratio = 15.9

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

2,5-Dimethyl-3,3,6,6-tetraphenyl-1,4-dioxa-2,5-diazonia-3,6-diboratacyclohexane, $C_{26}H_{28}B_2N_2O_2$, occupies a crystallographic inversion centre, and contains a six-membered BONBON heterocyclic ring, with a chair conformation and dative B—N bonds of length 1.640 (1) Å.

Comment

The BONBON heterocycle (I), a condensation product of *N*-methylhydroxylamine and diphenylborinic acid, is an example of substituted aminoxyboranes, the dimeric nature of which has often been postulated for aminoxy- or iminoxyboranes [references cited in Kliegel *et al.* (1992, 1994)]. A monocyclic BONBON ring was first established by an X-ray analysis of an oxime diarylborinate (Kliegel *et al.*, 1994). The same ring system is also present in several polycyclic compounds, which have been analysed by X-ray crystallography (Rettig & Trotter, 1983; Amt *et al.*, 1988; Kliegel *et al.*, 1991, 1992). Whereas in solution (^{1}H NMR) and gas phase (mass spectrum) of (I) the presence of the monomer Ph$_2$BONHMe is indicated, the crystals contain exclusively the BONBON dimer (I).



(I)

The molecule of (I) contains a centrosymmetric six-membered BONBON ring system, with a chair conformation [ring dihedral angles in a B—O—N—B section being 61.7 (1), −67.6 (1) and 61.1 (1)°], similar to that in related materials [*e.g.* Kliegel *et al.* (1994)]. The methyl substituents of the N atoms are in equatorial sites. The dative B—N bond is (typically) fairly long [1.640 (1) Å]. There is no N—H···O hydrogen bond, presumably as a result of the steric influence of the bulky phenyl substituents, the shortest intermolecular non-hydrogen contacts being C1···C12 and C13 = 3.46 and 3.48 Å.

Experimental

N-Methylhydroxylamine (0.19 g, 4 mmol) and oxybis(diphenylborane) (0.70 g, 2 mmol) were dissolved in 10 ml of ethanol and heated at boiling temperature for several minutes. Evaporation and cooling yielded 0.64 g (76%) of colorless crystals of (I) (m.p. 441–443 K). IR (KBr): 3145 cm^{-1} (N—H); ^{1}H NMR (400 MHz, CDCl$_3$/TMS), δ (p.p.m.): 1.40–1.90 (*s*, broad, exchangeable, 20% of 2H), 2.78

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(*d, J* = 6 Hz, 2CH$_3$), 6.33 (*q, J* = 6 Hz, partly exchangeable, 80% of 2H), 7.23–7.99 (*m*, 10 aromatic H); EI mass spectrum (70 eV, 513 K), *m*/*z*: 422 (1%, M^+), 211(51%, $\frac{1}{2}M^+$), 165 (100%, Ph$_2$B), 133 (28%, PhBONMe), 107 (21%), 105 (26%, PhBOH), 77 (52%, C$_6$H$_5$). Analysis calculated for C$_{26}$H$_{28}$B$_2$N$_2$O$_2$: C 73.96, H 6.70, N 6.64, B 5.12; found: C 74.03, H 6.62, N 6.40, B 4.98. The compound gives a deep-blue color reaction with diphenylcarbazone in methanolic solution, indicating the presence of a diphenylboron moiety (Neu, 1960; Friese & Umland, 1978). Crystals suitable for X-ray analysis were obtained by very slow crystallization from ethanol.

Crystal data

$C_{26}H_{28}B_2N_2O_2$
$M_r = 422.14$
Monoclinic, $P2_1/n$
$a = 6.9184\ (3)$ Å
$b = 8.8703\ (3)$ Å
$c = 18.7337\ (9)$ Å
$\beta = 95.444\ (3)°$
$V = 1144.47\ (8)$ Å^3
$Z = 2$

$D_x = 1.225$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6804 reflections
$\theta = 3.2–27.9°$
$\mu = 0.08$ mm^{-1}
$T = 173$ K
Block, colorless
0.50 × 0.30 × 0.25 mm

Data collection

Rigaku/ADSC CCD diffractometer
Area detector scans
Absorption correction: multi-scan (*d*TREK*; Molecular Structure Corporation, 1996–1998)
$T_{\min} = 0.96$, $T_{\max} = 0.98$
9941 measured reflections

2514 independent reflections
2059 reflections with $I > 3\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\max} = 27.9°$
$h = -8 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -23 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.058$
$S = 1.91$
2362 reflections
149 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2)]$
$(\Delta/\sigma)_{\max} = 0.005$
$\Delta\rho_{\max} = 0.32$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—N1	1.436 (1)	C5—C6	1.385 (2)
O1—B1	1.491 (1)	C6—C7	1.391 (2)
N1—C1	1.479 (1)	C8—C9	1.400 (2)
N1—B1$^{\rm i}$	1.640 (1)	C8—C13	1.397 (1)
C2—C3	1.400 (1)	C8—B1	1.609 (1)
C2—C7	1.395 (2)	C9—C10	1.391 (2)
C2—B1	1.616 (1)	C10—C11	1.384 (2)
C3—C4	1.393 (2)	C11—C12	1.384 (2)
C4—C5	1.372 (2)	C12—C13	1.391 (1)
N1—O1—B1	111.78 (6)	C9—C8—B1	120.25 (9)
O1—N1—C1	106.39 (7)	C13—C8—B1	122.68 (9)
O1—N1—B1$^{\rm i}$	110.95 (7)	C8—C9—C10	121.4 (1)
C1—N1—B1$^{\rm i}$	122.01 (7)	C9—C10—C11	120.2 (1)
C3—C2—C7	116.4 (1)	C10—C11—C12	119.6 (1)
C3—C2—B1	117.52 (9)	C11—C12—C13	119.9 (1)
C7—C2—B1	126.01 (9)	C8—C13—C12	121.8 (1)
C2—C3—C4	121.8 (1)	O1—B1—N1$^{\rm i}$	101.40 (7)
C3—C4—C5	120.4 (1)	O1—B1—C2	111.58 (8)
C4—C5—C6	119.3 (1)	O1—B1—C8	106.50 (7)
C5—C6—C7	120.1 (1)	N1$^{\rm i}$—B1—C2	113.50 (8)
C2—C7—C6	122.0 (1)	N1$^{\rm i}$—B1—C8	106.21 (7)
C9—C8—C13	117.08 (9)	C2—B1—C8	116.34 (8)

Symmetry code: (i) $1-x, 1-y, -z$.

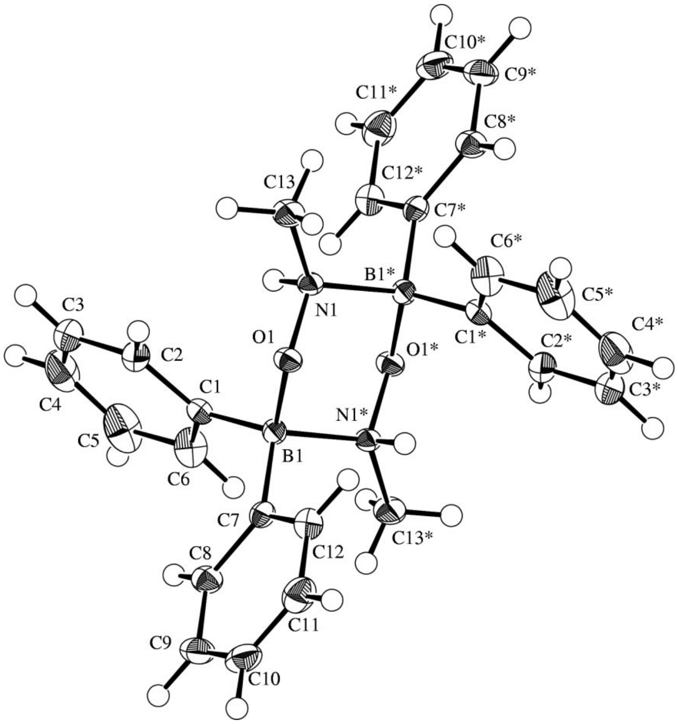

Figure 1
View of the title molecule (50% probability ellipsoids).

The H1 atom was refined isotropically, while all other H atoms were refined as riding on their attached atoms.

Data collection: *d*TREK* (Molecular Structure Corporation, 1996–1998); cell refinement: *d*TREK*; data reduction: *d*TREK*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *TEXSAN* (Molecular Structure Corporation, 1992–1997); software used to prepare material for publication: *TEXSAN*.

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