Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(2-Cyclohex-1-enylvinyl)[1,3,6,2]dioxazaborocane

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.102
Data-to-parameter ratio = 16.7

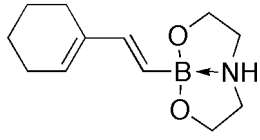
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{12}H_{20}BNO_2$, the B—N distance is 1.6720 (17) Å. Molecules are linked through intermolecular N—H···O hydrogen bonds to form infinite chains with an N···O distance of 2.8581 (13) Å.

Received 7 September 2001
Accepted 14 September 2001
Online 6 October 2001

Comment

The Diels–Alder (DA) reaction is a powerful method for the selective formation of functionalized cyclohexene derivatives. Dienylboronates have proved to be effective dienes for intermolecular DA reactions with reactive dienophiles (Vaultier *et al.*, 1987; Tailor & Hall, 2000) or intramolecular tethered DA reactions with unreactive dienophiles (Batey *et al.*, 1999). The synthetic utility of these dienes, though, can be limited due to their susceptibility to air and/or moisture. We are interested in developing stable dienylboronates that function as versatile equivalents for hetero-substituted dienes. The title compound, (I), fulfills the above criteria and is also amenable to long-term storage. Previously, it had been shown that compounds that are structurally related to (I) show promise in asymmetric Diels–Alder reactions (Wang, 1991).



(I)

The structure of the title compound is similar to that of the compound 4,5,7,8-tetrahydro-2-(2-propenyl)-6*H*-[1,3,6,2]dioxazaborocine, (II), which we have already determined (Thadani *et al.*, 2001). As in (II), moleclues of (I) are linked by intermolecular N—H···O hydrogen bonds to form infinite chains through glide-plane transformations along the *c* axis. The N···O distance in (I) is 2.8581 (13) Å for N1···O1 (see Fig. 2 and Table 2). The B1—N1 distance is 1.6720 (17) Å in (I) and 1.659 (4) Å in (II). A list of references for other dioxazaborocine compounds is included in our paper by Thadani *et al.* (2001).

Experimental

To a solution of dicyclohexyl (2-cyclohex-1-enylvinyl)boronate (Batey *et al.*, 1999) in a minimal amount of ^{i}PrOH was added

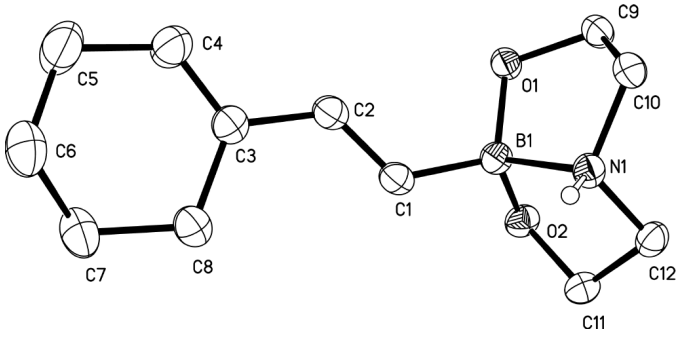


Figure 1
View of (I) showing the atom-labelling scheme. Ellipsoids are shown at the 50% probability level.

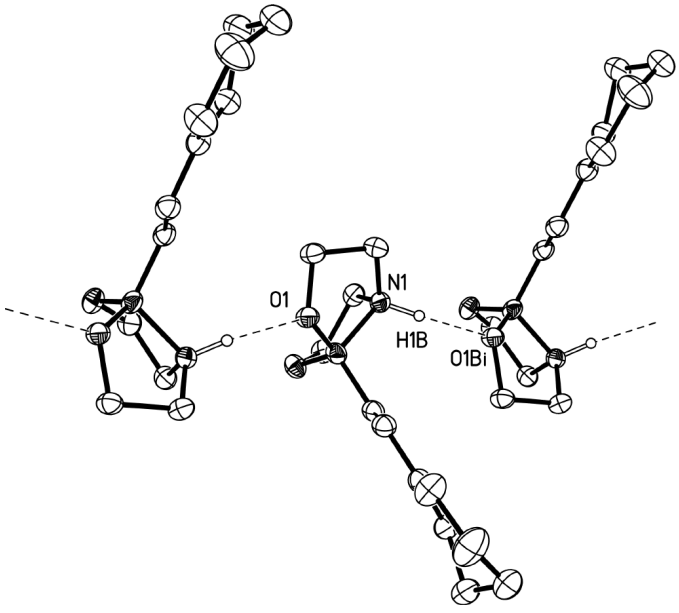


Figure 2
View of the hydrogen bonding in (I) showing the infinite chains in the *c* direction. Ellipsoids are at the 50% probability level.

diethanolamine (1 equivalent). The reaction mixture was stirred for 2 h at room temperature. The solvent was then removed under reduced pressure and the resulting solid recrystallized from acetonitrile. Compound (I) was obtained in 51% yield as clear colourless needles.

Crystal data

$C_{12}H_{20}BNO_2$
$M_r = 221.10$
Monoclinic, $P2_1/c$
$a = 11.6372\ (3)$ Å
$b = 10.4879\ (5)$ Å
$c = 9.9749\ (4)$ Å
$\beta = 100.657\ (3)^\circ$
$V = 1196.44\ (8)$ Å^3
$Z = 4$
$D_x = 1.227$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 11 487 reflections
$\theta = 4.2$–26.4°
$\mu = 0.08$ mm^{-1}
$T = 100\ (1)$ K
Needle, colourless
0.32 × 0.28 × 0.19 mm

Data collection

Nonius KappaCCD diffractometer
φ scans and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
$T_{min} = 0.975$, $T_{max} = 0.985$
11 487 measured reflections
2441 independent reflections
1870 reflections with $I > 2\sigma(I)$
$R_{int} = 0.056$
$\theta_{max} = 26.4^\circ$
$h = -14 \rightarrow 14$
$k = -13 \rightarrow 13$
$l = -12 \rightarrow 12$
Intensity decay: negligible

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.102$
$S = 1.05$
2441 reflections
146 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0485P)^2 + 0.1680P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—B1	1.4666 (17)	N1—B1	1.6720 (17)
O2—B1	1.4713 (17)	C1—B1	1.587 (2)
O1—B1—O2	113.23 (11)	O2—B1—N1	100.60 (10)
O1—B1—N1	101.66 (10)	C1—B1—N1	112.87 (10)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···O1^{i}	0.93	1.97	2.8581 (13)	158

Symmetry code: (i) $x, \frac{1}{2} - y, \frac{1}{2} + z$.

H atoms were included in calculated positions with C—H distances ranging from 0.95 to 0.99 Å and an N—H distance of 0.93 Å.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1999); program(s) used to refine structure: *SHELXTL/PC*; molecular graphics: *SHELXTL/PC*; software used to prepare material for publication: *SHELXTL/PC*.

The authors wish to acknowledge NSERC Canada and the University of Toronto.

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