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4,5,7,8-Tetrahydro-2-(2-propenyl)-6*H*-[1,3,6,2]dioxazaborocine

Avinash N. Thadani, Robert A. Batey and Alan J. Lough*

Department of Chemistry, University of Toronto, Toronto, Ontario, Canada M5S 3H6

Correspondence e-mail: alough@chem.utoronto.ca

Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.063
wR factor = 0.176
Data-to-parameter ratio = 16.5

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

In the title compound, $C_7H_{14}BNO_2$, the B—N distance is 1.659 (4) Å. Molecules are linked through intermolecular N—H···O hydrogen bonds to form infinite chains with an N···O distance of 2.907 (3) Å.

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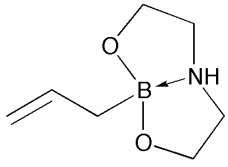
Comment

The reaction of allylboron reagents and their derivatives with carbonyl compounds is one of the most important reactions in organic synthesis. Many allylboron compounds, however, are sensitive to air and/or moisture and require special handling procedures. In view of this, the development of stable variants of allylboronic acid is an important task. We have recently synthesized potassium allyltrifluoroborate and demonstrated its synthetic utility in organic media (Batey *et al.*, 2000). The title compound, (I), is another stable variant that reacts with aldehydes to afford the corresponding homoallylic alcohols in moderate to good yields in dichloromethane. Both potassium allyltrifluoroborate and (I) are stable to air and/or moisture and can be stored for extended periods without any special precautions.



(I)

Molecules of (I) are linked by hydrogen bonds to form infinite chains through glide-plane transformations along the *c* axis, with a distance for N1···O1 of 2.903 (3) Å (see Fig. 2 and Table 2). A search of the April 2001 release of the Cambridge Structural Database (Allen & Kennard, 1993) revealed the structures of four other dioxazaborocine compounds [with refcodes COQRAB (Doidge-Harrison *et al.*, 1998), PBORXZ (Rettig & Trotter, 1975), PUTBUB (Caron & Hawkins, 1998) and SIBGIT (Howie *et al.*, 1997)]. The B—N distance in these compounds ranges from 1.657 to 1.672 Å and in (I), the B1—N1 distance is 1.659 (4) Å.

Experimental

To a solution of allylboronic acid (Brown *et al.*, 1990) (1 equivalent) in a minimal amount of ^{i}PrOH was added diethanolamine (1 equiva-

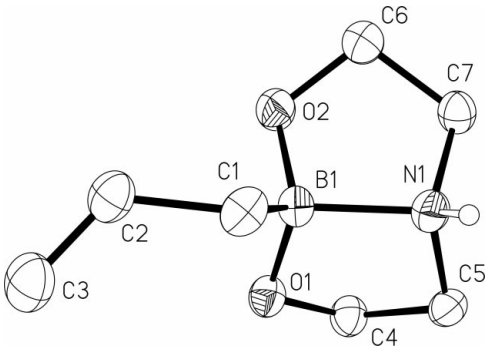


Figure 1
View of (I) showing the atom-labelling scheme. Ellipsoids are at the 50% probability level. H atoms bonded to C atoms have been removed for the sake of clarity.

lent). 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. The title compound was obtained in 66% yield as clear colourless needles.

Crystal data

$C_7H_{14}BNO_2$	D_x = 1.231 Mg m^{-3}
M_r = 155.00	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 6734 reflections
a = 21.1480 (17) Å	θ = 4.1–26.2°
b = 8.5399 (11) Å	μ = 0.09 mm^{-1}
c = 9.4229 (10) Å	T = 100 (1) K
β = 100.524 (7)°	Needle, colourless
V = 1673.2 (3) Å^3	0.40 × 0.10 × 0.07 mm
Z = 8	

Data collection

Nonius KappaCCD diffractometer	1668 independent reflections
φ scans, and ω scans with κ offsets	978 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)	R_{int} = 0.138
	θ_{max} = 26.2°
T_{min} = 0.966, T_{max} = 0.994	$h = -26 \rightarrow 26$
6735 measured reflections	$k = -10 \rightarrow 10$
	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.063	$w = 1/[\sigma^2(F_o^2) + (0.0903P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.176	$(\Delta/\sigma)_{max} < 0.001$
S = 0.99	$\Delta\rho_{max}$ = 0.28 e Å^{-3}
1668 reflections	$\Delta\rho_{min}$ = −0.33 e Å^{-3}
101 parameters	

Table 1
Selected geometric parameters (Å, °).

O1—B1	1.463 (4)	N1—B1	1.659 (4)
O2—B1	1.466 (4)	C1—B1	1.606 (4)
O1—B1—O2	111.3 (2)	O2—B1—N1	101.2 (2)
O1—B1—N1	102.2 (2)	C1—B1—N1	112.8 (2)

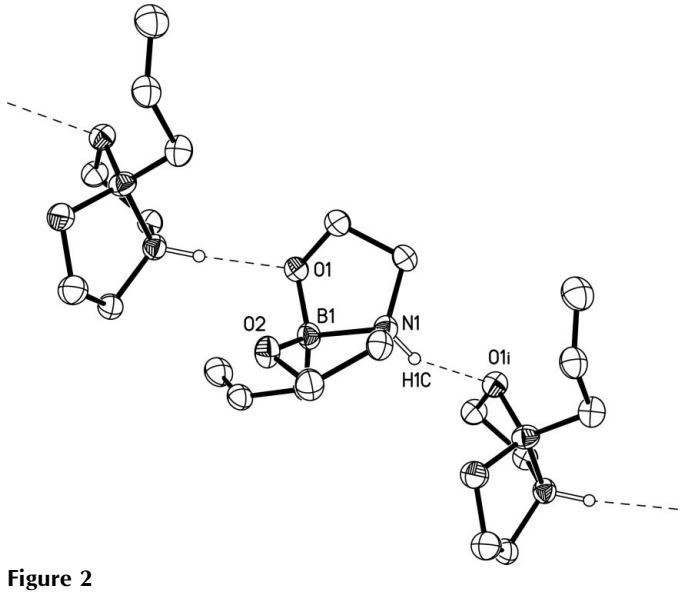


Figure 2
View of the hydrogen bonding in (I) showing infinite chains in the c-axis direction. Ellipsoids are at the 50% probability level. H atoms bonded to C atoms have been removed for the sake of clarity.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1C···O1^{i}	0.93	2.04	2.907 (3)	155

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

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, 1997–2001); 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*.

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