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(*E*)-2-Styryl-[1,3,6,2]dioxazaborolane

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.030
wR factor = 0.080
Data-to-parameter ratio = 8.4

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

In the title compound, $C_{12}H_{16}BNO_2$, the B—N distance is 1.662 (4) Å. Molecules are linked through intermolecular N—H···O hydrogen bonds to form infinite chains with an N···O distance of 2.810 (2) Å.

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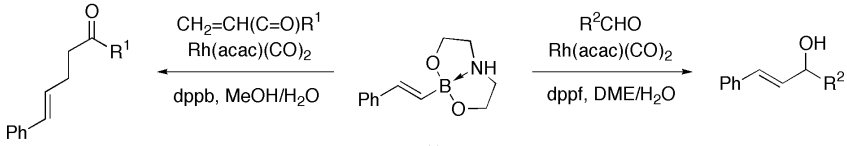
Comment

The title compound (I) shows high reactivity in rhodium(I)-catalysed 1,4-additions to α,β-unsaturated carbonyl compounds and additions to aldehydes [see Scheme below, in which dppb = 1,4-bis(diphenylphosphino)butane and dppf = 1,1′-bis(diphenylphosphino)ferrocene] (Batey & Smil, 2001). Use of (*E*)-2-phenyl-1-ethenylboronic acid, along with other aryl- and alkenylboronic acids, in 1,4-additions to α,β-unsaturated carbonyl compounds (Sakai *et al.*, 1997) and additions to aldehydes (Sakai *et al.*, 1998) has been reported, but the additional stability of diethanolamine boronate (I) to air and water makes its preparation, isolation, storage and handling more facile.



(I)

The structure of (I) is similar to that of the compounds 4,5,7,8-tetrahydro-2-(2-propenyl)-6*H*-[1,3,6,2]-dioxazaborocine (II) (Thadani *et al.*, 2001*a*) and 2-(2-cyclohex-1-enyl-vinyl)-[1,3,6,2] dioxazaborocane (III) (Thadani *et al.*, 2001*b*), which we have already determined. Molecules of (I) are linked by hydrogen bonds to form infinite chains through 2_1 screw axes in the **c** direction. The N···O distance in (I) is 2.810 (2) Å for N1···O1 (see Fig. 2 and Table 2). The B1—N1 distance is 1.662 (4) Å in (I), 1.6720 (17) Å in (II) and 1.659 (4) Å in (III). A list of references for other dioxazaborocine compounds is included in our earlier paper (Thadani *et al.*, 2001*a*).

Experimental

Crystals of (I) were obtained by treatment of (*E*)-2-phenyl-1-ethenylboronic acid, dissolved in a minimal amount of 2-propanol, with diethanolamine (1 equiv.). The mixture was stirred for 2 h at 298 K prior to collection of the precipitate by filtration. Recrystallization of the solid from acetonitrile gave (I) in 69% yield.

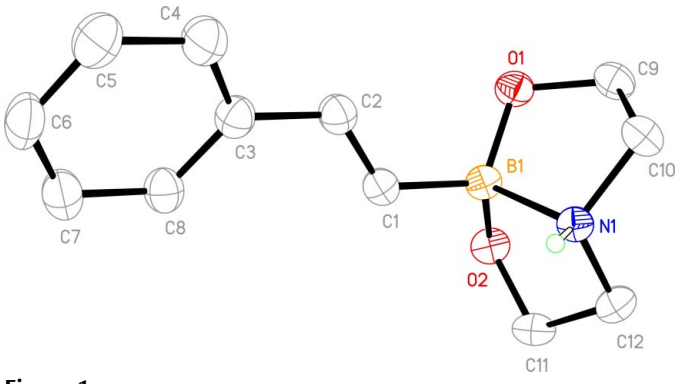


Figure 1
View of (I) showing the atom labelling scheme. Ellipsoids are drawn at the 50% probability level.

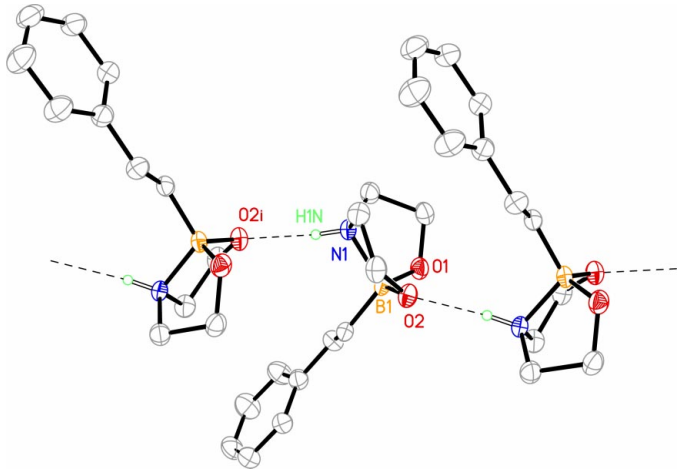


Figure 2
View of the hydrogen bonding in (I) showing infinite chains in the **c** direction. Ellipsoids are drawn at the 50% probability level.

Crystal data

$C_{12}H_{16}BNO_2$
$M_r = 217.07$
Orthorhombic, $Pca2_1$
$a = 13.3725\ (4)$ Å
$b = 9.7045\ (3)$ Å
$c = 8.9200\ (2)$ Å
$V = 1157.58\ (6)$ Å^3
$Z = 4$
$D_x = 1.246$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8659 reflections
$\theta = 4.2$–$26.4°$
$\mu = 0.08$ mm^{-1}
$T = 150\ (1)$ K
Needle, colourless
0.43 × 0.25 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans with κ offsets
8659 measured reflections
1259 independent reflections
1167 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 26.4°$
$h = -16 \rightarrow 16$
$k = -12 \rightarrow 12$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.080$
$S = 1.08$
1259 reflections
150 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0467P)^2 + 0.1149P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.026 (6)

Table 1
Selected geometric parameters (Å, °).

O1—B1	1.446 (2)	N1—B1	1.663 (2)
O2—B1	1.477 (2)	C1—B1	1.591 (3)
O1—B1—O2	113.45 (16)	O2—B1—C1	113.36 (14)
O1—B1—C1	113.99 (14)	C1—B1—N1	111.57 (15)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O2^{i}	0.84 (3)	1.97 (3)	2.810 (2)	175 (2)

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

H atoms were included in calculated positions with C—H distances ranging from 0.95 to 0.99 Å, and the H atom attached to N was refined with an isotropic thermal parameter.

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, 2001); 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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