C13	0.7807 (2)	0.1041 (2)	0.1586 (3)	0.037 (1)
C14	0.7044 (4)	0.2402 (2)	0.1552 (5)	0.065 (2)
C15	0.5774 (2)	0.0398 (2)	0.3245 (3)	0.035 (1)
C16	0.4086 (3)	0.0602 (3)	0.1919 (5)	0.062 (1)

Table 2. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å²) *for* (2*b*)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
S	0.64182 (8)	0.18083 (4)	0.85445 (4)	0.0600 (4)
O1	0.8484 (2)	0.0486 (1)	0.8058 (1)	0.052 (1)
O2	0.7124 (2)	0.0166 (1)	0.5708 (1)	0.064 (1)
O3	0.9789 (3)	−0.0161 (1)	0.6103 (1)	0.079 (1)
C1	1.0246 (3)	0.4209 (2)	0.7136 (1)	0.051 (1)
C2	1.1946 (4)	0.4453 (2)	0.7099 (2)	0.067 (1)
C3	1.3158 (3)	0.3814 (2)	0.6896 (2)	0.068 (1)
C4	1.2735 (3)	0.2903 (2)	0.6717 (2)	0.057 (1)
C4a	1.1054 (2)	0.2638 (1)	0.6773 (1)	0.041 (1)
C5	0.7783 (3)	0.1991 (1)	0.4601 (1)	0.046 (1)
C6	0.6985 (3)	0.2710 (2)	0.4139 (1)	0.052 (1)
C7	0.6490 (3)	0.3505 (2)	0.4587 (1)	0.049 (1)
C8	0.6775 (2)	0.3599 (1)	0.5505 (1)	0.040 (1)
C8a	0.7522 (2)	0.2876 (1)	0.5974 (1)	0.033 (1)
C9	0.8090 (2)	0.2841 (1)	0.6956 (1)	0.032 (1)
C9a	0.9821 (2)	0.3294 (1)	0.6978 (1)	0.037 (1)
C10	1.0311 (2)	0.1695 (1)	0.6771 (1)	0.039 (1)
C10a	0.8030 (2)	0.2077 (1)	0.5521 (1)	0.036 (1)
C11	0.8521 (2)	0.1804 (1)	0.7113 (1)	0.033 (1)
C12	0.8817 (2)	0.1407 (1)	0.6166 (1)	0.036 (1)
C13	0.7932 (2)	0.1248 (1)	0.7888 (1)	0.036 (1)
C14	0.6206 (5)	0.1016 (2)	0.9464 (2)	0.063 (1)
C15	0.8659 (3)	0.0381 (1)	0.5998 (1)	0.046 (1)
C16	0.6836 (6)	−0.0797 (2)	0.5466 (3)	0.087 (2)

Table 3. *Selected bond lengths* (Å) *and angles* (°)

	(2*a*)	(2*b*)
C═S	1.630 (3)	
C—SMe		1.753 (2)
S—Me		1.790 (3)
C—C (aromatic)	1.367 (5)–1.395 (4)	1.368 (4)–1.398 (3)
C—COSMe	1.458 (4)	1.483 (3)
C—CO_2Me	1.496 (4)	1.509 (3)
C═O	1.199 (3)	1.210 (2), 1.197 (3)
C—OMe	1.326 (3), 1.331 (3)	1.319 (3)
O—Me	1.448 (4), 1.446 (4)	1.455 (4)
C—S—C		101.5 (1)
C—O—C	119.6 (2), 117.4 (2)	116.5 (2)
C—C—C (external)	128.4 (2)–130.0 (3)	128.7 (2)–129.3 (2)

For both compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1990). Program(s) used to solve structures: *TEXSAN*. Program(s) used to refine structures: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

We thank the Natural Sciences and Engineering Research Council of Canada for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, together with synthesis and spectral details for compounds (1*a*), (1*b*), (2*a*) and (2*b*), have been deposited with the IUCr (Reference: FG1099). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Styrylboronic Acid

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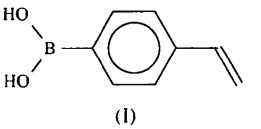
Abstract

Styrylboronic acid, $C_8H_9BO_2$, was one of several organoboron compounds investigated for their wood-preservation properties following *in situ* polymerization. The structure consists of independent monomeric molecules bound together by strong hydrogen bonds (B—O—H···O). The planes of the styryl and boronic acid moieties are twisted by 26 (1)° with respect to each other, apparently as a result of the hydrogen-bonding requirements.

Comment

The title compound, (I), was synthesized by reaction of the Grignard reagent prepared from 4-bromostyrene with tri-*n*-butyl borate, followed by hydrolysis (full details in supplementary material). Extremely thin (< 0.03 mm) large plates and intergrown needles were isolated by recrystallization from water. Recrystallization from other solvents gave unsatisfactory crystals.



(I)

One of the plates (Figs. 1 and 2, Table 1) consists of strongly hydrogen-bonded (B—O—H···O) molecules with a 26(1)° angle between the styryl and boronic acid [$B(OH)_2$] planes. This compares with 6.6 and 21.4° found in phenylboronic acid (Rettig & Trotter, 1977). Two types of hydrogen bonding are observed: between end-on pairs forming dimers across the crystallographic centres of symmetry along the *a* axis [O(1)···H(O2) 1.91 (10) Å, labelled *A* on Fig. 2], and between adjacent molecules along the short *b* axis [O(2)···H(O1) 2.21 (13) Å, labelled *B*]. The structure can be described, therefore, as hydrogen-bonded dimer chains running in the direction of the *b* axis. In phenylboronic acid, similar dimer units are bound by hydrogen bonds into an infinite array.

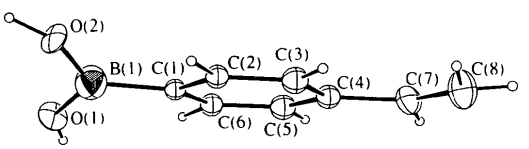


Fig. 1. The structure of styrylboronic acid showing atom labels, with displacement ellipsoids at the 30% probability level for non-H atoms.

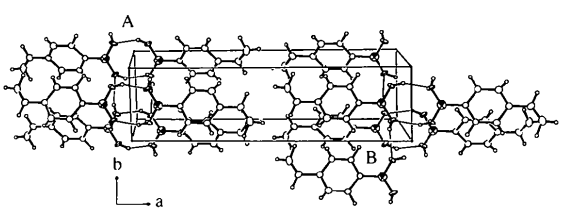


Fig. 2. View of the unit cell down the *c* axis; hydrogen bonds are shown with thin lines.

The 'unexpected' non-planarity of the molecule may be the result of the hydrogen-bonding requirements and these may play a part in *in situ* reactions such as polymerization. There are no significant deviations from expected bond lengths and angles in the molecule.

Experimental

A Grignard reagent is formed by the reaction of 4-bromostyrene with Mg in tetrahydrofuran. It is then reacted with tri-*n*-butyl borate to form the butyl ester of styrylboronic acid which is readily hydrolysed to styrylboronic acid. Thus, to a suspension of magnesium (1.5 g, 62.5 mmol) in anhydrous THF (10 ml) was added (after initiation with 1,2-dibromomethane) a solution of 4-bromostyrene (7.0 g, 38.3 mmol) in THF (15 ml). The rate of addition was controlled so as to maintain gentle reflux. After 2 h the solution was cooled to 195 K and tri-*n*-butyl borate (17.6 g, 76.6 mmol) added dropwise (*ca* 2 h) before being left to warm to ambient temperature overnight. The solution was diluted with ether (200 ml) and washed with hydrochloric acid (1 *M*, 50 ml), water (50 ml) and finally with sodium hydroxide solution (10%, 25 ml). The alkaline phase was acidified with dilute acid and extracted with ether (3 × 50 ml); the ether phase was then dried over anhydrous magnesium sulfate and concentrated to give a pale yellow solid. Two recrystallizations from water furnished lustrous off-white plates (2.82 g, 50%) that were identified by ^1H and ^{13}C NMR spectroscopy.

Crystal data

$C_8H_9BO_2$
$M_r = 148.0$
Monoclinic
$P2_1/c$
$a = 19.362\,(16)$ Å
$b = 5.128\,(6)$ Å
$c = 8.159\,(6)$ Å
$\beta = 100.11\,(6)°$
$V = 797.5\,(13)$ Å^3
$Z = 4$
$D_x = 1.232$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 21 reflections
$\theta = 2.0$–$18.0°$
$\mu = 0.09$ mm^{-1}
$T = 193$ K
Plate
0.52 × 0.20 × 0.03 mm
Colourless

Data collection

Nicolet *R3m* four-circle diffractometer
ω scans
Absorption correction: none
1543 measured reflections
1238 independent reflections
317 observed reflections [$I > 2.5\sigma(I)$]
$R_{int} = 0.065$
$\theta_{max} = 24°$
$h = -22 \rightarrow 22$
$k = 0 \rightarrow 5$
$l = 0 \rightarrow 9$
3 standard reflections monitored every 97 reflections
intensity decay: 2%

Refinement

Refinement on *F*
$R = 0.055$
$wR = 0.055$
$S = 1.36$
317 reflections
99 parameters
$w = 1/[\sigma^2(F) + 0.0006F^2]$
$(\Delta/\sigma)_{max} = 0.03$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2)

B_{iso} for C(1)–C(6) (phenyl); $B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$ for others.

	x	*y*	*z*	B_{eq}/B_{iso}
O(1)	0.0665 (5)	0.2207 (17)	0.4827 (13)	4.3 (5)
O(2)	0.0665 (4)	−0.2267 (16)	0.4443 (9)	3.6 (4)
C(1)	0.1802 (4)	0.004 (3)	0.4226 (11)	2.1 (2)
C(2)	0.2073 (4)	−0.188 (2)	0.3338 (12)	2.3 (2)
C(3)	0.2764 (5)	−0.193 (2)	0.3121 (13)	2.9 (3)
C(4)	0.3229 (4)	0.001 (3)	0.3841 (11)	2.6 (2)
C(5)	0.2961 (5)	0.191 (2)	0.4755 (13)	3.2 (3)
C(6)	0.2267 (5)	0.197 (2)	0.4937 (11)	2.6 (2)
C(7)	0.3977 (5)	0.003 (3)	0.3691 (15)	3.4 (7)
C(8)	0.4281 (6)	−0.163 (3)	0.2906 (18)	4.8 (7)
B(1)	0.1019 (6)	0.002 (4)	0.4514 (15)	5.5 (8)

Table 2. *Selected geometric parameters* (Å, °)

O(1)—B(1)	1.361 (19)	C(3)—C(4)	1.401 (15)
O(2)—B(1)	1.356 (19)	C(4)—C(5)	1.383 (16)
C(1)—C(2)	1.379 (15)	C(4)—C(7)	1.475 (13)
C(1)—C(6)	1.395 (14)	C(5)—C(6)	1.378 (13)
C(1)—B(1)	1.574 (13)	C(7)—C(8)	1.272 (21)
C(2)—C(3)	1.379 (13)		

C(2)—C(1)—C(6)	116.6 (8)	C(5)—C(4)—C(7)	120.4 (10)
C(2)—C(1)—B(1)	122.6 (10)	C(1)—C(6)—C(5)	120.6 (9)
C(6)—C(1)—B(1)	120.7 (10)	C(4)—C(7)—C(8)	125.8 (12)
C(1)—C(2)—C(3)	123.3 (9)	O(1)—B(1)—O(2)	116.9 (9)
C(2)—C(3)—C(4)	119.9 (9)	O(1)—B(1)—C(1)	123.6 (13)
C(3)—C(4)—C(5)	116.9 (8)	O(2)—B(1)—C(1)	119.5 (13)
C(3)—C(4)—C(7)	122.7 (10)		

B(1)—C(1)—C(2)—C(3)	−177.9 (12)	C(2)—C(1)—B(1)—O(2)	24.1 (5)
C(2)—C(1)—B(1)—O(1)	−155.6 (14)	C(6)—C(1)—B(1)—O(2)	−152.9 (14)
C(6)—C(1)—B(1)—O(1)	27.5 (6)		

Direct methods were used to solve the structure. H atoms and phenyl C atoms [C(1)–C(6)] were refined with isotropic displacement parameters. Two grouped displacement parameters for H atoms were employed: (i) for those attached to O atoms; (ii) for those attached to C atoms.

Data collection: *Nicolet R3m Software* (Siemens, 1983). Cell refinement: *Nicolet R3m Software*. Data reduction: *Nicolet R3m Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *NRCVAX* (Larson *et al.*, 1991). Molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1036). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5-Iodocytosine

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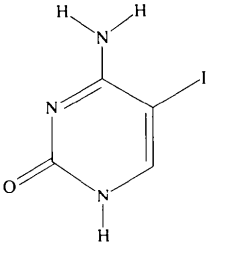
Abstract

There are two independent molecules in the asymmetric unit of the title compound, 4-amino-5-iodo-2(1*H*)-pyrimidinone, $C_4H_4IN_3O$. No significant conformational difference was found between the two molecules. The molecules are held together by N—H···O and N—H···N hydrogen bonds.

Comment

The crystal structures of 5-fluorocytosine (Louis, Low & Tollin, 1982) and 5-bromocytosine (Kato, Takenaka & Sasada, 1979) have been determined as the fundamental compounds for the investigation of the halogenation effect on the base moiety and the interaction between nucleic acids and proteins. Recently, it has been reported that cytosine deaminase can convert the antifungal agent 5-fluorocytosine into the antitumor agent 5-fluorouracil. This suggests importance of halogenated cytosines as medicinal substances (Wallace *et al.*, 1994). Thus, it seemed important to determine the structure of 5-iodocytosine, (I), one of the halogenated derivatives of cytosine.



(I)

The molecular packing is stabilized by intermolecular hydrogen bonds: N(1)—H(1)···N(3′)(x, 1+y, −1+z) 2.82 (1) Å, N(1′)—H(1′)···N(3)(1/2+x, −3/2+y, 1+z) 2.83 (1) Å, N(4)—H(41)···O(2′)(−1/2+x, 3/2+y, −1+z) 2.90 (1) Å and N(4′)—H(4′1)···O(2)(x, −1+y, 1+z) 2.91 (1) Å.

Note added in proof. Examination of the asymmetric set of atomic coordinates using *Xtal-GX* (Hall, 1995) indicated the presence of a screw-like relationship, although (at the time of going to press) it is uncertain whether this has space-group implications or is non-crystallographic.

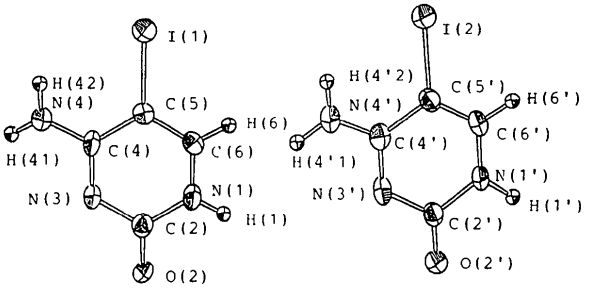


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound with the atomic numbering scheme, viewed along the *b* axis. Ellipsoids for non-H atoms correspond to 50% probability.