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5-Formyl-2-furanboronic acid at 100 K

Krzysztof Ejsmont,[a] Jacek Zaleski,[a]* Andrzej Sporzyński[b] and Michał Lewandowski[b]

[a]Institute of Chemistry, University of Opole, Oleska 48, 45-052 Opole, Poland, and [b]Faculty of Chemistry, Warsaw University of Technology, Noakowskiego 3, 00-664 Warsaw, Poland

Correspondence e-mail: zaleski@uni.opole.pl

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Key indicators

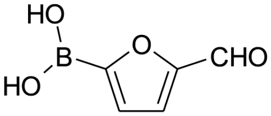
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.132
Data-to-parameter ratio = 10.1

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

The furan ring in the title compound, $C_5H_5BO_4$, is planar and each of the formyl and boronic groups makes a dihedral angle of *ca* 3° with this ring. The geometry of the furan ring is somewhat different to that found for substituted and unsubstituted furan structures. The molecules are connected to each other in the *bc* plane by C—H···O and O—H···O hydrogen bonds.

Comment

Arylboronic acids, ArB(OH)$_2$, have been known since the end of the nineteenth century. However, attention has recently been paid to these compounds owing to their new applications (Cuthbertson, 1998). The main synthetic application is in Suzuki coupling: reaction of aryl halides with arylboronic acids (Miyaura *et al.*, 1981; Miyaura & Suzuki, 1995; ChemFiles, 2003). Other applications are in asymmetric synthesis using chiral boronic acids (Currie *et al.*, 2000) or analytical use as molecular sensors (Ward *et al.*, 2002). These compounds are also used in medicine, *e.g.* for boron neutron-capture therapy (BNCT) (Soloway *et al.*, 1998) or as virus enzyme inhibitors (Priestley & Decicco, 2000). There are only a few examples of the crystal structures of boronic acids. In these structures, the B(OH)$_2$ group is attached to a phenyl ring (Feulner *et al.*, 1990; Gainsford *et al.*, 1995; Scouten *et al.*, 1994), pyridine ring (Parry *et al.*, 2002) or five-membered cyclopentadienyl ring (Norrild & Sotofte, 2001).



(I)

We present here the crystal structure of 5-formyl-2-furanboronic acid, (I), in which the boronic acid group is a substituent of a furan ring (Fig. 1 and Table 1). The B—O bond lengths are insignificantly different, the B6—O7 bond being shorter than the B6—O8 bond by about 0.02 Å. Similar differences have been reported for 4-carboxy-2-nitrobenzeneboronic acid (0.21 Å; Soundararajan *et al.*, 1993) and L-*p*-boronophenylalanine (0.31 Å; Shull *et al.*, 2000). The bond angles around atom B6 are distorted from the value of 120°; the O8—B6—C2 angle is 3.8° greater than 120°, whereas the O7—B6—C2 angle is 4.9° less than 120° and O7—B6—O8 is closest to the expected value. A similar geometry for the B(OH)$_2$ acid group is observed in 2-bromo-5-pyridylboronic acid structures (Parry *et al.*, 2002). The formyl and B(OH)$_2$

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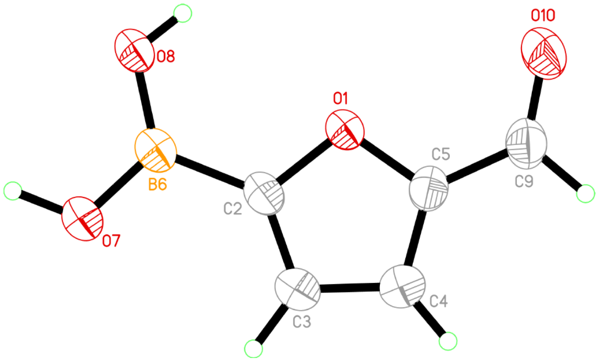

Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

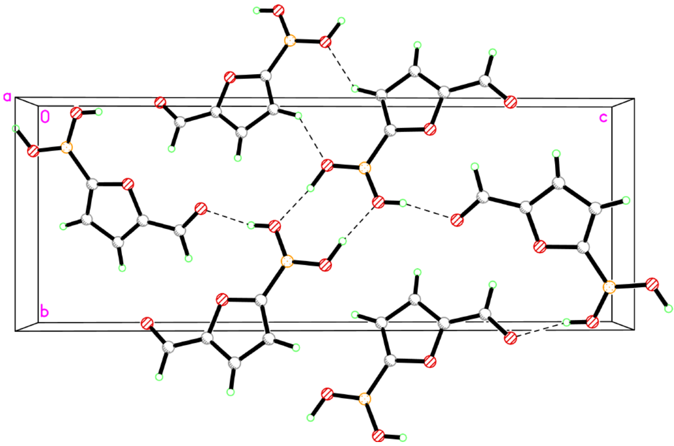

Figure 2
The packing diagram of the title compound, showing the hydrogen-bonding scheme (dashed lines).

groups in (I) are essentially coplanar with the furan ring. The dihedral angles between these planes and the furan ring are less than 3°. The five atoms of the furan ring are coplanar. The geometry of the furan ring in (I) is somewhat different to that of both unsubstituted furan rings [(II); Fourme, 1972] and substituted 5-nitro-2-furancarboxylic acid [(III); Alcock *et al.*, 1996]. The C3—C4 bond length is similar to that found in (III) but it is shorter than that observed in (II) by 0.25 Å. The remaining bond lengths *viz.* C—C$_\alpha$ (C2—C3 and C4—C5) and O—C$_\alpha$ (O1—C2 and O1—C5) are slightly different from those observed in (II) and (III). The O—C$_\alpha$—C and C$_\alpha$—O—C$_\alpha$ angles in the furan ring of (I) are closer to those in (II) than in (III).

The molecular network in the crystal consists of two nearly linear strong O—H$\cdots$O and one weaker C—H$\cdots$O hydrogen bond (Fig. 2 and Table 2). The O7—H7$\cdots$O8 hydrogen bond forms dimers of (I); this is characteristic of arylboronic acids in solid state (Alcock *et al.*, 1996; Feulner *et al.*, 1990; Gainsford *et al.*, 1995; Scouten *et al.*, 1994).

Experimental

5-Formyl-2-furanboronic acid was obtained from Aldrich.

Crystal data

$C_5H_5BO_4$
$M_r = 139.90$
Monoclinic, $P2_1/n$
$a = 3.7550\ (8)$ Å
$b = 7.758\ (2)$ Å
$c = 20.694\ (4)$ Å
$\beta = 91.31\ (3)°$
$V = 602.7\ (2)$ Å^3
$Z = 4$
$D_x = 1.542$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3391 reflections
$\theta = 3.3$–$25.5°$
$\mu = 0.13$ mm^{-1}
$T = 100.0\ (2)$ K
Cube, colourless
$0.20 \times 0.18 \times 0.16$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer
ω scans
Absorption correction: none
3391 measured reflections
1126 independent reflections
764 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.089$
$\theta_{\rm max} = 25.5°$
$h = -4 \rightarrow 3$
$k = -9 \rightarrow 9$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.132$
$S = 0.95$
1126 reflections
111 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0811P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.16$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C5	1.371 (2)	C4—C5	1.355 (3)
O1—C2	1.382 (2)	C5—C9	1.430 (3)
C2—C3	1.360 (3)	B6—O7	1.340 (3)
C2—B6	1.557 (3)	B6—O8	1.357 (3)
C3—C4	1.400 (3)	C9—O10	1.222 (3)
C5—O1—C2	106.6 (2)	C4—C5—C9	130.0 (2)
C3—C2—O1	108.8 (2)	O1—C5—C9	120.0 (2)
C3—C2—B6	131.3 (2)	O7—B6—O8	121.1 (2)
O1—C2—B6	119.9 (2)	O7—B6—C2	115.1 (2)
C2—C3—C4	107.7 (2)	O8—B6—C2	123.8 (2)
C5—C4—C3	106.8 (2)	O10—C9—C5	125.6 (2)
C4—C5—O1	110.0 (2)		
C5—O1—C2—B6	177.8 (2)	O1—C2—B6—O7	−178.8 (2)
B6—C2—C3—C4	−177.6 (2)	C3—C2—B6—O8	178.4 (2)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O7—H7$\cdots$O8$^{\rm i}$	0.98 (4)	1.82 (4)	2.799 (2)	173 (3)
O8—H8$\cdots$O10$^{\rm ii}$	0.82 (3)	1.93 (3)	2.729 (2)	164 (3)
C3—H3$\cdots$O7$^{\rm iii}$	0.99 (2)	2.55 (2)	3.355 (3)	139 (2)

Symmetry codes: (i) $1 - x, -y, -z$; (ii) $\frac{3}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iii) $-x, 1 - y, -z$.

Data collection: *CrysAlisCCD* (Oxford Diffraction, 2002); cell refinement: *CrysAlisRED* (Oxford Diffraction, 2002); data reduction: *CrysAlisRED*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXL*97.

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