Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

[3-(Propenamido)phenyl]boronic acid

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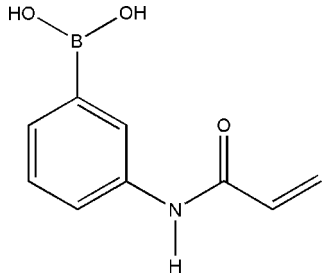
Received 28 August 2007; accepted 1 November 2007

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.108; data-to-parameter ratio = 9.6.

The title compound, $C_9H_{10}BNO_3$, was synthesized for investigation as a potential precursor for use in affinity chromatography and glucose sensing. Weak N—H···O and O—H···O hydrogen-bonding interactions are observed in the molecular structure. The dihedral angle between the benzene ring and the BO_2 plane is 35.1 (2)°.

Related literature

For related literature, see: Zhang *et al.* (2007); Chen *et al.* (2006); Deutsch & Osoling (1949).



Experimental

Crystal data

$C_9H_{10}BNO_3$
M_r = 190.99
Orthorhombic, *Fdd*2
a = 18.626 (4) Å
b = 42.112 (8) Å
c = 5.0536 (10) Å
V = 3963.9 (13) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 295 (2) K
0.43 × 0.18 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: numerical (*APEX2*; Bruker, 2006)
T_{min} = 0.87, T_{max} = 0.99
14806 measured reflections
1657 independent reflections
1279 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.108
S = 1.04
1657 reflections
172 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O2^{i}	0.77 (7)	2.02 (7)	2.759 (3)	163 (7)
O2—H2B···O3^{ii}	0.77 (8)	2.10 (9)	2.748 (3)	143 (7)
O3—H3A···O2^{iii}	0.75 (8)	2.24 (8)	2.748 (3)	126 (7)
O3—H3B···O3^{i}	0.87 (5)	1.88 (5)	2.746 (3)	173 (6)
N1—H1A···O1^{iv}	0.93 (3)	1.98 (3)	2.911 (3)	172 (2)

Symmetry codes: (i) $-x+1, -y, z$; (ii) $x, y, z-1$; (iii) $x, y, z+1$; (iv) $x-\frac{1}{4}, -y+\frac{1}{4}, z-\frac{1}{4}$.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXTL*.

We acknowledge the Canadian Government (NSERC) for funding.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KJ2069).

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  doi:10.1107/S1600536807055389

supplementary materials

Acta Cryst. (2007). E**63**, o4628 [**doi:10.1107/S1600536807055389**]

[3-(Propenamido)phenyl]boronic acid

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Comment

The title compound is a monomer containing a boronate group, which can form complexes with *cis* diols at pH values above 9 (Deutsch & Osoling, 1949). This interaction can be employed for affinity chromatography and glucose sensing (Chen *et al.*, 2006).

The molecular structure of the title compound is illustrated in Fig.1. Weak N—H···O and O—H···O hydrogen bonding interactions are observed in the molecular structure which help to stabilize the crystal structure and forming an infinite threedimensional network, as illustrated in the packing diagram displayed in Fig. 2. The dihedral angle between the benzene ring and the plane composed of O2—B1—O3 is 35.12(0.23)°.

Experimental

The synthesis of (I) was described previously (Zhang *et al.*, 2007). Single crystals suitable for X-ray analysis were obtained by slow evaporation of a water solution at room temperature.

Refinement

Friedel pairs were merged prior to refinement using *SHELXTL* (Bruker, 2000). The positions of the H atoms were determined using the difference map and were refined isotropically. The H atoms on O2 and O3 were disordered over 2 sites with 50% occupancy in each site. The disordered H atoms were refined with one common U_{iso}.

Figures

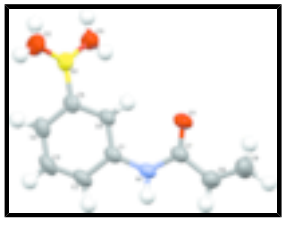

Fig. 1. The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size. Only one component of the disorder is shown.

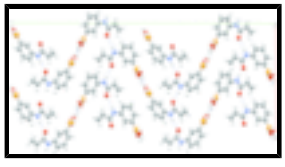

Fig. 2. The crystal packing of (II), viewed along the *c* axis. Only one component of the disorder is shown. H-bonds are shown as dashed lines.

[3-(Propenamido)phenyl]boronic acid

Crystal data

$C_9H_{10}BNO_3$	F_{000} = 1600
M_r = 190.99	D_x = 1.280 Mg m^{-3}
Orthorhombic, *Fdd*2	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: F 2 -2d	Cell parameters from 2853 reflections
a = 18.626 (4) Å	θ = 2.4–25.1°
b = 42.112 (8) Å	μ = 0.09 mm^{-1}
c = 5.0536 (10) Å	*T* = 295 (2) K
V = 3963.9 (13) Å^3	Needle, colourless
Z = 16	0.43 × 0.18 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer	1657 independent reflections
Radiation source: fine-focus sealed tube	1279 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.035
T = 295(2) K	θ_{max} = 30.5°
φ and ω scans	θ_{min} = 1.9°
Absorption correction: numerical (face correction with APEX2; Bruker, 2006)	*h* = −26→25
T_{min} = 0.87, T_{max} = 0.99	*k* = −59→59
14806 measured reflections	*l* = −7→2

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.039	$w = 1/[\sigma^2(F_o^2) + (0.0606P)^2 + 0.5557P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.108	$(\Delta/\sigma)_{max}$ = 0.007
S = 1.04	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
1657 reflections	$\Delta\rho_{min}$ = −0.12 e Å^{-3}
172 parameters	Extinction correction: none
1 restraint	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.34232 (7)	0.11569 (4)	1.1454 (4)	0.0653 (5)	
O2	0.43076 (12)	0.01158 (6)	0.3738 (4)	0.0779 (6)	
H2A	0.466 (3)	0.0024 (17)	0.394 (16)	0.082 (10)*	0.50
H2B	0.413 (4)	0.0131 (15)	0.237 (17)	0.082 (10)*	0.50
O3	0.43531 (9)	0.01566 (4)	0.8312 (3)	0.0569 (4)	
H3A	0.414 (4)	0.0220 (15)	0.944 (17)	0.082 (10)*	0.50
H3B	0.478 (3)	0.0072 (14)	0.827 (14)	0.082 (10)*	0.50
N1	0.23312 (9)	0.10547 (4)	0.9628 (3)	0.0476 (4)	
H1A	0.1859 (16)	0.1131 (6)	0.949 (6)	0.072 (8)*	
B1	0.39944 (13)	0.02160 (6)	0.6027 (5)	0.0469 (5)	
C1	0.24915 (10)	0.08023 (4)	0.7901 (4)	0.0442 (4)	
C2	0.19502 (12)	0.07111 (5)	0.6107 (5)	0.0567 (5)	
H2	0.1493 (13)	0.0830 (5)	0.624 (6)	0.059 (6)*	
C3	0.20685 (14)	0.04743 (6)	0.4318 (5)	0.0633 (6)	
H3	0.1704 (15)	0.0419 (6)	0.324 (7)	0.068 (7)*	
C4	0.27236 (13)	0.03202 (5)	0.4223 (4)	0.0543 (5)	
H4	0.2783 (13)	0.0162 (6)	0.288 (6)	0.060 (7)*	
C5	0.32638 (10)	0.03966 (4)	0.6037 (4)	0.0447 (4)	
C6	0.31372 (10)	0.06372 (5)	0.7880 (4)	0.0433 (4)	
H6	0.3479 (12)	0.0696 (5)	0.914 (5)	0.055 (6)*	
C7	0.27804 (10)	0.12142 (5)	1.1262 (4)	0.0478 (4)	
C8	0.24262 (14)	0.14649 (6)	1.2856 (6)	0.0646 (6)	
H8	0.1923 (18)	0.1488 (7)	1.258 (8)	0.085 (9)*	
C9	0.2770 (2)	0.16236 (8)	1.4653 (8)	0.0938 (11)	
H9A	0.2548 (17)	0.1772 (8)	1.613 (9)	0.101 (10)*	
H9B	0.322 (2)	0.1570 (8)	1.511 (9)	0.105 (12)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0357 (7)	0.0729 (9)	0.0873 (12)	0.0017 (6)	−0.0151 (8)	−0.0233 (9)
O2	0.0824 (15)	0.1155 (17)	0.0359 (9)	0.0226 (11)	0.0051 (9)	−0.0134 (10)

O3	0.0593 (10)	0.0778 (11)	0.0334 (7)	0.0152 (8)	−0.0019 (6)	−0.0005 (7)
N1	0.0334 (8)	0.0568 (9)	0.0526 (9)	0.0036 (7)	−0.0099 (7)	−0.0050 (8)
B1	0.0551 (12)	0.0549 (11)	0.0307 (8)	0.0020 (9)	0.0008 (8)	−0.0002 (9)
C1	0.0394 (9)	0.0505 (10)	0.0427 (9)	−0.0035 (8)	−0.0090 (7)	0.0036 (8)
C2	0.0463 (11)	0.0635 (12)	0.0602 (13)	−0.0005 (10)	−0.0215 (10)	−0.0013 (11)
C3	0.0607 (13)	0.0686 (14)	0.0606 (13)	−0.0068 (11)	−0.0281 (11)	−0.0065 (11)
C4	0.0648 (13)	0.0559 (11)	0.0421 (10)	−0.0038 (10)	−0.0128 (9)	−0.0034 (9)
C5	0.0507 (10)	0.0511 (10)	0.0324 (8)	−0.0022 (8)	−0.0017 (7)	0.0052 (8)
C6	0.0380 (9)	0.0550 (11)	0.0370 (9)	−0.0014 (8)	−0.0066 (7)	−0.0001 (7)
C7	0.0381 (9)	0.0530 (10)	0.0522 (10)	−0.0016 (8)	−0.0076 (8)	−0.0007 (9)
C8	0.0525 (13)	0.0653 (13)	0.0760 (17)	0.0114 (11)	−0.0125 (12)	−0.0159 (12)
C9	0.081 (2)	0.097 (2)	0.104 (3)	0.0237 (17)	−0.0317 (18)	−0.049 (2)

Geometric parameters (Å, °)

O1—C7	1.225 (2)	C2—C3	1.364 (4)
O2—B1	1.363 (3)	C2—H2	0.99 (2)
O2—H2A	0.76 (7)	C3—C4	1.383 (4)
O2—H2B	0.77 (8)	C3—H3	0.90 (3)
O3—B1	1.357 (3)	C4—C5	1.399 (3)
O3—H3A	0.74 (8)	C4—H4	0.96 (3)
O3—H3B	0.87 (5)	C5—C6	1.396 (3)
N1—C7	1.354 (3)	C6—H6	0.93 (3)
N1—C1	1.407 (3)	C7—C8	1.483 (3)
N1—H1A	0.94 (3)	C8—C9	1.297 (4)
B1—C5	1.559 (3)	C8—H8	0.95 (3)
C1—C6	1.389 (3)	C9—H9A	1.06 (4)
C1—C2	1.409 (3)	C9—H9B	0.89 (4)
B1—O2—H2A	114 (6)	C4—C3—H3	121.6 (17)
B1—O2—H2B	123 (6)	C3—C4—C5	120.3 (2)
H2A—O2—H2B	122 (8)	C3—C4—H4	116.9 (15)
B1—O3—H3A	109 (6)	C5—C4—H4	122.9 (15)
B1—O3—H3B	120 (5)	C6—C5—C4	118.86 (18)
H3A—O3—H3B	131 (7)	C6—C5—B1	120.24 (17)
C7—N1—C1	128.46 (17)	C4—C5—B1	120.90 (19)
C7—N1—H1A	117.1 (18)	C1—C6—C5	120.95 (16)
C1—N1—H1A	114.2 (18)	C1—C6—H6	117.0 (14)
O3—B1—O2	117.0 (2)	C5—C6—H6	122.0 (14)
O3—B1—C5	121.1 (2)	O1—C7—N1	123.7 (2)
O2—B1—C5	121.8 (2)	O1—C7—C8	122.2 (2)
C6—C1—N1	124.49 (16)	N1—C7—C8	114.19 (18)
C6—C1—C2	118.57 (19)	C9—C8—C7	121.8 (3)
N1—C1—C2	116.93 (18)	C9—C8—H8	122 (2)
C3—C2—C1	120.7 (2)	C7—C8—H8	116 (2)
C3—C2—H2	123.6 (16)	C8—C9—H9A	127.2 (19)
C1—C2—H2	115.7 (15)	C8—C9—H9B	121 (2)
C2—C3—C4	120.6 (2)	H9A—C9—H9B	109 (3)
C2—C3—H3	117.8 (18)		
C7—N1—C1—C6	10.7 (3)	O3—B1—C5—C4	−145.3 (2)

C7—N1—C1—C2	−170.2 (2)	O2—B1—C5—C4	35.9 (3)
C6—C1—C2—C3	−2.6 (3)	N1—C1—C6—C5	−177.70 (18)
N1—C1—C2—C3	178.2 (2)	C2—C1—C6—C5	3.2 (3)
C1—C2—C3—C4	−0.3 (4)	C4—C5—C6—C1	−0.9 (3)
C2—C3—C4—C5	2.6 (4)	B1—C5—C6—C1	179.45 (19)
C3—C4—C5—C6	−2.0 (3)	C1—N1—C7—O1	0.3 (3)
C3—C4—C5—B1	177.6 (2)	C1—N1—C7—C8	−178.9 (2)
O3—B1—C5—C6	34.3 (3)	O1—C7—C8—C9	−4.2 (4)
O2—B1—C5—C6	−144.5 (2)	N1—C7—C8—C9	174.9 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2A···O2^{i}	0.77 (7)	2.02 (7)	2.759 (3)	163 (7)
O2—H2B···O3^{ii}	0.77 (8)	2.10 (9)	2.748 (3)	143 (7)
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Symmetry codes: (i) −*x*+1, −*y*, *z*; (ii) *x*, *y*, *z*−1; (iii) *x*, *y*, *z*+1; (iv) *x*−1/4, −*y*+1/4, *z*−1/4.

Fig. 1

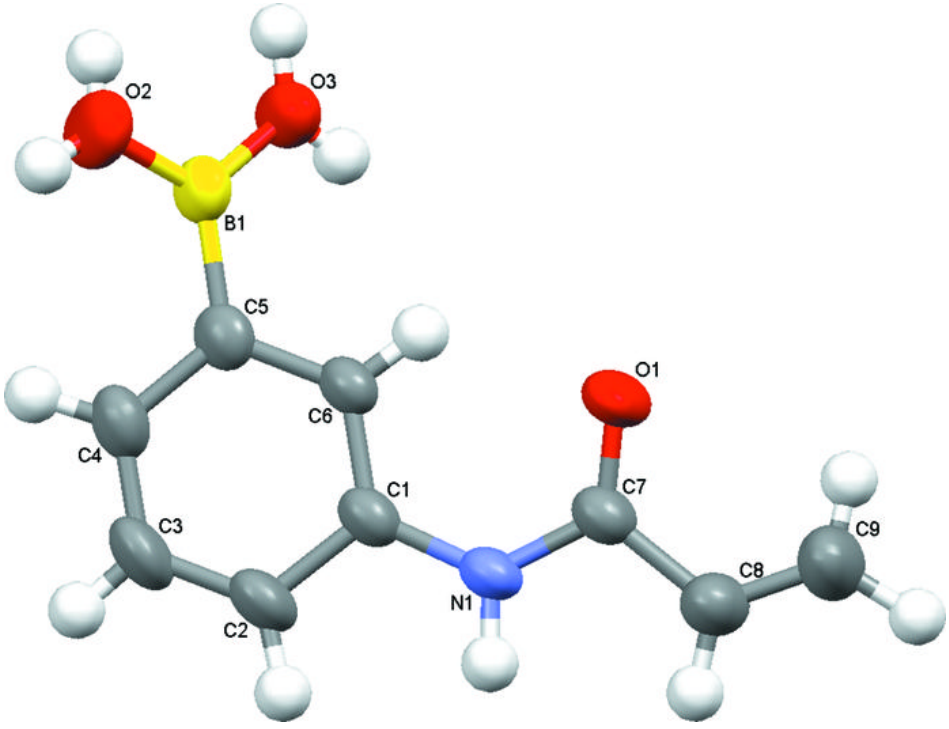

Fig. 2