Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

tert-Butyl 2-borono-1*H*-pyrrole-1-carboxylate

Zheng Zhong, Guo-Qiang Lin, Zhi-Hua Sun and Bing Wang*

Department of Chemistry, Fudan University, 220 Handan Road, Shanghai, 200433, People's Republic of China
Correspondence e-mail: wangbing@fudan.edu.cn

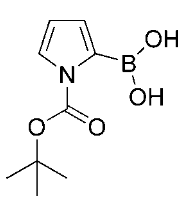
Received 16 February 2009; accepted 28 February 2009

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.042; wR factor = 0.106; data-to-parameter ratio = 11.5.

In the crystal structure of the title compound, $C_9H_{14}BNO_4$, the boronic acid group and carbamate groups are nearly co-planar with the pyrrole ring, making dihedral angles of 0.1 (2) and 2.2 (2)°, respectively. Intramolecular and intermolecular O—H···O hydrogen bonds help to stabilize the structure, the latter interaction leading to inversion dimers..

Related literature

For general background, see: Hall (2005); Kelly & Fuchs (1993).



Experimental

Crystal data

$C_9H_{14}BNO_4$
M_r = 211.02
Orthorhombic, *Pbca*
a = 13.014 (3) Å
b = 9.940 (2) Å
c = 17.417 (4) Å
V = 2252.9 (9) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.25 × 0.12 × 0.10 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: none
9542 measured reflections
2213 independent reflections
1208 reflections with $I > 2\sigma(I)$
R_{int} = 0.065

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.106
S = 0.85
2213 reflections
192 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O2	0.87 (2)	1.73 (2)	2.5819 (18)	164.7 (18)
O4—H4···O3^{i}	0.89 (3)	1.88 (3)	2.769 (3)	173 (3)

Symmetry code: (i) $-x+2, -y+1, -z+1$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

We thank Professor Lin-Hong Weng, Fudan University, for the X-ray analysis.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2484).

References

Bruker (2001). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Hall, D. G. (2005). Editor. *Boronic Acids*, Weinheim: Wiley-VCH.
Kelly, T. A. & Fuchs, V. U. (1993). *Tetrahedron* **49**, 1009–1016.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2009). E**65**, o687   [**doi:10.1107/S1600536809007375**]

tert-Butyl 2-borono-1*H*-pyrrole-1-carboxylate

Z. Zhong, G.-Q. Lin, Z.-H. Sun and B. Wang

Comment

Boronic acids are versatile compounds widely used in the synthesis of biaryls, as therapeutical agents, and as chemical sensors (Hall, 2005). The title compound is the key intermediate for the synthesis of (+)-pinanediol-*L*-boroproline (Kelly & Fuchs, 1993).

In the molecular structure of the title compound (Fig. 1), the pyrrole ring, the boronic acid group and the carboxyl groups are almost co-planar. The carbonyl links with the adjacent boronic acid group *via* O3—H3···O2 hydrogen bonding. Intermolecular hydrogen bond is also observed in the crystal structure (Table 1).

Experimental

All chemical reagents are commercial and used as received. Under -78°C and argon atmosphere, lithium diisopropylamide (1.0 *M* in THF, 5.0 ml, 5.0 mmol) was added dropwise to a solution of *tert*-butyl 1*H*-pyrrole-1-carboxylate (835 mg, 5.0 mmol) in dry THF (15 ml), and the solution was stirred at this temperature for 30 min. Trimethylborate (1.7 ml, 15 mmol) was added dropwise, and the mixture was allowed to warm to room temperature over 2 h and stirred overnight. After aqueous workup, the crude product was crystallized from hexanes. Single crystals suitable for X-ray analysis were obtained by recrystallization from a mixed solvent of ethyl acetate and hexane at ambient temperature (20–25°C).

Refinement

H atoms were located in a difference Fourier map and refined isotropically.

Figures

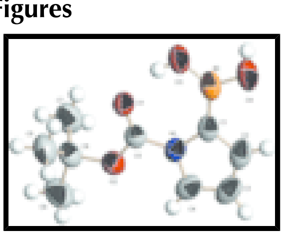


Fig. 1. The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

Tert-butyl 2-borono-1*H*-pyrrole-1-carboxylate

Crystal data

$C_9H_{14}BNO_4$	$F_{000} = 896$
$M_r = 211.02$	$D_x = 1.244$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation

	λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 810 reflections
a = 13.014 (3) Å	θ = 2.3–22.4°
b = 9.940 (2) Å	μ = 0.10 mm^{-1}
c = 17.417 (4) Å	T = 293 K
V = 2252.9 (9) Å^3	Prism, colorless
Z = 8	0.25 × 0.12 × 0.10 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	1208 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.065
Monochromator: graphite	θ_{max} = 26.0°
T = 293 K	θ_{min} = 2.3°
φ and ω scans	$h = -16 \rightarrow 13$
Absorption correction: none	$k = -12 \rightarrow 12$
9542 measured reflections	$l = -21 \rightarrow 19$
2213 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.042	All H-atom parameters refined
$wR(F^2)$ = 0.106	$w = 1/[\sigma^2(F_o^2) + (0.0588P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.85	$(\Delta/\sigma)_{max} < 0.001$
2213 reflections	$\Delta\rho_{max}$ = 0.12 e Å^{-3}
192 parameters	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > \sigma(F^2)$ 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}
B1	1.03949 (15)	0.6990 (2)	0.51802 (11)	0.0727 (6)
C1	1.09811 (14)	1.0494 (2)	0.59196 (12)	0.0767 (5)
C2	1.17519 (16)	1.0274 (3)	0.54329 (13)	0.0893 (7)
C3	1.16298 (14)	0.8995 (3)	0.51194 (12)	0.0821 (6)
C4	1.07732 (12)	0.83956 (18)	0.54116 (8)	0.0644 (5)
C5	0.94986 (12)	0.92187 (19)	0.63777 (9)	0.0635 (4)
C6	0.85439 (15)	1.03344 (19)	0.74024 (10)	0.0771 (5)
C7	0.75166 (18)	1.0282 (3)	0.70204 (16)	0.0955 (7)
C8	0.8711 (3)	0.9215 (3)	0.79700 (14)	0.1000 (7)
C9	0.8746 (3)	1.1693 (3)	0.7756 (2)	0.1119 (9)
N1	1.03646 (9)	0.93560 (14)	0.59221 (7)	0.0631 (4)
O1	0.93714 (8)	1.02801 (13)	0.68119 (7)	0.0754 (4)
O2	0.89519 (9)	0.82427 (13)	0.63724 (7)	0.0776 (4)
O3	0.95566 (10)	0.63485 (15)	0.54641 (8)	0.0921 (5)
O4	1.09809 (10)	0.6338 (2)	0.46635 (8)	0.0960 (5)
H3	1.2044 (14)	0.8510 (17)	0.4741 (10)	0.083 (5)*
H7A	0.7006 (17)	1.040 (2)	0.7397 (13)	0.104 (7)*
H7B	0.7430 (18)	1.106 (3)	0.6664 (14)	0.131 (10)*
H8A	0.8247 (16)	0.934 (2)	0.8403 (14)	0.109 (7)*
H1	1.0760 (14)	1.126 (2)	0.6241 (11)	0.089 (6)*
H9A	0.8259 (16)	1.184 (2)	0.8132 (14)	0.122 (8)*
H9B	0.8706 (15)	1.236 (2)	0.7359 (14)	0.109 (9)*
H7C	0.7384 (15)	0.942 (2)	0.6764 (13)	0.104 (7)*
H2	1.2262 (17)	1.090 (2)	0.5323 (11)	0.104 (7)*
H8B	0.941 (2)	0.928 (2)	0.8167 (14)	0.130 (9)*
H9C	0.949 (2)	1.169 (3)	0.7937 (16)	0.169 (12)*
H8C	0.8565 (17)	0.826 (3)	0.7735 (14)	0.135 (9)*
H3A	0.9265 (14)	0.6888 (19)	0.5791 (11)	0.087 (6)*
H4	1.080 (2)	0.548 (3)	0.4581 (16)	0.145 (12)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
B1	0.0572 (11)	0.1046 (17)	0.0563 (11)	0.0146 (11)	−0.0040 (9)	−0.0053 (11)
C1	0.0616 (11)	0.0916 (15)	0.0767 (12)	−0.0077 (11)	−0.0134 (10)	0.0135 (12)
C2	0.0590 (12)	0.120 (2)	0.0888 (15)	−0.0117 (12)	−0.0074 (11)	0.0335 (14)
C3	0.0565 (11)	0.1260 (19)	0.0638 (11)	0.0091 (12)	0.0003 (9)	0.0161 (13)
C4	0.0524 (9)	0.0915 (13)	0.0492 (8)	0.0113 (9)	−0.0034 (7)	0.0069 (9)
C5	0.0586 (10)	0.0744 (12)	0.0576 (9)	0.0032 (9)	−0.0030 (8)	0.0004 (9)
C6	0.0834 (13)	0.0832 (13)	0.0647 (11)	0.0110 (10)	0.0065 (9)	−0.0149 (10)
C7	0.0793 (15)	0.116 (2)	0.0913 (17)	0.0166 (14)	0.0054 (13)	−0.0170 (17)
C8	0.119 (2)	0.115 (2)	0.0655 (13)	0.0172 (16)	0.0126 (14)	0.0019 (14)
C9	0.129 (2)	0.103 (2)	0.103 (2)	0.0076 (16)	0.0117 (18)	−0.0376 (18)
N1	0.0494 (7)	0.0814 (10)	0.0585 (8)	−0.0007 (7)	−0.0031 (6)	0.0075 (7)

O1	0.0787 (8)	0.0766 (8)	0.0708 (7)	−0.0010 (6)	0.0069 (6)	−0.0128 (7)
O2	0.0698 (7)	0.0786 (9)	0.0844 (8)	−0.0067 (6)	0.0214 (6)	−0.0166 (7)
O3	0.0730 (8)	0.1037 (11)	0.0996 (10)	−0.0004 (7)	0.0200 (7)	−0.0384 (8)
O4	0.0799 (9)	0.1217 (14)	0.0863 (9)	0.0147 (8)	0.0190 (7)	−0.0238 (9)

Geometric parameters (Å, °)

B1—O4	1.346 (2)	C6—C7	1.494 (3)
B1—O3	1.357 (2)	C6—C8	1.504 (3)
B1—C4	1.535 (3)	C6—C9	1.507 (3)
C1—C2	1.331 (3)	C7—H7A	0.94 (2)
C1—N1	1.387 (2)	C7—H7B	0.99 (3)
C1—H1	0.99 (2)	C7—H7C	0.98 (2)
C2—C3	1.393 (3)	C8—H8A	0.97 (2)
C2—H2	0.93 (2)	C8—H8B	0.98 (3)
C3—C4	1.363 (3)	C8—H8C	1.05 (3)
C3—H3	0.978 (18)	C9—H9A	0.92 (2)
C4—N1	1.409 (2)	C9—H9B	0.96 (2)
C5—O2	1.2030 (19)	C9—H9C	1.02 (3)
C5—O1	1.309 (2)	O3—H3A	0.87 (2)
C5—N1	1.385 (2)	O4—H4	0.89 (3)
C6—O1	1.490 (2)		
O4—B1—O3	118.2 (2)	C6—C7—H7A	108.5 (13)
O4—B1—C4	115.60 (19)	C6—C7—H7B	110.6 (14)
O3—B1—C4	126.14 (17)	H7A—C7—H7B	105.0 (18)
C2—C1—N1	107.7 (2)	C6—C7—H7C	112.9 (12)
C2—C1—H1	135.0 (12)	H7A—C7—H7C	107.4 (18)
N1—C1—H1	117.2 (11)	H7B—C7—H7C	112 (2)
C1—C2—C3	108.3 (2)	C6—C8—H8A	109.2 (12)
C1—C2—H2	123.9 (13)	C6—C8—H8B	108.4 (15)
C3—C2—H2	127.8 (13)	H8A—C8—H8B	107 (2)
C4—C3—C2	110.3 (2)	C6—C8—H8C	112.7 (13)
C4—C3—H3	119.1 (11)	H8A—C8—H8C	107.5 (18)
C2—C3—H3	130.7 (10)	H8B—C8—H8C	111 (2)
C3—C4—N1	104.36 (18)	C6—C9—H9A	107.9 (16)
C3—C4—B1	124.22 (18)	C6—C9—H9B	108.3 (13)
N1—C4—B1	131.40 (15)	H9A—C9—H9B	112 (2)
O2—C5—O1	125.44 (15)	C6—C9—H9C	107.0 (17)
O2—C5—N1	123.79 (16)	H9A—C9—H9C	116 (2)
O1—C5—N1	110.77 (15)	H9B—C9—H9C	106 (2)
O1—C6—C7	109.75 (16)	C5—N1—C1	123.57 (16)
O1—C6—C8	108.80 (16)	C5—N1—C4	127.01 (15)
C7—C6—C8	113.4 (2)	C1—N1—C4	109.41 (16)
O1—C6—C9	100.86 (18)	C5—O1—C6	121.30 (13)
C7—C6—C9	111.6 (2)	B1—O3—H3A	107.4 (12)
C8—C6—C9	111.6 (2)	B1—O4—H4	114.5 (18)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O3^{i}	0.89 (3)	1.88 (3)	2.769 (3)	173 (3)
O3—H3A···O2	0.87 (2)	1.73 (2)	2.5819 (18)	164.7 (18)

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1.

Fig. 1