Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(*p*-Tolyl)-1,3,2-benzodioxaborole

George Bramham,[a] Andrei S. Batsanov,[b]* Todd B. Marder[b] and Nicholas C. Norman[a]

[a]School of Chemistry, University of Bristol, Bristol BS8 1TS, England, and [b]Department of Chemistry, University of Durham, South Road, Durham DH1 3LE, England

Correspondence e-mail: a.s.batsanov@durham.ac.uk

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.128
Data-to-parameter ratio = 13.2

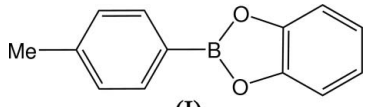
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title molecule, $C_{13}H_{11}BO_2$, adopts a planar conformation and a stack/herringbone packing motif in the solid state.

Received 31 January 2006
Accepted 2 February 2006

Comment

Compound (I) was obtained *via* cobalt-mediated borylation of 4-iodotoluene, observed during our studies of the synthesis and reactivity of cobalt boryl complexes (Dai *et al.*, 1996; Adams *et al.*, 2006).



(I)

The asymmetric unit comprises one molecule (Fig. 1), which is nearly planar (r.m.s. deviation for all non-H atoms 0.057 Å), like its prototype 2-phenyl-1,3,2-benzodioxaborole (Zettler *et al.*, 1974). The B atom is trigonal-planar; its coordination plane is inclined by 2.9 (1)° to the catechol arene ring (i) and by 3.7 (1)° to the tolyl arene ring (ii). Molecules related *via* the *b* translation form a stack with a mean interplanar separation of 3.52 (5) Å. Stacks are packed in a herringbone motif, in which planes of adjacent molecules are nearly perpendicular [dihedral angle 89.7 (1)°].

Experimental

To a stirred light-yellow solution of $[Co(PMe_3)_3(BO_2C_6H_4)_2]$ (Dai *et al.*, 1996) (0.110 g, 0.21 mmol) in hexane (2.0 ml), 4-iodotoluene (0.054 g, 0.25 mmol) was added at room temperature, resulting in a brown solution. After heating at 343 K overnight, the mixture became pink in colour. The solvent was then removed *in vacuo* and the residues were redissolved in THF (10 ml) to which was added excess $CoCl_2$. The mixture was stirred for a further 15 min before being reduced to dryness *in vacuo*. The residues were then extracted with hexane and the resulting solution was concentrated *in vacuo*, during which a colourless solid appeared. This was redissolved by gentle heating, after which the solution was cooled slowly to give colourless crystals of (I) (0.015 g). ^{11}B NMR: δ 31.9. EI–MS *m*/*z* 210 (M^+).

Crystal data

$C_{13}H_{11}BO_2$
M_r = 210.03
Monoclinic, $P2_1/c$
a = 17.7405 (10) Å
b = 4.9935 (4) Å
c = 12.3989 (16) Å
β = 100.80 (1)°
V = 1078.93 (17) Å^3
Z = 4
D_x = 1.293 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 687 reflections
θ = 10.3–24.0°
μ = 0.09 mm^{-1}
T = 120 (2) K
Plate, colourless
0.22 × 0.15 × 0.05 mm

Data collection

Bruker SMART 6000 CCD area-detector diffractometer	1654 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.071$
ω scans	$\theta_{max} = 27.5^\circ$
Absorption correction: none	$h = -23 \rightarrow 17$
9171 measured reflections	$k = -6 \rightarrow 6$
2486 independent reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	All H-atom parameters refined
$R[F^2 > 2\sigma(F^2)] = 0.040$	$w = 1/[\sigma^2(F_o^2) + (0.0681P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.128$	
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
2486 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
189 parameters	$\Delta\rho_{min} = -0.19$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.384 (2)	O2—B	1.393 (2)
O1—B	1.389 (2)	C7—B	1.533 (2)
O2—C2	1.384 (2)		
O1—B—O2	111.00 (14)	O2—B—C7	124.66 (13)
O1—B—C7	124.33 (14)		
O1—B—C7—C8	−3.0 (2)	O2—B—C7—C12	−3.4 (2)

All H atoms were refined isotropically, yielding the following distances: Csp^3—H = 0.98 (2) to 1.01 (2) Å and Csp^2—H = 0.95 (2) to 1.00 (2) Å.

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

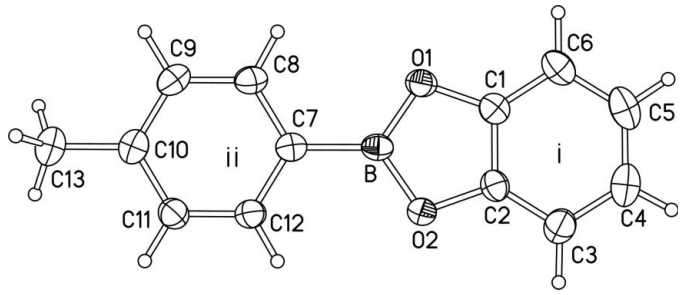


Figure 1
Molecular structure of (I). Atomic displacement ellipsoids are drawn at the 50% probability level.

The authors thank the EPSRC for financial support and Frontier Scientific Inc. for generous gifts of diborane(4) compounds.

References

Adams, C. J., Baber, R. A., Batsanov, A. S., Bramham, G., Charmant, J. P. H., Haddow, M. F., Howard, J. A. K., Lam, W. H., Lin, Z. Y., Marder, T. B., Norman, N. C. & Orpen, A. G. (2006). *Dalton Trans.* In the press (DOI 10 1039/b516594f).

Bruker (2001). *SMART* (Version 5.625), *SAINT* (Version 6.02A) and *SHELXTL* (Version 6.12). Bruker AXS Inc., Madison, Wisconsin, USA.

Dai, C. Y., Stringer, G., Corrigan, J. F., Taylor, N. J., Marder, T. B. & Norman, N. C. (1996). *J. Organomet. Chem.* **513**, 273–275.

Zettler, F., Hausen, H. D. & Hess, H. (1974). *Acta Cryst.* B**30**, 1876–1878.

supporting information

Acta Cryst. (2006). E**62**, o972–o973 [https://doi.org/10.1107/S1600536806004089]

2-(*p*-Tolyl)-1,3,2-benzodioxaborole

George Bramham, Andrei S. Batsanov, Todd B. Marder and Nicholas C. Norman

2-(*p*-tolyl)-1,3,2-benzodioxaborole

Crystal data

$C_{13}H_{11}BO_2$
$M_r = 210.03$
Monoclinic, $P2_1/c$
$a = 17.7405$ (10) Å
$b = 4.9935$ (4) Å
$c = 12.3989$ (16) Å
$\beta = 100.80$ (1)°
$V = 1078.93$ (17) Å^3
$Z = 4$
$F(000) = 440$
$D_x = 1.293$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 687 reflections
$\theta = 10.3$–24.0°
$\mu = 0.09$ mm^{-1}
$T = 120$ K
Plate, colourless
0.22 × 0.15 × 0.05 mm

Data collection

Bruker SMART 6000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 5.6 pixels mm^{-1}
ω scans
9171 measured reflections
2486 independent reflections
1654 reflections with $I > 2\sigma(I)$
$R_{int} = 0.071$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.2°$
$h = -23 \rightarrow 17$
$k = -6 \rightarrow 6$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.128$
$S = 1.02$
2486 reflections
189 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0681P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

Special details

Experimental. The data collection nominally covered over 3/4 of the full sphere of reciprocal space, by a combination of 3 sets of ω scans; each set at different φ angles and each scan (20 sec exposure) covering 0.3° in ω. Crystal to detector distance 4.84 cm.

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.26370 (6)	0.5110 (2)	0.65710 (8)	0.0304 (3)
O2	0.32209 (6)	0.5032 (2)	0.50706 (8)	0.0317 (3)
C1	0.32220 (9)	0.6985 (3)	0.67077 (12)	0.0287 (4)
C2	0.35703 (9)	0.6944 (3)	0.58054 (12)	0.0297 (4)
C3	0.41590 (10)	0.8648 (4)	0.57084 (15)	0.0408 (4)
H3	0.4396 (11)	0.861 (4)	0.5035 (15)	0.047 (5)*
C4	0.43888 (11)	1.0432 (4)	0.65723 (16)	0.0436 (5)
H4	0.4779 (11)	1.172 (4)	0.6538 (14)	0.048 (5)*
C5	0.40358 (11)	1.0468 (3)	0.74747 (15)	0.0421 (5)
H5	0.4171 (11)	1.172 (4)	0.8068 (15)	0.049 (5)*
C6	0.34387 (10)	0.8728 (3)	0.75651 (13)	0.0367 (4)
H6	0.3182 (10)	0.877 (3)	0.8214 (15)	0.041 (5)*
C7	0.20757 (9)	0.1786 (3)	0.50309 (11)	0.0273 (3)
C8	0.15251 (9)	0.0748 (3)	0.55850 (13)	0.0316 (4)
H8	0.1495 (9)	0.140 (3)	0.6324 (14)	0.035 (4)*
C9	0.10013 (10)	−0.1151 (3)	0.51049 (13)	0.0341 (4)
H9	0.0606 (11)	−0.190 (4)	0.5502 (14)	0.049 (5)*
C10	0.10028 (9)	−0.2085 (3)	0.40431 (13)	0.0311 (4)
C11	0.15523 (9)	−0.1058 (3)	0.34862 (13)	0.0322 (4)
H11	0.1546 (10)	−0.172 (3)	0.2721 (14)	0.043 (5)*
C12	0.20784 (9)	0.0831 (3)	0.39697 (12)	0.0309 (4)
H12	0.2439 (10)	0.159 (3)	0.3550 (14)	0.041 (5)*
C13	0.04401 (11)	−0.4166 (3)	0.35259 (16)	0.0393 (4)
H131	0.0568 (12)	−0.597 (4)	0.3802 (17)	0.058 (6)*
H132	−0.0076 (13)	−0.384 (4)	0.3710 (18)	0.065 (7)*
H133	0.0387 (14)	−0.416 (4)	0.270 (2)	0.074 (7)*
B	0.26424 (10)	0.3944 (3)	0.55553 (13)	0.0274 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0311 (6)	0.0353 (6)	0.0255 (5)	0.0005 (5)	0.0074 (4)	−0.0008 (4)
O2	0.0331 (6)	0.0381 (6)	0.0247 (5)	−0.0070 (5)	0.0076 (4)	−0.0030 (4)
C1	0.0278 (8)	0.0291 (8)	0.0278 (7)	0.0047 (6)	0.0017 (6)	0.0018 (6)
C2	0.0295 (9)	0.0309 (8)	0.0269 (7)	0.0000 (7)	0.0006 (6)	−0.0005 (6)
C3	0.0367 (10)	0.0450 (10)	0.0404 (10)	−0.0074 (8)	0.0067 (8)	0.0005 (8)
C4	0.0366 (11)	0.0353 (9)	0.0545 (11)	−0.0063 (8)	−0.0023 (8)	−0.0005 (8)
C5	0.0430 (11)	0.0340 (9)	0.0428 (10)	0.0069 (8)	−0.0082 (8)	−0.0098 (8)
C6	0.0380 (10)	0.0383 (9)	0.0311 (8)	0.0077 (7)	0.0000 (7)	−0.0048 (7)
C7	0.0262 (8)	0.0295 (8)	0.0262 (7)	0.0036 (6)	0.0045 (6)	0.0035 (6)
C8	0.0330 (9)	0.0360 (8)	0.0269 (8)	0.0006 (7)	0.0080 (7)	0.0042 (6)

C9	0.0309 (9)	0.0363 (8)	0.0362 (9)	−0.0024 (7)	0.0086 (7)	0.0080 (7)
C10	0.0274 (9)	0.0268 (7)	0.0376 (8)	0.0020 (6)	0.0019 (6)	0.0051 (6)
C11	0.0321 (9)	0.0330 (8)	0.0317 (8)	0.0003 (7)	0.0060 (7)	−0.0033 (6)
C12	0.0298 (9)	0.0345 (8)	0.0296 (8)	−0.0008 (7)	0.0084 (7)	−0.0007 (6)
C13	0.0361 (11)	0.0322 (9)	0.0473 (11)	−0.0042 (8)	0.0021 (8)	0.0040 (7)
B	0.0290 (10)	0.0306 (9)	0.0232 (8)	0.0037 (7)	0.0063 (7)	0.0034 (6)

Geometric parameters (Å, º)

O1—C1	1.384 (2)	C7—C12	1.400 (2)
O1—B	1.389 (2)	C7—B	1.533 (2)
O2—C2	1.384 (2)	C8—C9	1.382 (2)
O2—B	1.393 (2)	C8—H8	0.983 (16)
C1—C6	1.372 (2)	C9—C10	1.397 (2)
C1—C2	1.376 (2)	C9—H9	1.001 (18)
C2—C3	1.370 (2)	C10—C11	1.394 (2)
C3—C4	1.394 (3)	C10—C13	1.499 (2)
C3—H3	1.003 (18)	C11—C12	1.382 (2)
C4—C5	1.381 (3)	C11—H11	1.002 (17)
C4—H4	0.95 (2)	C12—H12	0.974 (18)
C5—C6	1.391 (3)	C13—H131	0.98 (2)
C5—H5	0.962 (19)	C13—H132	1.00 (2)
C6—H6	0.996 (18)	C13—H133	1.01 (2)
C7—C8	1.395 (2)		
C1—O1—B	105.19 (12)	C9—C8—H8	118.5 (10)
C2—O2—B	105.09 (11)	C7—C8—H8	120.2 (10)
C6—C1—C2	122.41 (15)	C8—C9—C10	121.00 (15)
C6—C1—O1	128.22 (14)	C8—C9—H9	121.3 (10)
C2—C1—O1	109.34 (13)	C10—C9—H9	117.7 (10)
C3—C2—C1	121.89 (15)	C11—C10—C9	117.99 (15)
C3—C2—O2	128.74 (14)	C11—C10—C13	120.96 (15)
C1—C2—O2	109.36 (13)	C9—C10—C13	121.04 (15)
C2—C3—C4	116.59 (16)	C12—C11—C10	120.95 (15)
C2—C3—H3	120.5 (11)	C12—C11—H11	121.4 (10)
C4—C3—H3	122.9 (11)	C10—C11—H11	117.7 (10)
C5—C4—C3	121.26 (17)	C11—C12—C7	121.26 (15)
C5—C4—H4	118.1 (11)	C11—C12—H12	119.4 (10)
C3—C4—H4	120.6 (11)	C7—C12—H12	119.2 (10)
C4—C5—C6	121.68 (16)	C10—C13—H131	113.5 (12)
C4—C5—H5	122.8 (11)	C10—C13—H132	110.8 (12)
C6—C5—H5	115.5 (11)	H131—C13—H132	103.4 (17)
C1—C6—C5	116.17 (16)	C10—C13—H133	111.1 (13)
C1—C6—H6	122.5 (11)	H131—C13—H133	109.2 (17)
C5—C6—H6	121.4 (10)	H132—C13—H133	108.3 (19)
C8—C7—C12	117.52 (15)	O1—B—O2	111.00 (14)
C8—C7—B	121.09 (13)	O1—B—C7	124.33 (14)
C12—C7—B	121.37 (13)	O2—B—C7	124.66 (13)

C9—C8—C7	121.28 (15)		
O1—B—C7—C8	−3.0 (2)	O2—B—C7—C12	−3.4 (2)