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(*E*)-2-[2-(4-Methylphenyl)ethenyl]-1,3,2-benzodioxaborole

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Key indicators

Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.108
Data-to-parameter ratio = 16.3

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

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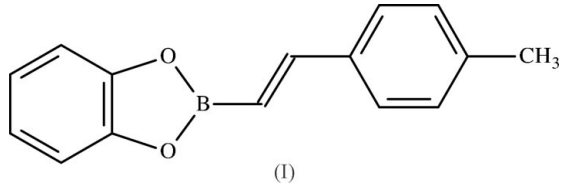
Molecules of the title compound, $C_{15}H_{13}BO_2$, are essentially planar with a high degree of conjugation. Pairs of molecules related by inversion symmetry show π-stacking interactions, and the overall packing is a herring-bone pattern. The molecular geometry is similar to that of closely related analogues.

Comment

The title compound, (I), is one of a series of 2-styrylboronate esters prepared in a study of hydroboration reactions of alkynes, with a variety of *para* substituents (Wiesauer, 1997). We have previously reported the structure of the parent compound with no substituent in the *para* position (Clegg *et al.*, 2001). The title compound is the methyl analogue. Structures have also been determined for the SMe (Yuan *et al.*, 1990), OMe (Nguyen *et al.*, 2002) and CF_3 (Clegg *et al.*, 2004) derivatives.



(I)

The molecule of the title compound (Fig. 1) is essentially completely planar except for the H atoms of the methyl group, with a high degree of conjugation. The r.m.s. deviation of all non-H atoms from their mean plane is 0.071 Å. All torsion angles for non-H atoms are close to 0 and 180°, the largest corresponding to a twist of about 7° around the B—C bond linking the alkene double bond to the benzodioxaborole (Bcat) group (Table 1). This almost completely planar arrangement is found also for the other derivatives mentioned above.

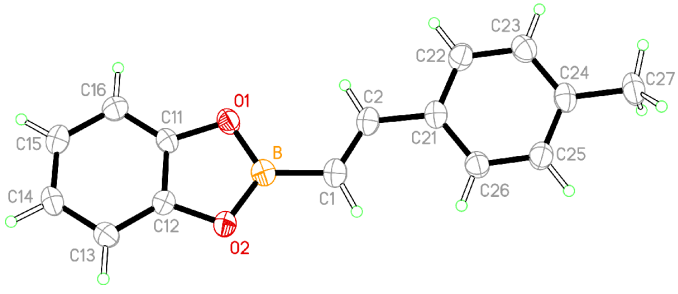


Figure 1
The molecular structure, with atom labels and 50% probability ellipsoids for non-H atoms.

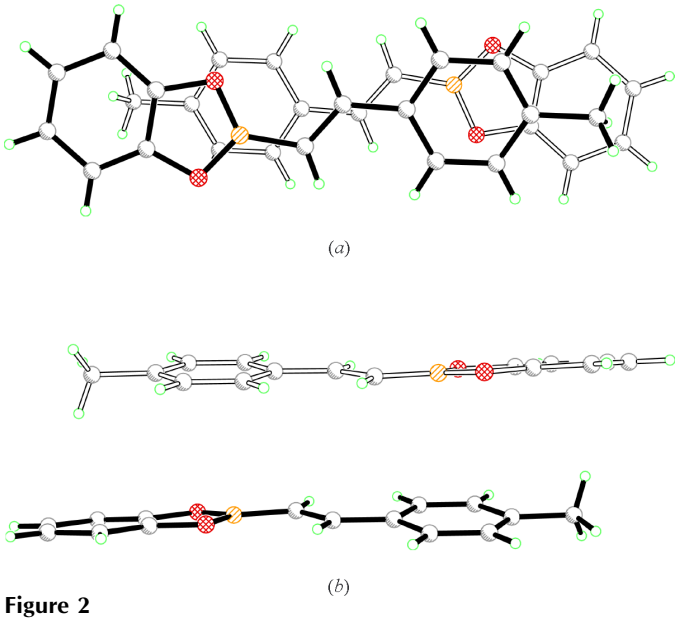


Figure 2
The overlap of two parallel molecules related by inversion symmetry, seen (*a*) from above and (*b*) from the side. One molecule is shown with filled bonds, and the other with hollow bonds.

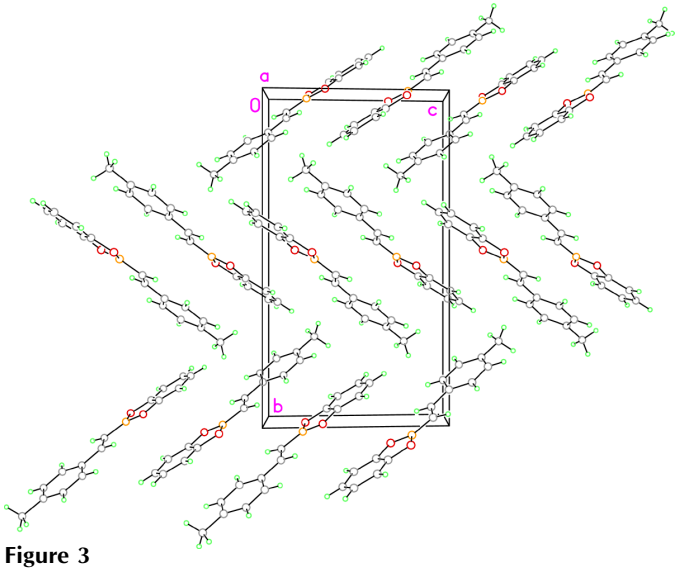


Figure 3
The crystal packing, viewed along the *a* axis.

Bond lengths and angles are typical of compounds in which Bcat is attached to an alkene double bond; these include not only the derivatives with different *para* substituents, but also the compounds (Bcat)CH═C(*R*)(Cl), where *R* is either Me or Et (Bayer *et al.*, 2002), the symmetrically substituted alkene $(Bcat)_2C{=}C(Bcat)_2$ (Gu *et al.*, 2001), two Bcat-substituted cyclopentadienes (Avent *et al.*, 2003), and several alkenes and dialkenes with two or more Bcat groups (Lesley *et al.*, 1996; Clegg *et al.*, 1996). Steric interaction, particularly between Bcat groups, forces some of these molecules to adopt non-planar forms, which has minor effects on the degree of conjugation and hence on some bond lengths.

Centrosymmetric pairs of molecules of the title compound show extensive overlap (Fig. 2) and a separation of about 3.57 Å, indicating some π-stacking interaction. These dimeric units are further assembled into a herring-bone pattern in the overall crystal packing (Fig. 3), as is commonly found for planar organic molecules.

Experimental

4-Methylphenylethyne (0.565 g, 4.86 mmol) and catecholborane (0.643 g, 5.36 mmol) were heated at 343 K for 2 h in a scintillation vial under a nitrogen atmosphere. The resulting yellow solid was recrystallized twice from *n*-hexane, in a final yield of 67%. Analysis calculated: C 76.32, H 5.55%; found: C 76.78, H 5.41%. Mass spectrum: 236 (M^+, 100%), 221 (12.7%), 209 (6.3%), 143 (7.7%), 118 (27.6%), 117 (29.2%), 116 (26.3%), 115 (29.3%), 105 (10.6%), 91 (27.8%). ^{1}H NMR (200 MHz): δ 2.36 (*s*, 3H, CH_3), 6.41 (*d*, J = 18.5 Hz, 1H, H2), 7.07 (*m*, 2H, two of H13–H16), 7.18 (*d*, J = 7.9 Hz, 2H, H23 and H25), 7.24 (*m*, 2H, two of H13–H16), 7.47 (*d*, J = 7.9 Hz, 2H, H22 and H26), 7.74 (*d*, J = 18.5 Hz, 1H, H1) (using the crystallographic numbering scheme of Fig. 1). ^{13}C{^{1}H} NMR (50 MHz): δ 21.4 (1C, C27), 112.3 (2C, C13 and C16), 122.6 (2C, C14 and C15), 127.4 and 129.5 (2 × 2C, C22, C23, C25, C26), 134.3 (1C, C24), 139.9 (1C, C21), 148.4 (2C, C11 and C12), 152.0 (1C, C2), resonance of C1 too broad to be observed. ^{11}B{^{1}H} NMR (64 MHz): δ 31.7.

Crystal data

$C_{15}H_{13}BO_2$	D_x = 1.293 Mg m^{-3}
M_r = 236.06	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 5025 reflections
a = 6.4402 (9) Å	θ = 2.0–28.4°
b = 18.455 (3) Å	μ = 0.08 mm^{-1}
c = 10.2790 (14) Å	T = 160 (2) K
β = 96.901 (3)°	Block, colourless
V = 1212.8 (3) Å^3	0.60 × 0.58 × 0.40 mm
Z = 4	

Data collection

Bruker SMART 1K CCD diffractometer	2420 reflections with $I > 2\sigma(I)$
Thin-slice ω scans	R_{int} = 0.029
Absorption correction: none	θ_{max} = 28.6°
6815 measured reflections	$h = -8 \rightarrow 6$
2697 independent reflections	$k = -14 \rightarrow 24$
	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.042P)^2 + 0.4808P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.042	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.108	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
S = 1.06	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
2697 reflections	Extinction correction: *SHELXTL*
165 parameters	Extinction coefficient: 0.0146 (18)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.3370 (19)	B—O1	1.3911 (17)
C1—B	1.5343 (19)	B—O2	1.3907 (17)
C2—C21	1.4709 (17)		
C2—C1—B	122.11 (12)	O1—B—O2	111.27 (11)
C1—C2—C21	127.71 (12)	B—O1—C11	105.17 (10)
C1—B—O1	123.97 (12)	B—O2—C12	105.16 (10)
C1—B—O2	124.75 (12)		
B—C1—C2—C21	177.86 (12)	C1—C2—C21—C22	−177.24 (13)
C2—C1—B—O1	6.1 (2)	C1—C2—C21—C26	3.2 (2)
C2—C1—B—O2	−172.12 (12)		

H atoms were positioned geometrically and refined with a riding model, including torsional freedom around the C—C bond, with C—

H = 0.95 (aromatic and olefinic) or 0.98 Å (methyl), and with U_{iso}(H) = 1.2U_{eq}(C) (1.5U_{eq} for methyl groups).

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

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supporting information

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$h = -8 \rightarrow 6$
$k = -14 \rightarrow 24$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.108$
$S = 1.06$
2697 reflections
165 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.042P)^2 + 0.4808P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: SHELXTL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0146 (18)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7050 (2)	−0.04966 (7)	0.89091 (13)	0.0313 (3)
H1	0.8030	−0.0617	0.9643	0.038*
C2	0.5083 (2)	−0.07351 (7)	0.88687 (12)	0.0297 (3)

H2	0.4144	−0.0588	0.8133	0.036*
B	0.7775 (2)	−0.00340 (7)	0.78084 (14)	0.0284 (3)
O1	0.64428 (14)	0.02313 (5)	0.67502 (9)	0.0307 (2)
O2	0.98406 (14)	0.01587 (5)	0.77180 (8)	0.0297 (2)
C11	0.77191 (19)	0.06104 (6)	0.60009 (12)	0.0266 (3)
C12	0.9775 (2)	0.05603 (6)	0.65746 (11)	0.0260 (3)
C13	1.1399 (2)	0.08712 (7)	0.60257 (13)	0.0319 (3)
H13	1.2810	0.0821	0.6406	0.038*
C14	1.0856 (2)	0.12663 (7)	0.48735 (13)	0.0335 (3)
H14	1.1925	0.1498	0.4465	0.040*
C15	0.8799 (2)	0.13274 (7)	0.43139 (13)	0.0353 (3)
H15	0.8486	0.1605	0.3537	0.042*
C16	0.7173 (2)	0.09903 (8)	0.48652 (13)	0.0343 (3)
H16	0.5762	0.1022	0.4474	0.041*
C21	0.4201 (2)	−0.11989 (6)	0.98286 (12)	0.0278 (3)
C22	0.2122 (2)	−0.14172 (7)	0.95868 (13)	0.0341 (3)
H22	0.1305	−0.1267	0.8802	0.041*
C23	0.1217 (2)	−0.18513 (8)	1.04736 (14)	0.0357 (3)
H23	−0.0203	−0.1996	1.0280	0.043*
C24	0.2356 (2)	−0.20760 (6)	1.16357 (12)	0.0311 (3)
C25	0.4438 (2)	−0.18621 (7)	1.18733 (13)	0.0340 (3)
H25	0.5252	−0.2014	1.2658	0.041*
C26	0.5356 (2)	−0.14312 (7)	1.09922 (13)	0.0324 (3)
H26	0.6781	−0.1293	1.1182	0.039*
C27	0.1371 (3)	−0.25333 (8)	1.26094 (14)	0.0412 (4)
H27A	0.2251	−0.2958	1.2844	0.062*
H27B	−0.0018	−0.2693	1.2220	0.062*
H27C	0.1237	−0.2247	1.3398	0.062*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0367 (7)	0.0277 (6)	0.0306 (6)	0.0024 (5)	0.0087 (5)	0.0021 (5)
C2	0.0373 (7)	0.0260 (6)	0.0273 (6)	0.0028 (5)	0.0095 (5)	0.0003 (5)
B	0.0313 (7)	0.0243 (6)	0.0306 (7)	0.0007 (5)	0.0074 (6)	−0.0024 (5)
O1	0.0265 (5)	0.0326 (5)	0.0339 (5)	−0.0018 (3)	0.0071 (4)	0.0049 (4)
O2	0.0302 (5)	0.0315 (5)	0.0276 (4)	−0.0005 (4)	0.0040 (4)	0.0055 (3)
C11	0.0254 (6)	0.0264 (6)	0.0290 (6)	−0.0017 (4)	0.0076 (5)	−0.0008 (5)
C12	0.0292 (6)	0.0243 (5)	0.0245 (6)	0.0012 (4)	0.0038 (5)	0.0001 (4)
C13	0.0270 (6)	0.0342 (6)	0.0345 (7)	−0.0024 (5)	0.0040 (5)	0.0014 (5)
C14	0.0347 (7)	0.0332 (7)	0.0346 (7)	−0.0054 (5)	0.0125 (6)	0.0026 (5)
C15	0.0427 (8)	0.0344 (7)	0.0292 (6)	0.0013 (6)	0.0061 (6)	0.0060 (5)
C16	0.0304 (7)	0.0392 (7)	0.0325 (7)	0.0012 (5)	0.0004 (5)	0.0046 (5)
C21	0.0346 (7)	0.0235 (6)	0.0271 (6)	0.0018 (5)	0.0105 (5)	−0.0015 (4)
C22	0.0339 (7)	0.0378 (7)	0.0310 (6)	0.0032 (5)	0.0059 (5)	0.0047 (5)
C23	0.0334 (7)	0.0377 (7)	0.0370 (7)	−0.0035 (5)	0.0090 (6)	0.0016 (5)
C24	0.0438 (8)	0.0218 (6)	0.0296 (6)	−0.0020 (5)	0.0118 (5)	−0.0024 (5)
C25	0.0428 (8)	0.0311 (6)	0.0282 (6)	−0.0026 (5)	0.0039 (6)	0.0025 (5)

C26	0.0353 (7)	0.0323 (6)	0.0300 (6)	−0.0045 (5)	0.0058 (5)	−0.0004 (5)
C27	0.0561 (10)	0.0327 (7)	0.0369 (7)	−0.0111 (6)	0.0144 (7)	0.0027 (6)

Geometric parameters (Å, º)

C1—H1	0.950	C15—C16	1.395 (2)
C1—C2	1.3370 (19)	C16—H16	0.950
C1—B	1.5343 (19)	C21—C22	1.3913 (19)
C2—H2	0.950	C21—C26	1.3979 (18)
C2—C21	1.4709 (17)	C22—H22	0.950
B—O1	1.3911 (17)	C22—C23	1.3931 (19)
B—O2	1.3907 (17)	C23—H23	0.950
O1—C11	1.3822 (14)	C23—C24	1.3882 (19)
O2—C12	1.3857 (14)	C24—C25	1.390 (2)
C11—C12	1.3863 (17)	C24—C27	1.5065 (18)
C11—C16	1.3710 (18)	C25—H25	0.950
C12—C13	1.3723 (18)	C25—C26	1.3898 (18)
C13—H13	0.950	C26—H26	0.950
C13—C14	1.3990 (19)	C27—H27A	0.980
C14—H14	0.950	C27—H27B	0.980
C14—C15	1.384 (2)	C27—H27C	0.980
C15—H15	0.950		
H1—C1—C2	118.9	C11—C16—H16	121.8
H1—C1—B	118.9	C15—C16—H16	121.8
C2—C1—B	122.11 (12)	C2—C21—C22	119.31 (12)
C1—C2—H2	116.1	C2—C21—C26	122.93 (12)
C1—C2—C21	127.71 (12)	C22—C21—C26	117.76 (12)
H2—C2—C21	116.1	C21—C22—H22	119.4
C1—B—O1	123.97 (12)	C21—C22—C23	121.27 (12)
C1—B—O2	124.75 (12)	H22—C22—C23	119.4
O1—B—O2	111.27 (11)	C22—C23—H23	119.5
B—O1—C11	105.17 (10)	C22—C23—C24	121.01 (13)
B—O2—C12	105.16 (10)	H23—C23—C24	119.5
O1—C11—C12	109.31 (10)	C23—C24—C25	117.73 (12)
O1—C11—C16	128.64 (11)	C23—C24—C27	121.17 (13)
C12—C11—C16	122.05 (12)	C25—C24—C27	121.10 (12)
O2—C12—C11	109.07 (10)	C24—C25—H25	119.2
O2—C12—C13	128.70 (11)	C24—C25—C26	121.65 (12)
C11—C12—C13	122.23 (11)	H25—C25—C26	119.2
C12—C13—H13	121.9	C21—C26—C25	120.58 (12)
C12—C13—C14	116.15 (12)	C21—C26—H26	119.7
H13—C13—C14	121.9	C25—C26—H26	119.7
C13—C14—H14	119.2	C24—C27—H27A	109.5
C13—C14—C15	121.58 (12)	C24—C27—H27B	109.5
H14—C14—C15	119.2	C24—C27—H27C	109.5
C14—C15—H15	119.3	H27A—C27—H27B	109.5
C14—C15—C16	121.49 (12)	H27A—C27—H27C	109.5

H15—C15—C16	119.3	H27B—C27—H27C	109.5
C11—C16—C15	116.46 (12)		
B—C1—C2—C21	177.86 (12)	C12—C13—C14—C15	−1.0 (2)
C2—C1—B—O1	6.1 (2)	C13—C14—C15—C16	−0.8 (2)
C2—C1—B—O2	−172.12 (12)	O1—C11—C16—C15	179.70 (12)
C1—B—O1—C11	−179.67 (12)	C12—C11—C16—C15	−0.1 (2)
O2—B—O1—C11	−1.20 (13)	C14—C15—C16—C11	1.4 (2)
C1—B—O2—C12	178.97 (12)	C1—C2—C21—C22	−177.24 (13)
O1—B—O2—C12	0.51 (13)	C1—C2—C21—C26	3.2 (2)
B—O1—C11—C12	1.42 (13)	C2—C21—C22—C23	−179.45 (12)
B—O1—C11—C16	−178.44 (13)	C26—C21—C22—C23	0.15 (19)
B—O2—C12—C11	0.40 (13)	C21—C22—C23—C24	0.5 (2)
B—O2—C12—C13	−179.13 (13)	C22—C23—C24—C25	−0.9 (2)
O1—C11—C12—O2	−1.17 (14)	C22—C23—C24—C27	178.79 (13)
O1—C11—C12—C13	178.40 (11)	C23—C24—C25—C26	0.73 (19)
C16—C11—C12—O2	178.70 (11)	C27—C24—C25—C26	−179.00 (12)
C16—C11—C12—C13	−1.7 (2)	C24—C25—C26—C21	−0.1 (2)
O2—C12—C13—C14	−178.27 (12)	C2—C21—C26—C25	179.22 (12)
C11—C12—C13—C14	2.26 (19)	C22—C21—C26—C25	−0.36 (19)