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A new polymorph of dimesitylborinic acid

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Key indicators

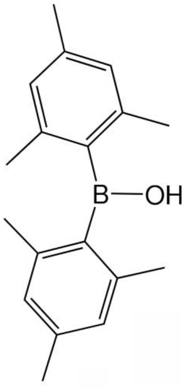
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.043
wR factor = 0.109
Data-to-parameter ratio = 18.7

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

A new polymorph of dimesitylborinic acid, $C_{18}H_{23}BO$, closely resembles the one previously reported in molecular conformation, crystal packing and symmetry. The asymmetric unit contains two molecules. The structure contains tetramers of crystallographic C_2 symmetry, held together by a ring of four O—H···O hydrogen bonds.

Comment

In the course of our studies (Yuan *et al.*, 2006) on the synthesis, crystal structures, linear and non-linear optical properties of *p*-*R*-phenyl-, *p*-*R*-phenylethynyl- and (*E*)-2-(*p*-*R*-phenyl)ethenyl-dimesitylboranes, we obtained a new (β) polymorph of the title compound (I). It is distinct from the form (α) reported by Weese *et al.* (1987), although it has the same space group ($C2/c$), broadly similar packing motifs (Fig. 1) and lattice parameters [for (α-I) at T = 130 K: a = 22.934 (12), b = 15.248 (9), c = 21.993 (11) Å, β = 125.53 (3)°, V = 6260 (6) Å^3]. The asymmetric unit comprises two molecules (Fig. 2 and Table 1) which, together with their equivalents generated by a twofold axis, form a hydrogen-bonded tetramer. The centroid of the tetramer has the coordinates ($\frac{1}{2}$, 0.5413, $\frac{1}{4}$), compared with ($\frac{1}{2}$, 0.2916, $\frac{1}{4}$) in (α-I). The four O atoms of the tetramer form a flattened tetrahedron, and each hydroxyl H atom is disordered over two positions; hence we have a superposition of two systems of hydrogen bonds, running along the O_4 ring in opposite directions (Fig. 3 and Table 2). A similar type of disorder may exist in (α-I), for which the hydroxyl H atoms were not located.



(I)

The B atoms have a trigonal-planar geometry, the arene rings (see Fig. 2) are inclined to their coordination planes (BC_2O) by 59.53 (4) (ring bz1), 53.37 (5) (bz2), 56.40 (5) (bz3) and 48.02 (6)° (bz4). The corresponding angles in (α-I) are 53.9, 53.5, 47.9 and 56.5°, respectively.

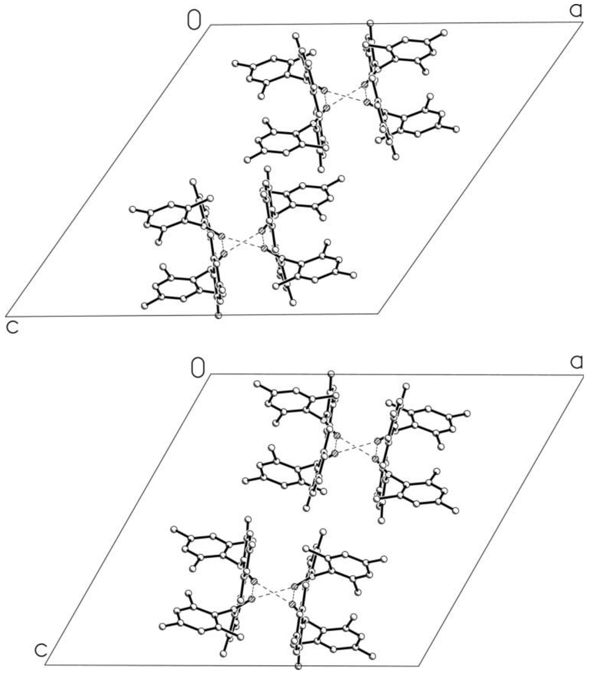


Figure 1
Crystal packing of α- (top) and β-forms of (I), projected on the (010) planes.

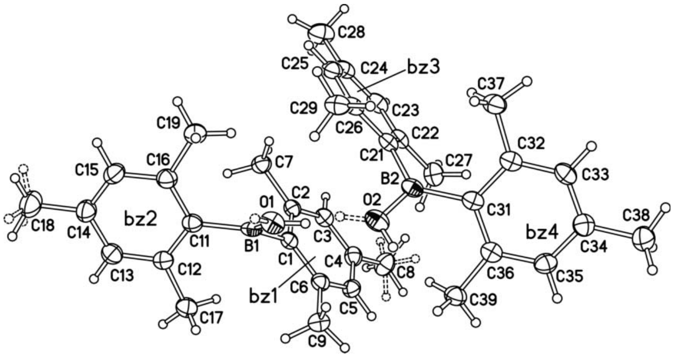


Figure 2
The asymmetric unit of (β-I), showing displacement ellipsoids at the 50% probability level.

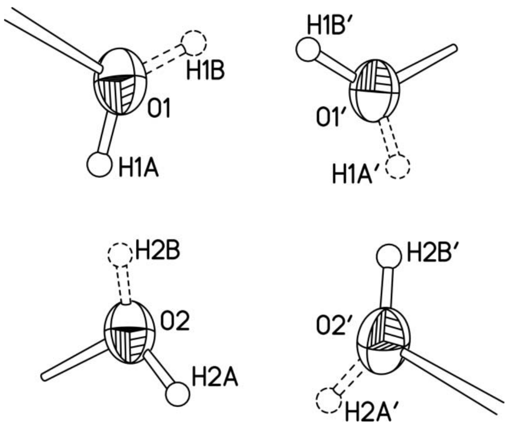


Figure 3
Hydrogen bonding in the tetramer. Primed atoms are generated by the twofold axis.

Compound (I) has been studied extensively, both chemically and crystallographically, as a ligand in transition-metal complexes, *e.g.* by Chisholm *et al.* (1993), Anulewicz-Ostrowska *et al.* (2000, 2002), Cole *et al.* (2005) and Zhao *et al.* (2007).

Experimental

Compound (I) was isolated as a hydrolysis by-product of the synthesis of (*E*)-1-{bis(mesityl)boryl}-2-(1,2-dicarba-closo-dodecaboranyl)-ethene (Yuan *et al.*, 2006) and obtained during recrystallization of the sample by slow evaporation of a DCM/hexane solution.

Crystal data

$C_{18}H_{23}BO$	$V = 6237.1\ (13)\ \text{Å}^3$
$M_r = 266.17$	$Z = 16$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
$a = 22.890\ (3)$ Å	$\mu = 0.07\ \text{mm}^{-1}$
$b = 15.233\ (2)$ Å	$T = 120\ (2)$ K
$c = 20.552\ (2)$ Å	0.33 × 0.25 × 0.04 mm
$\beta = 119.50\ (1)^\circ$	

Data collection

Bruker ProteumM APEX CCD area-detector diffractometer	7157 independent reflections
Absorption correction: none	4443 reflections with $I > 2\sigma(I)$
34987 measured reflections	$R_{\text{int}} = 0.067$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$	383 parameters
$wR(F^2) = 0.109$	H-atom parameters constrained
$S = 0.90$	$\Delta\rho_{\max} = 0.23$ e Å^{-3}
7157 reflections	$\Delta\rho_{\min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—B1	1.3647 (19)	O2—B2	1.3663 (18)
B1—C11	1.574 (2)	B2—C31	1.577 (2)
B1—C1	1.588 (2)	B2—C21	1.583 (2)
O1—B1—C11	117.53 (13)	O2—B2—C31	117.42 (13)
O1—B1—C1	116.49 (13)	O2—B2—C21	116.14 (13)
C11—B1—C1	125.97 (13)	C31—B2—C21	126.43 (13)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*B*···O1^{i}	0.84	2.04	2.7400 (19)	140
O1—H1*A*···O2	0.84	1.92	2.7071 (15)	155
O2—H2*B*···O1	0.84	1.88	2.7071 (15)	168
O2—H2*A*···O2^{i}	0.84	2.24	2.7370 (19)	118

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

All H atoms were approximately located in a difference Fourier map. Methyl groups were refined as rigid bodies rotating around the C—C bonds, with C—H = 0.98 Å and a common refined U_{iso} value for all H atoms of each group. Methyl groups $C8H_3$ and $C18H_3$ were treated as rotationally disordered (in a 2:1 ratio between 2 orientations). Hydroxyl atoms H1 and H2 were treated as disordered equally between positions *A* and *B*, riding on the oxygen atoms O1 and O2, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. The directions of O—H bonds were taken

o2640 Entwistle *et al.* • $C_{18}H_{23}BO$ *Acta Cryst.* (2007). E**63**, o2639–o2641

from the difference Fourier map, but their lengths were idealized to 0.84 Å. Aryl H atoms were treated as riding on the C atoms [C—H = 0.95 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C)].

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; 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*.

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Acta Cryst. (2007). E**63**, o2639–o2641 Entwistle *et al.* • $C_{18}H_{23}BO$ **o2641**