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A ‘tetrameric’ 3,4-di-*p*-tolyl-1,2,5-oxadiborole derivative

Michael J. Bayer, Hans Pritzkow and Walter Siebert*

Anorganisch-Chemisches Institut, Universität Heidelberg, Im Neuenheimer Feld 270, D-69120 Heidelberg, Germany
Correspondence e-mail: d61@ix.urz.uni-heidelberg.de

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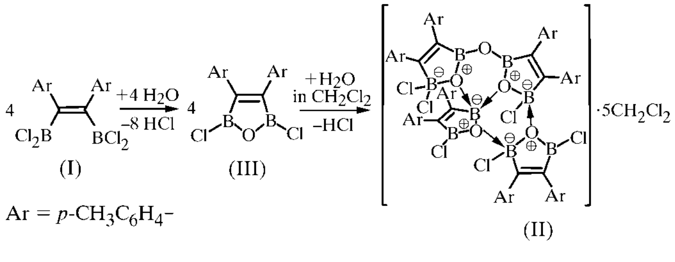
In the title compound, 1,1,6a,7,9a,10-hexachloro-2,3,5,6,8,9,11,12-octa-*p*-tolyl-1,6a,9a,12a-tetraborata-3a,4a,7,10-tetrabora-4a^1,6b,9b,12b-tetraoxonia-4-oxatetracyclopenta[1,2-*a*:2,1,5-*de*:1,2-*g*:1,2-*i*]naphthalene dichloromethane pentasolvate, $C_{64}H_{56}B_8Cl_6O_5\cdot5CH_2Cl_2$, two condensed oxadiborole rings are attached to two further oxadiborole rings in a type of donor–acceptor bonding, thus forming a ten-membered alternating $(B-O)_5$ naphthalene-like arrangement as the central building block.

Comment

In an attempt to synthesize 1,4-dichloro-2,3,5,6-tetra-*p*-tolyl-1,4-diboracyclohexa-2,5-diene, $[(p\text{-}CH_3C_6H_4)_2C_2BCl]_2$, *via* thermolysis of 1,2-bis(dichloroboryl)-1,2-di-*p*-tolylethene, (I), in toluene, we obtained the title compound, (II), as yellow crystals. The formation of (II) can be explained by traces of water originating from toluene that had not been rigorously dried.



The ‘tetramer’ (II) consists of two oxadiborole rings, (III), that are connected by an oxygen bridge (B—O—B). In addition, two further oxadiborole rings are attached to the diboryloxane derivative, thus forming donor–acceptor bonding systems in which the Cl atom on B3 (from the B3–B6 ring) migrates to atom B5. Compound (II) consists of a bicyclic $(B-O)_5$ building block with three $[(p\text{-tolyl-C})_2BCl]$ bridges and one di-*p*-tolylethene bridge. Compound (II) crystallizes with five CH_2Cl_2 solvate molecules.

In the molecular structure of (II), depicted in Fig. 1, different B—O bond lengths are observed. The B—O distances between atom O1 and the sp^2-hybridized atoms B1 and B2 [1.353 (2) and 1.363 (2) Å, respectively] lie in the range of comparable B—O bond lengths found in catecholborylalkanes, $C_6H_4O_2BR$ (B—O = 1.39 Å; Bayer *et al.*, 2002), and in oxadiboroles [B—O = 1.374 (3) Å; Köster *et al.*, 1994]. The B2—O2, B1—O3, B6—O4 and B8—O5 bond lengths in (II) are elongated [1.414 (2)–1.433 (2) Å] because of the threefold coordination of the O atoms. Bonds to the sp^3-hybridized B atoms are elongated compared with the corresponding bonds of sp^2-hybridized B atoms [B—C = 1.589–1.598 (3) *versus* 1.521–1.533 (3) Å and B—Cl = 1.814–1.840 (2) *versus* 1.728–1.734 (2) Å]. For B—O bonds between sp^3 B and threefold-coordinated O atoms the following tendency is observed: B atoms without neighboring Cl atoms form shorter B—O bonds [B3—O2 = 1.497 (2) Å and B3—O3 = 1.495 (2) Å] than B atoms with one chlorine substituent [B4—O3, B4—O5, B7—O4 and B7—O5 fall in the range 1.531 (2)–1.557 (2) Å], while the B—O bond at B5, which bears two Cl atoms, is even longer [1.623 (2) Å]. This observation is explained by the electron-withdrawing properties of the Cl atoms.

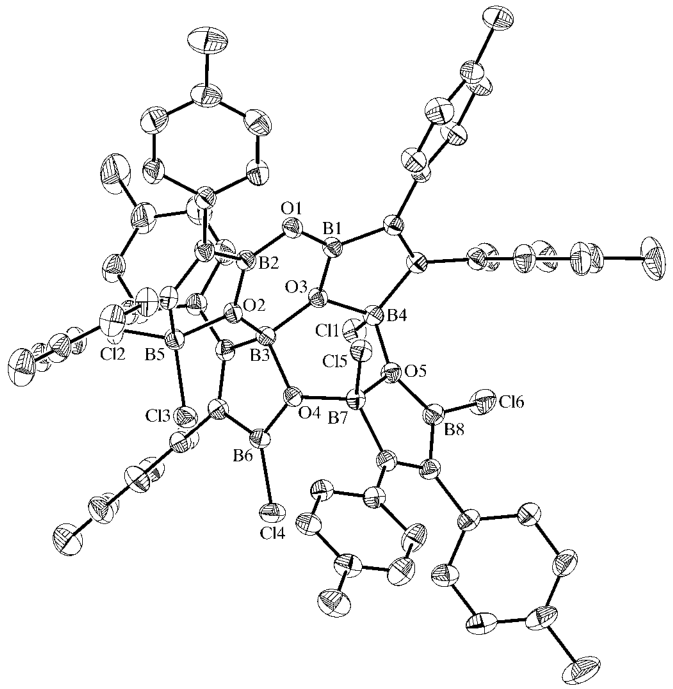


Figure 1
The molecular structure of (II). H atoms and solvent molecules have been omitted for clarity, and displacement ellipsoids are shown at the 50% probability level.

Experimental

Compound (I) was synthesized *via* diboronation of di-*p*-tolylacetylene with B_2Cl_4 according to the literature method of Siebert *et al.* (1989). Compound (I) (950 mg, 2.57 mmol) was dissolved in toluene (20 ml), and the resulting solution refluxed for 4 h. The solvent was removed in a vacuum to yield a red–brown oily residue. Recrystallization from pentane afforded yellow crystals of (II) at room temperature, which were completely insoluble in common organic solvents once formed.

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Crystal data

$C_{64}H_{56}B_8Cl_6O_5\cdot5CH_2Cl_2$
$M_r = 1628.90$
Triclinic, $P\overline{1}$
$a = 13.7073$ (8) Å
$b = 16.5780$ (9) Å
$c = 20.0237$ (11) Å
$\alpha = 68.135$ (1)°
$\beta = 82.695$ (1)°
$\gamma = 73.925$ (1)°
$V = 4056.3$ (4) Å^3
$Z = 2$
$D_x = 1.334$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6409 reflections
$\theta = 2.3$–31.1°
$\mu = 0.59$ mm^{-1}
$T = 190$ (2) K
Irregular, yellow
0.45 × 0.32 × 0.31 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1999)
$T_{\rm min} = 0.747$, $T_{\rm max} = 0.834$
68 074 measured reflections
25 625 independent reflections
17 850 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.034$
$\theta_{\rm max} = 31.0°$
$h = -19 \rightarrow 19$
$k = -21 \rightarrow 24$
$l = 0 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.214$
$S = 1.17$
25 625 reflections
841 parameters
Only H-atom U values refined
$w = 1/[\sigma^2(F_o^2) + (0.1264P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.009$
$\Delta\rho_{\rm max} = 1.06$ e Å^{-3}
$\Delta\rho_{\rm min} = -1.34$ e Å^{-3}

There are five dichloromethane molecules in the structure, two of which are strongly disordered. As it was not possible to model these disordered molecules, we used the SQUEEZE option in *PLATON* (Spek, 2003). H atoms were inserted at calculated positions, and a common isotropic displacement parameter was refined for each type. A residual electron density of about 1 e Å^{-3} was observed around the solvent molecules.

Data collection: *SMART* (Bruker, 1997–1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1549). Services for accessing these data are described at the back of the journal.

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