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N-(10-Bromoanthracen-9-ylmethyl)-*N*-[2-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)benzyl]methylamine at 240 K

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Key indicators

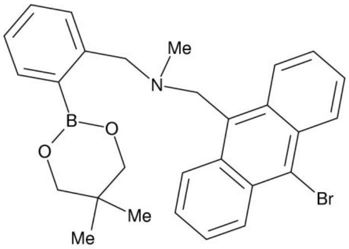
Single-crystal X-ray study
T = 240 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.085
Data-to-parameter ratio = 25.9

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

Features of the structure of the title compound, $C_{28}H_{29}BBrNO_2$, are the planar coordination of B and the intramolecular B···N contact distance of 3.204 (3) Å. The molecules form layers parallel to (010), with the creation of π–π and C—H···π contacts.

Comment

The synthesis and structure determination of the title compound, (I), is part of a continuing study, following on from the work of James *et al.* (1994), of compounds of potential value for use as sensors for sugar-like species. The molecular structure of (I) is shown in Fig. 1, and selected bond lengths and angles are given in Table 1. Bond lengths in the aryl fragments are in the ranges 1.351 (3)–1.445 (2) Å and 1.364 (3)–1.403 (3) Å for the bromoanthracene (C1–C14) and benzene ring (C18–C23) moieties, respectively. The C7—Br1 bond length [1.909 (2) Å] is, like all of the bond lengths found in this structure, as expected for a molecule of this kind. Particularly significant, however, is the intramolecular B1···N1 distance of 3.204 (3) Å. The dihedral angle between the least-squares planes (unit weights applied to the constituent atoms) of the anthracene and benzene ring groups as defined above is 69.64 (8)°. The relevant torsion angles in Table 1 are clearly compatible with the description of the conformation of the six-membered dioxaborinane ring as an envelope, with atom C25 at the point of the flap and with puckering parameters (Cremer & Pople, 1975) $Q(2)$, $Q(3)$ and $\varphi(2)$ of 0.384 (2) Å, −0.273 (2) Å and 359.3 (3)°, respectively [overall Q = 0.471 (2) Å, θ = 125.5 (2)° and $\varphi = \varphi(2)$ for the ring atoms in the order B1—O1—C24—C25—C28—O2]. The only axial non-H-atom substituent is atom C26.



(I)

In the unit cell, the molecules form layers in such a way as to induce two kinds of intermolecular contacts between pairs of centrosymmetrically related molecules. This situation is illustrated in Fig. 2, where these interactions are shown as dashed lines. The first is a π–π interaction between anthracene fragments, which is most conveniently assessed in terms of the

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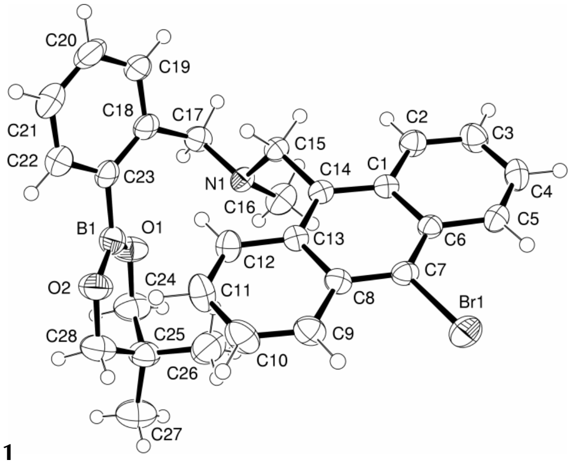

Figure 1
The molecular structure of (I), showing the labelling scheme. Non-H atoms are shown as 50% probability displacement ellipsoids and H atoms are shown as small spheres of arbitrary radii.

overlap of the rings of the form C1/C6–C8/C13/C14. These rings are related to one another (symmetry code: $2 - x$, $-y$, $1 - z$) by crystallographic centres of symmetry and their least-squares planes are then, by definition, precisely parallel to one another. In this circumstance, the overlap between the rings can be completely specified in terms of the distance between the centroids [3.950 (1) Å] and the perpendicular distance between the overlapping rings [3.483 (1) Å]. These values can be treated as two sides of a right-angled triangle of which the third side [1.863 (1) Å] is the lateral displacement or slippage of the overlapping rings in a direction parallel to their least-squares planes. The overlap of the anthracene fragments is shown more fully in Fig. 3. The second intermolecular contact in the layer of molecules is of the C—H$\cdots\pi$ type and involves atoms C28 and H28*A* and the benzene ring, defined by C18–23, with centroid *Cg* (symmetry code: $1 - x$, y, $\frac{1}{2} - z$). This interaction is characterized by C—H, H$\cdots$*Cg*, H_{perp} (the perpendicular distance of H28*A* from the plane of the benzene ring) and C$\cdots$*Cg* distances of 0.98, 3.32, 3.19 and 4.224 (3) Å, respectively. The C28—H28*A*$\cdots$*Cg* angle and the angle at H28*A* between H28*A*$\cdots$*Cg* and H_{perp} are 154 and 16°, respectively.

Experimental

Compound (I) was synthesized according to the procedure of James *et al.* (1995) but with 10-bromo-9-bromomethylanthracene as starting material. The product was recrystallized from methanol in a refrigerator at 283 K (m.p. 340–342 K). ^{1}H NMR (300 MHz, CDCl$_3$): δ 7.29–8.56 (*m*, 12H, ArH), 4.35 (*s*, 2H, ArCH$_2$), 3.92 (*s*, 2H, ArCH$_2$), 3.54 (*s*, 4H, OCH$_2$), 2.18 (*s*, 3H, NCH$_3$), 0.89 [*s*, 6H, C(CH$_3$)$_2$].

Crystal data

$C_{28}H_{29}BBrNO_2$
M_r = 502.24
Monoclinic, $C2/c$
a = 12.854 (7) Å
b = 14.457 (9) Å
c = 26.686 (11) Å
β = 100.06 (4)°
V = 4883 (5) Å^3
Z = 8

D_x = 1.366 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 22 217 reflections
θ = 4.3–31.9°
μ = 1.71 mm^{-1}
T = 240 (2) K
Block, yellow
0.40 × 0.40 × 0.35 mm

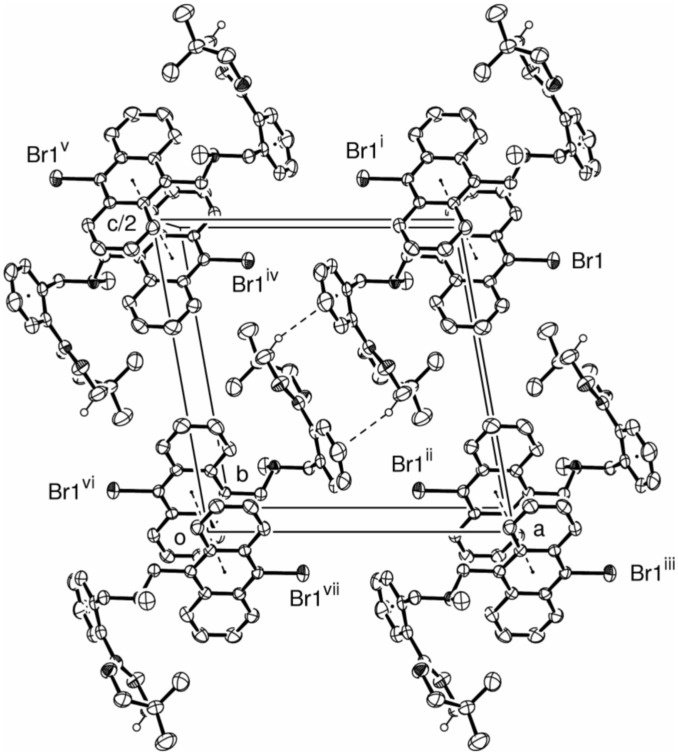

Figure 2
Part of a layer, parallel to (010), of molecules of (I). Intermolecular C—H$\cdots\pi$ and π–π contacts as explained in the text are denoted by dashed lines. The representation is otherwise the same as in Fig. 1, except that only selected atoms are labelled and H atoms not used in forming intermolecular contacts have been omitted. [Symmetry codes: (i) $2 - x$, $-y$, $1 - z$; (ii) $2 - x$, y, $\frac{1}{2} - z$; (iii) x, $-y$, $z - \frac{1}{2}$; (iv) $x - 1$, y, z; (v) $1 - x$, $-y$, $1 - z$; (vi) $1 - x$, y, $\frac{1}{2} - z$; (vii) $x - 1$, $-y$, $z - \frac{1}{2}$.]

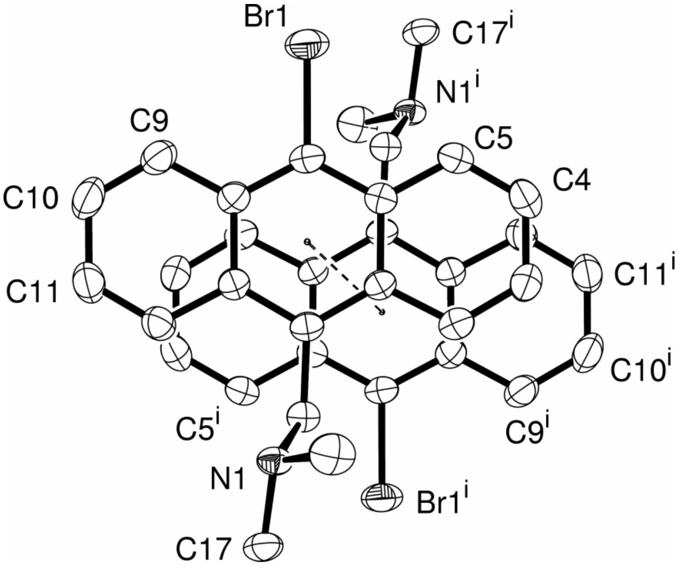

Figure 3
A pair of incomplete molecules of (I), displaying the overlap of the anthracene fragments. Non-H atoms are shown as 50% probability displacement ellipsoids and H atoms have been omitted for clarity. Selected atoms are labelled. The dashed line joins the ring centroids mentioned in the text. [Symmetry code: (i) $2 - x$, $-y$, $1 - z$.]

Data collection

Oxford Diffraction Excalibur2 CCD area-detector diffractometer
$\omega/2\theta$ scans
Absorption correction: multi-scan (Blessing, 1995, 1997)
$T_{\min} = 0.907$, $T_{\max} = 1.000$
22 217 measured reflections
7799 independent reflections
3964 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.038$
$\theta_{\max} = 31.9°$
$h = -17 \rightarrow 18$
$k = -20 \rightarrow 18$
$l = -39 \rightarrow 39$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.085$
$S = 0.87$
7799 reflections
301 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.35$ e Å^{-3}
$\Delta\rho_{\min} = -0.35$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C7—Br1	1.909 (2)	B1—O2	1.357 (3)
C15—N1	1.465 (2)	O1—C24	1.438 (2)
N1—C16	1.454 (2)	C24—C25	1.515 (3)
N1—C17	1.465 (2)	C25—C28	1.511 (3)
C23—B1	1.576 (3)	C28—O2	1.440 (3)
B1—O1	1.353 (3)		
C16—N1—C15	111.55 (15)	O1—B1—O2	123.2 (2)
C16—N1—C17	109.82 (14)	B1—O1—C24	119.76 (17)
C15—N1—C17	109.82 (14)	O1—C24—C25	112.36 (17)
O1—B1—C23	121.02 (19)	C28—C25—C24	107.60 (19)
O2—B1—C23	115.7 (2)	O2—C28—C25	112.76 (18)
		B1—O2—C28	119.39 (18)
O2—B1—O1—C24	−0.8 (3)	C23—B1—O1—C24	−178.20 (18)
B1—O1—C24—C25	−27.9 (3)	C23—B1—O2—C28	178.75 (18)
O1—C24—C25—C28	52.9 (3)	O1—C24—C25—C26	−68.2 (2)
C24—C25—C28—O2	−52.7 (3)	O1—C24—C25—C27	170.64 (19)
C25—C28—O2—B1	27.3 (3)	C26—C25—C28—O2	67.5 (3)
O1—B1—O2—C28	1.2 (3)	C27—C25—C28—O2	−170.9 (2)

In the final stages of refinement, H atoms were placed in calculated positions, with C—H = 0.94, 0.97 and 0.98 Å for aryl, methyl and methylene H atoms, respectively, and refined using a riding model, with $U_{\rm iso}$(H) values set at $1.5U_{\rm eq}$(C) for methyl H atoms and $1.2U_{\rm eq}$(C) for other H atoms.

Data collection: *CrysAlisCCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlisCCD*; data reduction: *CrysAlisRED* (Oxford Diffraction, 2003); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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