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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.

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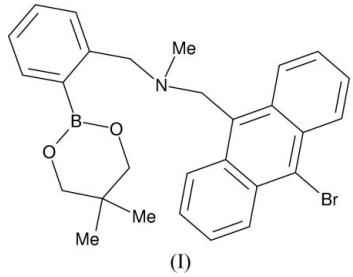
Features of the structure of the title compound, $C_{28}H_{29}BBrNO_2$, are the planar coordination of B and the intramolecular B···N contact distance of 3.204 (3) Å. The molecules form layers parallel to (010), with the creation of π–π and C—H···π contacts.

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 φ(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 φ = φ(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

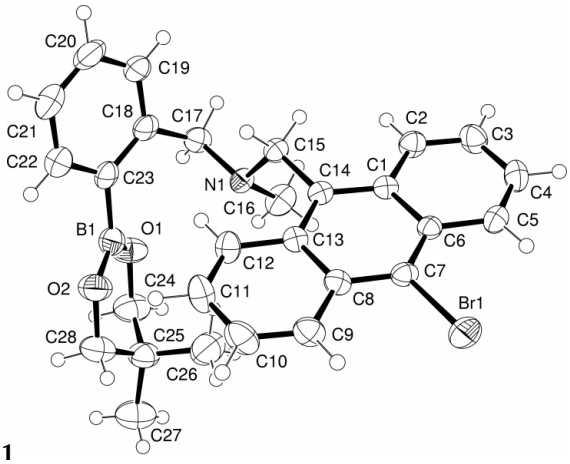


Figure 1
The molecular structure of (I), showing the labelling scheme. Non-H atoms are shown as 50% probability displacement ellipsoids and H atoms are shown as small spheres of arbitrary radii.

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

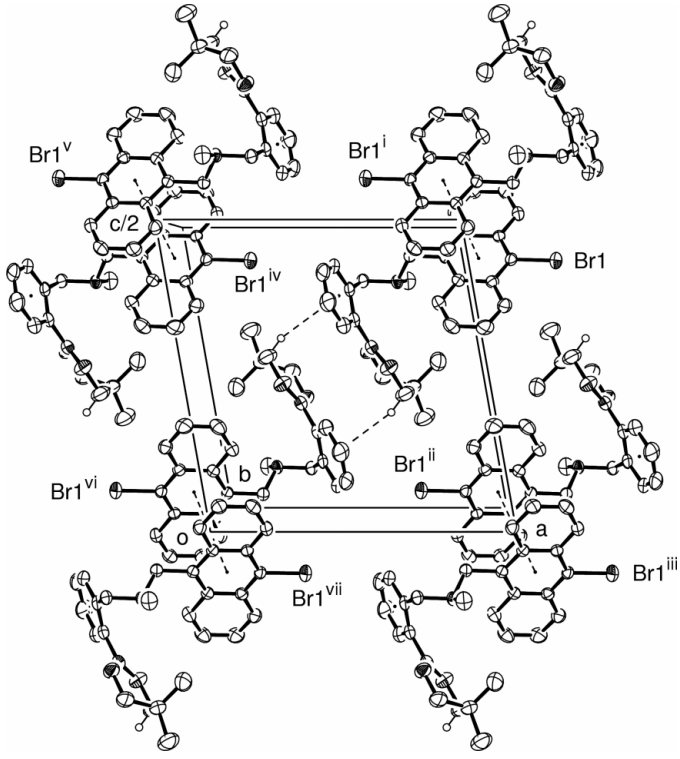


Figure 2
Part of a layer, parallel to (010), of molecules of (I). Intermolecular C—H$\cdots\pi$ and π–π contacts as explained in the text are denoted by dashed lines. The representation is otherwise the same as in Fig. 1, except that only selected atoms are labelled and H atoms not used in forming intermolecular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

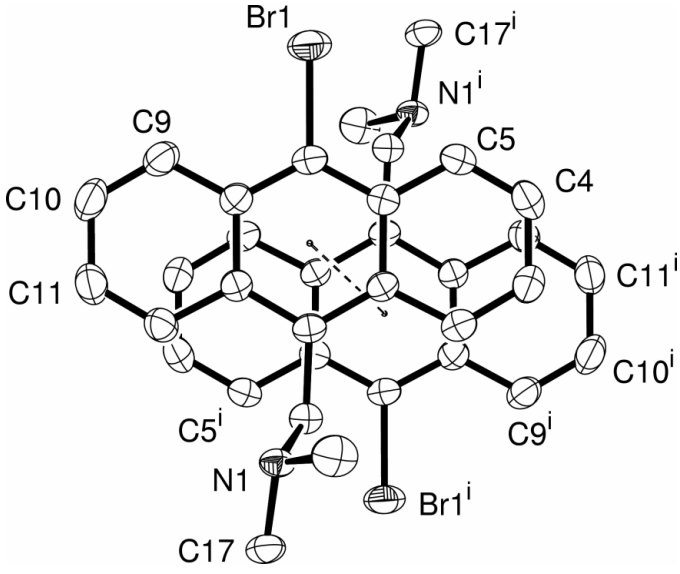


Figure 3
A pair of incomplete molecules of (I), displaying the overlap of the anthracene fragments. Non-H atoms are shown as 50% probability displacement ellipsoids and H atoms have been omitted for clarity. Selected atoms are labelled. The dashed line joins the ring centroids mentioned in the text. [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_{int} = 0.038
θ_{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_{iso}(H) values set at $1.5U_{eq}$(C) for methyl H atoms and $1.2U_{eq}$(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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supporting information

Acta Cryst. (2004). E**60**, o571–o573 [https://doi.org/10.1107/S1600536804005410]

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_N_-(10-Bromoanthracen-9-ylmethyl)-_N_-[2-(5,5-dimethyl-1,3,2-dioxaborinan-2- yl)benzyl]methylamine

Crystal data

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$M_r = 502.24$
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$c = 26.686\ (11)$ Å
$\beta = 100.06\ (4)°$
$V = 4883\ (5)$ Å^3
$Z = 8$
$F(000) = 2080$
$D_x = 1.366$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 22217 reflections
$\theta = 4.3–31.9°$
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$T = 240$ K
Block, yellow
0.40 × 0.40 × 0.35 mm

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Graphite monochromator
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Absorption correction: multi-scan (Blessing, 1995, 1997)
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22217 measured reflections
7799 independent reflections
3964 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 31.9°$, $\theta_{min} = 4.3°$
$h = -17\rightarrow18$
$k = -20\rightarrow18$
$l = -39\rightarrow39$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.085$
$S = 0.87$
7799 reflections
301 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
$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2]$ 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}

Special details

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. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	1.00768 (13)	0.11827 (11)	0.47589 (7)	0.0263 (4)
C2	0.98551 (15)	0.17985 (13)	0.51471 (7)	0.0357 (5)
H2	0.9147	0.1937	0.5163	0.043*
C3	1.06275 (16)	0.21884 (14)	0.54909 (8)	0.0410 (5)
H3	1.0451	0.2588	0.5741	0.049*
C4	1.16931 (16)	0.19984 (14)	0.54764 (8)	0.0406 (5)
H4	1.2225	0.2268	0.5719	0.049*
C5	1.19588 (14)	0.14315 (12)	0.51166 (7)	0.0335 (4)
H5	1.2676	0.1314	0.5112	0.040*
C6	1.11789 (13)	0.10103 (12)	0.47450 (7)	0.0275 (4)
C7	1.14230 (14)	0.04195 (12)	0.43666 (7)	0.0300 (4)
C8	1.06619 (14)	−0.00107 (12)	0.40060 (7)	0.0298 (4)
C9	1.09059 (16)	−0.06173 (13)	0.36229 (7)	0.0390 (5)
H9	1.1617	−0.0740	0.3606	0.047*
C10	1.01365 (18)	−0.10229 (14)	0.32809 (8)	0.0481 (6)
H10	1.0317	−0.1420	0.3031	0.058*
C11	0.90665 (17)	−0.08475 (14)	0.33011 (8)	0.0459 (5)
H11	0.8536	−0.1126	0.3061	0.055*
C12	0.87942 (15)	−0.02842 (12)	0.36609 (7)	0.0361 (5)
H12	0.8075	−0.0185	0.3669	0.043*
C13	0.95661 (13)	0.01626 (12)	0.40287 (7)	0.0276 (4)
C14	0.92829 (13)	0.07521 (12)	0.44044 (7)	0.0269 (4)
C15	0.81203 (13)	0.08650 (12)	0.44265 (7)	0.0296 (4)
H15A	0.7797	0.0251	0.4423	0.035*
H15B	0.8052	0.1163	0.4749	0.035*
N1	0.75375 (11)	0.14110 (10)	0.40070 (6)	0.0287 (3)
C16	0.78962 (16)	0.23665 (13)	0.40270 (9)	0.0469 (5)
H16A	0.7725	0.2663	0.4328	0.070*
H16B	0.7548	0.2692	0.3726	0.070*
H16C	0.8655	0.2382	0.4040	0.070*
C17	0.64047 (13)	0.13793 (13)	0.40230 (7)	0.0362 (5)
H17A	0.6037	0.1839	0.3786	0.043*
H17B	0.6292	0.1542	0.4366	0.043*
C18	0.59414 (13)	0.04339 (14)	0.38841 (7)	0.0357 (5)
C19	0.55765 (15)	−0.00943 (15)	0.42532 (8)	0.0448 (5)

H19	0.5571	0.0161	0.4577	0.054*
C20	0.52202 (16)	−0.09926 (17)	0.41514 (10)	0.0568 (6)
H20	0.4977	−0.1342	0.4405	0.068*
C21	0.52252 (17)	−0.13653 (17)	0.36825 (10)	0.0586 (6)
H21	0.4991	−0.1976	0.3613	0.070*
C22	0.55737 (16)	−0.08461 (16)	0.33091 (9)	0.0496 (6)
H22	0.5569	−0.1111	0.2987	0.060*
C23	0.59334 (14)	0.00611 (13)	0.33980 (8)	0.0365 (5)
B1	0.63288 (17)	0.05875 (18)	0.29493 (9)	0.0407 (6)
O1	0.61759 (11)	0.15084 (10)	0.28833 (5)	0.0502 (4)
C24	0.65182 (19)	0.19649 (17)	0.24618 (9)	0.0573 (6)
H24A	0.6718	0.2603	0.2559	0.069*
H24B	0.5929	0.1987	0.2174	0.069*
C25	0.74475 (17)	0.14806 (15)	0.22963 (8)	0.0474 (5)
C26	0.84277 (18)	0.15812 (18)	0.27099 (10)	0.0655 (7)
H26A	0.8282	0.1327	0.3027	0.098*
H26B	0.8608	0.2231	0.2757	0.098*
H26C	0.9014	0.1250	0.2608	0.098*
C27	0.7649 (2)	0.19040 (18)	0.17960 (9)	0.0727 (8)
H27A	0.8238	0.1589	0.1687	0.109*
H27B	0.7815	0.2555	0.1847	0.109*
H27C	0.7022	0.1836	0.1537	0.109*
C28	0.7151 (2)	0.04756 (17)	0.22043 (9)	0.0603 (7)
H28A	0.6594	0.0429	0.1904	0.072*
H28B	0.7767	0.0134	0.2133	0.072*
O2	0.67864 (12)	0.00534 (10)	0.26306 (5)	0.0508 (4)
Br1	1.287387 (16)	0.017499 (16)	0.434454 (9)	0.04864 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0281 (9)	0.0231 (9)	0.0281 (10)	0.0037 (7)	0.0059 (8)	0.0033 (8)
C2	0.0321 (11)	0.0366 (11)	0.0394 (12)	0.0041 (8)	0.0089 (9)	−0.0025 (9)
C3	0.0439 (12)	0.0423 (13)	0.0368 (12)	0.0033 (10)	0.0065 (10)	−0.0107 (10)
C4	0.0386 (12)	0.0405 (12)	0.0392 (12)	−0.0068 (9)	−0.0029 (10)	−0.0052 (10)
C5	0.0260 (10)	0.0350 (11)	0.0388 (11)	−0.0007 (8)	0.0040 (8)	0.0019 (9)
C6	0.0277 (9)	0.0282 (10)	0.0269 (10)	−0.0003 (7)	0.0052 (8)	0.0041 (8)
C7	0.0267 (9)	0.0341 (11)	0.0302 (10)	0.0052 (8)	0.0079 (8)	0.0051 (8)
C8	0.0315 (9)	0.0300 (11)	0.0278 (10)	0.0047 (8)	0.0050 (8)	0.0026 (8)
C9	0.0392 (11)	0.0408 (12)	0.0379 (12)	0.0097 (9)	0.0095 (10)	−0.0029 (10)
C10	0.0582 (15)	0.0458 (13)	0.0407 (13)	0.0101 (11)	0.0095 (11)	−0.0146 (11)
C11	0.0465 (13)	0.0413 (13)	0.0448 (13)	0.0028 (10)	−0.0061 (10)	−0.0121 (10)
C12	0.0324 (10)	0.0315 (11)	0.0419 (12)	0.0017 (8)	0.0000 (9)	−0.0038 (9)
C13	0.0297 (9)	0.0234 (9)	0.0291 (9)	0.0022 (7)	0.0036 (8)	0.0024 (8)
C14	0.0266 (9)	0.0250 (10)	0.0296 (10)	0.0037 (7)	0.0059 (8)	0.0053 (8)
C15	0.0279 (10)	0.0293 (10)	0.0318 (11)	0.0005 (7)	0.0059 (8)	0.0016 (8)
N1	0.0234 (8)	0.0270 (8)	0.0354 (9)	0.0036 (6)	0.0045 (7)	0.0037 (7)
C16	0.0471 (13)	0.0310 (12)	0.0624 (15)	0.0014 (9)	0.0094 (11)	0.0092 (11)

C17	0.0279 (10)	0.0445 (12)	0.0365 (11)	0.0114 (9)	0.0068 (8)	0.0040 (9)
C18	0.0179 (9)	0.0486 (13)	0.0403 (12)	0.0040 (8)	0.0044 (8)	0.0051 (10)
C19	0.0263 (10)	0.0697 (16)	0.0393 (12)	−0.0052 (10)	0.0080 (9)	0.0008 (11)
C20	0.0372 (12)	0.0731 (17)	0.0614 (16)	−0.0174 (12)	0.0126 (11)	0.0146 (14)
C21	0.0500 (14)	0.0575 (15)	0.0691 (17)	−0.0245 (12)	0.0124 (13)	−0.0047 (14)
C22	0.0369 (12)	0.0643 (16)	0.0472 (14)	−0.0099 (10)	0.0060 (10)	−0.0023 (12)
C23	0.0202 (9)	0.0476 (13)	0.0401 (11)	−0.0008 (8)	0.0014 (8)	0.0022 (10)
B1	0.0285 (12)	0.0543 (16)	0.0375 (14)	0.0034 (11)	0.0011 (10)	0.0008 (12)
O1	0.0523 (9)	0.0562 (10)	0.0457 (9)	0.0183 (7)	0.0185 (7)	0.0183 (8)
C24	0.0596 (15)	0.0642 (16)	0.0494 (14)	0.0135 (12)	0.0133 (12)	0.0249 (12)
C25	0.0501 (13)	0.0565 (15)	0.0372 (12)	0.0007 (11)	0.0120 (10)	0.0097 (11)
C26	0.0503 (15)	0.0789 (18)	0.0679 (17)	−0.0034 (13)	0.0116 (13)	0.0139 (14)
C27	0.085 (2)	0.0823 (19)	0.0568 (17)	−0.0016 (15)	0.0291 (15)	0.0153 (14)
C28	0.0704 (17)	0.0725 (18)	0.0427 (14)	−0.0069 (13)	0.0232 (12)	0.0006 (12)
O2	0.0604 (10)	0.0521 (10)	0.0440 (9)	−0.0048 (7)	0.0207 (8)	−0.0002 (7)
Br1	0.02980 (11)	0.06577 (16)	0.05168 (14)	0.00946 (10)	0.01076 (9)	−0.00139 (12)

Geometric parameters (Å, º)

C1—C14	1.410 (2)	C16—H16C	0.9700
C1—C2	1.432 (3)	C17—C18	1.511 (3)
C1—C6	1.445 (2)	C17—H17A	0.9800
C2—C3	1.352 (3)	C17—H17B	0.9800
C2—H2	0.9400	C18—C19	1.391 (3)
C3—C4	1.404 (3)	C18—C23	1.403 (3)
C3—H3	0.9400	C19—C20	1.388 (3)
C4—C5	1.351 (3)	C19—H19	0.9400
C4—H4	0.9400	C20—C21	1.364 (3)
C5—C6	1.418 (2)	C20—H20	0.9400
C5—H5	0.9400	C21—C22	1.383 (3)
C6—C7	1.400 (2)	C21—H21	0.9400
C7—C8	1.393 (3)	C22—C23	1.397 (3)
C7—Br1	1.909 (2)	C22—H22	0.9400
C8—C9	1.423 (3)	C23—B1	1.576 (3)
C8—C13	1.442 (3)	B1—O1	1.353 (3)
C9—C10	1.356 (3)	B1—O2	1.357 (3)
C9—H9	0.9400	O1—C24	1.438 (2)
C10—C11	1.409 (3)	C24—C25	1.515 (3)
C10—H10	0.9400	C24—H24A	0.9800
C11—C12	1.351 (3)	C24—H24B	0.9800
C11—H11	0.9400	C25—C28	1.511 (3)
C12—C13	1.423 (3)	C25—C26	1.530 (3)
C12—H12	0.9400	C25—C27	1.532 (3)
C13—C14	1.411 (2)	C26—H26A	0.9700
C14—C15	1.515 (2)	C26—H26B	0.9700
C15—N1	1.465 (2)	C26—H26C	0.9700
C15—H15A	0.9800	C27—H27A	0.9700
C15—H15B	0.9800	C27—H27B	0.9700

N1—C16	1.454 (2)	C27—H27C	0.9700
N1—C17	1.465 (2)	C28—O2	1.440 (3)
C16—H16A	0.9700	C28—H28A	0.9800
C16—H16B	0.9700	C28—H28B	0.9800
C14—C1—C2	123.22 (16)	N1—C17—H17A	109.2
C14—C1—C6	120.31 (16)	C18—C17—H17A	109.2
C2—C1—C6	116.47 (16)	N1—C17—H17B	109.2
C3—C2—C1	122.34 (18)	C18—C17—H17B	109.2
C3—C2—H2	118.8	H17A—C17—H17B	107.9
C1—C2—H2	118.8	C19—C18—C23	119.66 (19)
C2—C3—C4	120.27 (19)	C19—C18—C17	119.34 (18)
C2—C3—H3	119.9	C23—C18—C17	120.88 (17)
C4—C3—H3	119.9	C20—C19—C18	121.0 (2)
C5—C4—C3	120.49 (18)	C20—C19—H19	119.5
C5—C4—H4	119.8	C18—C19—H19	119.5
C3—C4—H4	119.8	C21—C20—C19	119.7 (2)
C4—C5—C6	121.45 (17)	C21—C20—H20	120.2
C4—C5—H5	119.3	C19—C20—H20	120.2
C6—C5—H5	119.3	C20—C21—C22	120.1 (2)
C7—C6—C5	123.12 (16)	C20—C21—H21	120.0
C7—C6—C1	117.90 (16)	C22—C21—H21	120.0
C5—C6—C1	118.97 (16)	C21—C22—C23	121.8 (2)
C8—C7—C6	123.51 (16)	C21—C22—H22	119.1
C8—C7—Br1	117.90 (13)	C23—C22—H22	119.1
C6—C7—Br1	118.59 (14)	C22—C23—C18	117.81 (18)
C7—C8—C9	123.71 (17)	C22—C23—B1	117.73 (19)
C7—C8—C13	117.82 (16)	C18—C23—B1	124.45 (18)
C9—C8—C13	118.46 (17)	O1—B1—C23	121.02 (19)
C10—C9—C8	121.57 (19)	O2—B1—C23	115.7 (2)
C10—C9—H9	119.2	O1—B1—O2	123.2 (2)
C8—C9—H9	119.2	B1—O1—C24	119.76 (17)
C9—C10—C11	119.89 (19)	O1—C24—C25	112.36 (17)
C9—C10—H10	120.1	O1—C24—H24A	109.1
C11—C10—H10	120.1	C25—C24—H24A	109.1
C12—C11—C10	120.77 (19)	O1—C24—H24B	109.1
C12—C11—H11	119.6	C25—C24—H24B	109.1
C10—C11—H11	119.6	H24A—C24—H24B	107.9
C11—C12—C13	121.86 (18)	C28—C25—C24	107.60 (19)
C11—C12—H12	119.1	C28—C25—C26	111.17 (19)
C13—C12—H12	119.1	C24—C25—C26	109.73 (19)
C14—C13—C12	121.93 (16)	C28—C25—C27	108.59 (19)
C14—C13—C8	120.64 (16)	C24—C25—C27	109.36 (19)
C12—C13—C8	117.43 (16)	C26—C25—C27	110.3 (2)
C1—C14—C13	119.82 (15)	C25—C26—H26A	109.5
C1—C14—C15	122.00 (15)	C25—C26—H26B	109.5
C13—C14—C15	118.12 (16)	H26A—C26—H26B	109.5
N1—C15—C14	113.88 (14)	C25—C26—H26C	109.5

N1—C15—H15A	108.8	H26A—C26—H26C	109.5
C14—C15—H15A	108.8	H26B—C26—H26C	109.5
N1—C15—H15B	108.8	C25—C27—H27A	109.5
C14—C15—H15B	108.8	C25—C27—H27B	109.5
H15A—C15—H15B	107.7	H27A—C27—H27B	109.5
C16—N1—C15	111.55 (15)	C25—C27—H27C	109.5
C16—N1—C17	109.82 (14)	H27A—C27—H27C	109.5
C15—N1—C17	109.82 (14)	H27B—C27—H27C	109.5
N1—C16—H16A	109.5	O2—C28—C25	112.76 (18)
N1—C16—H16B	109.5	O2—C28—H28A	109.0
H16A—C16—H16B	109.5	C25—C28—H28A	109.0
N1—C16—H16C	109.5	O2—C28—H28B	109.0
H16A—C16—H16C	109.5	C25—C28—H28B	109.0
H16B—C16—H16C	109.5	H28A—C28—H28B	107.8
N1—C17—C18	111.86 (14)	B1—O2—C28	119.39 (18)

C14—C1—C2—C3	178.46 (18)	C8—C13—C14—C15	−176.70 (15)
C6—C1—C2—C3	−1.3 (3)	C1—C14—C15—N1	110.71 (18)
C1—C2—C3—C4	0.2 (3)	C13—C14—C15—N1	−72.1 (2)
C2—C3—C4—C5	0.6 (3)	C14—C15—N1—C16	−65.43 (19)
C3—C4—C5—C6	−0.1 (3)	C14—C15—N1—C17	172.57 (15)
C4—C5—C6—C7	180.00 (17)	C16—N1—C17—C18	167.01 (16)
C4—C5—C6—C1	−1.0 (3)	C15—N1—C17—C18	−69.96 (19)
C14—C1—C6—C7	1.0 (2)	N1—C17—C18—C19	112.94 (19)
C2—C1—C6—C7	−179.30 (16)	N1—C17—C18—C23	−63.2 (2)
C14—C1—C6—C5	−178.08 (16)	C23—C18—C19—C20	1.1 (3)
C2—C1—C6—C5	1.7 (2)	C17—C18—C19—C20	−175.04 (17)
C5—C6—C7—C8	178.75 (16)	C18—C19—C20—C21	−0.1 (3)
C1—C6—C7—C8	−0.2 (3)	C19—C20—C21—C22	−0.7 (3)
C5—C6—C7—Br1	−0.5 (2)	C20—C21—C22—C23	0.4 (3)
C1—C6—C7—Br1	−179.53 (12)	C21—C22—C23—C18	0.6 (3)
C6—C7—C8—C9	−179.51 (17)	C21—C22—C23—B1	179.1 (2)
Br1—C7—C8—C9	−0.2 (2)	C19—C18—C23—C22	−1.3 (3)
C6—C7—C8—C13	−0.3 (3)	C17—C18—C23—C22	174.77 (17)
Br1—C7—C8—C13	178.97 (12)	C19—C18—C23—B1	−179.76 (18)
C7—C8—C9—C10	179.77 (18)	C17—C18—C23—B1	−3.7 (3)
C13—C8—C9—C10	0.6 (3)	C22—C23—B1—O1	145.6 (2)
C8—C9—C10—C11	−0.1 (3)	C18—C23—B1—O1	−36.0 (3)
C9—C10—C11—C12	−0.6 (3)	C22—C23—B1—O2	−32.0 (3)
C10—C11—C12—C13	0.8 (3)	C18—C23—B1—O2	146.40 (19)
C11—C12—C13—C14	179.93 (18)	O2—B1—O1—C24	−0.8 (3)
C11—C12—C13—C8	−0.3 (3)	B1—O1—C24—C25	−27.9 (3)
C7—C8—C13—C14	0.2 (2)	O1—C24—C25—C28	52.9 (3)
C9—C8—C13—C14	179.42 (16)	C24—C25—C28—O2	−52.7 (3)
C7—C8—C13—C12	−179.62 (16)	C25—C28—O2—B1	27.3 (3)
C9—C8—C13—C12	−0.4 (2)	O1—B1—O2—C28	1.2 (3)
C2—C1—C14—C13	179.18 (16)	C23—B1—O1—C24	−178.20 (18)
C6—C1—C14—C13	−1.1 (2)	C23—B1—O2—C28	178.75 (18)

C2—C1—C14—C15	−3.7 (3)	O1—C24—C25—C26	−68.2 (2)
C6—C1—C14—C15	176.01 (15)	O1—C24—C25—C27	170.64 (19)
C12—C13—C14—C1	−179.68 (16)	C26—C25—C28—O2	67.5 (3)
C8—C13—C14—C1	0.5 (2)	C27—C25—C28—O2	−170.9 (2)
C12—C13—C14—C15	3.1 (2)		