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3-Benzyl-6-bromo-3,4-dihydro-2*H*-benzo[e][1,3]oxazine

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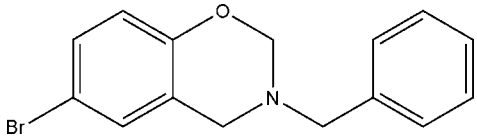
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.005 Å; R factor = 0.041; wR factor = 0.120; data-to-parameter ratio = 15.7.

In the title compound, $C_{14}H_{15}BrNO$, the dihedral angle between the two benzene rings is 77.1 (3)°. In the crystal structure, the molecules are linked through C—H···π(arene) interactions.

Related literature

For related literature, see: Barker *et al.* (2006); Gentles *et al.* (1991); Peglion *et al.* (1997); Petterson *et al.* (1990); Ren *et al.* (2001).



Experimental

Crystal data

$C_{15}H_{14}BrNO$
M_r = 304.18
Triclinic, $P\overline{1}$
a = 6.2944 (6) Å
b = 10.7466 (10) Å
c = 10.9927 (10) Å
α = 65.554 (1)°
β = 78.674 (1)°
γ = 86.678 (2)°
V = 663.51 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.09 mm^{-1}
T = 291 (2) K
0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.577, T_{max} = 0.748
4516 measured reflections
2567 independent reflections
2029 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.121
S = 1.11
2567 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*2 and *Cg*3 are the centroids of benzene rings C1–C6 and C10–C15, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8A···$Cg3^{i}$	0.97	2.75	3.706 (6)	178
C12—H12···$Cg2^{ii}$	0.93	2.71	3.632 (8)	173

Symmetry codes: (i) $x+1, y, z$; (ii) $-x, -y, -z+1$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2090).

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supplementary materials

Acta Cryst. (2007). E**63**, o3580 [**doi:10.1107/S1600536807035039**]

3-Benzyl-6-bromo-3,4-dihydro-2*H*-benzo[*e*][1,3]oxazine

X.-L. Chen, X.-J. Diao and M.-H. Wu

Comment

Benzoxazine, which has been widely used as a potential agonist trigger, antipsychotic agent (Barker *et al.*, 2006), antimalarial agent (Ren *et al.*, 2001), and a serotonin and dopamine receptor (Gentles *et al.*, 1991; Petterson *et al.*, 1990; Peglion *et al.*, 1997), is a useful heterocyclic compound. The title compound (I) was prepared by reaction of 4-bromophenol, formaldehyde and benzyl amine. The crystal structure of (I) is described here.

In the molecule (Fig. 1), the dihedral angle between the two benzene rings is 77.1 (3)°. N1 and C8 deviate markedly from the O1—C4—C5—C7—N1—C8 plane, with deviations of −0.320 (4) and 0.282 (7) Å, respectively.

In the crystal structure, molecules are linked by two C—H···π(arene) interactions, which connect H12 to the centroid of C1–C6, *Cg*2, in an adjacent molecule (symmetry code: –*X*, –Y, 1-*Z*), and H8A to the centroid of C2–C7, *Cg*3, in a second adjacent molecule (symmetry code: 1+*X*, Y, *Z*).

Experimental

Formaldehyde (8 ml, 40%, 0.1 mol) was added slowly with stirring to a mixture of methanol (35 ml), benzylamine (10.7 g, 0.1 mol) and 4-bromophenol (10.8 g, 0.1 mol) over 2 h. The mixture was stirred for an additional 60 h at room temperature. The resulting bright yellow solid was filtered and washed with methanol. The solid residue was recrystallized from methanol to give colorless crystals of the title compound (I) suitable for X-ray analysis, with a yield of 85%. ^{1}HNMR($CDCl_3$, 400 MHz), 7.36 (m, 8H, aromatic), 4.87 (s, 2H, N—CH_2—O), 3.94 (s, 2H, N—CH_2-heterocyclic), 3.90 (s, 2H, *N*—CH_2-benzyl).

Refinement

All H atoms were placed in calculated positions (C—H = 0.93 Å) and included in the riding model approximation, with U_{iso}(H) = 1.2U_{iso}(C).

Figures

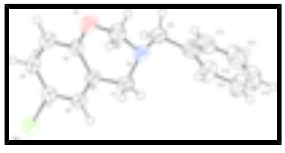

Fig. 1. View of the molecule of (I) with the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

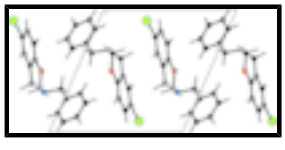

Fig. 2. The crystal packing of (I), viewed down the *c* axis.

3-Benzyl-6-bromo-3,4-dihydro-2*H*-benzo[e][1,3]oxazine

Crystal data

$C_{15}H_{14}BrNO$	$Z = 2$
$M_r = 304.18$	$F_{000} = 308$
Triclinic, $P\overline{1}$	$D_x = 1.523$ Mg m^{-3}
$a = 6.2944$ (6) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 10.7466$ (10) Å	Cell parameters from 1924 reflections
$c = 10.9927$ (10) Å	$\theta = 3.3$–24.8º
$\alpha = 65.554$ (1)º	$\mu = 3.09$ mm^{-1}
$\beta = 78.674$ (1)º	$T = 291$ (2) K
$\gamma = 86.678$ (2)º	Block, colourless
$V = 663.51$ (11) Å^3	0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2567 independent reflections
Radiation source: fine-focus sealed tube	2029 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.022$
$T = 291$(2) K	$\theta_{max} = 26.0$º
φ and ω scans	$\theta_{min} = 2.1$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	$h = -7 \rightarrow 7$
$T_{min} = 0.577$, $T_{max} = 0.748$	$k = -13 \rightarrow 11$
4516 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	H-atom parameters constrained
$wR(F^2) = 0.121$	$w = 1/[\sigma^2(F_o^2) + (0.0651P)^2 + 0.1104P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.11$	$(\Delta/\sigma)_{max} = 0.001$
2567 reflections	$\Delta\rho_{max} = 0.48$ e Å^{-3}
163 parameters	$\Delta\rho_{min} = -0.35$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.14407 (6)	0.63851 (4)	0.09288 (4)	0.0672 (2)
C1	0.3047 (5)	0.5198 (3)	0.2233 (3)	0.0460 (8)
C2	0.5220 (5)	0.4985 (4)	0.1810 (4)	0.0511 (8)
H2	0.5885	0.5429	0.0902	0.061*
C3	0.6370 (5)	0.4108 (4)	0.2755 (4)	0.0536 (9)
H3	0.7807	0.3937	0.2477	0.064*
C4	0.5405 (5)	0.3479 (3)	0.4114 (3)	0.0452 (8)
C5	0.3205 (5)	0.3674 (3)	0.4537 (3)	0.0407 (7)
C6	0.2055 (5)	0.4537 (3)	0.3573 (3)	0.0428 (7)
H6	0.0593	0.4670	0.3836	0.051*
C7	0.2163 (5)	0.2921 (4)	0.6014 (3)	0.0478 (8)
H7A	0.2058	0.3538	0.6463	0.057*
H7B	0.0706	0.2625	0.6072	0.057*
C8	0.5623 (6)	0.2128 (4)	0.6429 (4)	0.0607 (10)
H8A	0.6417	0.1349	0.6944	0.073*
H8B	0.5749	0.2838	0.6742	0.073*
C9	0.3010 (5)	0.0537 (4)	0.6479 (4)	0.0502 (8)
H9A	0.4166	−0.0100	0.6733	0.060*
H9B	0.3053	0.0818	0.5514	0.060*
C10	0.0872 (5)	−0.0181 (3)	0.7260 (3)	0.0463 (8)
C11	−0.0042 (7)	−0.1055 (4)	0.6850 (5)	0.0651 (10)
H11	0.0647	−0.1173	0.6077	0.078*
C12	−0.1982 (8)	−0.1754 (4)	0.7594 (6)	0.0817 (14)
H12	−0.2574	−0.2346	0.7320	0.098*
C13	−0.3018 (7)	−0.1584 (5)	0.8710 (5)	0.0799 (14)
H13	−0.4321	−0.2054	0.9194	0.096*
C14	−0.2166 (7)	−0.0724 (5)	0.9135 (4)	0.0724 (12)
H14	−0.2881	−0.0609	0.9905	0.087*
C15	−0.0217 (6)	−0.0023 (4)	0.8403 (4)	0.0574 (9)
H15	0.0362	0.0564	0.8689	0.069*
O1	0.6632 (3)	0.2629 (3)	0.4997 (3)	0.0621 (7)
N1	0.3396 (4)	0.1730 (3)	0.6717 (3)	0.0459 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0550 (3)	0.0711 (3)	0.0505 (3)	0.00478 (19)	−0.00503 (17)	−0.00313 (19)
C1	0.0474 (17)	0.0404 (17)	0.0429 (18)	−0.0006 (14)	−0.0033 (14)	−0.0122 (14)
C2	0.0452 (17)	0.050 (2)	0.0427 (19)	−0.0100 (15)	0.0083 (14)	−0.0098 (16)
C3	0.0326 (16)	0.059 (2)	0.057 (2)	−0.0032 (15)	0.0062 (14)	−0.0176 (18)
C4	0.0331 (15)	0.0449 (18)	0.0473 (19)	−0.0071 (13)	−0.0014 (13)	−0.0106 (15)
C5	0.0356 (15)	0.0382 (17)	0.0442 (17)	−0.0033 (13)	0.0001 (13)	−0.0158 (14)
C6	0.0380 (15)	0.0371 (17)	0.0455 (19)	−0.0017 (13)	0.0026 (13)	−0.0135 (14)
C7	0.0414 (16)	0.0505 (19)	0.0434 (19)	0.0055 (14)	0.0017 (14)	−0.0162 (15)
C8	0.0441 (18)	0.073 (3)	0.052 (2)	−0.0128 (18)	−0.0074 (16)	−0.0115 (19)
C9	0.0424 (17)	0.054 (2)	0.0463 (19)	0.0057 (15)	−0.0020 (14)	−0.0164 (16)
C10	0.0479 (18)	0.0422 (18)	0.0440 (19)	0.0046 (14)	−0.0116 (14)	−0.0123 (15)
C11	0.082 (3)	0.046 (2)	0.069 (3)	0.003 (2)	−0.012 (2)	−0.026 (2)
C12	0.092 (3)	0.047 (2)	0.106 (4)	−0.016 (2)	−0.028 (3)	−0.025 (3)
C13	0.066 (3)	0.064 (3)	0.081 (3)	−0.023 (2)	−0.023 (2)	0.006 (2)
C14	0.059 (2)	0.097 (3)	0.042 (2)	−0.015 (2)	−0.0064 (18)	−0.008 (2)
C15	0.0487 (19)	0.078 (3)	0.0404 (19)	−0.0148 (18)	−0.0062 (15)	−0.0183 (19)
O1	0.0344 (11)	0.0765 (18)	0.0554 (15)	0.0014 (11)	−0.0043 (10)	−0.0092 (13)
N1	0.0378 (13)	0.0516 (16)	0.0418 (15)	−0.0008 (12)	−0.0058 (11)	−0.0132 (13)

Geometric parameters (Å, °)

Br1—C1	1.901 (3)	C8—H8A	0.9700
C1—C6	1.374 (4)	C8—H8B	0.9700
C1—C2	1.394 (5)	C9—N1	1.453 (4)
C2—C3	1.377 (5)	C9—C10	1.504 (5)
C2—H2	0.9300	C9—H9A	0.9700
C3—C4	1.383 (5)	C9—H9B	0.9700
C3—H3	0.9300	C10—C15	1.379 (5)
C4—O1	1.361 (4)	C10—C11	1.389 (5)
C4—C5	1.404 (4)	C11—C12	1.389 (7)
C5—C6	1.384 (4)	C11—H11	0.9300
C5—C7	1.509 (5)	C12—C13	1.350 (7)
C6—H6	0.9300	C12—H12	0.9300
C7—N1	1.463 (4)	C13—C14	1.369 (7)
C7—H7A	0.9700	C13—H13	0.9300
C7—H7B	0.9700	C14—C15	1.392 (5)
C8—N1	1.425 (4)	C14—H14	0.9300
C8—O1	1.455 (4)	C15—H15	0.9300
C6—C1—C2	120.6 (3)	H8A—C8—H8B	107.6
C6—C1—Br1	120.1 (2)	N1—C9—C10	113.1 (3)
C2—C1—Br1	119.3 (3)	N1—C9—H9A	109.0
C3—C2—C1	119.1 (3)	C10—C9—H9A	109.0
C3—C2—H2	120.4	N1—C9—H9B	109.0
C1—C2—H2	120.4	C10—C9—H9B	109.0

C2—C3—C4	120.6 (3)	H9A—C9—H9B	107.8
C2—C3—H3	119.7	C15—C10—C11	118.1 (3)
C4—C3—H3	119.7	C15—C10—C9	122.0 (3)
O1—C4—C3	117.7 (3)	C11—C10—C9	120.0 (3)
O1—C4—C5	122.0 (3)	C12—C11—C10	120.1 (4)
C3—C4—C5	120.3 (3)	C12—C11—H11	119.9
C6—C5—C4	118.6 (3)	C10—C11—H11	119.9
C6—C5—C7	122.2 (3)	C13—C12—C11	120.8 (4)
C4—C5—C7	119.2 (3)	C13—C12—H12	119.6
C1—C6—C5	120.8 (3)	C11—C12—H12	119.6
C1—C6—H6	119.6	C12—C13—C14	120.5 (4)
C5—C6—H6	119.6	C12—C13—H13	119.8
N1—C7—C5	111.9 (2)	C14—C13—H13	119.8
N1—C7—H7A	109.2	C13—C14—C15	119.3 (4)
C5—C7—H7A	109.2	C13—C14—H14	120.3
N1—C7—H7B	109.2	C15—C14—H14	120.3
C5—C7—H7B	109.2	C10—C15—C14	121.2 (4)
H7A—C7—H7B	107.9	C10—C15—H15	119.4
N1—C8—O1	114.4 (3)	C14—C15—H15	119.4
N1—C8—H8A	108.7	C4—O1—C8	115.3 (2)
O1—C8—H8A	108.7	C8—N1—C9	114.3 (3)
N1—C8—H8B	108.7	C8—N1—C7	109.1 (3)
O1—C8—H8B	108.7	C9—N1—C7	113.9 (3)
C6—C1—C2—C3	−0.4 (5)	C15—C10—C11—C12	−0.8 (6)
Br1—C1—C2—C3	−179.0 (3)	C9—C10—C11—C12	178.0 (4)
C1—C2—C3—C4	−2.0 (6)	C10—C11—C12—C13	0.8 (7)
C2—C3—C4—O1	−179.7 (3)	C11—C12—C13—C14	−0.4 (7)
C2—C3—C4—C5	3.2 (6)	C12—C13—C14—C15	0.1 (7)
O1—C4—C5—C6	−178.9 (3)	C11—C10—C15—C14	0.5 (6)
C3—C4—C5—C6	−1.9 (5)	C9—C10—C15—C14	−178.2 (4)
O1—C4—C5—C7	0.0 (5)	C13—C14—C15—C10	−0.2 (6)
C3—C4—C5—C7	177.0 (3)	C3—C4—O1—C8	172.8 (3)
C2—C1—C6—C5	1.7 (5)	C5—C4—O1—C8	−10.1 (5)
Br1—C1—C6—C5	−179.7 (2)	N1—C8—O1—C4	42.2 (5)
C4—C5—C6—C1	−0.6 (5)	O1—C8—N1—C9	66.8 (4)
C7—C5—C6—C1	−179.4 (3)	O1—C8—N1—C7	−62.0 (4)
C6—C5—C7—N1	158.9 (3)	C10—C9—N1—C8	157.9 (3)
C4—C5—C7—N1	−19.9 (4)	C10—C9—N1—C7	−75.8 (3)
N1—C9—C10—C15	−21.0 (4)	C5—C7—N1—C8	48.8 (4)
N1—C9—C10—C11	160.3 (3)	C5—C7—N1—C9	−80.2 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8A···Cg3^{i}	0.97	2.75	3.706 (6)	178
C12—H12···Cg2^{ii}	0.93	2.71	3.632 (8)	173

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*, −*y*, −*z*+1.

Fig. 1

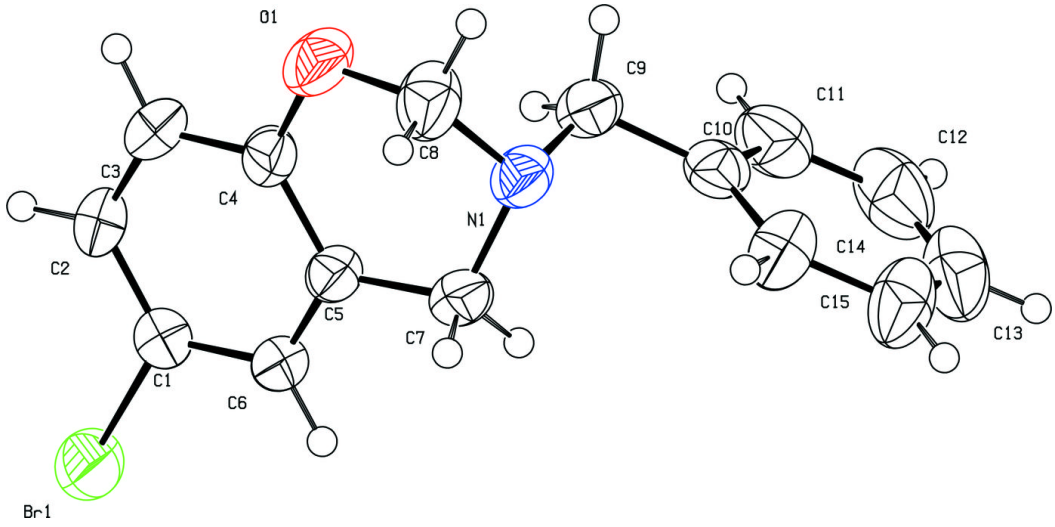

Fig. 2