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3-Benzyl-6-methyl-3,4-dihydro-2*H*-1,3-benzoxazine

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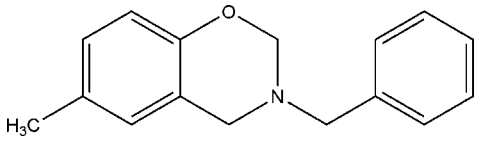
Key indicators: single-crystal X-ray study; T = 290 K; mean σ(C–C) = 0.003 Å; R factor = 0.056; wR factor = 0.150; data-to-parameter ratio = 14.7.

In the title molecule, $C_{16}H_{17}NO$, the dihedral angle between the phenyl and benzene rings is 28.4 (1)°. The six-membered 3,4-dihydro-2*H*-1,3-oxazine ring adopts a screw boat conformation. In the crystal structure, weak C—H···π(arene) interactions are present.

Related literature

For background information, see: Holly & Cope (1944); Ren *et al.* (2001); Gentles *et al.* (1991); Petterson *et al.* (1990); Peglion *et al.* (1997).



Experimental

Crystal data

$C_{16}H_{17}NO$
M_r = 239.31
Monoclinic, $P2_1/n$
a = 13.4112 (11) Å
b = 5.1816 (4) Å
c = 19.4220 (17) Å
β = 105.007 (1)°
V = 1303.63 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 290 (2) K
0.20 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.985, T_{max} = 0.993
8116 measured reflections
2418 independent reflections
1721 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.150
S = 1.05
2418 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 1
C—H···π interactions (Å, °).

*Cg*1 and *Cg*2 are the centroids defined by ring atoms C11–C16 and C2–C7, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···*Cg*1^{i}	0.93	2.93	3.820 (3)	159
C8—H8*B*···*Cg*2^{ii}	0.97	2.98	3.907 (2)	159

Symmetry codes: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x, y-1, z$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; 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: LH2465).

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supplementary materials

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3-Benzyl-6-methyl-3,4-dihydro-2*H*-1,3-benzoxazine

X.-L. Chen and M.-H. Wu

Comment

Benzoxazine is a unique heterocyclic compound obtained from a cyclization reaction involving phenol, formaldehyde, and amine (Holly & Cope, 1944). It has generated interest as use in antipsychotic agents and antimalarial agents (Ren *et al.*, 2001) and in the research of serotonin and dopamine receptors (Gentles *et al.*, 1991; Petterson *et al.*, 1990; Peglion *et al.*, 1997). The title compound was prepared by reaction of 4-methylphenol, formaldehyde and benzyl amine and it's crystal structure has been determined herein.

In the molecule (Fig. 1), the dihedral angle between the phenyl and benzene rings is 28.4 (1)°. The atoms C11/C10/N1/C8 are nearly planar with the torsion angle for C11—C10—N1—C8 being 176.8 (2)°, and the dihedral angle between the C11—C10—N1—C8 mean plane and phenyl ring (C11—C16) is 65.6 (2)°.

In the crystal structure, molecules are linked by two types of weak C—H···(arene) interactions, involving atom H4 and the centroid of atoms C11–16, *Cg*1^{i}, (symmetry code: (i) 3/2 − *x*,1/2 + *y*,3/2 − *z*), and H8B and the centroid of C2—C7, *Cg*2^{ii}, (symmetry code: (ii) *x*,-1 + *y*, *z*) (Fig. 2).

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-methylphenol (10.8 g, 0.1 mol) over 2 h. The mixture was stirred for 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 in yield 88%, which were suitable for X-ray analysis.

Refinement

All H atoms were placed in calculated positions (C—H = 0.93–0.97 Å) and included in the riding model approximation, with U_{iso} (H) = 1.2U_{iso} (C) or 1.5U_{eq}(methyl C).

Figures

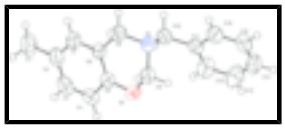

Fig. 1. View of the molecule with the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

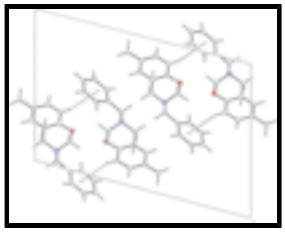

Fig. 2. The packing viewed down the *b* axis. Intermolecular C—H···π interactions are shown as dashed lines.

3-Benzyl-6-methyl-3,4-dihydro-2*H*-1,3-benzoxazine

Crystal data

$C_{16}H_{17}NO$	F_{000} = 512
M_r = 239.31	D_x = 1.219 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 1790 reflections
a = 13.4112 (11) Å	θ = 3.2–22.2º
b = 5.1816 (4) Å	μ = 0.08 mm^{-1}
c = 19.4220 (17) Å	T = 290 (2) K
β = 105.007 (1)º	Block, colorless
V = 1303.63 (19) Å^3	0.20 × 0.20 × 0.10 mm
Z = 4	

Data collection

Bruker SMART CCD diffractometer	2418 independent reflections
Radiation source: fine-focus sealed tube	1721 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.034
T = 290(2) K	θ_{max} = 25.5º
φ and ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	h = −16→16
T_{min} = 0.985, T_{max} = 0.993	k = −5→6
8116 measured reflections	l = −21→23

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.056	H-atom parameters constrained
$wR(F^2)$ = 0.150	$w = 1/[\sigma^2(F_o^2) + (0.0644P)^2 + 0.2864P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.001
2418 reflections	$\Delta\rho_{max}$ = 0.15 e Å^{-3}

164 parameters	$\Delta\rho_{min} = -0.14$ 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}
C1	0.6350 (2)	0.7043 (6)	1.07179 (14)	0.0944 (9)
H1A	0.6472	0.8870	1.0741	0.142*
H1B	0.6759	0.6244	1.1144	0.142*
H1C	0.5633	0.6714	1.0676	0.142*
C2	0.66457 (17)	0.5940 (5)	1.00776 (13)	0.0629 (6)
C3	0.61450 (16)	0.6715 (5)	0.93952 (14)	0.0685 (7)
H3	0.5613	0.7914	0.9332	0.082*
C4	0.64120 (15)	0.5760 (5)	0.88051 (12)	0.0618 (6)
H4	0.6066	0.6323	0.8351	0.074*
C5	0.71923 (14)	0.3970 (4)	0.88896 (10)	0.0489 (5)
C6	0.77210 (14)	0.3136 (4)	0.95650 (10)	0.0467 (5)
C7	0.74327 (16)	0.4150 (4)	1.01466 (11)	0.0565 (6)
H7	0.7782	0.3605	1.0601	0.068*
C8	0.85903 (16)	0.1211 (4)	0.96335 (11)	0.0544 (5)
H8A	0.9140	0.1620	1.0053	0.065*
H8B	0.8339	−0.0506	0.9696	0.065*
C9	0.81595 (15)	0.1013 (4)	0.83811 (11)	0.0536 (6)
H9A	0.7788	−0.0572	0.8412	0.064*
H9B	0.8433	0.0885	0.7966	0.064*
C10	0.96489 (15)	0.3522 (4)	0.89932 (10)	0.0519 (5)
H10A	1.0192	0.3587	0.9434	0.062*
H10B	0.9230	0.5062	0.8970	0.062*
C11	1.01278 (14)	0.3519 (4)	0.83739 (10)	0.0480 (5)
C12	0.98730 (19)	0.5350 (4)	0.78431 (13)	0.0702 (7)
H12	0.9385	0.6603	0.7864	0.084*
C13	1.0325 (2)	0.5370 (5)	0.72802 (14)	0.0847 (8)
H13	1.0144	0.6634	0.6930	0.102*
C14	1.10317 (19)	0.3551 (5)	0.72380 (13)	0.0738 (7)
H14	1.1343	0.3572	0.6862	0.089*
C15	1.12840 (18)	0.1695 (6)	0.77481 (14)	0.0792 (8)

H15	1.1760	0.0425	0.7715	0.095*
C16	1.08396 (17)	0.1677 (5)	0.83165 (12)	0.0692 (7)
H16	1.1024	0.0404	0.8664	0.083*
N1	0.90020 (12)	0.1235 (3)	0.90035 (8)	0.0484 (4)
O1	0.74424 (10)	0.3150 (3)	0.82815 (7)	0.0597 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0824 (18)	0.121 (2)	0.094 (2)	−0.0074 (17)	0.0484 (16)	−0.0340 (18)
C2	0.0527 (13)	0.0740 (15)	0.0689 (15)	−0.0104 (11)	0.0278 (12)	−0.0150 (12)
C3	0.0454 (12)	0.0731 (16)	0.0891 (19)	0.0073 (11)	0.0212 (12)	−0.0068 (14)
C4	0.0438 (11)	0.0787 (16)	0.0608 (14)	0.0053 (11)	0.0100 (10)	0.0053 (12)
C5	0.0418 (10)	0.0559 (12)	0.0495 (12)	−0.0029 (9)	0.0127 (9)	−0.0003 (10)
C6	0.0455 (11)	0.0509 (12)	0.0454 (12)	−0.0044 (9)	0.0148 (9)	0.0027 (9)
C7	0.0551 (12)	0.0681 (14)	0.0490 (12)	−0.0105 (11)	0.0183 (10)	0.0000 (10)
C8	0.0574 (12)	0.0553 (13)	0.0507 (12)	0.0028 (10)	0.0142 (10)	0.0075 (10)
C9	0.0532 (12)	0.0547 (13)	0.0549 (13)	−0.0072 (10)	0.0174 (10)	−0.0106 (10)
C10	0.0486 (11)	0.0532 (13)	0.0533 (12)	−0.0022 (9)	0.0122 (9)	−0.0082 (10)
C11	0.0430 (11)	0.0462 (11)	0.0533 (12)	−0.0061 (9)	0.0098 (9)	−0.0050 (10)
C12	0.0850 (17)	0.0537 (14)	0.0780 (16)	0.0071 (12)	0.0323 (14)	0.0064 (12)
C13	0.114 (2)	0.0709 (18)	0.0777 (18)	−0.0083 (16)	0.0406 (17)	0.0111 (14)
C14	0.0683 (15)	0.093 (2)	0.0680 (16)	−0.0250 (14)	0.0311 (13)	−0.0078 (15)
C15	0.0533 (14)	0.106 (2)	0.0810 (18)	0.0149 (14)	0.0229 (13)	−0.0101 (16)
C16	0.0594 (14)	0.0826 (17)	0.0672 (15)	0.0217 (12)	0.0192 (12)	0.0082 (13)
N1	0.0499 (9)	0.0460 (10)	0.0502 (10)	0.0005 (7)	0.0146 (8)	−0.0014 (8)
O1	0.0562 (8)	0.0794 (11)	0.0420 (8)	0.0072 (8)	0.0102 (7)	−0.0010 (7)

Geometric parameters (Å, °)

C1—C2	1.512 (3)	C9—O1	1.447 (2)
C1—H1A	0.9600	C9—H9A	0.9700
C1—H1B	0.9600	C9—H9B	0.9700
C1—H1C	0.9600	C10—N1	1.472 (2)
C2—C3	1.381 (3)	C10—C11	1.503 (2)
C2—C7	1.385 (3)	C10—H10A	0.9700
C3—C4	1.378 (3)	C10—H10B	0.9700
C3—H3	0.9300	C11—C16	1.375 (3)
C4—C5	1.376 (3)	C11—C12	1.377 (3)
C4—H4	0.9300	C12—C13	1.381 (3)
C5—O1	1.376 (2)	C12—H12	0.9300
C5—C6	1.389 (3)	C13—C14	1.354 (4)
C6—C7	1.388 (3)	C13—H13	0.9300
C6—C8	1.514 (3)	C14—C15	1.359 (3)
C7—H7	0.9300	C14—H14	0.9300
C8—N1	1.467 (2)	C15—C16	1.384 (3)
C8—H8A	0.9700	C15—H15	0.9300
C8—H8B	0.9700	C16—H16	0.9300
C9—N1	1.430 (2)		

C2—C1—H1A	109.5	O1—C9—H9A	108.8
C2—C1—H1B	109.5	N1—C9—H9B	108.8
H1A—C1—H1B	109.5	O1—C9—H9B	108.8
C2—C1—H1C	109.5	H9A—C9—H9B	107.7
H1A—C1—H1C	109.5	N1—C10—C11	112.67 (15)
H1B—C1—H1C	109.5	N1—C10—H10A	109.1
C3—C2—C7	117.16 (19)	C11—C10—H10A	109.1
C3—C2—C1	120.9 (2)	N1—C10—H10B	109.1
C7—C2—C1	121.9 (2)	C11—C10—H10B	109.1
C4—C3—C2	121.8 (2)	H10A—C10—H10B	107.8
C4—C3—H3	119.1	C16—C11—C12	117.43 (19)
C2—C3—H3	119.1	C16—C11—C10	121.01 (19)
C5—C4—C3	119.8 (2)	C12—C11—C10	121.56 (18)
C5—C4—H4	120.1	C11—C12—C13	121.5 (2)
C3—C4—H4	120.1	C11—C12—H12	119.2
O1—C5—C4	116.91 (18)	C13—C12—H12	119.2
O1—C5—C6	122.45 (17)	C14—C13—C12	120.0 (2)
C4—C5—C6	120.59 (18)	C14—C13—H13	120.0
C7—C6—C5	117.98 (18)	C12—C13—H13	120.0
C7—C6—C8	123.24 (18)	C13—C14—C15	119.6 (2)
C5—C6—C8	118.77 (17)	C13—C14—H14	120.2
C2—C7—C6	122.7 (2)	C15—C14—H14	120.2
C2—C7—H7	118.6	C14—C15—C16	120.6 (2)
C6—C7—H7	118.6	C14—C15—H15	119.7
N1—C8—C6	111.64 (16)	C16—C15—H15	119.7
N1—C8—H8A	109.3	C11—C16—C15	120.8 (2)
C6—C8—H8A	109.3	C11—C16—H16	119.6
N1—C8—H8B	109.3	C15—C16—H16	119.6
C6—C8—H8B	109.3	C9—N1—C8	108.58 (15)
H8A—C8—H8B	108.0	C9—N1—C10	113.14 (16)
N1—C9—O1	113.82 (15)	C8—N1—C10	111.64 (15)
N1—C9—H9A	108.8	C5—O1—C9	115.02 (15)
C7—C2—C3—C4	0.0 (3)	C16—C11—C12—C13	−1.1 (3)
C1—C2—C3—C4	179.1 (2)	C10—C11—C12—C13	179.0 (2)
C2—C3—C4—C5	0.5 (3)	C11—C12—C13—C14	0.5 (4)
C3—C4—C5—O1	−178.12 (18)	C12—C13—C14—C15	0.7 (4)
C3—C4—C5—C6	−0.7 (3)	C13—C14—C15—C16	−1.2 (4)
O1—C5—C6—C7	177.70 (17)	C12—C11—C16—C15	0.5 (3)
C4—C5—C6—C7	0.4 (3)	C10—C11—C16—C15	−179.5 (2)
O1—C5—C6—C8	−1.3 (3)	C14—C15—C16—C11	0.6 (4)
C4—C5—C6—C8	−178.59 (18)	O1—C9—N1—C8	63.2 (2)
C3—C2—C7—C6	−0.2 (3)	O1—C9—N1—C10	−61.4 (2)
C1—C2—C7—C6	−179.4 (2)	C6—C8—N1—C9	−50.5 (2)
C5—C6—C7—C2	0.1 (3)	C6—C8—N1—C10	74.9 (2)
C8—C6—C7—C2	179.00 (19)	C11—C10—N1—C9	−60.3 (2)
C7—C6—C8—N1	−157.35 (18)	C11—C10—N1—C8	176.84 (15)
C5—C6—C8—N1	21.6 (3)	C4—C5—O1—C9	−171.58 (17)
N1—C10—C11—C16	−65.1 (2)	C6—C5—O1—C9	11.0 (3)

N1—C10—C11—C12	114.8 (2)	N1—C9—O1—C5	−43.0 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···Cg1^{i}	0.93	2.93	3.820 (3)	159
C8—H8B···Cg2^{ii}	0.97	2.98	3.907 (2)	159

Symmetry codes: (i) −*x*+3/2, *y*+1/2, −*z*+3/2; (ii) *x*, *y*−1, *z*.

Fig. 1

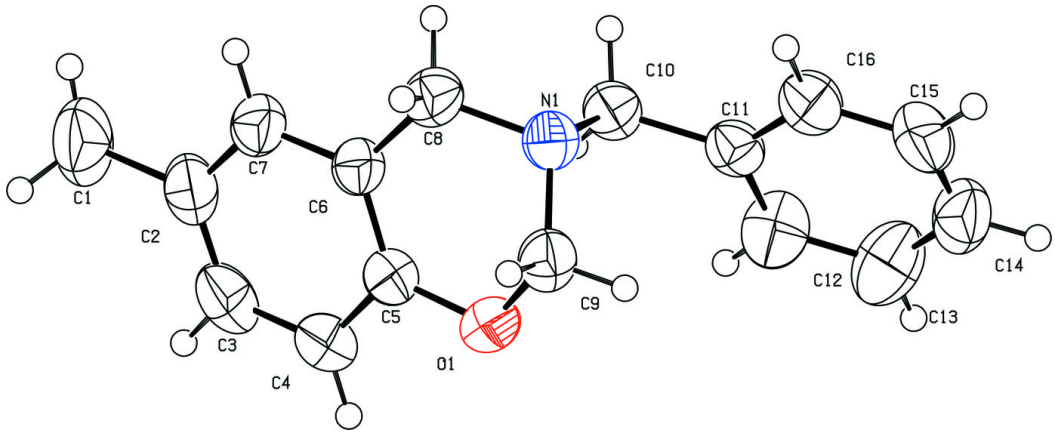

Fig. 2