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4-Benzyl-2H-1,4-benzoxazin-3(4H)-one

Yu-Liang Zhang[a]* and Xiao-Hui Li[b]

[a]Department of Chemistry, East China University of Science and Technology, Shanghai 200237, People's Republic of China, and [b]Department of Chemistry and Chemical Engineering, Liaoning Normal University, Dalian 116029, People's Republic of China

Correspondence e-mail:
yuliang_zhang@sohu.com

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.118
Data-to-parameter ratio = 14.3

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

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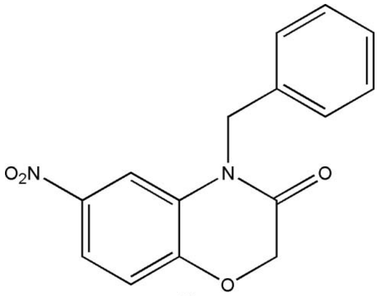
In the title compound, $C_{15}H_{12}N_2O_4$, the dihedral angle between the two benzene rings is 73.6 (2)°. In the crystal structure, molecules are linked through intermolecular C—H⋯O hydrogen-bonding interactions, forming dimers.

Comment

3-Oxo-3,4-dihydro-2H-1,4-benzoxazine and its derivatives are important building blocks for peptidomimic and tricyclic imidazoline drugs. Dihydro-1,4-benzoxazine can, to some extent, be considered a bioisostere of dihydro[1,4]-benzodioxine. Numerous benzoxazine derivatives possess various pharmacological properties (Gade, 2002; Linton & Hamilton, 1997; Yin *et al.*, 2004). These facts encouraged us to explore new synthetic methods and investigate the solid-state structures of dihydro-1,4-benzodioxine derivatives. Usually, benzoxazine derivatives are prepared using 2-aminophenol. In this paper, 4-nitrophenol was used as the starting material in a nucleophilic substitution reaction to obtain the title benzoxazine derivative, (I).



(I)

The molecular structure of compound (I) is shown in Fig. 1. The dihedral angle between the C3–C8 and C10–C15 rings is 73.6 (2)°, and the dihedral angle between the C3–C8 benzene ring and the O3/N2/O4 nitro plane is 8.5 (2)°. All the bond lengths and bond angles in (I) are in normal ranges (Allen *et al.*, 1987).

In the crystal structure, molecules are linked through intermolecular C—H⋯O hydrogen-bonding interactions (Table 1), forming dimers (Fig. 2).

Experimental

A suspension of 2-nitrophenol (2.78 g, 20.0 mmol), potassium carbonate (6.91 g, 50.0 mmol) and hydrated ytterbium(III) nitrate (0.47 g, 1.0 mmol) in N,N-dimethylformamide (40 ml) was heated at 383 K overnight. The reaction mixture was poured into ice-cooled water (100 ml) and the precipitated product was filtered off. The crude product was purified by column chromatography using petroleum ether/ethyl acetate(3:1 *v*/*v*) as eluent, giving a yellow compound. Single crystals of (I) were obtained by recrystallization from methanol. ^{1}H NMR (200 MHz, $CDCl_3$, TMS internal reference): δ 4.65 (*s*, 2H, $N-CH_2$), 4.95 (*s*, 2H, $-CH_2CO-$), 6.98–7.22 (*m*, 6H, Ar–H), 8.10–8.16 (*m*, 2H, *p*-$NO_2-C_6H_4$). Elemental analysis found: C 63.46, H 4.38, N 9.90%; calculated: C 63.38, H 4.25, N 9.85%.

Crystal data

$C_{15}H_{12}N_2O_4$
$M_r = 284.27$
Monoclinic, $P2_1/n$
$a = 7.933$ (2) Å
$b = 9.615$ (3) Å
$c = 17.514$ (3) Å
$\beta = 97.973$ (5)°
$V = 1323.0$ (6) Å^3
$Z = 4$
$D_x = 1.427$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.11$ mm^{-1}
$T = 298$ (2) K
Block, yellow
0.45 × 0.37 × 0.32 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.954$, $T_{\max} = 0.967$
7586 measured reflections
2728 independent reflections
1377 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.046$
$\theta_{\max} = 26.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.118$
$S = 1.02$
2728 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0334P)^2 + 0.2803P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0142 (16)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2*A*···O1^{i}	0.97	2.49	3.249 (2)	135
C9—H9*B*···O1	0.97	2.36	2.740 (2)	102

Symmetry code: (i) $-x+1, -y+1, -z$.

H atoms were positioned geometrically and refined as riding, with C—H = 0.93–0.97Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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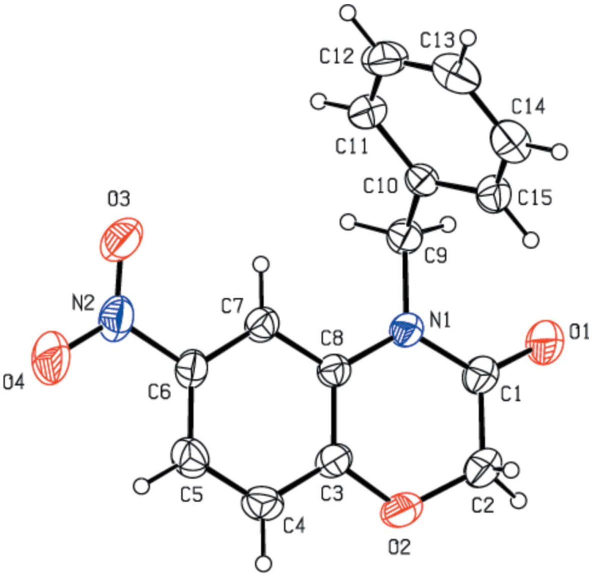

Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

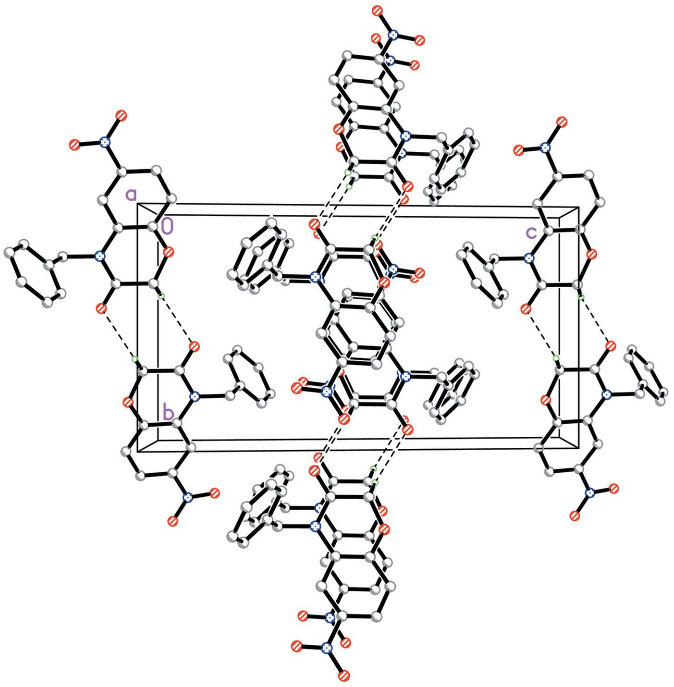

Figure 2
View of the molecular packing of (I) along the *a* axis. Intermolecular C—H···O hydrogen-bonding interactions forming dimers are shown as dashed lines.

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