

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

4-Benzyl-7-chloro-2*H*-1,4-benzoxazin-3(4*H*)-oneZhu-Bo Li,^{a*} Hao Yang,^b Yong-Sheng Xie^c and Chen-Guang Zhao^a

^aCollege of Pharmaceutical Sciences, Southwest University, Chongqing 400715, People's Republic of China, ^bCollege of Horticulture and Landscape Architecture, Southwest University, Chongqing 400715, People's Republic of China, and ^cSchool of Chemistry & Chemical Engineering, Shandong University, Jinan 250100, People's Republic of China

Correspondence e-mail: lizhubo2007@163.com

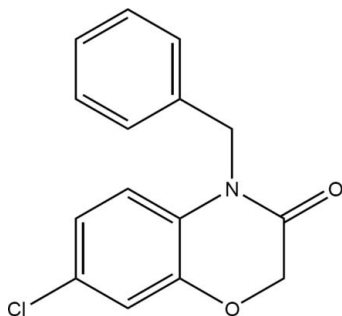
Received 13 March 2009; accepted 3 April 2009

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; disorder in main residue; R factor = 0.044; wR factor = 0.121; data-to-parameter ratio = 12.5.

In the title compound, $\text{C}_{15}\text{H}_{12}\text{ClNO}_2$, the two benzene rings are nearly perpendicular to each other [dihedral angle = $89.99(13)^\circ$]. The O atom of the six-membered heterocyclic ring is disordered over two sites in a ratio of 0.46 (4):0.54 (4) and is displaced from the mean plane formed by other five atoms, resulting an envelope conformation of the six-membered heterocycle ring.

Related literature

For the biological activity of benzo[*b*][1,4]oxazin-3(4*H*)-ones, see: Frechette & Weidner-Wells (1997); Maag *et al.* (2004). For the synthesis of benzo[*b*][1,4]oxazin-3(4*H*)-ones, see: Zuo *et al.* (2008). For a related structure, see: Cao *et al.* (2004).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{12}\text{ClNO}_2$	$V = 1289.4(5) \text{ \AA}^3$
$M_r = 273.71$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 5.0894(11) \text{ \AA}$	$\mu = 0.29 \text{ mm}^{-1}$
$b = 12.630(3) \text{ \AA}$	$T = 298 \text{ K}$
$c = 20.070(4) \text{ \AA}$	$0.16 \times 0.14 \times 0.10 \text{ mm}$
$\beta = 91.833(4)^\circ$	

Data collection

Bruker SMART CCD area-detector diffractometer	6606 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2005)	2268 independent reflections
$T_{\min} = 0.955$, $T_{\max} = 0.971$	1402 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$	182 parameters
$wR(F^2) = 0.121$	H-atom parameters constrained
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$
2268 reflections	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$

Data collection: SMART (Bruker 2005); cell refinement: SAINT (Bruker 2005); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: XP in SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXL97.

This study was supported by the Key Program Projects of the Municipal Natural Science Foundation of Chongqing, China (grant No. CSTC,2007AC1042).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2496).

References

- Bruker (2005). SMART, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Cao, D.-Y., Chen, L.-G., Song, J., Feng, R.-X. & Lou, K.-X. (2004). *Acta Cryst.* **E60**, o1788–o1789.
- Frechette, R. & Weidner-Wells, M. A. (1997). WO Patent No. 9 717 333.
- Maag, H., Sui, M. & Zhao, S. H. (2004). WO Patent No. 20 04 041 792.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Zuo, H., Meng, L. J., Ghate, M., Hwang, K., Cho, Y. K., Chandrasekhar, S., Raji Reddy, Ch. & Shin, D. S. (2008). *Tetrahedron Lett.* **49**, 3827–3830.

supplementary materials

Acta Cryst. (2009). E65, o1019 [doi:10.1107/S1600536809012586]

4-Benzyl-7-chloro-2*H*-1,4-benzoxazin-3(4*H*)-one

Z.-B. Li, H. Yang, Y.-S. Xie and C.-G. Zhao

Comment

Benzo[*b*][1,4]oxazin-3(4*H*)-ones are an important class of heteroaromatic ring systems that have shown to exhibit a wide range of biological activities such as antimicrobial (Frechette & Weidner-Wells, 1997) and selective 5-HT₆ antagonistic activities (Maag *et al.*, 2004). Due to its extensive biological application, the efficient synthesis of benzo[*b*][1,4]oxazin-3(4*H*)-ones has recently received much attention (Zuo *et al.*, 2008). We report here the crystal structure of the title compound (Fig. 1). All bond lengths and angles are normal. The dihedral angle between the two benzene rings is 89.99 (13)°. The crystal packing is mainly stabilized by van der Waals interactions. In the title compound the six-membered heterocyclic ring is envelope conformation while in the related compound (Cao *et al.*, 2004) it adopts a screw-boat conformation.

Experimental

To the solution of *N*-benzyl-2-(2,4-dichlorophenoxy)acetamide (0.620 g, 2.0 mmol) in DMF (20 ml), caesium carbonate (0.787 g, 2.4 mmol) was added. The mixture was refluxed for 2 h. After completion of the reaction (by TLC monitoring), the DMF was removed under vacuum. Water (20 ml) was added into the residue to obtain a turbid solution and it was extracted by ethyl acetate (20 ml \times 4). The combined organic layer was washed by 1 mol/L of hydrochloric acid (10 ml \times 3) and saturated sodium chloride solution (10 ml \times 3), dried over MgSO₄. And then the mixture was filtered and the filtrate was concentrated under reduced pressure to obtain the corresponding crude product. The product was purified by column chromatography on silica gel using ethyl acetate/hexane = 1/5 as eluent (yield 70%). Crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution of the solid dissolved in ethyl acetate/hexane at room temperature for 10 days.

Refinement

H atoms were positioned geometrically with C—H = 0.93–0.97 Å and refined in the riding-model approximation with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The O2 atom is disordered over two sites with a ratio of 0.46 (4):0.54 (4).

Figures

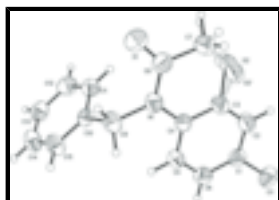


Fig. 1. The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

4-Benzyl-7-chloro-2H-1,4-benzoxazin-3(4H)-one

Crystal data

$C_{15}H_{12}ClNO_2$	$F_{000} = 568$
$M_r = 273.71$	$D_x = 1.410 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
Hall symbol: -P 2ybc	$\lambda = 0.71073 \text{ \AA}$
$a = 5.0894 (11) \text{ \AA}$	Cell parameters from 1156 reflections
$b = 12.630 (3) \text{ \AA}$	$\theta = 2.6\text{--}20.8^\circ$
$c = 20.070 (4) \text{ \AA}$	$\mu = 0.29 \text{ mm}^{-1}$
$\beta = 91.833 (4)^\circ$	$T = 298 \text{ K}$
$V = 1289.4 (5) \text{ \AA}^3$	Block, colorless
$Z = 4$	$0.16 \times 0.14 \times 0.10 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer	2268 independent reflections
Radiation source: fine-focus sealed tube	1402 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.031$
$T = 298 \text{ K}$	$\theta_{\text{max}} = 25.1^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.9^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$h = -6 \rightarrow 6$
$T_{\text{min}} = 0.955$, $T_{\text{max}} = 0.971$	$k = -15 \rightarrow 10$
6606 measured reflections	$l = -23 \rightarrow 22$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H-atom parameters constrained
$wR(F^2) = 0.121$	$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 0.1942P]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
2268 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
182 parameters	$\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$
	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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C11	1.04449 (14)	0.70775 (6)	0.03801 (4)	0.0845 (3)	
O1	-0.0247 (4)	0.38796 (16)	0.23334 (10)	0.0903 (6)	
O2	0.459 (4)	0.4090 (11)	0.1102 (14)	0.079 (4)	0.46 (4)
O2'	0.358 (5)	0.4386 (16)	0.0928 (5)	0.085 (4)	0.54 (4)
N1	0.2417 (4)	0.52888 (17)	0.21372 (9)	0.0620 (5)	
C1	0.8090 (5)	0.6568 (2)	0.09015 (12)	0.0620 (7)	
C2	0.6971 (5)	0.5605 (2)	0.07550 (13)	0.0723 (7)	
H2	0.7454	0.5233	0.0378	0.087*	
C3	0.5134 (5)	0.5196 (2)	0.11685 (13)	0.0679 (7)	
C4	0.4348 (5)	0.57338 (19)	0.17315 (11)	0.0567 (6)	
C5	0.5528 (6)	0.6694 (2)	0.18658 (13)	0.0715 (8)	
H5	0.5058	0.7070	0.2242	0.086*	
C6	0.7390 (6)	0.7116 (2)	0.14561 (14)	0.0765 (8)	
H6	0.8161	0.7767	0.1556	0.092*	
C7	0.2299 (8)	0.3790 (2)	0.13820 (16)	0.0967 (10)	
H7A	0.2446	0.3040	0.1481	0.116*	0.46 (4)
H7B	0.0913	0.3865	0.1042	0.116*	0.46 (4)
H7'A	0.3253	0.3162	0.1528	0.116*	0.54 (4)
H7'B	0.0757	0.3544	0.1131	0.116*	0.54 (4)
C8	0.1376 (6)	0.4322 (2)	0.19949 (14)	0.0713 (7)	
C9	0.1563 (5)	0.5849 (2)	0.27351 (12)	0.0673 (7)	
H9A	-0.0118	0.5564	0.2862	0.081*	
H9B	0.1306	0.6591	0.2626	0.081*	
C10	0.3484 (4)	0.57652 (19)	0.33223 (11)	0.0534 (6)	
C11	0.5208 (5)	0.4939 (2)	0.34046 (13)	0.0684 (7)	
H11	0.5248	0.4407	0.3085	0.082*	
C12	0.6897 (6)	0.4889 (2)	0.39610 (16)	0.0849 (9)	
H12	0.8071	0.4328	0.4010	0.102*	
C13	0.6842 (6)	0.5657 (3)	0.44339 (14)	0.0797 (8)	
H13	0.7962	0.5616	0.4808	0.096*	
C14	0.5167 (6)	0.6478 (3)	0.43615 (14)	0.0819 (9)	
H14	0.5142	0.7007	0.4683	0.098*	
C15	0.3484 (5)	0.6531 (2)	0.38073 (14)	0.0748 (8)	

supplementary materials

H15 0.2327 0.7098 0.3763 0.090*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0817 (5)	0.0935 (6)	0.0780 (5)	-0.0153 (4)	0.0008 (4)	0.0149 (4)
O1	0.0999 (15)	0.0831 (13)	0.0889 (14)	-0.0188 (12)	0.0197 (12)	0.0042 (11)
O2	0.081 (6)	0.055 (5)	0.103 (8)	-0.012 (4)	0.024 (6)	-0.024 (5)
O2'	0.111 (9)	0.085 (6)	0.062 (3)	-0.030 (6)	0.014 (4)	-0.019 (3)
N1	0.0640 (13)	0.0662 (13)	0.0556 (12)	-0.0020 (11)	-0.0015 (10)	-0.0049 (10)
C1	0.0630 (16)	0.0635 (16)	0.0585 (15)	-0.0031 (13)	-0.0109 (13)	0.0075 (13)
C2	0.0794 (18)	0.0759 (19)	0.0618 (16)	-0.0103 (15)	0.0068 (14)	-0.0117 (14)
C3	0.0764 (18)	0.0629 (16)	0.0642 (16)	-0.0124 (14)	-0.0004 (14)	-0.0120 (14)
C4	0.0628 (15)	0.0569 (15)	0.0497 (14)	-0.0002 (13)	-0.0074 (12)	0.0003 (12)
C5	0.098 (2)	0.0594 (16)	0.0570 (15)	-0.0021 (15)	0.0018 (15)	-0.0067 (13)
C6	0.099 (2)	0.0597 (16)	0.0709 (18)	-0.0122 (16)	-0.0035 (16)	0.0018 (15)
C7	0.135 (3)	0.0709 (19)	0.085 (2)	-0.024 (2)	0.028 (2)	-0.0136 (18)
C8	0.0770 (19)	0.0689 (18)	0.0678 (17)	-0.0049 (16)	-0.0007 (15)	0.0044 (15)
C9	0.0586 (15)	0.0778 (18)	0.0653 (16)	0.0050 (13)	0.0013 (13)	-0.0107 (14)
C10	0.0501 (14)	0.0603 (15)	0.0501 (13)	0.0006 (12)	0.0070 (11)	0.0018 (12)
C11	0.0787 (18)	0.0624 (16)	0.0640 (17)	0.0060 (14)	0.0017 (14)	0.0003 (13)
C12	0.094 (2)	0.075 (2)	0.085 (2)	0.0196 (16)	-0.0067 (18)	0.0158 (17)
C13	0.086 (2)	0.094 (2)	0.0594 (17)	-0.0009 (18)	-0.0062 (14)	0.0101 (17)
C14	0.083 (2)	0.097 (2)	0.0655 (18)	-0.0023 (18)	-0.0002 (16)	-0.0212 (17)
C15	0.0673 (17)	0.0811 (19)	0.0758 (18)	0.0175 (14)	0.0001 (15)	-0.0147 (16)

Geometric parameters (\AA , $^\circ$)

C11—C1	1.739 (3)	C7—H7A	0.9700
O1—C8	1.222 (3)	C7—H7B	0.9700
O2—C7	1.363 (11)	C7—H7'A	0.9700
O2—C3	1.429 (11)	C7—H7'B	0.9700
O2'—C7	1.363 (9)	C9—C10	1.511 (3)
O2'—C3	1.372 (9)	C9—H9A	0.9700
N1—C8	1.357 (3)	C9—H9B	0.9700
N1—C4	1.413 (3)	C10—C11	1.370 (3)
N1—C9	1.471 (3)	C10—C15	1.372 (3)
C1—C6	1.368 (4)	C11—C12	1.389 (4)
C1—C2	1.370 (3)	C11—H11	0.9300
C2—C3	1.371 (3)	C12—C13	1.357 (4)
C2—H2	0.9300	C12—H12	0.9300
C3—C4	1.388 (3)	C13—C14	1.348 (4)
C4—C5	1.376 (3)	C13—H13	0.9300
C5—C6	1.381 (4)	C14—C15	1.384 (4)
C5—H5	0.9300	C14—H14	0.9300
C6—H6	0.9300	C15—H15	0.9300
C7—C8	1.491 (4)		
C7—O2—C3	113.6 (7)	O2'—C7—H7'A	114.1

C7—O2'—C3	117.4 (6)	C8—C7—H7'A	106.6
C8—N1—C4	120.7 (2)	H7B—C7—H7'A	129.7
C8—N1—C9	118.7 (2)	O2—C7—H7'B	124.2
C4—N1—C9	120.5 (2)	O2'—C7—H7'B	103.0
C6—C1—C2	120.4 (3)	C8—C7—H7'B	107.6
C6—C1—C11	120.3 (2)	H7A—C7—H7'B	81.4
C2—C1—C11	119.3 (2)	H7'A—C7—H7'B	106.6
C1—C2—C3	119.4 (2)	O1—C8—N1	124.1 (3)
C1—C2—H2	120.3	O1—C8—C7	119.3 (3)
C3—C2—H2	120.3	N1—C8—C7	116.6 (3)
C2—C3—O2'	117.7 (5)	N1—C9—C10	113.70 (19)
C2—C3—C4	121.9 (2)	N1—C9—H9A	108.8
O2'—C3—C4	118.2 (8)	C10—C9—H9A	108.8
C2—C3—O2	116.5 (5)	N1—C9—H9B	108.8
C4—C3—O2	119.5 (8)	C10—C9—H9B	108.8
C5—C4—C3	117.1 (2)	H9A—C9—H9B	107.7
C5—C4—N1	123.0 (2)	C11—C10—C15	117.8 (2)
C3—C4—N1	119.9 (2)	C11—C10—C9	122.9 (2)
C4—C5—C6	121.8 (3)	C15—C10—C9	119.3 (2)
C4—C5—H5	119.1	C10—C11—C12	120.6 (3)
C6—C5—H5	119.1	C10—C11—H11	119.7
C1—C6—C5	119.4 (3)	C12—C11—H11	119.7
C1—C6—H6	120.3	C13—C12—C11	120.2 (3)
C5—C6—H6	120.3	C13—C12—H12	119.9
O2—C7—C8	120.6 (7)	C11—C12—H12	119.9
O2'—C7—C8	118.1 (8)	C14—C13—C12	120.1 (3)
O2—C7—H7A	107.2	C14—C13—H13	120.0
O2'—C7—H7A	129.9	C12—C13—H13	120.0
C8—C7—H7A	107.2	C13—C14—C15	119.8 (3)
O2—C7—H7B	107.2	C13—C14—H14	120.1
O2'—C7—H7B	80.1	C15—C14—H14	120.1
C8—C7—H7B	107.2	C10—C15—C14	121.5 (3)
H7A—C7—H7B	106.8	C10—C15—H15	119.3
O2—C7—H7'A	85.8	C14—C15—H15	119.3
C6—C1—C2—C3	0.2 (4)	C4—C5—C6—C1	0.0 (4)
C11—C1—C2—C3	179.3 (2)	C3—O2—C7—O2'	60.5 (13)
C1—C2—C3—O2'	163.7 (15)	C3—O2—C7—C8	-34 (3)
C1—C2—C3—C4	0.6 (4)	C3—O2'—C7—O2	-69.5 (14)
C1—C2—C3—O2	-163.1 (15)	C3—O2'—C7—C8	34 (3)
C7—O2'—C3—C2	162.2 (16)	C4—N1—C8—O1	178.0 (2)
C7—O2'—C3—C4	-34 (3)	C9—N1—C8—O1	0.1 (4)
C7—O2'—C3—O2	66.7 (14)	C4—N1—C8—C7	-2.3 (4)
C7—O2—C3—C2	-162.9 (15)	C9—N1—C8—C7	179.8 (2)
C7—O2—C3—O2'	-62.9 (13)	O2—C7—C8—O1	-160.9 (16)
C7—O2—C3—C4	33 (3)	O2'—C7—C8—O1	164.1 (14)
C2—C3—C4—C5	-1.1 (4)	O2—C7—C8—N1	19.4 (17)
O2'—C3—C4—C5	-164.0 (13)	O2'—C7—C8—N1	-15.6 (15)
O2—C3—C4—C5	162.1 (14)	C8—N1—C9—C10	100.5 (3)
C2—C3—C4—N1	179.0 (2)	C4—N1—C9—C10	-77.4 (3)

supplementary materials

O2'—C3—C4—N1	16.0 (14)	N1—C9—C10—C11	-25.9 (3)
O2—C3—C4—N1	-17.9 (14)	N1—C9—C10—C15	155.7 (2)
C8—N1—C4—C5	-177.8 (2)	C15—C10—C11—C12	-0.3 (4)
C9—N1—C4—C5	0.1 (3)	C9—C10—C11—C12	-178.7 (3)
C8—N1—C4—C3	2.1 (3)	C10—C11—C12—C13	0.6 (4)
C9—N1—C4—C3	-180.0 (2)	C11—C12—C13—C14	-0.8 (5)
C3—C4—C5—C6	0.8 (4)	C12—C13—C14—C15	0.7 (5)
N1—C4—C5—C6	-179.3 (2)	C11—C10—C15—C14	0.2 (4)
C2—C1—C6—C5	-0.5 (4)	C9—C10—C15—C14	178.6 (2)
C11—C1—C6—C5	-179.6 (2)	C13—C14—C15—C10	-0.4 (4)

Fig. 1

