Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 7-(Benzyloxy)spiro[2*H*-1,3-benzoxazine-2,1′-cyclohexan]-4(3*H*)-one

**Shan-Zhong Jian and Ming Lei***

Department of Chemistry, Zhejiang University, Hangzhou 310027, People's Republic of China

Correspondence e-mail: minglei701@yahoo.com.cn

**Key indicators**

Single-crystal X-ray study
*T* = 295 K
Mean σ(C–C) = 0.002 Å
*R* factor = 0.039
*wR* factor = 0.086
Data-to-parameter ratio = 17.2

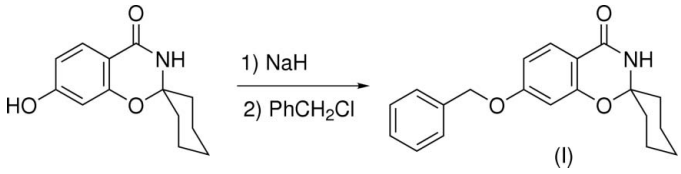
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{20}H_{21}NO_3$, the dihedral angle between the two benzene rings is 8.79 (7)° and the cyclohexane ring adopts a chair conformation. The molecules are linked by paired N—H···O hydrogen bonds into centrosymmetric $R_2^2(8)$ dimers.

Received 5 September 2005
Accepted 9 September 2005
Online 14 September 2005

## Comment

The molecular structure of (I) is shown in Fig. 1. The dihedral angle between the benzene ring of the 2*H*-benzoxazine system and the phenyl ring is 8.79 (7)°. The bulky, six-membered cyclohexane chair ring in the compound could be used in asymmetric induction, as we have reported previously (Jian *et al.*, 2005).




(I)

In the crystal structure, atom N1 acts as a hydrogen donor to atom O1 of a symmetry-related molecule (Table 1), leading to the formation of centrosymmetric $R_2^2(8)$ dimers (Fig. 2).

## Experimental

To a mixture of 7-hydroxy-[2*H*-1,3-benzoxazine-2,1′-cyclohexan]-4(3*H*)-one (2.0 g, 8.6 mmol) and NaH (0.40 g, 11.7 mmol, 70%) in dimethylformamide (30 ml), benzyl chloride (0.9 ml, 8 mmol) was added dropwise. This mixture was stirred at room temperature for 30 min and then at 333 K overnight. $CH_2Cl_2$ (30 ml) and water

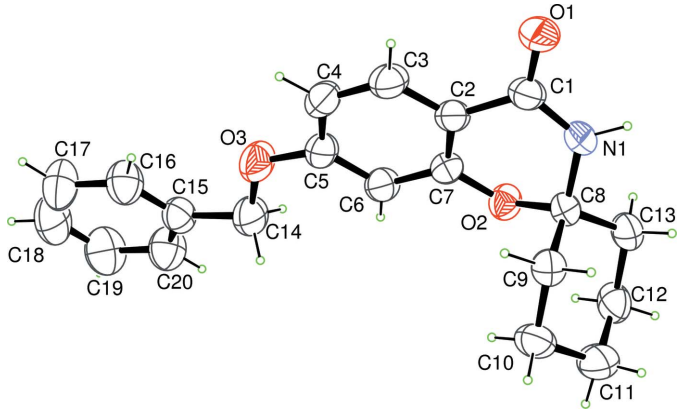



**Figure 1**
The molecule of compound (I) in the crystal. Displacement ellipsoids are drawn at the 50% probability level.

(10 ml) were then added to the reaction mixture. The organic layer was washed successively with water (3 × 10 ml), dried over anhydrous $Na_2SO_4$ and evaporated *in vacuo*. Recrystallization of the resulting white solid from MeOH gave colourless crystals of (I) (m.p. 470–471 K). Spectroscopic analysis: $^1$H NMR (500 MHz, $CDCl_3$, $\delta$, p.p.m.): 1.39–2.11 (*m*, 10H), 5.08 (*s*, 2H), 6.52 (*s*, 1H), 6.66 (*brs*, 1H), 6.67 (*dd*, 1H), 7.35–7.44 (*m*, 5H), 7.83 (*d*, 1H).

*Crystal data*

$C_{20}H_{21}NO_3$
$M_r$ = 323.38
Monoclinic, $P2_1/c$
$a$ = 6.0904 (2) Å
$b$ = 8.9171 (3) Å
$c$ = 30.8699 (8) Å
$\beta$ = 93.1470 (10)°
$V$ = 1673.98 (9) Å$^3$
$Z$ = 4
$D_x$ = 1.283 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 6953 reflections
$\theta$ = 2.6–27.5°
$\mu$ = 0.09 mm$^{-1}$
$T$ = 295 (2) K
Prism, colourless
0.26 × 0.25 × 0.2 mm

*Data collection*

Rigaku R-AXIS RAPID diffractometer
$\omega$ scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min}$ = 0.976, $T_{max}$ = 0.983
7297 measured reflections
3819 independent reflections
2271 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.026
$\theta_{max}$ = 27.5°
$h = -7 \rightarrow 7$
$k = -11 \rightarrow 11$
$l = -39 \rightarrow 40$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.086
$S$ = 1
3819 reflections
222 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.037P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.13 e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.0164 (11)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O1$^{i}$ | 0.87 (1) | 1.98 (1) | 2.8541 (15) | 177 (1) |

Symmetry code: (i) $-x+2, -y+1, -z+1$.

Atom H1 was found in a difference Fourier map and refined freely. The H atoms of the methylene groups and of the aromatic ring were placed in calculated positions, with C—H distances of 0.97 and 0.93 Å, respectively, and were included in the final cycles of the least-squares refinement as riding on their carrier atoms, with $U_{iso}$(H) = 1.2$U_{eq}$ of the corresponding carrier atom.

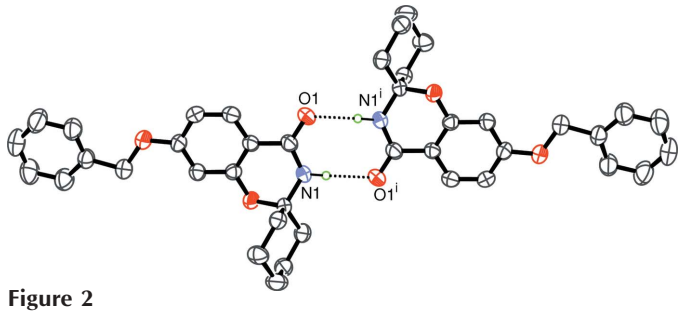



**Figure 2**
A view of the dimer formed by paired N—H···O hydrogen bonds (dashed lines) in the crystal structure of (I). H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry code: (i) $2-x$, $1-y$, $1-z$.]

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2003).

We thank the National Natural Science Foundation of China (grant No. 20272051) and the Teaching and Research Award Programme for Outstanding Young Teachers in Higher Education Institutions of the MoE, China.

## References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Jian, S. Z., Ma, C. & Wang, Y. G. (2005). *Synthesis*, pp. 725–730.
Rigaku (1998). *PROCESS-AUTO. Version*, **1**, 06. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2004). *CrystalStructure. Version*, **3**, 6.0. Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381–5209, USA.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.