Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-(2-Bromobutanoyl)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

Received 5 September 2005
Accepted 6 September 2005
Online 14 September 2005

In the title compound, $C_{17}H_{20}BrNO_3$, synthesized from spiro[2*H*-1,3-benzoxazine-2,1′-cyclohexan]-4(3*H*)-one and 2-bromobutanoyl bromide, the chair cyclohexane ring in the molecule shows high asymmetric induction in the synthesis of *trans* β-lactams.

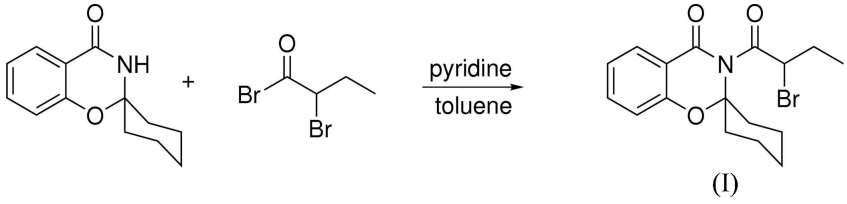
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.036
wR factor = 0.096
Data-to-parameter ratio = 17.3

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

Comment

As we previously reported, the title compound (I) can be used to synthesize *trans* β-lactams with high diastereoselectivity (Jian *et al.*, 2005). The bulky chair cyclohexane ring in the compound plays an important role in efficient asymmetric induction, which leads to *trans* β-lactams exclusively.



(I)

Fig. 1 shows the structure of (I). The compound crystallizes in the monoclinic space group *C*2/*c* with one molecule in the asymmetric unit. Selected molecular parameters are listed in Table 1; these may be considered normal (Table 1). There are no π–π stacking or other weak intermolecular interactions in (I), and the crystal packing (Fig. 2) is controlled by van der Waals forces.

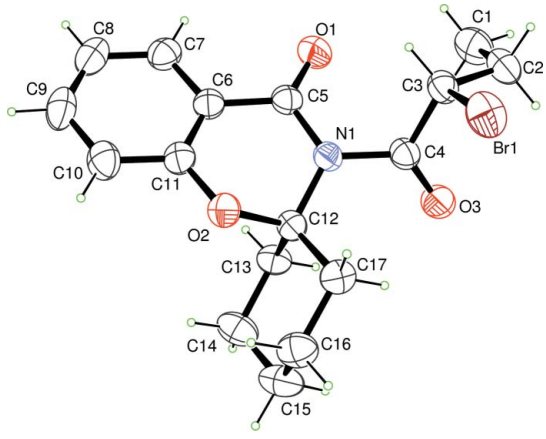


Figure 1
The molecule of (I). Displacement ellipsoids are drawn at the 50% probability level for non-H atoms.

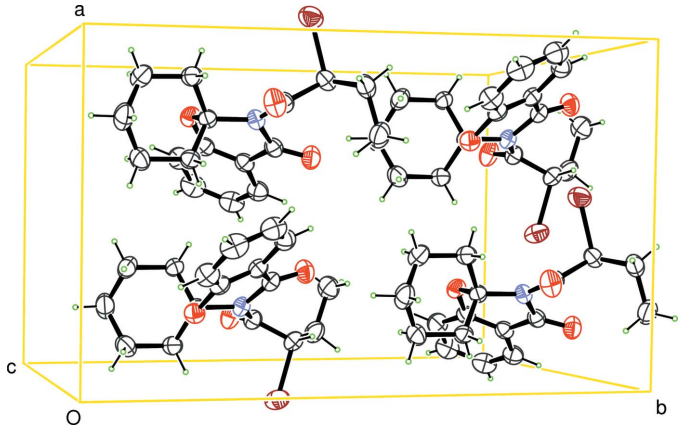


Figure 2
A packing diagram, viewed approximately along the *c* axis.

Experimental

To a mixture of spiro[2*H*-1,3-benzoxazine-2,1′-cyclohexan]-4(3*H*)-one (217 mg, 1 mmol), pyridine (95 mg, 1.2 mmol) and toluene (10 ml) was added 2-bromobutanoyl bromide (276 mg, 1.2 mmol) dropwise at 278–288 K. This mixture was stirred at the same temperature for 30 min and then at 298 K for 20 h. The reaction mixture was poured into water (10 ml). The organic layer was washed successively with saturated aqueous $NaHCO_3$ (5 ml) and brine (5 ml), dried over anhydrous Na_2SO_4, and evaporated *in vacuo*. The residue was dissolved in 2-propanol (3 ml) at 323–325 K, gradually cooled to 283 K and stirred at the same temperature for 1 h. The resulting crystals were collected, washed with 2-propanol (3 ml) and dried at 313 K for 20 h to afford 300 mg (82% yield) of (I). Colourless crystals were obtained from a CH_2Cl_2/EtOH (1:10 *v*/*v*) solution after leaving it to stand for 4 d (m.p. 342–344 K). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.13 (*t*, *J* = 7.3 Hz), 1.29 (*m*, 1H), 1.53–2.41 (*m*, 10H), 4.98 (*dd*, 1H, *J* = 5.2 and 8.8 Hz), 7.01 (*m*, 1H), 7.11 (*m*, 1H), 7.55 (*m*, 1H), 7.93 (*m*, 1H). ESI–MS: *m*/*z* 366 ($[M+1]^+$).

Crystal data

$C_{17}H_{20}BrNO_3$
M_r = 366.25
Monoclinic, *C*2/*c*
a = 10.9022 (7) Å
b = 17.636 (1) Å
c = 16.8688 (8) Å
β = 92.795 (2)°
V = 3239.5 (3) Å^3
Z = 8
D_x = 1.502 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 10907 reflections
θ = 2.2–27.3°
μ = 2.55 mm^{-1}
T = 295 (1) K
Block, colourless
0.35 × 0.3 × 0.2 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.444, T_{max} = 0.600
14339 measured reflections
3481 independent reflections
2919 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 27°
h = −12 → 13
k = −22 → 22
l = −21 → 21

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.096
S = 1.07
3481 reflections
201 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 2.5231P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0024 (3)

Table 1
Selected geometric parameters (Å, °).

Br1—C3	1.981 (2)	N1—C5	1.446 (3)
O1—C5	1.208 (3)	N1—C4	1.462 (3)
O2—C11	1.421 (3)	N1—C12	1.494 (3)
O3—C4	1.191 (3)		
C11—O2—C12	118.96 (16)	C4—C3—Br1	102.64 (16)
C5—N1—C4	119.45 (17)	C12—C13—C14	113.07 (19)
C5—N1—C12	118.42 (16)	C13—C14—C15	109.8 (2)
C4—N1—C12	119.75 (17)	C15—C16—C17	112.1 (2)
C2—C3—Br1	106.02 (16)	C16—C17—C12	109.52 (19)

The methyl groups were constrained to an ideal geometry [C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C)] and were allowed to rotate freely about the C—C bonds. The other H atoms were placed in calculated positions, with U_{iso}(H) = 1.2U_{eq}(C) and C—H = 0.93–0.96 Å, and included in the final cycles of refinement in the riding-model approximation.

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.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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, People's Republic of China.

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., 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.