Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(+)-*N*-[(3*S*)-3-(2,4-Dichlorophenyl-4-methyl)-pentanoyl]bornane-10,2-sultam

Xiu-Fang Cao, Guang-Ao Yu,* Wen-Chang Lu, Xiang-Gao Meng and Sheng-Hua Liu

Key Laboratory of Pesticides & Chemical Biology, Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, People's Republic of China

Correspondence e-mail: yuguang@mail.ccnu.edu.cn

Received 23 May 2006
Accepted 21 June 2006

Key indicators

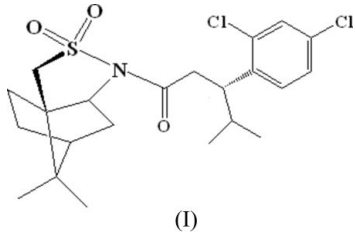
Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.005 Å
R factor = 0.042
wR factor = 0.126
Data-to-parameter ratio = 15.9

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

In the crystal structure of the title compound, $C_{22}H_{29}Cl_2NO_3S$, molecules are linked *via* C—H···O interactions, forming chains along the *a* axis.

Comment

The readily available enantiomers of bornane-10,2-sultam serve as efficient, versatile and practical chiral auxiliaries (Oppolzer, 1990), and we have focused our attention on this area. In this paper, we present the X-ray crystallographic analysis of the title compound, (I).



(I)

In (I), the six-membered ring of the sultam has a boat form (Fig. 1 and Table 21). The C4/C5/C6/C7 and C4/C9/C8/C7 planes form a dihedral angle of 110.1 (3)°. The C4/C3/C7 plane forms almost equal dihedral angles of 124.2 (3) and 125.6 (3)°, respectively, with the C4/C5/C6/C7 and C4/C9/C8/C7 planes. The molecules are linked *via* C—H···O interactions (Table 2), forming chains along the *a* axis.

Experimental

N-[3-(2,4-Dichlorophenyl)propenoyl]bornane-(+)-10,2-sultam (3.314 g, 8.0 mmol) was reacted with *i*-PrMgCl (16.0 mmol) in dry thf (100 ml) at 195 K to obtain compound (I) (yield 2.338 g, 64%). $[\alpha]_D^{23}$ = +69.70° (*c* = 1.03, $CHCl_3$). Crystals appropriate for X-ray data collection were grown by slow evaporation of a dichloromethane solution at 292 K.

Crystal data

$C_{22}H_{29}Cl_2NO_3S$
M_r = 458.42
Monoclinic, $P2_1$
a = 7.8570 (9) Å
b = 16.6100 (19) Å
c = 8.9443 (10) Å
β = 97.134 (2)°
V = 1158.2 (2) Å^3
Z = 2
D_x = 1.314 Mg m^{-3}
Mo *Kα* radiation
μ = 0.39 mm^{-1}
T = 292 (2) K
Block, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
5917 measured reflections
4227 independent reflections
4024 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 26.0°

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.081P)^2 + 0.0828P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.042$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.126$	$\Delta\rho_{max} = 0.36$ e Å^{-3}
$S = 1.11$	$\Delta\rho_{min} = -0.25$ e Å^{-3}
4227 reflections	Absolute structure: Flack (1983), 1860 Friedel pairs
266 parameters	Flack parameter: −0.09 (6)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C9—N1	1.472 (4)	C20—Cl2	1.743 (3)
C10—S1	1.783 (3)	N1—S1	1.699 (2)
C11—O3	1.207 (4)	O1—S1	1.418 (2)
C11—N1	1.389 (4)	O2—S1	1.431 (3)
C18—Cl1	1.744 (3)		
C4—C10—S1	107.7 (2)	N1—S1—C10	95.79 (13)
C4—C5—C6—C7	4.6 (4)	C12—C13—C17—C22	−26.9 (4)
C7—C8—C9—C4	7.9 (3)	C4—C9—N1—S1	−29.1 (3)
C10—C4—C9—N1	32.1 (4)	C9—N1—S1—C10	14.7 (2)
C9—C4—C10—S1	−21.7 (4)	C4—C10—S1—N1	4.8 (3)
C11—C12—C13—C14	172.7 (2)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10A···O3^{i}	0.97	2.45	3.366 (4)	158

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

All H atoms were constrained to an ideal geometry, with C—H = 0.93–0.98 Å and U_{iso}(H)= 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The absolute configuration of (I), based on the Flack (1983) parameter, is consistent with the known absolute configuration of (+)-2,10-sultam (Boiadjiev *et al.*, 2001)

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

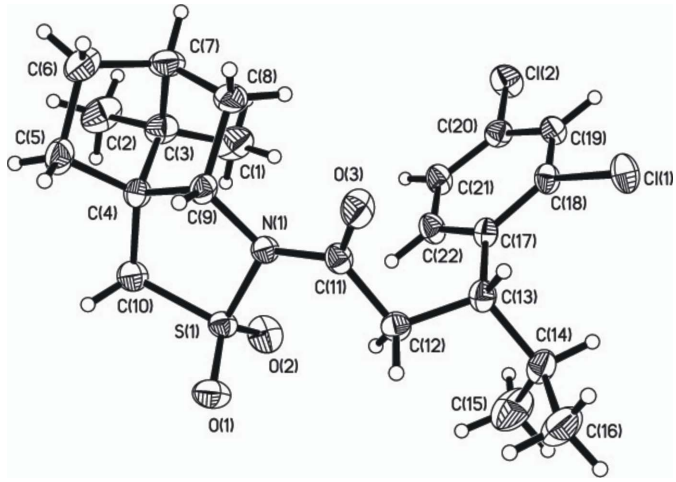


Figure 1
The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

The authors acknowledge financial support from the Scientific Research Foundation for Returned Overseas Chinese Scholars, State Education Ministry, the Hubei Province Science Fund for Distinguished Young Scholars (No. 2003ABB006) and the Natural Science Foundation of Hubei Province (No. 2005ABA038).

References

Boiadjiev, S. E. & Lightner, D. A. (2001). *Tetrahedron Asymmetry*, **12**, 2551–2564.
Bruker (2001). *SAINT-Plus* (Version 6.45), *SMART* (Version 5.628) and *SHELXTL* (Version 6.14). Bruker AXS, Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Oppolzer, W. (1990). *Pure Appl. Chem.* **62**, 1241–1250.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.