Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-(*p*-Bromophenyl)-5-*p*-tosylperhydropyrrolo-[3,4-*b*]pyrrole

S. Selvanayagam,[a] D. Velmurugan,[a]* K. Ravikumar,[b] M. Poornachandran[c] and R. Raghunathan[c]

[a]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, [b]Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and [c]Department of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India

Correspondence e-mail: d_velu@yahoo.com

Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
R factor = 0.039
wR factor = 0.118
Data-to-parameter ratio = 19.3

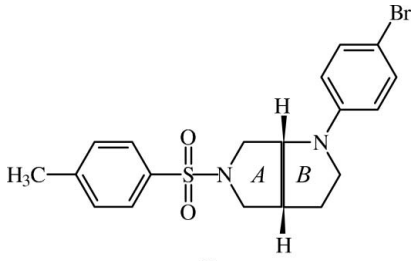
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{19}H_{21}BrN_2O_2S$, the fused pyrrolidine rings adopt envelope conformations. The molecular packing is stabilized by weak intermolecular C—H···O interactions and van der Waals forces.

Received 10 June 2005
Accepted 6 July 2005
Online 13 July 2005

Comment

Pyrrole derivatives have good *in vitro* activity against mycobacteria and candidae (Biava *et al.*, 2005). These derivatives also possess anti-inflammatory (Fernandes *et al.*, 2004) and antiviral (Borthwick *et al.*, 2003) activities. In view of its medicinal importance, the crystal structure determination of the title compound, (I), was carried out by X-ray diffraction.



(I)

Compound (I) (Fig. 1) consists of fused pyrrolidine rings (*A* and *B*), bromophenyl and toluenesulfonyl groups. The H atoms attached to the junction of rings *A* and *B* adopt a *cis* configuration with a torsion angle of −14.4 (3)°. The dihedral angle between the fused pyrrolidine rings *A* and *B* is 63.2 (1)° (*i.e* the angle between the mean planes through atoms N1/C8/C9/C10 and N2/C9/C10/C12). The C—Br, C—S, N—S and S—

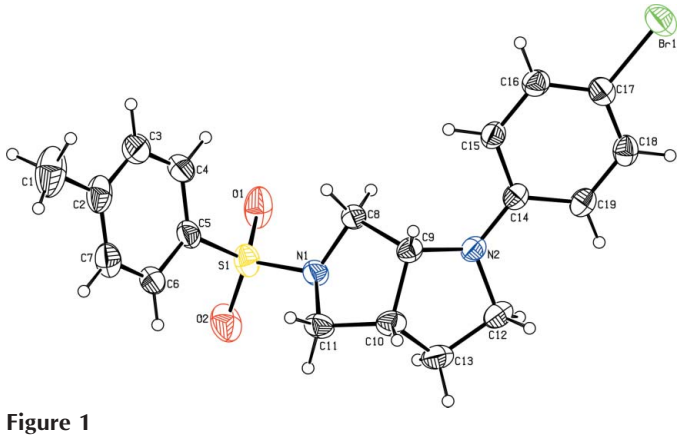


Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

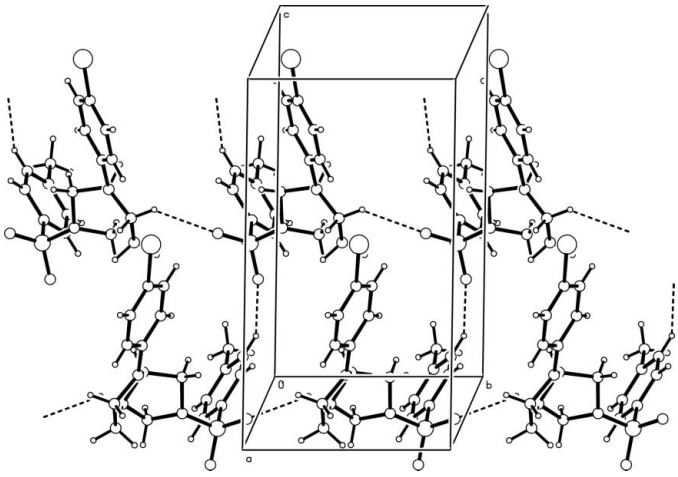


Figure 2
The molecular packing of (I), showing the intermolecular C—H···O hydrogen bonds (dashed lines).

O bond lengths (Table 1) are comparable to those reported in the literature (Allen *et al.*, 1987).

Atom S1 has a distorted tetrahedral configuration, with the angles O2—S1—O1 [120.2 (2)°] and C5—S1—N1 [107.4 (1)°] deviating significantly from ideal tetrahedral values. Similar distortions in the sulfonyl group were reported and attributed to the repulsive interaction between the short S═O bonds (Ravishankar *et al.*, 2003). The sums of the angles at atoms N1 and N2 of the pyrrolidine rings (346.7 and 354.2°, respectively) are in accordance with sp^3 hybridization. The torsion angles C1—C2—C3—C4 [−179.7 (3)°] and C1—C2—C7—C6 [179.7 (3)°] indicate that the methyl group does not deviate significantly from the plane of the attached benzene ring. The bromophenyl group is essentially planar, with a maximum deviation of 0.078 (1) Å for atom Br1. The mean planes of the bromophenyl and methylphenyl groups make a dihedral angle of 38.2 (1)°.

Pyrroldine rings *A* and *B* both adopt envelope conformations, with puckering parameters q_2 = 0.388 (3) (ring *A*) and 0.295 (2) Å (ring *B*), and φ = 159.2 (3) (ring *A*) and 102.4 (4) (ring *B*) (Cremer & Pople, 1975). For ring *A*, atom C11 deviates by 0.590 (3) Å from the least-squares plane through the remaining four atoms, whereas for ring *B*, atom C13 deviates by 0.454 (3) Å from the corresponding least-squares plane. The molecular packing (Fig. 2) is stabilized by weak intermolecular C—H···O hydrogen bonds (Table 2) and van der Waals forces.

Experimental

A solution of *N*-allyl-*N*-(2-oxoethyl)-4-methylbenzenesulfonamide (1 mmol) and *p*-bromobenzylglycine (1.2 mmol) in dry toluene (20 ml) was refluxed for 4 h. After completion of the reaction, the solvent was evaporated under vacuum and the residue was chromatographed using ethyl acetate and hexane mixture (9:1) to yield the title compound. Single crystals suitable for X-ray diffraction were obtained from an ethyl acetate and hexane (1:1) mixture by slow evaporation.

Crystal data

$C_{19}H_{21}BrN_2O_2S$	D_x = 1.502 Mg m^{-3}
M_r = 421.35	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 6484 reflections
a = 17.7114 (13) Å	θ = 2.3–22.9°
b = 7.8569 (6) Å	μ = 2.33 mm^{-1}
c = 14.3048 (10) Å	T = 273 (2) K
β = 110.589 (1)°	Block, colourless
V = 1863.5 (2) Å^3	0.22 × 0.20 × 0.18 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3239 reflections with $I > 2\sigma(I)$
ω scans	R_{int} = 0.024
Absorption correction: none	θ_{max} = 28.0°
20629 measured reflections	$h = -23 \rightarrow 22$
4384 independent reflections	$k = -10 \rightarrow 10$
	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0686P)^2 + 0.4426P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.039	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.118	$\Delta\rho_{max}$ = 0.99 e Å^{-3}
S = 1.02	$\Delta\rho_{min}$ = −0.46 e Å^{-3}
4384 reflections	
227 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Br1—C17	1.903 (2)	S1—N1	1.621 (2)
S1—O1	1.425 (2)	S1—C5	1.758 (3)
S1—O2	1.428 (2)		
O1—S1—O2	120.2 (2)	C8—N1—S1	120.3 (1)
N1—S1—C5	107.4 (1)	C14—N2—C12	121.2 (2)
C11—N1—C8	107.5 (2)	C14—N2—C9	120.9 (2)
C11—N1—S1	118.9 (2)	C12—N2—C9	112.1 (2)
C1—C2—C3—C4	−179.7 (3)	C1—C2—C7—C6	179.7 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O2^{i}	0.93	2.47	3.206 (3)	137
C10—H10···O1^{ii}	0.98	2.52	3.337 (3)	140

Symmetry codes: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $x, y-1, z$.

The H atoms were positioned geometrically and treated as riding on their parent C atoms, with C—H distances of 0.93–0.98 Å and $U_{iso} = 1.5U_{eq}$(C) for methyl H and $1.2U_{eq}$(C) for the remaining H atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

S*S* thanks the Council of Scientific and Industrial Research (CSIR) for providing a Senior Research Fellowship. DV

acknowledges the University Grants Commission (UGC) and the Department of Bio-Technology (DBT), India, for providing computing facilities under major Research projects and also thanks the Department for financial support under the UGC–SAP and DST–FIST programs.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, S1–19.
Biava, M., Porretta, G. C., Poce, G., Deidda, D., Pompei, R., Tafi, A. & Manetti, F. (2005). *Bioorg. Med. Chem.* **13**, 1221–1230.
Borthwick, A. D., Davies, D. E., Erti, P. F., Exall, A. M., Haley, T. M., Hart, G. J., Jackson, D. L., Parry, N. R., Patikis, A., Trivedi, N., Weingarten, C. G. & Woolven, J. M. (2003). *J. Med. Chem.* **46**, 4428–4449.
Bruker (2001). *SAINT* (Version 6.28a) and *SMART* (Version 5.625). Bruker AXS Inc., Madison, Wisconsin, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Fernandes, E., Costa, D., Toste, S. A., Lima, J. L. & Reis, S. (2004). *Free Radical Biol. Med.* **37**, 1895–1905.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Ravishankar, T., Chinnakali, K., Arumugam, N., Srinivasan, P. C., Usman, A. & Fun, H.-K. (2003). *Acta Cryst.* E**59**, o1903–o1906.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.