Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(2*R*,3a*R*,4*S*,6a*R*,7a*R*)-*cis*-4-Benzyl-2-(4-bromophenyl)-5-(4-methylphenylsulfonyl)perhydrothiazolidino[3,4-*a*]pyrrolo[4,5-*c*]pyrrole

Navin V. Narayanan,[a] D. Gayathri,[a] D. Velmurugan,[a]* K. Ravikumar[b] and M. Poornachandran[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

Received 15 March 2007
Accepted 25 March 2007

In the title compound, $C_{28}H_{29}BrN_2O_2S_2$, the thiazolidine and the two pyrrolidine rings adopt envelope conformations. The crystal packing is stabilized by weak C—H$\cdots\pi$ interactions.

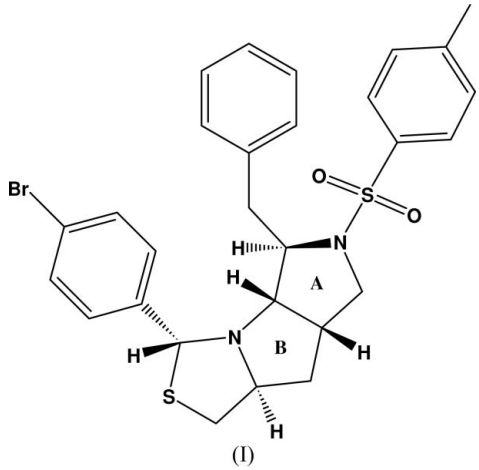
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.046
wR factor = 0.130
Data-to-parameter ratio = 19.6

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

Comment

Pyrrolidine derivatives are extensively studied for their important medicinal properties. Pyrrolidine occurs widely in nature and is a structural component of porphyrin heme, chlorophyll and vitamin B12. Pyrrolidine compounds have antifungal and antimicrobial activities (Amal Raj *et al.*, 2003). Thiazolidine derivatives possess antidiabetic and adipogenic properties (Norisada *et al.*, 2004).



(I)

In the title compound, (I), the S2—C6 bond length is long compared with S2—C5, while the N2—C6 bond is short compared with N2—C4 and N2—C7 (Table 1). These long and short bonds may be a result of the bromophenyl substituent at C6. The sums of the bond angles around N1 (353.8°) and N2 (334.9°) indicate sp^2- and sp^3-hybridization, respectively. The C23–C28 phenyl ring makes dihedral angles of 21.1 (2) and 79.6 (2)°, respectively, with the C9–C14 and C15–C20 benzene rings. Atom Br1 deviates by 0.094 (1) Å from the mean plane of the benzene ring C9–C14.

The two fused pyrrolidine rings (N1/C1/C2/C7/C8, *A* and C2—C4/N2/C7, *B*) adopt envelope conformations, with flap atom C2 deviating by 0.506 (2) and 0.552 (3) Å, respectively, from the N1/C1/C7/C8 and C3/C4/N2/C7 planes. The thiazolidine ring also adopts an envelope conformation, with atom S2 deviating by 0.888 (1) Å from the other atoms in the ring, whereas in a similar structure, 3-(4-bromophenyl)-6-(*p*-tosyl)perhydrothiazolidino[3,4-*a*]pyrrolo[4,5-*c*]pyrrole (Kavitha *et al.*, 2006), the thiazolidine ring adopts a twist conformation.

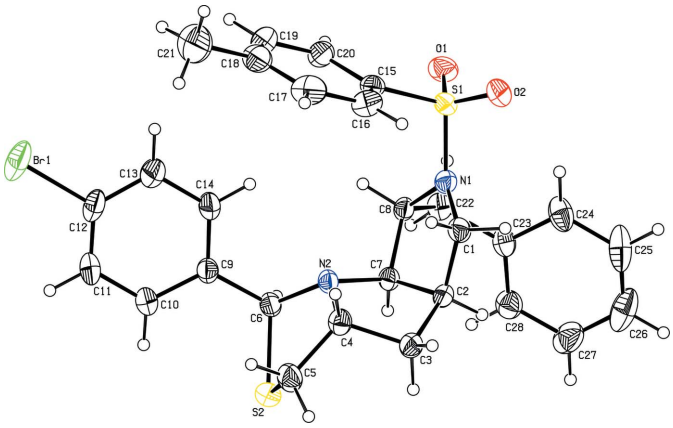


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids.

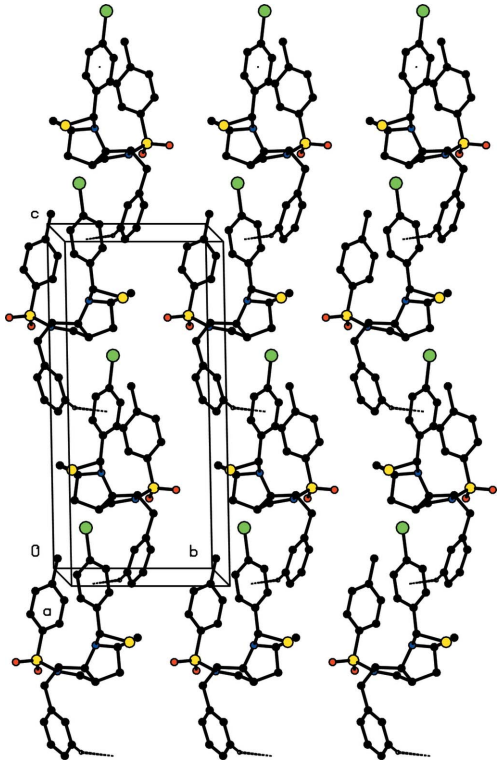


Figure 2
The molecular packing of (I), viewed approximately down the *a* axis. For clarity, H atoms not involved in the C—H···π interactions (dashed lines) have been omitted.

The puckering parameters (Cremer & Pople, 1975) and the smallest displacement asymmetry parameters (Nardelli, 1983) are q_2 = 0.326 (3) Å, φ = 257.4 (4)° and $\Delta_s(C_2)$ = 3.4 (2)° for ring *A*, q_2 = 0.358 (3) Å, φ = 105.9 (4)° and $\Delta_s(C_2)$ = 1.5 (3)° for ring *B*, and q_2 = 0.522 (2) Å, φ = 357.6 (3)° and $\Delta_s(S_2)$ = 3.8 (2)° for the thiazolidine ring.

The crystal packing is stabilized by van der Waals and weak C—H···π intermolecular interactions. Atom C27 acts as a donor to the C9–C14 benzene ring at (x, $\frac{1}{2} - y$, $-\frac{1}{2} + z$) (centroid *Cg*), with H···*Cg* and C···*Cg* distances of 2.85 and 3.716 (4) Å and a C—H···*Cg* angle of 156° (Fig. 2).

Experimental

A mixture of (*S*)-2-(*N*-allyl-*N*-tosylamino)-3-phenylpropanal (1.0 mmol) and 2-(*p*-bromophenyl)thiazolidine-4-carboxylic acid (1.5 mmol) in toluene (30 ml) was refluxed under Dean–Stark conditions till the completion of the reaction. The reaction mixture was then concentrated *in vacuo* and extracted with dichloromethane (2 × 20 ml) and water (2 × 20 ml). The organic layer was washed with brine (2 × 20 ml), dried with anhydrous sodium sulfate and concentrated *in vacuo*. The residue was then subjected to column chromatography (silica gel, 100–200 mesh), eluting with a hexane–ethyl acetate (8:2) mixture, to give the title compound. The title compound was crystallized from a hexane–ethyl acetate (4:1) solution by slow evaporation.

Crystal data

$C_{28}H_{29}BrN_2O_2S_2$
M_r = 569.56
Monoclinic, $P2_1/c$
a = 14.7207 (8) Å
b = 9.1692 (5) Å
c = 20.6819 (11) Å
β = 109.461 (1)°
V = 2632.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.75 mm^{-1}
T = 293 (2) K
0.25 × 0.24 × 0.22 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
29550 measured reflections
6207 independent reflections
4428 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.130
S = 0.97
6207 reflections
317 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.80 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C4—N2	1.490 (3)	C6—S2	1.847 (2)
C5—S2	1.804 (3)	C7—N2	1.478 (3)
C6—N2	1.449 (3)		
C1—N1—C8	112.7 (2)	C6—N2—C7	114.8 (2)
C1—N1—S1	119.9 (2)	C6—N2—C4	110.7 (2)
C8—N1—S1	121.2 (2)	C7—N2—C4	109.4 (2)

H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.5U_{eq}(methyl C) or 1.2U_{eq}(C).

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: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

DV thanks DST, India, for a major research project and DG thanks CSIR, India, for the award of a senior research fellowship. Department of Science and Technology (DST–

FIST) and University Grants Commission (UGC), Government of India, are acknowledged by DV for providing facilities to the department.

References

Amal Raj, A., Raghunathan, R., Sridevi Kumari, M. R. & Raman, N. (2003). *Bioorg. Med. Chem.* **11**, 407–409.
Bruker (2001). *SMART* (Version 5.625/NT/2000) and *SAINT* (Version 6.28a). Bruker AXS Inc., Madison, Wisconsin, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Kavitha, V., Gayathri, D. R., Velmurugan, D., Ravikumar, K. & Poornachandran, M. (2006). *Acta Cryst.* E**62**, o2146–o2148.
Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Norisada, N., Masuzaki, H., Fujimoto, M., Inoue, G., Hosoda, K., Hayashi, T., Watanabe, M., Muraoka, S., Yoneda, F. & Nakao, K. (2004). *Metabolism*, **53**, 1532–1537.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.