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cis-1-(4-Bromophenyl)-6-ethyl-5-tosylperhydropyrrolo[3,4-*b*]pyrrole

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.006 Å
R factor = 0.057
wR factor = 0.147
Data-to-parameter ratio = 23.8

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

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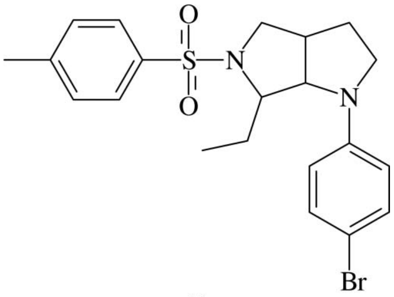
In the title molecule, $C_{21}H_{25}BrN_2O_2S$, one of the fused pyrrolidine rings adopts an envelope conformation, while the other is in a twist conformation. In the crystal structure, molecules related by translation along the *a* axis are linked by π–π interactions into a chain.

Comment

Inhibitors of human cytomegalovirus (HCMV) protease have been designed, based on the 5-oxohexahydropyrrolo[3,2-*b*]pyrrole ring system (Borthwick *et al.*, 2000). Pyrrolo[1,2-*a*]pyrrole compounds are used as anti-inflammatory and analgesic agents (Muchowski *et al.*, 1989). Certain pyrrolo[3,4-*c*]pyrroles act as potent and selective orphanin FQ/nociceptin (N/OFQ) receptor (NOP) agonists (Kolczewski *et al.*, 2003). We report here the structure of the title compound, (I), containing a pyrrolo[3,4-*b*]pyrrole unit.



(I)

Bond lengths and angles in (I) (Table 1) agree with those observed in a similar structure, 1-(*p*-bromophenyl)-5-*p*-tosylperhydropyrrolo[3,4-*b*]pyrrole (Selvanayagam *et al.*, 2005). The sums of the bond angles around atoms N1 (350.9°) and N2 (356.9°) indicate sp^2 hybridization. However, N1 deviates from the S1/C1/C4 plane by 0.268 (4) Å and N2 deviates from the C3/C6/C7 plane by 0.146 (4) Å, indicating slight pyramidalization. The C7–C12 and C13–C18 benzene rings are almost parallel to each other [dihedral angle 3.1 (2)°], but no π–π interaction is observed as they are displaced away from each other with a centroid–centroid distance of 4.734 (2) Å. The N1/C1—C4 pyrrolidine ring adopts an envelope conformation, with atom C3 at the flap (Fig. 1). The deviation of atom C3 from the N1/C1/C2/C4 plane is 0.450 (6) Å. The other pyrrolidine ring (N2/C3/C2/C5/C6) adopts a twist conformation, with Cremer & Pople (1975) puckering parameters q_2 and φ of 0.339 (4) Å and 272.4 (7)°, respectively. The ethyl substituent is attached axially to the N1/C1–C4 ring.

In the crystal structure of (I), molecules related by translation along the *a* axis are linked by π–π interactions into a chain (Fig. 2). The interactions involve the C7–C12 (centroid *Cg*1) and C13–C18 (centroid *Cg*2) benzene rings, with a $Cg1\cdots Cg2^{i}$ distance of 3.726 (2) Å [symmetry code: (i) $x + 1$, y, z].

Experimental

A mixture of 2(*N*-allyl-*N*-tosylamino)butanal (1 mmol) and *N*-(4-bromo)phenylglycine (1 mmol) in toluene (20 ml) was refluxed until the disappearance of the starting materials, as indicated by thin-layer chromatography. After completion of the reaction, the solvent was evaporated under vacuum and the residue was chromatographed using a hexane–ethyl acetate (8:2 *v*/*v*) mixture to yield the title compound. The compound was recrystallized from an ethyl acetate solution by slow evaporation.

Crystal data

$C_{21}H_{25}BrN_2O_2S$
$M_r = 449.40$
Orthorhombic, $Pna2_1$
$a = 7.7131\ (3)$ Å
$b = 19.6640\ (8)$ Å
$c = 12.9518\ (4)$ Å
$V = 1964.40\ (13)$ Å^3
$Z = 4$
$D_x = 1.520$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 2.22$ mm^{-1}
$T = 100.0\ (1)$ K
Plate, colourless
$0.28 \times 0.27 \times 0.07$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.531$, $T_{max} = 0.869$
21230 measured reflections
5855 independent reflections
4509 reflections with $I > 2\sigma(I)$
$R_{int} = 0.081$
$\theta_{max} = 30.3°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.147$
$S = 1.02$
5855 reflections
246 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0868P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 2.61$ e Å^{-3}
$\Delta\rho_{min} = -0.92$ e Å^{-3}
Absolute structure: Flack (1983), with 2795 Friedel pairs
Flack parameter: $-0.005\ (11)$

H atoms were positioned geometrically, with C—H = 0.95–1.00 Å, and treated as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C). A rotating-group model was used for the methyl groups. The highest residual electron density peak is located 0.91 Å from atom Br1.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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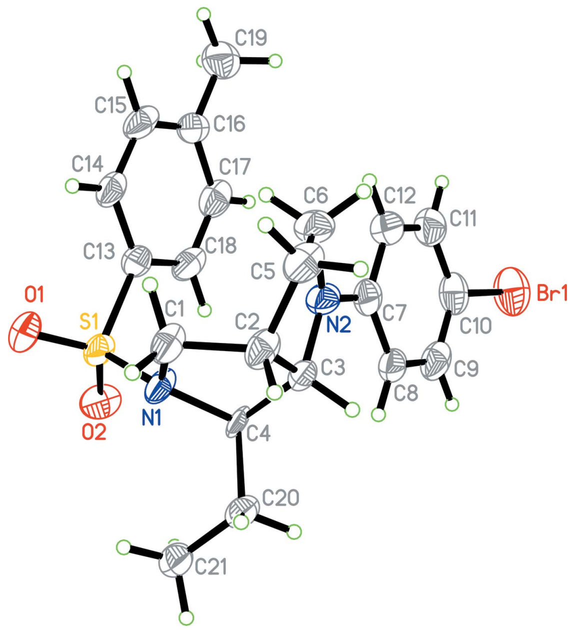

Figure 1
The molecular structure of (I), showing 80% probability displacement ellipsoids and the atomic numbering scheme.

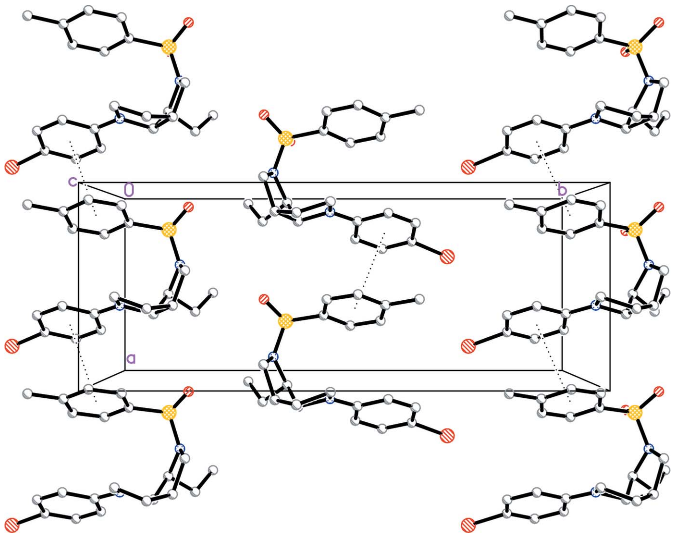

Figure 2
A view of the molecular chains in (I) formed by π–π stacking interactions (dotted lines). H atoms have been omitted for clarity.

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