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(3a*R*,6*S*,6a*R*)-*cis*-6-Benzyl-1-(4-chlorophenyl)-5-tosylperhydropyrrolo[3,4-*b*]pyrrole

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.001 Å
R factor = 0.037
wR factor = 0.094
Data-to-parameter ratio = 48.9

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

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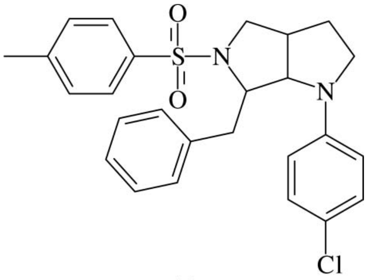
In the title molecule, $C_{26}H_{27}ClN_2O_2S$, the fused pyrrolidine rings adopt twist conformations. C—H⋯O and C—H⋯π interactions link the molecules into a three-dimensional network.

Comment

The background to this study has been described in the preceding paper (Chinnakali *et al.*, 2007). We report here the crystal structure of the title compound, (I) (Fig. 1).



(I)

The geometric parameters in (I) are comparable with those observed in a similar structure, 1-(*p*-bromophenyl)-5-*p*-tosylperhydropyrrolo[3,4-*b*]pyrrole (Selvanayagam *et al.*, 2005). N1 deviates from the S1/C1/C4 plane by 0.306 (1) Å and N2 deviates from the C3/C6/C7 plane by 0.162 (1) Å, indicating slight pyramidalization. The C13–C18 and C21–C26 rings make dihedral angles of 9.81 (5) and 81.59 (3)°, respectively, with the C7–C8 ring. Both N1/C1–C4 and N2/C3/C2/C5/C6 pyrrolidine rings adopt twist conformations. The Cremer & Pople (1975) puckering parameters q_2 and φ_2 are 0.3128 (9) Å and 89.25 (16)°, respectively, for the N1/C1–C4 ring, and 0.3429 (11) Å and 262.40 (17)°, respectively, for the N2/C3/C2/C5/C6 ring. The benzyl substituent is attached axially to the N1/C1–C4 ring, while the chlorophenyl group is attached equatorially to the N2/C3/C2/C5/C6 ring.

In the crystal structure of (I), C—H⋯O intermolecular hydrogen bonds and C—H⋯π interactions involving the C21–C26 phenyl ring (centroid *Cg*1) link the molecules into a three-dimensional framework (Table 1).

Experimental

A mixture of (*S*)-2-(*N*)-allyl-*N*-tosylamino-3-phenylpropanal (1 mmol) and *N*-(4-chloro)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

$C_{26}H_{27}ClN_2O_2S$
$M_r = 467.01$
Orthorhombic, $P2_12_12_1$
$a = 10.3604$ (2) Å
$b = 10.7859$ (2) Å
$c = 21.0024$ (4) Å
$V = 2346.94$ (8) Å^3
$Z = 4$
$D_x = 1.322$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.28$ mm^{-1}
$T = 100.0$ (1) K
Block, colourless
0.65 × 0.64 × 0.45 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.797$, $T_{max} = 0.885$
58953 measured reflections
14228 independent reflections
12745 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 40.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.094$
$S = 1.05$
14228 reflections
291 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0516P)^2 + 0.1841P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}
Absolute structure: Flack (1983), with 6271 Friedel pairs
Flack parameter: 0.31 (3)

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of the C21–C26 phenyl ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C17—H17···O2^{i}	0.95	2.57	3.4684 (11)	159
C23—H23···O1^{ii}	0.95	2.53	3.4750 (12)	176
C12—H12···*Cg*1	0.95	2.76	3.6558 (9)	157
C15—H15···*Cg*1^{iii}	0.95	2.56	3.4547 (10)	157

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

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. During the refinement, the structure was treated as an inversion twin.

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).

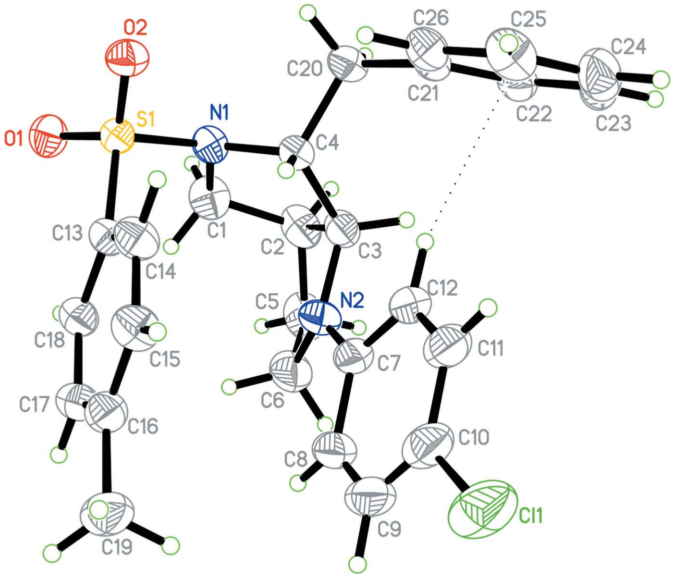


Figure 1
The molecular structure of (I), showing 80% probability displacement ellipsoids and the atomic numbering scheme. The dotted line indicates a C—H···π interaction.

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