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

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.116
Data-to-parameter ratio = 13.1

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

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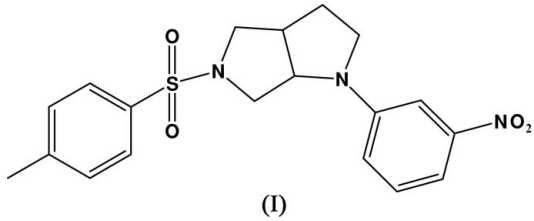
In the title compound, $C_{19}H_{21}N_3O_4S$, one of the pyrrolidine rings adopt a half-chair conformation, while the other is in an envelope conformation. The molecules are linked into *C*(9) chains by C—H···O hydrogen bonds.

Comment

The pyrrolidine motif occurs in many families of biologically important compounds. Owing to the ease of substitution and modifications at several positions, many derivatives of pyrrolidine have been synthesized with varying properties (Baldwin *et al.*, 1994). The derivatives of pyrrolidine have been found to exhibit antifungal and antimicrobial activities (Amal Raj *et al.*, 2003). It has been shown that *N*-substituted pyrrole derivatives inhibit human immunodeficiency virus type-I (HIV-I) (Jiang *et al.*, 2004). We report here the crystal structure of the title compound, (I).



(I)

The bond lengths in (I) (Fig. 1) show normal values (Allen *et al.*, 1987). The O—S—O angle deviates significantly from the ideal tetrahedral value compared to N—S—C, N—S—O and C—S—O angles (Table 1). The sums of the bond angles around N1 (343.6°) and N2 (359.4°) indicate that N1 is sp^3-hybridized and N2 is sp^2-hybridized. The N1/C1–C4 pyrrolidine ring adopts a half-chair conformation with an asymmetry parameter (Nardelli, 1983) ΔC_2(C2) of 4.1 (2)°, and puckering parameters (Cremer & Pople, 1975) q_2 of 0.342 (2) Å and φ of −23.1 (3)°. The other pyrrolidine ring (N2/C3/C2/C5/C6) is in an envelope conformation, with ΔC_s(C5), q_2 and φ values of 7.8 (2)°, 0.343 (3) Å and 116.4 (4)°, respectively. The deviation of atom C5 from the mean plane defined by atoms N2, C3, C2 and C6 is 0.522 (4) Å. The dihedral angle between the N2/C3/C2/C6 and C7–C12 planes is 8.25 (6)°.

C—H···O hydrogen bonds (Table 2) link the molecules into chains; atom C4 in the molecule at (*x*, *y*, *z*) acts as a hydrogen-bond donor to atom O4 in the molecule at (*x*, *y*, 1 + *z*), forming a *C*(9) chain running along the *c* axis (Fig. 2).

Experimental

A solution of *N*-allyl-*N*-(2-oxoethyl)-4-methylbenzene sulfonamide (1 mmol) and *m*-nitrophenylglycine (1.2 mmol) in dry toluene (20 ml) was refluxed for 3 h. After completion of the reaction, the solvent was

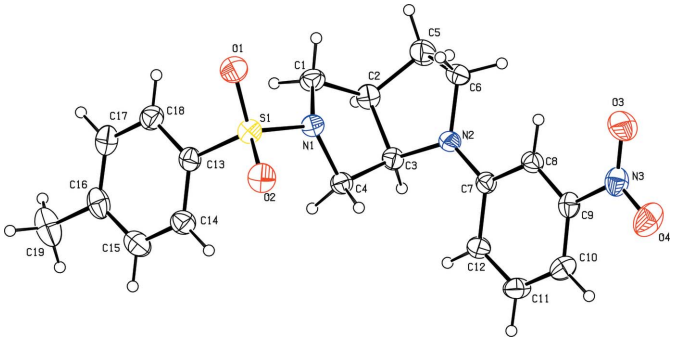


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids.

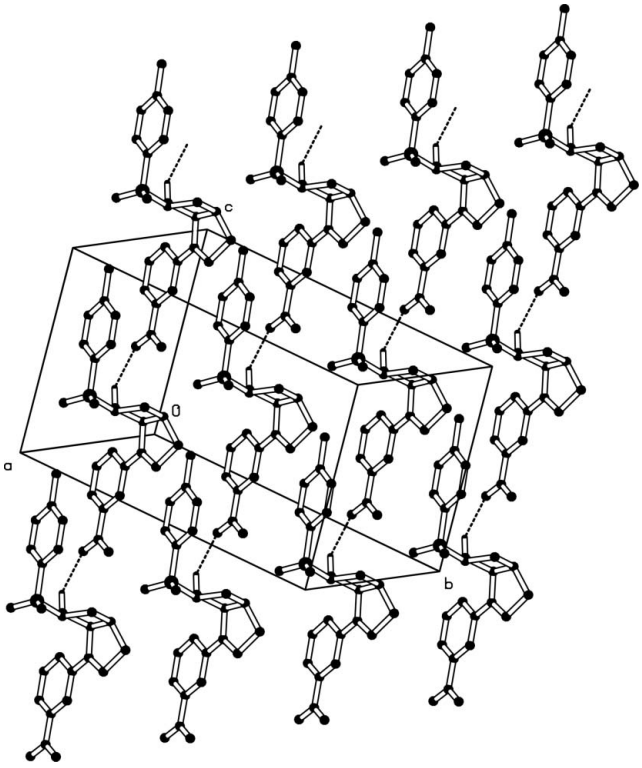


Figure 2
The crystal packing of (I). For the sake of clarity, H atoms not involved in hydrogen bonds (dashed lines) have been omitted.

evaporated under vacuum and the residue was chromatographed using a hexane and ethyl acetate (9:1) mixture to yield the title compound. The compound was recrystallized from ethyl acetate by slow evaporation.

Crystal data

$C_{19}H_{21}N_3O_4S$
M_r = 387.45
Monoclinic, $P2_1/c$
a = 7.4456 (4) Å
b = 25.2680 (15) Å
c = 10.0594 (6) Å
β = 104.286 (1)°
V = 1834.00 (18) Å^3
Z = 4
D_x = 1.403 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 273 (2) K
Block, colourless
0.25 × 0.22 × 0.21 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
17515 measured reflections
3217 independent reflections
2942 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.116
S = 1.14
3217 reflections
245 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0504P)^2 + 0.7696P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.475 (3)	C13—S1	1.767 (2)
C3—N2	1.463 (2)	N1—S1	1.631 (2)
C4—N1	1.474 (2)	N3—O3	1.212 (3)
C6—N2	1.447 (3)	N3—O4	1.221 (2)
C7—N2	1.367 (2)	O1—S1	1.428 (2)
C9—N3	1.468 (3)	O2—S1	1.425 (2)
C4—N1—C1	107.7 (2)	O4—N3—C9	118.2 (2)
C4—N1—S1	118.3 (1)	O2—S1—O1	120.2 (1)
C1—N1—S1	117.6 (1)	O2—S1—N1	106.6 (1)
C7—N2—C6	123.6 (2)	O1—S1—N1	106.2 (1)
C7—N2—C3	123.7 (2)	O2—S1—C13	108.0 (1)
C6—N2—C3	112.1 (2)	O1—S1—C13	107.9 (1)
O3—N3—O4	122.9 (2)	N1—S1—C13	107.3 (1)
O3—N3—C9	118.9 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4A···O4^{i}	0.97	2.56	3.431 (3)	150

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

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

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