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2-(4-Fluorophenyl)-5-(phenylsulfonyl)perhydro-1,3-thiazolo[3,4-*a*]pyrrolo[4,5-*c*]pyrrole

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.001 Å
Disorder in main residue
R factor = 0.037
wR factor = 0.100
Data-to-parameter ratio = 38.4

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

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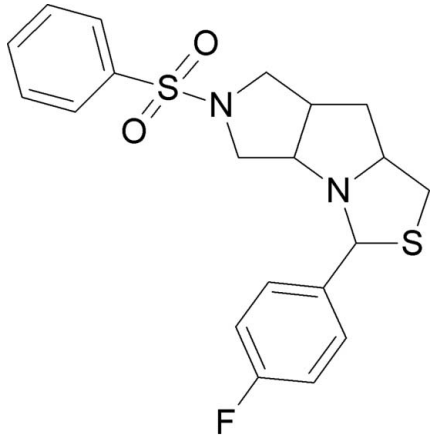
In the title compound, $C_{20}H_{21}FN_2O_2S_2$, the thiazolidine ring is disordered and both conformers adopt envelope conformations. One of the pyrrolidine rings adopts an envelope conformation, while the other is in a twist conformation. C—H···O hydrogen bonds link the molecules into a chain along the *a* axis, and inversion-related molecules in adjacent chains are interconnected *via* C—H···O and C—H···π interactions to form a double-stranded chain.

Comment

Pyrrolidine derivatives have been found to exhibit antifungal and antimicrobial activities (Amal Raj *et al.*, 2003). Pyrrolopyrrole compounds exhibit anti-inflammatory and analgesic activities (Rooks *et al.*, 1982; Muchowski *et al.*, 1989). Inhibitors of human cytomegalovirus (HCMV) protease have been designed based on the pyrrolopyrrole ring system (Borthwick *et al.*, 2000). Pyrrolothiazole derivatives show antileukaemic activity (Anderson & Mach, 1987) and some of them are used as platelet-activating factor (PAF) antagonists (Weissman *et al.*, 1993; Le Naour *et al.*, 1994). We report here the structure of the title compound, (I).



(I)

The molecular structure of (I) is illustrated in Fig. 1. Bond lengths and angles in (I) agree with those observed in the bromo- and chloro-analogues (Kumar *et al.*, 2006; Senthil Kumar *et al.*, 2006). The configuration around atom N1 is nearly planar [N1 deviates from the C1/C4/S2 plane by 0.225 (1) Å], whereas atom N2 exhibits a pyramidal geometry. Both the major and the minor conformers of the disordered thiazolidine ring adopt envelope conformations. In the major conformer, the deviation of atom C8 from the S1/C7*B*/C6/N2

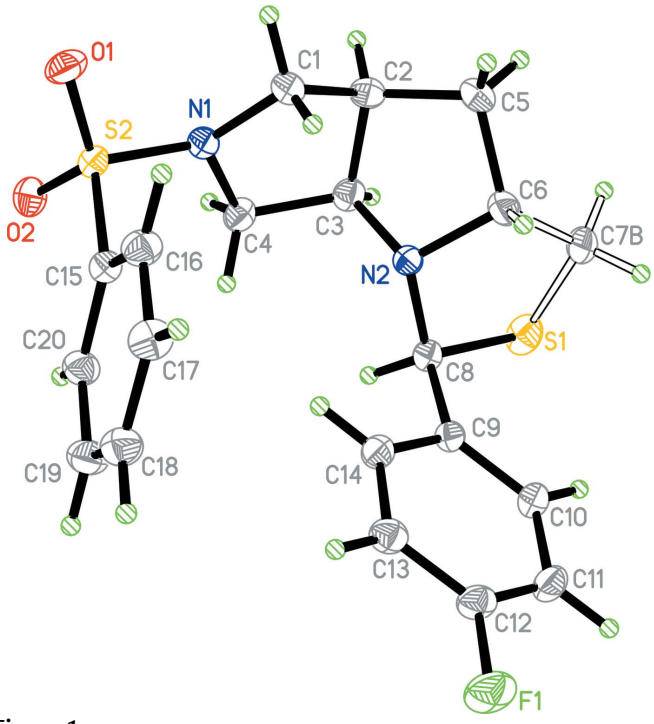


Figure 1
The structure of (I), showing 60% probability displacement ellipsoids and the atomic numbering scheme. Only the major component of the disordered C7 atom is shown.

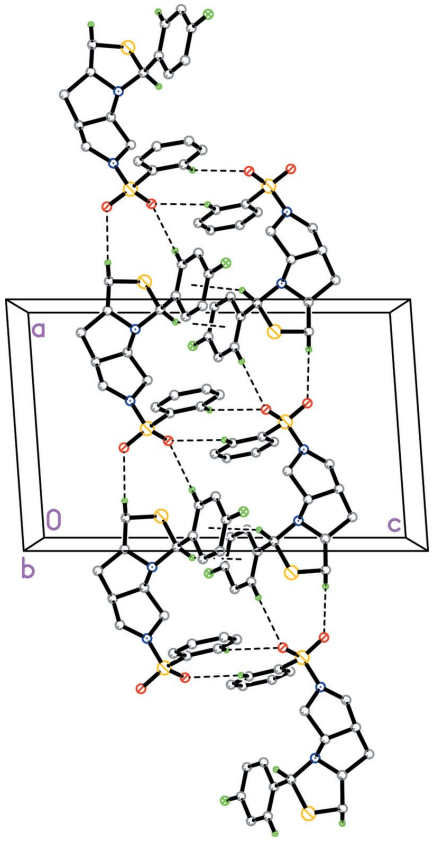


Figure 2
A view of a hydrogen-bonded (dashed lines) double-stranded chain in (I). Only the H atoms involved in hydrogen bonding are shown. Only the major component of the disordered C7 atom is shown.

plane is 0.619 (4) Å, and in the minor conformer the deviation of atom N2 from the S1/C6/C7*A*/C8 ring is 0.544 (2) Å. One of the pyrrolidine rings (N1/C1–C4) adopts an envelope conformation, with atom C2 at the flap position [deviation 0.559 (1) Å], while the other pyrrolidine ring (N2/C3/C2/C5/C6) is twisted on the N2—C3 bond.

Molecules translated by one unit cell along the *a* axis are linked by intermolecular C7*B*—H7*BA*···O1^{i} and C10—H10···O2^{i} hydrogen bonds (symmetry codes as in Table 1) to form a chain. Inversion-related molecules in adjacent chains are interconnected *via* C20—H20···O2^{ii} hydrogen bonds and C8—H8···*Cg*1^{iii} interactions, involving the C9–C14 benzene rings (centroid *Cg*1), to form a double-stranded chain along the *a* axis (Fig. 2).

A superimposed fit of the non-H atoms of (I) and the corresponding atoms in the bromo-analogue (Kumar *et al.*, 2006) gives an r.m.s. deviation of 0.113 Å. A similar fit with the chloro-analogue (Senthil Kumar *et al.*, 2006) gives an r.m.s. deviation of 0.102 Å. The conformations of the thiazolidine and N1/C1–C4 pyrrolidine rings in (I) are different from the bromo- and chloro-analogues, in which the rings adopt twist conformations. The patterns of intermolecular C—H···π hydrogen bonding in the crystal structures of (I) and its bromo- and chloro-analogues are identical but those of other hydrogen bonds differ.

Experimental

A solution of *N*-allyl-*N*-(2-oxoethyl)benzenesulfonamide (1 mmol) and 2-(*p*-fluorophenyl)thiazolidine-4-carboxylic acid (1.2 mmol) in dry toluene (30 ml) was refluxed for 4.5 h. After completion of the reaction, the solvent was evaporated under vacuum and the residue was chromatographed (SiO_2) using a hexane–ethyl acetate (8:2) mixture, to yield the title compound. Compound (I) was recrystallized from ethyl acetate.

Crystal data

$C_{20}H_{21}FN_2O_2S_2$
M_r = 404.51
Monoclinic, $P2_1/c$
a = 10.4806 (1) Å
b = 10.6106 (1) Å
c = 16.8245 (2) Å
β = 94.055 (1)°
V = 1866.29 (3) Å^3
Z = 4
D_x = 1.440 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 100.0 (1) K
Block, colourless
0.48 × 0.42 × 0.30 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.864, T_{max} = 0.912
62572 measured reflections
9762 independent reflections
8289 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 37.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.100
S = 1.02
9762 reflections
254 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0516P)^2 + 0.4746P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.63 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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

*Cg*1 is the centroid of the ring C9–C14.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7*B*—H7*BA*···O1^{i}	0.99	2.59	3.558 (6)	165
C10—H10···S1	0.95	2.67	3.1147 (9)	109
C10—H10···O2^{i}	0.95	2.54	3.4353 (11)	158
C20—H20···O2	0.95	2.55	2.9095 (11)	103
C20—H20···O2^{ii}	0.95	2.57	3.2247 (11)	127
C8—H8···*Cg*1^{iii}	1.00	2.55	3.4629 (9)	151

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, -y, -z$; (iii) $-x, -y, -z$.

Atom C7 was found to be disordered over two positions, C7*A* and C7*B*, with occupancy factors of 0.45 (2) and 0.55 (2). The S—C and C—C bond lengths involving the disordered atoms were restrained to be equal. H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H = 0.95–1.00 Å and U_{iso}(H) = 1.2U_{eq}(C).

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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