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2-(4-Methylphenyl)-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
R factor = 0.031
wR factor = 0.091
Data-to-parameter ratio = 33.8

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

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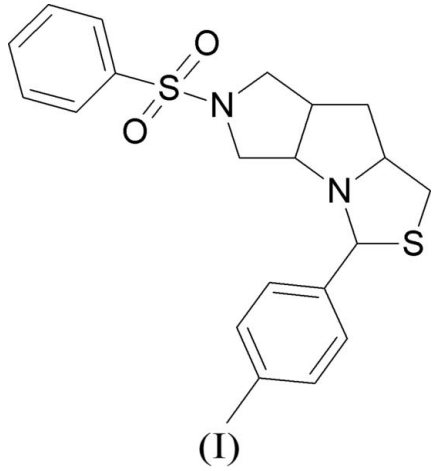
In the title molecule, $C_{21}H_{24}N_2O_2S_2$, the two pyrrolidine rings adopt twisted conformations, while the thiazolidine ring is in an envelope conformation with the N atom at the flap position. In the crystal structure, molecules translated by a unit cell along the *a* axis are linked by intermolecular O—H···O hydrogen bonds into chains. Adjacent chains are interconnected *via* π–π and C—H···π interactions to form sheets parallel to the *ab* plane.

Comment

Inhibitors of human cytomegalovirus (HCMV) protease have been designed based on the 5-oxo-hexahydro-pyrrolo[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). Some of the pyrrolo[1,2-*c*]thiazole derivatives are used as platelet-activating factor (PAF) antagonists (Weissman *et al.*, 1993; Le Naour *et al.*, 1994). They also inhibit cytokine-dependent induction of human immunodeficiency virus (HIV) expression in chronically infected promonocytic cells (Weissman *et al.*, 1993). Since the title compound, (I), also contains a pyrrolopyrrole and a pyrrolothiazole unit it may also exhibit some biological activity.



(I)

Bond lengths and angles in (I) (Fig. 1) agree with those observed in a similar structure, 2-(4-chlorophenyl)-5-(phenylsulfonyl)perhydrothiazolo[3,4-*a*]pyrrolo[4,5-*c*]pyrrole, (II) (Senthil Kumar *et al.*, 2006). The sums of the bond angles around atoms N1 (351.6°) and N2 (330.1°) indicate sp^2 and sp^3 hybridization, respectively. However, atom N1 is slightly out of the plane [deviation 0.257 (1) Å] defined by atoms S2, C1 and C4, indicating a slight degree of pyramidalization. The

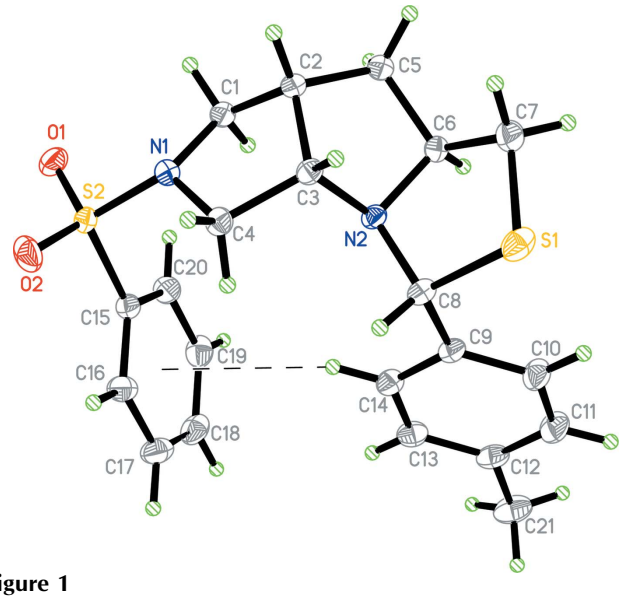


Figure 1
The structure of (I), showing 60% probability displacement ellipsoids and the atomic numbering scheme. The dashed line indicates a C—H···π interaction.

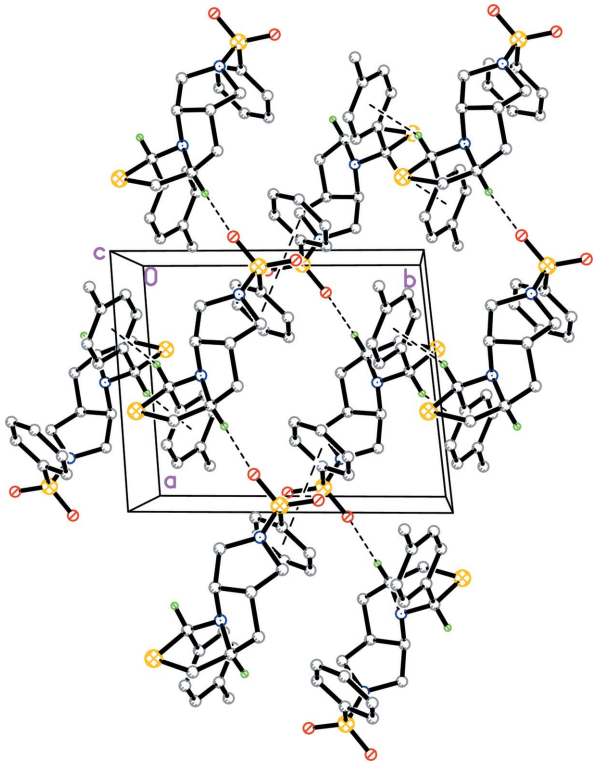


Figure 2
A view of a hydrogen-bonded (dashed lines) sheet in (I). Only the H atoms involved in hydrogen bonding are shown.

thiazolidine ring adopts an envelope conformation, with atom N2 at the flap position. The deviation of atom N2 from the S1/C6–C8 plane is 0.544 (1) Å. The two pyrrolidine rings (N1/C1–C4 and N2/C3/C2/C5/C6) adopt twisted conformations with Cremer & Pople (1975) puckering parameters q_2 and φ of 0.361 (1) Å and 55.5 (1)°, respectively, for the pyrrolidine ring N1/C1–C4, and 0.417 (1) Å and 192.7 (1)°, respectively, for the pyrrolidine ring N2/C3/C2/C5/C6.

The molecular conformation of (I) is stabilized by weak C—H···S and C—H···O intramolecular hydrogen bonds, and also by C—H···π interactions (Table 1) involving the C15–C20 phenyl ring (centroid *Cg*1).

A superimposed fit of the non-H atoms of (I) and the corresponding atoms in (II) gives an r.m.s. deviation of 0.963 Å. The conformation of (I) is slightly different from that of (II), as the thiazolidine ring in the latter adopts a twisted conformation, compared with the envelope conformation in (I).

In the crystal structure of (I), molecules translated by a unit cell along the *a* axis are linked by intermolecular C—H···O hydrogen bonds (Table 1) into chains. The C15–C20 phenyl rings of inversion-related molecules in adjacent chains are stacked with a $Cg1\cdots Cg1^{i}$ distance of 3.7539 (5) Å [symmetry code: (i) $-x, 1-y, -z$], indicating π–π interactions. These interactions link adjacent chains to form double-stranded chains along the *a* axis. The double-chains are interconnected *via* C—H···π interactions involving C9–C14 benzene rings (centroid *Cg*2) to form a sheet-like structure parallel to the *ab* plane (Fig. 2). The patterns of intermolecular hydrogen bonding are different in the crystal structures of (I) and (II).

Experimental

A solution of *N*-allyl-*N*-(2-oxoethyl)benzenesulfonamide (1 mmol) and 2-(*p*-methylphenyl)thiazolidine-4-carboxylic acid (1.2 mmol) in dry toluene (30 ml) was refluxed for 3.5 h. After completion of the reaction, the solvent was evaporatedunder 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

$C_{21}H_{24}N_2O_2S_2$
M_r = 400.54
Triclinic, $P\overline{1}$
a = 8.9672 (1) Å
b = 10.4040 (1) Å
c = 10.6794 (1) Å
α = 88.311 (1)°
β = 72.555 (1)°
γ = 83.761 (1)°
V = 944.87 (2) Å^3
Z = 2
D_x = 1.408 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 100 (2) K
Block, colourless
0.69 × 0.63 × 0.43 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.782, T_{max} = 0.881
23928 measured reflections
8275 independent reflections
7477 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 35.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.091
S = 1.04
8275 reflections
245 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0473P)^2 + 0.2769P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

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

*Cg*1 and *Cg*2 are the centroids of the rings C15–C20 and C9–C14, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10···S1	0.95	2.66	3.1166 (8)	110
C16—H16···O2	0.95	2.54	2.9152 (11)	103
C14—H14···*Cg*1	0.95	2.98	3.8372 (9)	152
C6—H6···O2^{i}	1.00	2.38	2.9791 (10)	118
C8—H8···*Cg*2^{ii}	1.00	2.69	3.6178 (9)	154

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

The H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C—H = 0.95–1.00 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.2U_{eq}(methyl C). A rotating-group model was used for the methyl group.

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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References

Borthwick, A. D., Angier, S. J., Crame, A. J., Exall, A. M., Haley, T. M., Hart, G. J., Mason, A. M., Pennell, A. M. K. & Weingarten, G. G. (2000). *J. Med. Chem.* **43**, 4452–4464.
Bruker (2005). *APEX2* (Version 1.27), *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Le Naour, R., Clayette, P., Henin, Y., Mabondzo, A., Raoul, H., Bousseau, A. & Dormont, D. (1994). *J. Gen. Virol.* **75**, 1379–1388.
Muchowski, J. M., Galeazzi, E., Greenhouse, R., Guzman, A., Perez, V., Ackerman, N., Ballaron, S. A., Rovito, J. R., Tomolonis, A. J. & Young, J. M. (1989). *J. Med. Chem.* **32**, 1202–1207.
Senthil Kumar, G., Chinnakali, K., Poornachandran, M., Raghunathan, R. & Fun, H. K. (2006). *Acta Cryst.* E**62**, o3799–o3801.
Sheldrick, G. M. (1998). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Weissman, D., Poli, G., Bousseau, A. & Fauci, A. S. (1993). *Proc. Natl Acad. Sci. USA*, **90**, 2537–2541.