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5-(Phenylsulfonyl)perhydrothiazolo[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 = 0.036
wR factor = 0.106
Data-to-parameter ratio = 48.8

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

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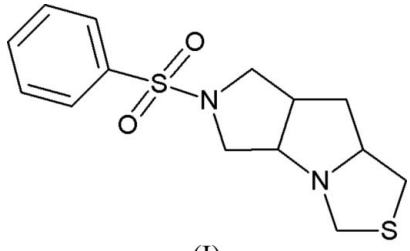
All the five-membered rings of the title molecule, $C_{14}H_{18}N_2O_2S_2$, adopt envelope conformations. Intermolecular C—H···O hydrogen bonds link the molecules into a two-dimensional network parallel to the *ab* plane.

Comment

Pyrrolopyrrole compounds exbhit 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 5-oxo-hexahydropyrrolo[3,2-*b*]pyrrole ring system (Borthwick *et al.*, 2000). Pyrrolothiazole derivatives show antileukemic 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). They also inhibit cytokine-dependent induction of human immunodeficiency virus (HIV) expression in chronically infected promonocytic cells (Weissman *et al.*, 1993). We report here the structure of the title compound, (I).



(I)

The molecular structure of (I) is illustrated in Fig. 1. All N—Csp^3 bond lengths (Table 1) are comparable to the reported mean value of 1.469 (14) Å (Allen *et al.*, 1987) except the N2—C8 bond which is shorter in length. Atom N1 is slightly out of the plane [deviation = 0.356 (1) Å] defined by atoms S2, C1 and C4, indicating a slight degree of pyramidalization. The sum of the bond angles around the atom N2 (329.7°) indicates sp^3 hybridization.

The thiazolidine ring and the two pyrrolidine rings (N1/C1–C4 and N2/C3/C2/C5/C6) adopt envelope conformations, with atoms N2, N1 and C6 deviating from the S1/C6–C8, C1–C4 and N2/C2/C3/C5 planes by 0.541 (1), 0.602 (1) and 0.586 (1) Å, respectively. The Cremer & Pople (1975) puckering parameters q_2 and φ are 0.381 (1) Å and 284.3 (1)° for the thiazolidine ring, 0.406 (1) Å and 358.1 (1)° for the pyrrolidine ring (N1/C1–C4), and 0.386 (1) Å and 329.0 (1)° for the pyrrolidine ring (N2/C3/C2/C5/C6).

As seen in Fig. 2, molecules translated by one unit along the *a* axis are linked by intermolecular C1—H1*A*···O1^{i} hydrogen

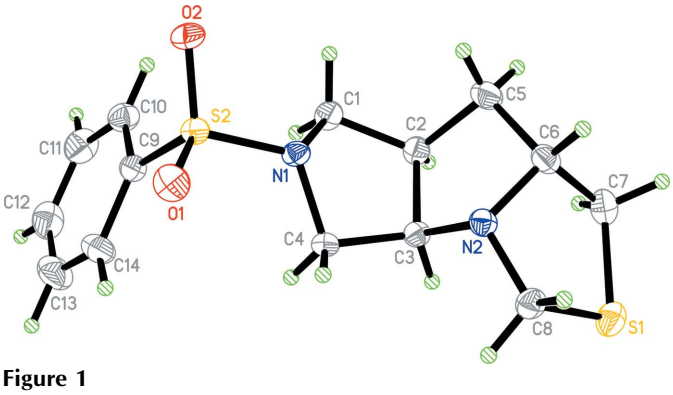


Figure 1
A view of (I), showing the atomic numbering. Displacement ellipsoids are drawn at the 60% probability level.

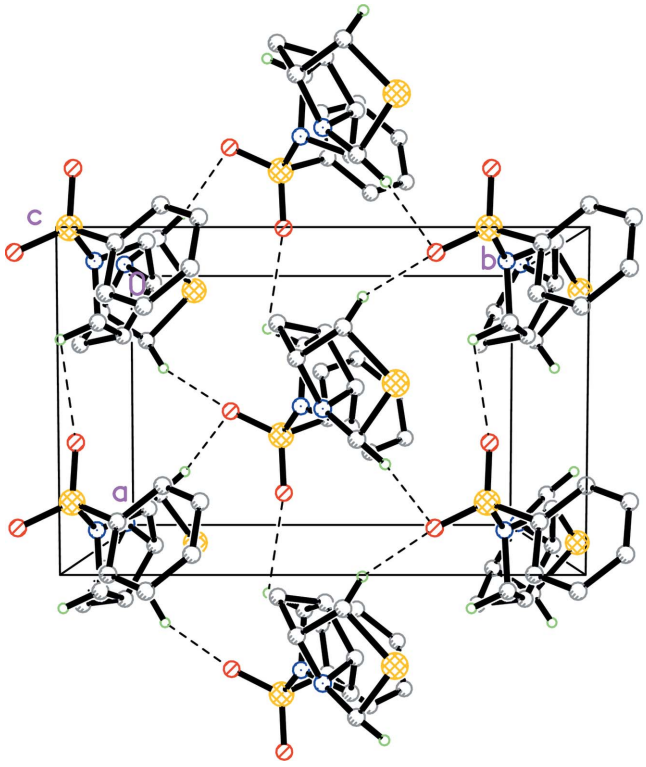


Figure 2
View of a hydrogen-bonded (dashed lines) sheet in (I). Only the H atoms involved in hydrogen bonding are shown.

bonds (see Table 1 for details and symmetry code), forming a chain. Glide-related molecules in adjacent chains are connected *via* C7—H7*B*···O2^{ii} and C8—H8*A*···O2^{iii} interactions (Table 1), generating a two-dimensional network parallel to the *ab* plane.

Experimental

A solution of *N*-allyl-*N*-(2-oxoethyl)benzenesulfonamide (1 mmol) and thiazolidine-4-carboxylic acid (1.2 mmol) in dry toluene (30 ml) was refluxed for 3 h. After completion of the reaction, the solvent was evaporated under vacuum and the residue was chromatographed using a hexane–ethyl acetate (9:1) mixture, to yield the title compound. It was recrystallized from ethyl acetate.

Crystal data

$C_{14}H_{18}N_2O_2S_2$
M_r = 310.42
Monoclinic, $P2_1/n$
a = 6.5439 (1) Å
b = 9.8794 (1) Å
c = 23.0360 (3) Å
β = 97.451 (1)°
V = 1476.70 (3) Å^3
Z = 4
D_x = 1.396 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 100.0 (1) K
Block, colorless
0.39 × 0.33 × 0.26 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.845, T_{max} = 0.912
71353 measured reflections
8831 independent reflections
6960 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 39.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.106
S = 1.07
8831 reflections
181 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.056P)^2 + 0.1806P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C7	1.8210 (10)	N1—C4	1.4762 (10)
S1—C8	1.8665 (8)	N1—C1	1.4773 (9)
S2—O1	1.4346 (6)	N2—C8	1.4348 (10)
S2—O2	1.4376 (6)	N2—C6	1.4670 (10)
S2—N1	1.6253 (7)	N2—C3	1.4789 (10)
S2—C9	1.7651 (8)		
C4—N1—C1	106.58 (6)	C8—N2—C6	108.69 (6)
C4—N1—S2	119.24 (5)	C8—N2—C3	114.18 (6)
C1—N1—S2	118.23 (5)	C6—N2—C3	106.80 (6)

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1*A*···O1^{i}	0.99	2.50	2.9355 (9)	106
C7—H7*B*···O2^{ii}	0.99	2.38	3.3247 (11)	158
C8—H8*A*···O2^{iii}	0.99	2.54	3.5031 (10)	165

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

H atoms were positioned geometrically (C—H = 0.95–1.00 Å) and were treated as riding on their parent C atoms, with 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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