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2-(4-Bromophenyl)-5-(phenylsulfonyl)perhydrothiazolo[3,4-*a*]pyrrolo[4,5-*c*]pyrrole

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.107
Data-to-parameter ratio = 19.4

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

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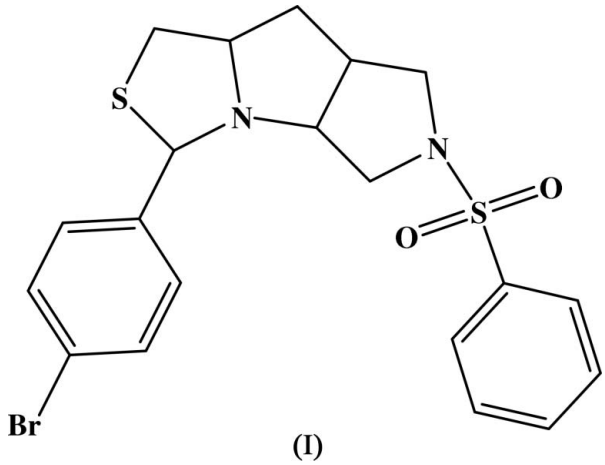
The two pyrrolizidine rings and the thiazolidine ring in the title compound, $C_{20}H_{21}BrN_2O_2S_2$, adopt twist conformations. The molecular conformation is stabilized by weak C—H···S and C—H···O intramolecular interactions. The crystal packing is stabilized by C—H···O and C—H···π intermolecular interactions.

Comment

The structural and therapeutic diversity of small heterocycles has fascinated organic and medicinal chemists. Pyrrolidine and thiazolidine derivatives have gained much importance in the pharmaceutical industry due to their high medicinal value. Pyrrolidine derivatives are useful in preventing and treating rheumatoid arthritis, asthma, allergies, rhinitis and related diseases, as they inhibit the production of prostaglandin E2 and intracellular phospholipase A2 (Mitsuaki *et al.*, 1997). Thiazolidine and its derivatives have biological importance, such as antiradiation, anti-oxidant, anti-amoebic, antidiabetic and anti-inflammatory agents (Eswaramoorthy *et al.*, 1991). We present here the crystal structure of the title compound, (I).



(I)

In compound (I), the sums of the bond angles around atoms N1 (353.1°) and N2 (330.6°) indicate sp^2 and sp^3 hybridization, respectively. The thiazolidine ring and the two pyrrolidine rings (N1/C1–C4 and N2/C3/C2/C5/C6) adopt twist conformations, with the pseudo-twofold axes passing through the N2—C8, C1—C2 and N2—C3 bonds, respectively. The puckering parameters (Cremer & Pople, 1975) and the smallest displacement asymmetry parameters (Nardelli, 1983) are, for the thiazolidine ring, q_2 = 0.391 (2) Å, φ = 306.0 (3)° and $\Delta_2(C_7)$ = 7.9 (2); for the pyrrolidine ring (N1/C1–C4), q_2 =

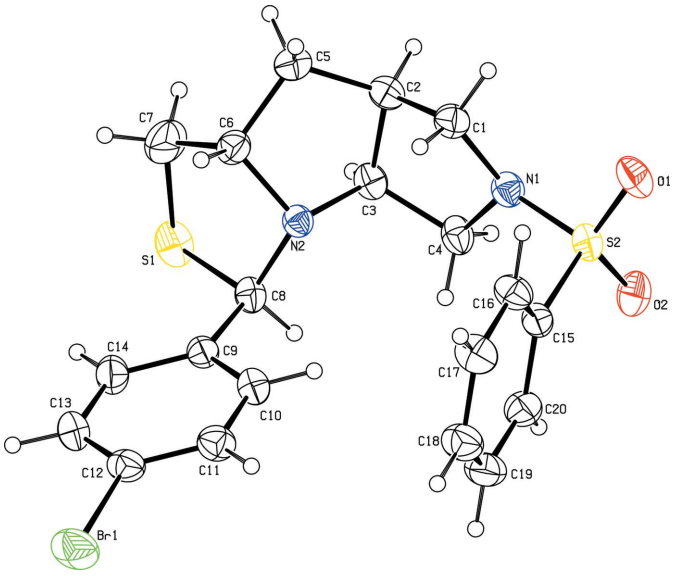


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids.

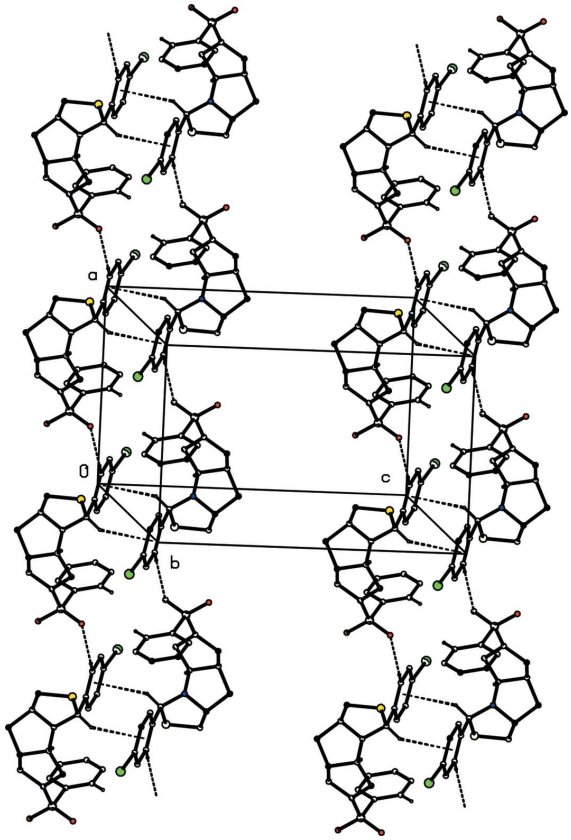


Figure 2
The molecular packing of (I), viewed somewhat inclined to the *b* axis. Hydrogen bonds are shown as dashed lines.

0.356 (2) Å, φ = 240.9 (4)° and $\Delta_2(C_4)$ = 5.9 (2); and for the pyrrolidine ring N2/C3/C2/C5/C6, q_2 = 0.411 (2) Å, φ = 18.6 (3)° and $\Delta_2(C_5)$ = 1.4 (2).

The molecular conformation of (I) is stabilized by weak C—H···S and C—H···O intramolecular interactions. The crystal packing is stabilized by C—H···O and C—H···π intermolecular interactions (Table 2).

Experimental

A solution of *N*-allyl-*N*-(2-oxoethyl)benzenesulfonamide (1 mmol) and 2-(*p*-bromophenyl)thiazolidine-4-carboxylic acid (1.2 mmol) in dry toluene (20 ml) was refluxed for 4 h. After completion of the reaction, the solvent was evaporated under vacuum and the residue was chromatographed using a hexane–ethyl acetate mixture (9:1), to yield the title compound. It was recrystallized from ethyl acetate.

Crystal data

$C_{20}H_{21}BrN_2O_2S_2$
M_r = 465.42
Monoclinic, $P2_1/c$
a = 10.5926 (6) Å
b = 11.4144 (6) Å
c = 16.6896 (9) Å
β = 94.102 (1)°
V = 2012.74 (19) Å^3
Z = 4
D_x = 1.536 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 2.27 mm^{-1}
T = 293 (2) K
Block, colourless
0.24 × 0.22 × 0.21 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
22489 measured reflections
4728 independent reflections
3843 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 28.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.107
S = 0.95
4728 reflections
244 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 1.1249P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.07 e Å^{-3}
$\Delta\rho_{min}$ = −0.70 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1—C12	1.890 (2)	S2—C15	1.768 (2)
S1—C7	1.801 (3)	N1—C4	1.478 (3)
S1—C8	1.852 (2)	N1—C1	1.473 (3)
S2—O2	1.431 (2)	N2—C8	1.439 (2)
S2—O1	1.433 (2)	N2—C3	1.472 (2)
S2—N1	1.613 (2)	N2—C6	1.475 (3)
C7—S1—C8	91.3 (1)	C4—N1—C1	111.5 (2)
O2—S2—O1	120.5 (1)	C4—N1—S2	119.7 (2)
O2—S2—N1	106.2 (1)	C1—N1—S2	121.9 (2)
O1—S2—N1	107.0 (1)	C8—N2—C3	115.5 (2)
O2—S2—C15	107.2 (1)	C8—N2—C6	109.0 (2)
O1—S2—C15	107.3 (1)	C3—N2—C6	106.1 (2)
N1—S2—C15	108.2 (1)		
C15—S2—N1—C4	75.7 (2)	N2—C8—C9—C10	54.6 (2)
C15—S2—N1—C1	−72.6 (2)	S1—C8—C9—C10	174.4 (2)
N2—C8—C9—C14	−128.2 (2)	N1—S2—C15—C16	86.7 (2)
S1—C8—C9—C14	−8.4 (3)	N1—S2—C15—C20	−93.7 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···S1	0.98	2.86	3.237 (2)	104
C14—H14···S1	0.93	2.65	3.099 (2)	110
C14—H14···O2^{i}	0.93	2.56	3.403 (2)	151
C20—H20···O2	0.93	2.57	2.920 (2)	103
C8—H8···$Cg1^{ii}$	0.97	2.77	3.627 (2)	146

Symmetry codes: (i) $x-1, y, z$; (ii) $-x, -y+1, -z$. $Cg1$ is the centroid of the C9–C14 ring.

H atoms were positioned geometrically and allowed to ride on their parent C atoms, with C—H distances constrained in the range 0.93–0.98 Å and with U_{iso}(H) = 1.2 U_{eq}(C). The highest peak is located 0.83 Å from atom Br1.

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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References

Bruker (2001). *SMART* (Version. 5.625/NT/2000) and *SAINT* (Version 6.28a). Bruker AXS Inc., Madison, Wisconsin, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Eswaramoorthy, S., Ponnuswamy, M. N., Raju, K. S. & Czerwinski, E. W. (1991). *Acta Cryst.* C**47**, 171–173.
Mitsuaki, O., Toshiyuki, K., Fumihiko, W. & Koaru, S. (1997). *Chem. Abstr.* **17**, 22529u, 126, 578.
Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.