Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4′-(4-Methoxyphenyl)-1′-methyl-5-diphenyl-5,6,7,8,9,10-hexahydro-1,3-cycloheptapyrimidino-[2,3-*b*]thiazole-2-spiro-3′-pyrrolidine-2′-spiro-3″-1*H*-indole-2″,3(2*H*,3″*H*)-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.048
wR factor = 0.139
Data-to-parameter ratio = 18.3

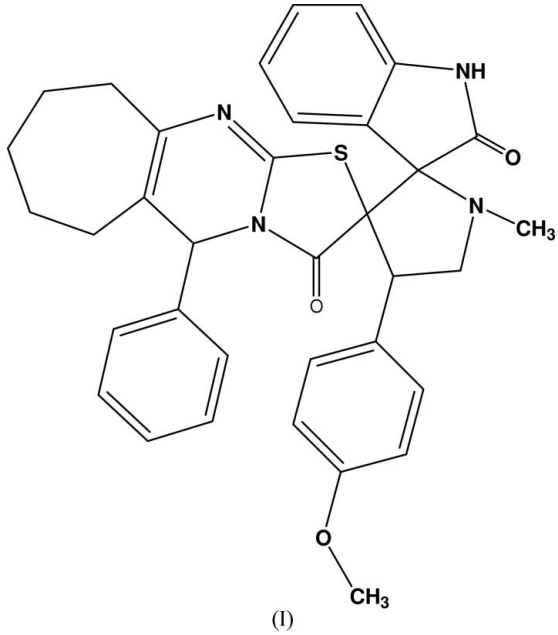
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 22 November 2006
Accepted 24 November 2006

The pyrrolidine ring in the title compound, $C_{35}H_{34}N_4O_3S$, adopts an envelope conformation. The thiazolidine ring is planar. The molecule is stabilized by weak C—H···O interactions, and the crystal packing is stabilized by intermolecular N—H···N and C—H···O interactions.

Comment

The pyrrolidine alkaloids mimicking the structures of pentose with nitrogen in the ring are known to be inhibitors of glycosidases (Mizushina *et al.*, 2003). Thaizolidine derivatives possess antidiabetic and adipogenic properties (Norisada *et al.*, 2004). Indole and its derivatives represent one of the most active classes of compounds, possessing a wide spectrum of biological activity (Hiremath *et al.*, 1988). In view of the above biological importance, we have undertaken the structure determination of the title compound, (I), by X-ray diffraction (Fig. 1).



(I)

The molecular geometry of (I) is comparable to those of related structures reported earlier (Gayathri *et al.*, 2005, 2006*a*,*b*). The sums of the bond angles around N1 (337.3°) and N3 (359.8°) indicate sp^3- and sp^2-hybridization, respectively.

The methyl atom C5 lies 0.561 (4) Å below the plane of atoms C1–C4 and atom O3 lies 0.032 (2) Å below the plane of the benzene ring C29–C34. Atom O1 deviates by 0.061 (1) Å from the plane of the five-membered ring in the indane group. The six-membered ring N3/C22/N4/C21/C15/C14 is slightly non-planar, with atom C14 deviating by 0.179 (2) Å from the

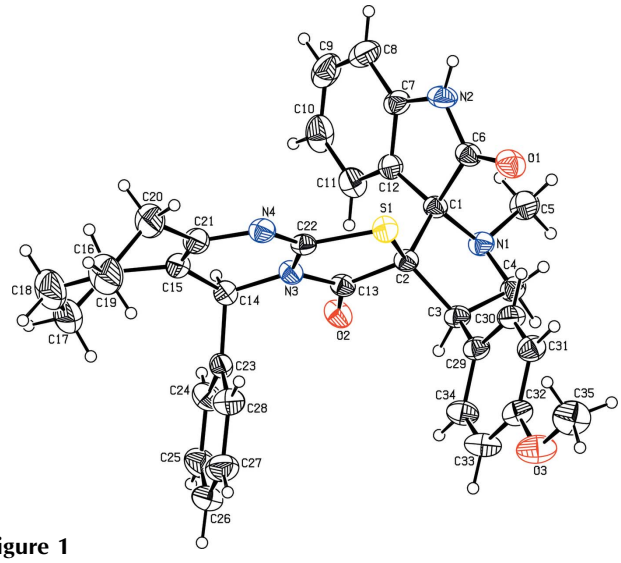


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids.

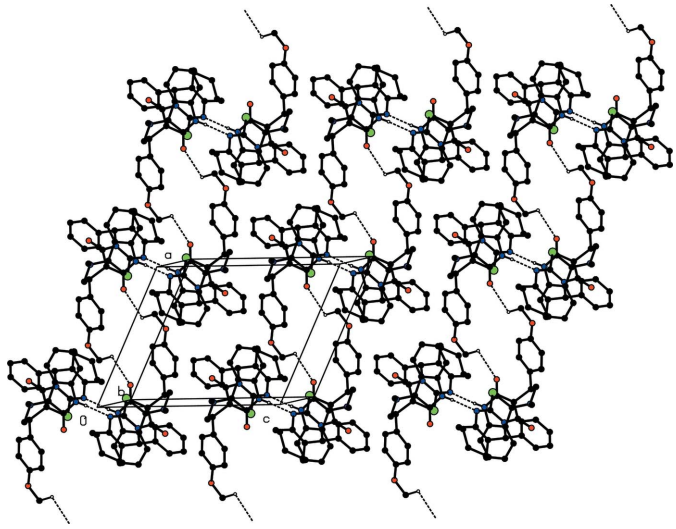


Figure 2
The packing of (I), viewed approximately down the *b* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

plane of the other atoms, and is thus an envelope, because of the phenyl (C23–C28) substituent at atom C14. The dihedral angle between the two benzene rings (C23–C28 and C29–C34) is 19.0 (1)° and that between the five- (C1/C6/N2/C7/C12) and six-membered (C7–C12) rings in the indane system is 5.2 (1)°. The seven-membered ring has a chair conformation.

The pyrrolidine ring adopts an envelope conformation, with atom N1 deivating by 0.593 (1) Å from the plane of the other atoms. The puckering parameters (Cremer & Pople, 1975) and the smallest displacement asymmetry parameter (Nardelli, 1983) for the pyrrolidine ring are q_2 = 0.409 (2) Å, φ = 352.0 (2)° and Δ_s(N1) = 7.9 (2)°.

The molecule is stabilized by weak C—H···O intramolecular interactions. The crystal packing is stabilized by N—H···N and C—H···O intermolecular interactions (Table 1). The N2—H2···N4^{ii} intermolecular interaction generates a centrosymmetric dimer of $R_2^2(16)$ motif centred at (0, 0, 0) (Fig. 2). The N—H···N hydrogen-bonded dimers are linked along the *a* axis by the paired C35—H35*C*···O1^{i} intermolecular interactions which generate a centrosymmetric dimer of $R_2^2(24)$ motif centred at ($\frac{1}{2}$, 0, 0). The symmetry codes (i) and (ii) are given in Table 1.

Experimental

A mixture of isatin (1.2 mmol), sarcosine (1.2 mmol) and 5-phenyl-2-(*p*-methoxy)phenylmethylene-5,6,7,8,9,10-hexahydrocyclohepta[*d*]-thiazolo[3,2-*a*]pyrimidin-3(2*H*)-one (1 mmol) in methanol–dioxane (1:1, 20 ml) was refluxed until the disappearance of the starting materials (5.5 h) as shown by thin-layer chromatography. The reaction mixture was then concentrated *in vacuo* and extracted with water (50 ml) and dichloromethane (50 ml). The organic layer was washed with brine, dried with anhydrous sodium sulfate and concentrated *in vacuo*. The residue was purified by column chromatography (silica gel, 100–200 mesh), eluting with a hexane–ethyl acetate (8:2) mixture, to give the title compound, which was recrystallized from methanol by slow evaporation.

Crystal data

$C_{35}H_{34}N_4O_3S$
M_r = 590.72
Triclinic, $P\overline{1}$
a = 11.4338 (7) Å
b = 11.9540 (8) Å
c = 13.5843 (11) Å
α = 70.300 (2)°
β = 65.143 (1)°
γ = 72.179 (1)°
V = 1556.46 (19) Å^3
Z = 2
D_x = 1.260 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 293 (2) K
Block, colourless
0.26 × 0.24 × 0.23 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
18120 measured reflections
7146 independent reflections
5793 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 28.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.139
S = 0.99
7146 reflections
390 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0813P)^2 + 0.2597P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O2	0.98	2.46	2.949 (2)	111
C4—H4*A*···O1	0.97	2.47	3.044 (2)	118
C11—H11···O2	0.93	2.41	3.016 (2)	123
C35—H35*C*···O1^{i}	0.96	2.58	2.998 (3)	106
N2—H2···N4^{ii}	0.86	2.10	2.951 (2)	169

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

All H atoms were refined using a riding model, with C—H distances of 0.93 Å for aromatic H, 0.98 Å for methine H, 0.97 Å for methylene H and 0.96 Å for methyl H, and N—H = 0.86 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N), or 1.5U_{eq}(C) for methyl groups.

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).

The Department of Science and Technology (DST–FIST), Government of India, is acknowledged by DG and DV for providing facilities to the department. DV thanks DST, India, for a major research project and DG thanks CSIR, India, for the award of a Senior Research Fellowship.

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