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1′-Methyl-5-diphenyl-4′-*p*-tolyl-5,6,7,8,9,10-hexahydro-1,3-cyclooctapyrimidino[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 = 0.037
wR factor = 0.104
Data-to-parameter ratio = 14.0

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

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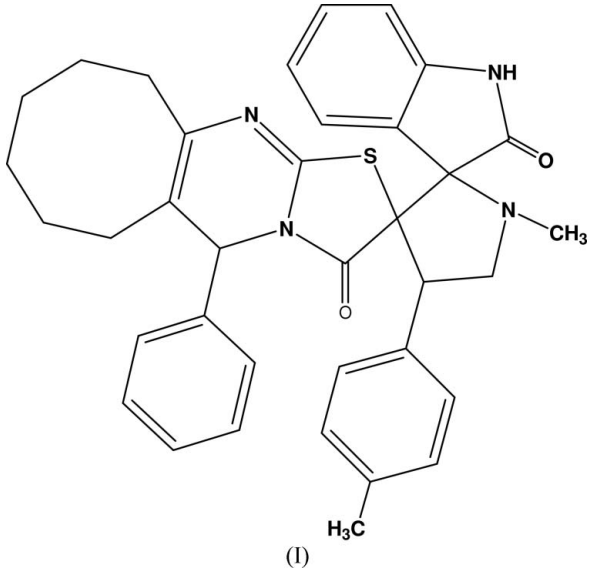
The pyrrolidine ring in the title compound, $C_{36}H_{36}N_4O_2S$, adopts an envelope conformation. The thiazolidine ring adopts a twisted conformation, with a pseudo-twofold axis passing through the N atom and the opposite S—C bond. The crystal packing is stabilized by N—H···N intermolecular interactions.

Comment

Pyrrolidine, a basic intermediate used in a wide range of applications in organic synthesis, has gained much attention in the pharmacological industry for its medicinal value. Derivatives of pyrrolidine are found to have anticonvulsant (Obniska *et al.*, 2002), antimicrobial and antifungal activity against various pathogens, except Bacillus subtilis (Amal Raj *et al.*, 2003). Oxindole derivatives help to treat and prevent diabetic complications arising from elevated levels of sorbitol and act as aldose reductase inhibitors (Rajeswaran *et al.*, 1999). Thiazolidines and their derivatives have diverse biological importance with properties such as local anaesthetic, anti-seizure, antitubercular, antibacterial, anti-amoebic, anti-diabetic and anti-inflammatory (Chaurasia, 1971; Zimenkovsky *et al.*, 1999), and anti-oxidant (Rachinskii, 1964). The spiro ring system is a frequently encountered structural motif in many pharmacologically relevant alkaloids.



(I)

As spiropyrrolidine compounds are of great medicinal importance, we have undertaken the three-dimensional structure determination of the title compound, (I), by X-ray diffraction (Fig. 1).

The molecular geometry of (I) is comparable with that of a similar structure reported earlier (Gayathri *et al.*, 2005). The sums of the bond angles around N1 (337.0°) and N3 (359.4°) indicate sp^3 and sp^2 hybridization, respectively.

The methyl atom C5 lies 0.542 (2) Å below the plane of atoms C1–C4 and atom C36 lies 0.094 (3) Å above the plane of the benzene ring C30–C35. The five- (C1/C6/N2/C7/C12) and six- (C7–C12) membered rings in the indane group are planar, with a dihedral angle of 5.8 (1)° between these rings. Atom O1 deviates by 0.073 (1) Å from the plane of the five-membered ring in the indane group. The six-membered ring N3/C14/C21/C28/N4/C29 is slightly non-planar, with atom C14 deviating by 0.230 (2) Å from the plane of the other atoms, because of the phenyl (C15–C20) substituent at atom C14. The dihedral angle between the two benzene rings (C15–C20 and C30–C35) is 19.6 (1)°. The pyrrolidine ring adopts an envelope conformation, with atom N1 deviating by 0.588 (1) Å from the mean plane of atoms C1–C4, and the thiazolidine ring adopts a twisted conformation with a pseudo-twofold axis passing through N3 and the S1—C2 bond.

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 = 355.3 (3)° and $\Delta_s(N_1)$ = 5.0 (2), and for the thiazolidine ring are q_2 = 0.084 (1) Å, φ = 18.5 (11)° and $\Delta_2(N_3)$ = 0.9 (2).

The molecule of (I) is stabilized by weak C—H···O intramolecular interactions. The crystal packing is stabilized by N—H···N intermolecular interactions, generating a centrosymmetric dimer of $R_2^2(16)$ motif (Bernstein *et al.*, 1995) (Table 1 and Fig. 2).

These results, except for the different conformation of the thiazolidine ring, are very similar to those for the closely related compound reported in the preceding paper (Gayathri *et al.*, 2006).

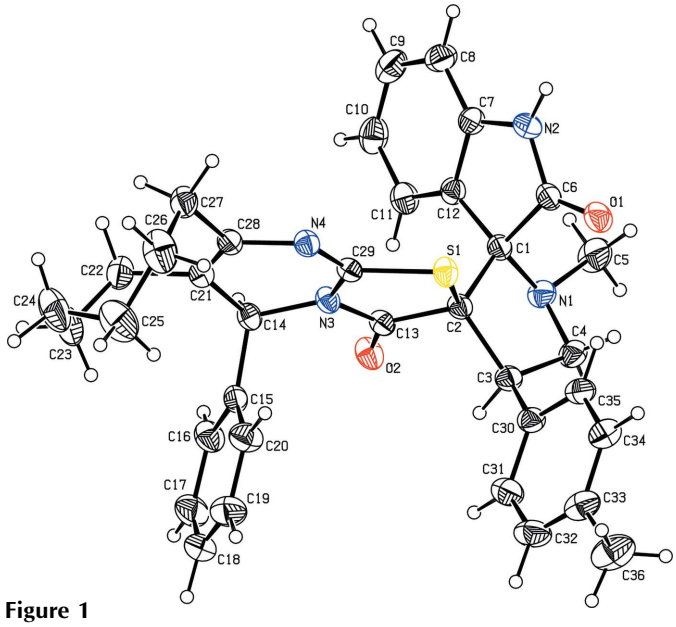


Figure 1
The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

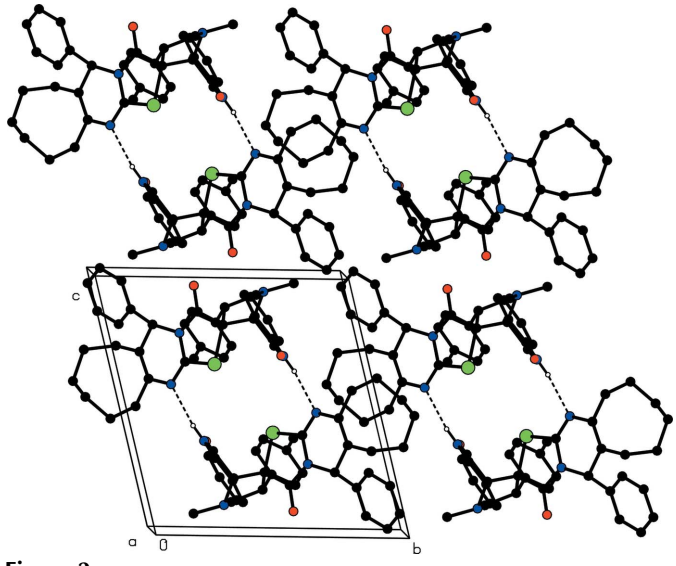


Figure 2
The packing of (I), viewed approximately down the *a* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

Experimental

A mixture of isatin (1.2 mmol), sarcosine (1.2 mmol) and 5-phenyl-2-(*p*-methyl)phenylmethylene-5,6,7,8,9,10,11-heptahydrocycloocta-[*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 (4 h) as shown by thin-layer chromatography analysis. 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) with a hexane–ethyl acetate (8:2) mixture to give the title compound, which was recrystallized from methanol by slow evaporation.

Crystal data

$C_{36}H_{36}N_4O_2S$
M_r = 588.75
Triclinic, $P\overline{1}$
a = 11.3531 (8) Å
b = 12.1973 (9) Å
c = 13.2117 (9) Å
α = 94.181 (1)°
β = 111.683 (1)°
γ = 111.377 (1)°
V = 1536.88 (19) Å^3
Z = 2
D_x = 1.272 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 293 (2) K
Block, colourless
0.25 × 0.22 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
14952 measured reflections
5422 independent reflections
4781 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.104
S = 1.03
5422 reflections
388 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0549P)^2 + 0.3799P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···N4^{i}	0.86	2.13	2.975 (2)	170
C3—H3···O2	0.98	2.43	2.932 (2)	111
C4—H4A···O1	0.97	2.50	3.069 (2)	117
C11—H11···O2	0.93	2.49	3.097 (2)	123
C35—H35···O1	0.93	2.58	3.426 (3)	152

Symmetry code: (i) $-x+2, -y+1, -z+1$.

All H atoms were treated using a riding model, with C—H = 0.93 for aromatic H, 0.98 for methine H, 0.97 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).

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