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1′-Methyl-5-diphenyl-4′-*p*-tolyl-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 = 0.041
wR factor = 0.107
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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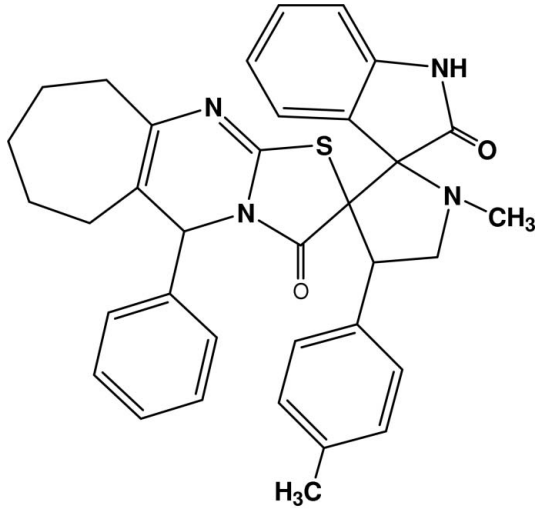
The pyrrolidine ring in the title compound, $C_{35}H_{34}N_4O_2S$, adopts an envelope conformation, with the N atom deviating by 0.586 (1) Å from the plane of the other atoms. The molecule is stabilized by weak C—H···O interactions and the crystal packing is stabilized by N—H···N intermolecular interactions, generating a dimer with an $R_2^2(16)$ motif.

Comment

Heterocyclic compounds, especially five- and six-membered rings, have occupied an important place among organic compounds for their great pharmaceutical importance, especially pyrrolidine and thiazolidine derivatives. Synthetic spiro-pyrrolidine derivatives show activity against the aldose reductase enzyme which controls influenza (Stylianakis *et al.*, 2003). Indole and its derivatives represent one of the most active classes of compounds, possessing a wide range of biological activities (Hiremath *et al.*, 1988). Owing to the ease of substitution and modifications at several positions, many derivatives of pyrrolidine have been synthesized with different properties (Baldwin *et al.*, 1994). In view of the importance of these compounds, we have undertaken the structure determination of the title compound, (I) (Fig. 1).



(I)

The molecular geometry of (I) is comparable with that of a related structure reported earlier (Gayathri *et al.*, 2005). The sums of the bond angles around N1 (337.2°) and N3 (359.9°) indicate sp^3 and sp^2 hybridization, respectively.

The methyl atom C5 lies 0.545 (2) Å below the plane of atoms C1–C4, and atom C35 lies 0.087 (3) Å above the plane of the benzene ring C29–C34. The five- (C1/C6/N2/C7/C12) and six- (C7–C12) membered rings in the indane group are

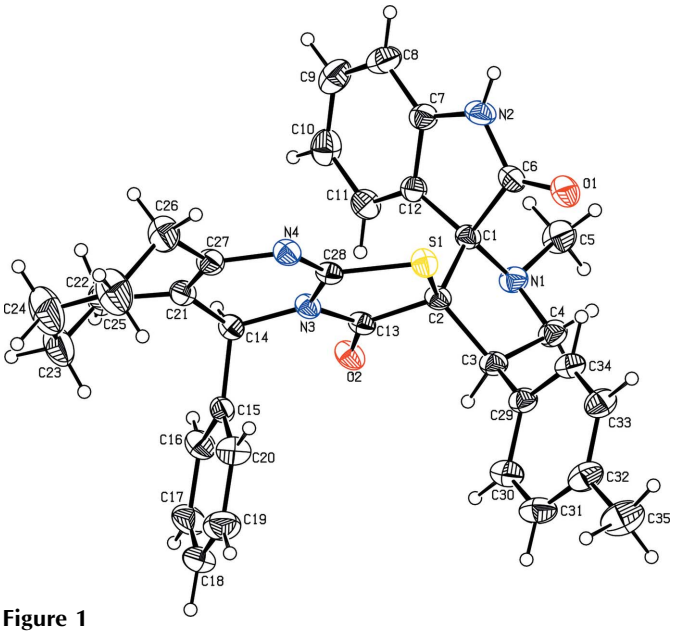


Figure 1
The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

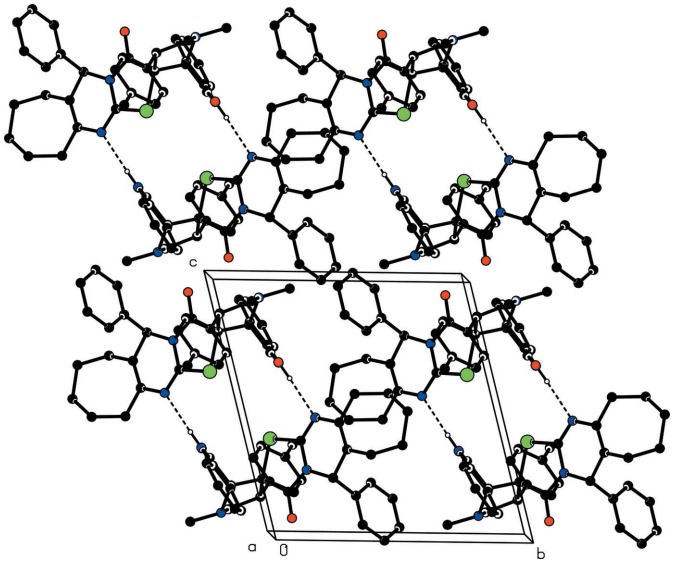


Figure 2
The packing of (I), viewed approximately down the *a* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

planar, with a dihedral angle of 6.3 (1)° between these rings. Atom O1 deviates by 0.105 (1) Å from the plane of the five-membered ring in the indane group. The six-membered ring N3/C14/C21/C27/N4/C28 is slightly non-planar, with atom C14 deviating by 0.159 (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 C29–C34) is 21.9 (1)°.

The pyrrolidine ring adopts an envelope conformation, with atom N1 deviating by 0.586 (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 = 0.406 (2) Å, φ = 353.4 (3)° and $\Delta_s(N_1)$ = 6.7 (2).

The molecule is stabilized by weak C—H···O intramolecular interactions and 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).

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-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 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) eluted with a hexane–ethyl acetate (8:2) mixture to give the title compound, which was recrystallized from methanol by slow evaporation.

Crystal data

$C_{35}H_{34}N_4O_2S$
M_r = 574.72
Triclinic, $P\overline{1}$
a = 11.3427 (8) Å
b = 12.0148 (9) Å
c = 13.0102 (10) Å
α = 95.604 (1)°
β = 110.887 (1)°
γ = 109.567 (1)°
V = 1512.67 (19) Å^3
Z = 2
D_x = 1.262 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 293 (2) K
Block, colourless
0.24 × 0.21 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
14702 measured reflections
5317 independent reflections
4625 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.107
S = 1.04
5317 reflections
379 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 0.4411P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 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
C3—H3···O2	0.98	2.45	2.944 (2)	111
C4—H4A···O1	0.97	2.50	3.070 (2)	118
C11—H11···O2	0.93	2.46	3.045 (3)	121
N2—H2···N4^{i}	0.86	2.13	2.988 (2)	177

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

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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