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Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.053
wR factor = 0.141
Data-to-parameter ratio = 17.8

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

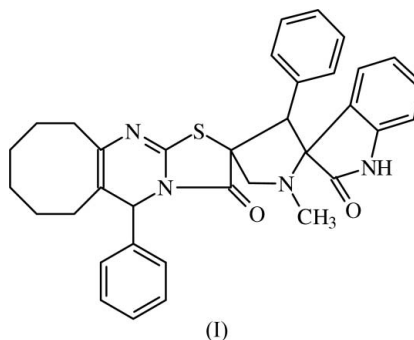
1'-Methyl-3',5''-diphenyl-6'',7'',8'',9'',10'',11''-hexahydro-1H-indole-3-spiro-2'-pyrrolidine-4'-spiro-2''-5''H-cycloocteno[1,2-d]thiazolo[3,2-a]pyrimidine-2,3''(3H,2''H)-dione

In the title compound, $\text{C}_{35}\text{H}_{34}\text{N}_4\text{O}_2\text{S}$, the five-membered pyrrolidine and thiazole rings adopt envelope and twist conformations, respectively. The pyrimidine ring adopts a twist-boat conformation. The molecular structure is stabilized by $\text{C}-\text{H}\cdots\text{O}$ and $\pi-\pi$ intramolecular interactions, and strong $\text{N}-\text{H}\cdots\text{N}$ intermolecular hydrogen bonds stabilize the packing, in addition to van der Waals interactions.

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Comment

Heterocyclic compounds, particularly five- and six-membered ring compounds, have occupied a prominent place among various classes of organic compounds for their diverse biological activities. In particular, spiro[indole-pyrrolidine] ring systems acquired a special place in the heterocyclic field because this is a frequently encountered structural motif in many pharmacologically relevant alkaloids (Amal Raj *et al.*, 2003). Spiro-indole derivatives exhibit antibacterial and antifungal properties (Dandia *et al.*, 1993; Joshi *et al.*, 1990). In view of its importance and to obtain more detailed information on the structure and conformation of the molecule, the crystal structure of the title compound, (I), was determined.



The molecular structure of (I) is illustrated in Fig. 1. Selected geometric parameters are presented in Table 1. The geometry is comparable with that of the structure reported by Gayathri *et al.* (2005). In the current structure, a cyclooctenone ring is attached to the pyrimidine ring, whereas in the previous structure a cycloheptanone ring is attached to the pyrimidine ring. Fig. 2. shows a superposition of the pyrrolidine ring of these two structures, using *Qmol* (Gans & Shalloy, 2001); the r.m.s. deviation is 0.008 Å.

The oxindole group is planar, with a maximum deviation of 0.050 (2) Å for atom C29. Atom O1 deviates from the mean plane of this ring system by 0.126 (2) Å. The dihedral angle between the fused six- and five-membered rings of the oxindole group is 4.3 (1)°.

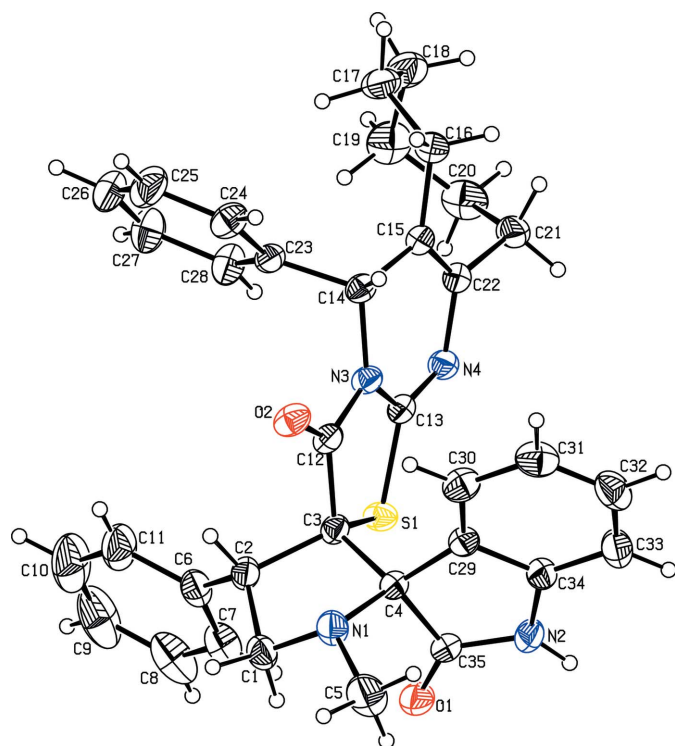


Figure 1

The molecular structure and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

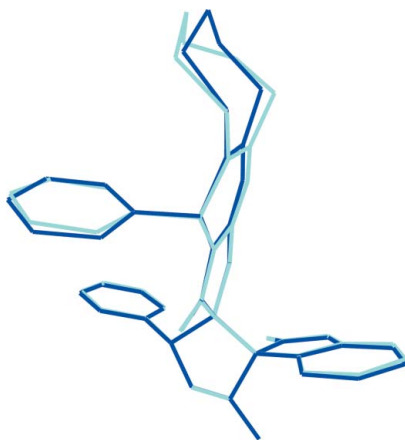


Figure 2

Superposition of (I) (cyan) with the similar reported structure of Gayathri *et al.* (2005) (blue).

The sum of the angles at N1 of the pyrrolidine ring (337.2°) is in accordance with sp^3 -hybridization and that at N3 of the pyrimidine ring (359.5°) is in accordance with sp^2 -hybridization. The methyl group is attached equatorially to the pyrrolidine ring. The pyrrolidine ring adopts an envelope conformation with puckering parameters $q_2 = 0.409$ (2) Å and $\varphi = 150.5$ (3) $^\circ$ (Cremer & Pople, 1975). Atom N1 deviates by 0.586 (2) Å from the least-squares plane through the remaining four atoms (C1–C4) of the ring. The thiazole ring adopts a twist conformation, with puckering parameters $q_2 =$

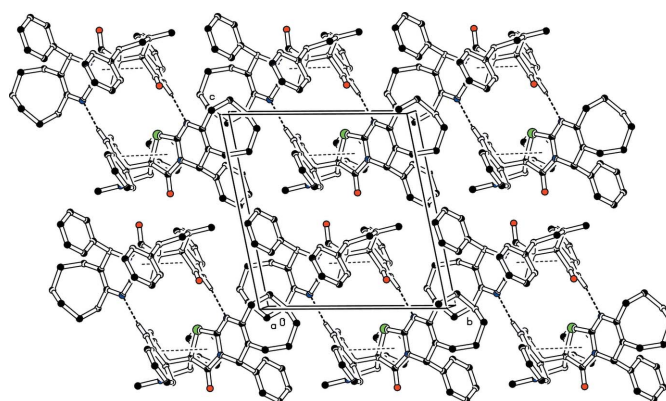


Figure 3

A view of the molecular packing of (I), showing N–H...N hydrogen bonds and π – π intramolecular interactions as dashed lines. H atoms not involved in the interactions shown have been omitted.

0.108 (2) Å, $\varphi = -19.9$ (9) $^\circ$ and the displacement asymmetry parameter $\Delta_2(\text{N3}) = 0.002$ (1) (Nardelli, 1983). The pyrimidine ring adopts a twist-boat conformation, with puckering parameters $q_2 = 0.151$ (2) Å, $q_3 = -0.056$ (2) Å, $Q_T = 0.161$ (2) Å and $\varphi = -85.0$ (7) $^\circ$. The eight-membered ring adopts a boat-chair conformation.

The molecular structure is influenced by C–H...O intramolecular interactions (Table 2) and also a π – π interaction between the thiazole ring (S1/C3/C12/N3/C13) and the five-membered ring of the oxindole group (C4/C29/C34/N2/C35), with a centroid separation of 3.191 (1) Å. In the molecular packing, in addition to van der Waals interactions, N–H...N hydrogen bonds link inversion-related molecules to form $R_2^2(8)$ graph-set dimers (Fig. 2 and Table 2).

Experimental

A mixture of 2-(phenylmethylene)-5-phenyl-5,6,7,8,9,10,11-hepta-hydro-1,3-cyclooctapyrimidino[2,3-*b*]thiazol-3-one (1 mmol), isatin (1.2 mmol) and sarcosine (1.2 mmol) was refluxed in 20 ml of methanol–dioxane solvent (1:1) for 4 h. After completion of the reaction, the residue was chromatographed with a hexane–ethyl acetate mixture (8:2) to obtain the title compound, which was recrystallized from methanol.

Crystal data

$\text{C}_{35}\text{H}_{34}\text{N}_4\text{O}_2\text{S}$
 $M_r = 574.72$
 Triclinic, $P\bar{1}$
 $a = 10.6368$ (8) Å
 $b = 12.1783$ (9) Å
 $c = 13.1467$ (10) Å
 $\alpha = 95.259$ (1) $^\circ$
 $\beta = 109.133$ (1) $^\circ$
 $\gamma = 108.604$ (1) $^\circ$

$V = 1487.79$ (19) Å³
 $Z = 2$
 $D_x = 1.283$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.15$ mm⁻¹
 $T = 293$ (2) K
 Block, colourless
 $0.24 \times 0.21 \times 0.18$ mm

Data collection

Bruker SMART APEX
 diffractometer
 ω scans
 Absorption correction: none
 17008 measured reflections

6766 independent reflections
 5455 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$
 $\theta_{\text{max}} = 28.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.141$
 $S = 1.04$
 6766 reflections
 380 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0721P)^2 + 0.3938P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.34 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.15 \text{ e } \text{\AA}^{-3}$

Table 1

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1–H1B \cdots O1	0.97	2.49	3.069 (3)	118
C2–H2A \cdots O2	0.98	2.42	2.927 (2)	112
C30–H30 \cdots O2	0.93	2.48	3.094 (2)	123
N2–H2 \cdots N4 ⁱ	0.86	2.11	2.965 (2)	172

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

H atoms were positioned geometrically and were treated as riding on their parent atoms, with C–H = 0.93–0.98 \AA , N–H = 0.86 \AA , and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2U_{\text{eq}}(\text{C}, \text{N})$ for other H atoms.

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:

ORTEP-3 (Farrugia, 1997) and PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97 and PARST (Nardelli, 1995).

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