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3-Benzylidene-3′,7′-diphenylcyclohexanespiro-6′-(perhydro-2-thiapyrrolizidine)-5′-spiro-3″-1*H*-indole-2,2″-dione

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In the title compound, $C_{37}H_{32}N_2O_2S$, the two rings of the thiapyrrolizidine residues each adopt an envelope conformation, whereas the cyclohexanone ring adopts a chair conformation. N—H···O hydrogen bonds connect the molecules into centrosymmetric dimers.

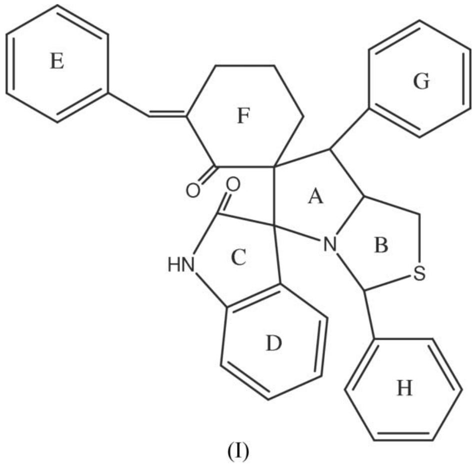
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.051
wR factor = 0.142
Data-to-parameter ratio = 17.0

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

Comment

The spiro-indolepyrrolidine ring system is encountered in many pharmacologically important alkaloids, *e.g.* vincrinstine, vinblastine and spiro-typostatins (Cordel, 1981; Bindra, 1973). Substituted pyrrolidine compounds have been found to have antimicrobial and antifungal activity against various pathogens (Amalraj *et al.*, 2003). Oxindole derivatives are found to be potent aldose reductase inhibitors (ARIs), which help to treat and prevent diabetic complications arising from elevated levels of sorbitol (Rajeswaran *et al.*, 1999). In view of this medicinal importance, and as a continuation of our studies, an X-ray crystallographic analysis of the title compound, (I), has been carried out and the results are presented here.



(I)

Fig. 1 shows a displacement ellipsoid plot of (I), with the atom-numbering scheme. Selected geometric parameters are given in Table 1. Compound (I) consists of a thiapyrrolizidine system (rings *A* and *B*) connected to an oxindole group (rings *C* and *D*) at C2, a benzylidene-cyclohexanone residue (rings *E* and *F*) at C3, and two phenyl rings (*G* and *H*) at C4 and C8.

The pyrrolidine ring (*A*) adopts an envelope conformation, with puckering parameters $q_2 = 0.381$ (2) Å and $\varphi = -75.8$ (2)° (Cremer & Pople, 1975); the displacement asymmetry parameter (Nardelli, 1983) is $\Delta_S(C4) = 0.016$ (1). Ring *B* adopts an envelope conformation, with puckering parameters $q_2 = 0.469$ (2) Å and $\varphi = -74.2$ (2)° and a lowest displacement

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asymmetry parameter Δ_S(S7) = 0.012 (1). The cyclohexanone ring (*F*) adopts a chair conformation.

The crystal structure of (I) is stabilized by intermolecular N—H···O hydrogen bonds, forming centrosymmetric dimers denoted as $R_2^2(8)$ (Bernstein *et al.*, 1995).

Experimental

A mixture of 2,6-bis-acylidine-cyclohaxanone, 2-phenylthiazolidine-4-carboxylic acid and benzaldehyde (1:1:1 mole ratio) in dimethylformamide–toluene (3:1) was refluxed until the disappearance of the starting material, as evidenced by thin-layer chromatography. After completion of the reaction, the solvent was evaporated under vacuum. The resulting crude product was purified by column chromatography (silica gel; hexane–ethyl acetate 9:1). The compound was recrystallized from methanol to yield crystals of (I) (m.p. 507 K).

Crystal data

$C_{37}H_{32}N_2O_2S$
M_r = 568.71
Monoclinic, $P2_1/c$
a = 10.0312 (6) Å
b = 20.8599 (13) Å
c = 14.1305 (8) Å
β = 100.973 (1)°
V = 2902.7 (3) Å^3
Z = 4
D_x = 1.301 Mg m^{-3}
Mo *Kα* radiation
μ = 0.15 mm^{-1}
T = 293 (2) K
Block, colourless
0.35 × 0.24 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: None
17503 measured reflections
6460 independent reflections
4985 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.022
$\theta_{\rm max}$ = 28.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.142
S = 1.02
6460 reflections
379 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0816P)^2 + 0.4571P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ = 0.001
$\Delta\rho_{\rm max}$ = 0.38 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.20 e Å^{-3}

Table 1
Selected torsion angles (°).

C17—C18—C22—C23	−177.4 (2)	C18—C22—C23—C24	134.5 (2)
C19—C18—C22—C23	−2.0 (3)	C3—C4—C29—C34	−93.9 (2)
C18—C22—C23—C28	−45.9 (3)	S7—C8—C35—C36	44.4 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O2	0.98	2.41	2.804 (2)	103
C20—H20*B*···O1	0.97	2.50	3.134 (2)	122
C21—H21*B*···O1	0.97	2.44	2.975 (2)	114
C40—H40···O1	0.93	2.50	3.384 (2)	159
N10—H10···O1$^{\rm i}$	0.86	2.04	2.851 (2)	157
C25—H25···O1$^{\rm ii}$	0.93	2.51	3.236 (3)	135

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

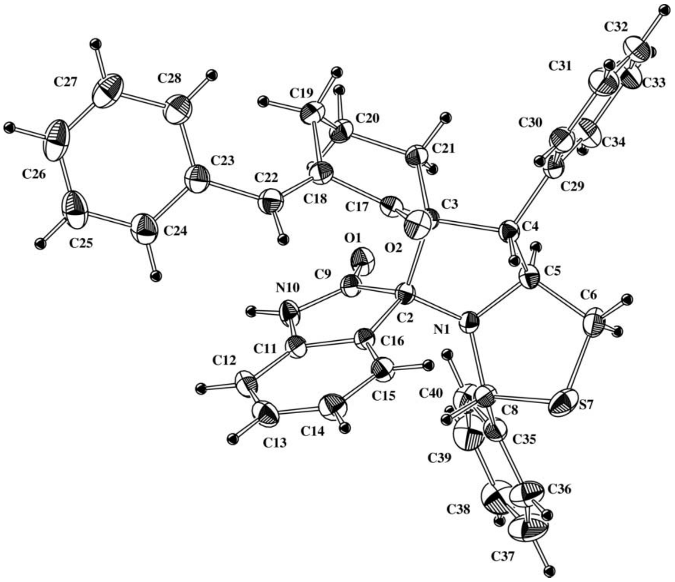

Figure 1
A perspective view of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

All H atoms were positioned geometrically and treated as riding on their parent C atoms, with N—H = 0.86 Å and C—H ranging from 0.93 to 0.98 Å, and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C,N).

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: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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