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1″-Benzyl-5″-benzylidene-1′-methyl-4′-phenyl-1*H*-indole-3-spiro-2′-pyrrolidine-3′-spiro-3″-piperidine-2(3*H*),3″-dione

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The title compound, $C_{36}H_{33}N_3O_2$, was synthesized by the intermolecular [3 + 2]-cycloaddition of azomethine ylide, derived from isatin and sarcosine by a decarboxylative route, and 1-benzyl-3,5-dibenzylidene-piperidin-4-one. There are two spiro junctions in the molecule, which consists of a nearly planar 2-oxindole ring, an envelope pyrrolidine ring and a chair piperidone ring. There is an intermolecular N—H···O hydrogen bond, the N···O distance being 2.835 (3) Å.

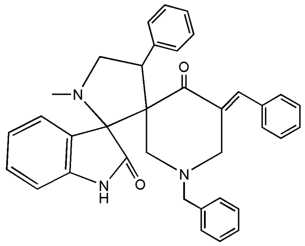
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.063
wR factor = 0.181
Data-to-parameter ratio = 13.8

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

Comment

Spiro compounds represent an important class of naturally occurring substances characterized by highly pronounced biological properties (Kobayashi *et al.*, 1991; James *et al.*, 1991). 1,3-Dipolar cycloaddition reactions are important processes for the construction of spiro compounds (Caramella & Grunanger, 1984). In this paper, the structure of the title compound, (I), is reported.



(I)

There are two spiro junctions in the molecule, which consists of 2-oxindole, pyrrolidine and piperidone rings. The pyrrolidine ring (N2/C7/C6/C5/C8) displays an envelope conformation. The N2/C8/C5/C6 moiety is nearly planar and the mean deviation from this plane is 0.03 (1) Å. Atom C7 is displaced from the N2/C8/C5/C6 plane by 0.64 (2) Å, forming the flap of the envelope. The dihedral angle between the N2/C7/C6 and N2/C8/C5/C6 planes is 43.4 (4)°. The dihedral angle between the C30–C35 phenyl plane and the N2/C8/C5/C6 plane is 63.9 (3)°. The 2-oxindole ring system (C8/C9/N3/C10/C15/C14/C13/C12/C11) is nearly planar and the mean deviation from this plane is 0.03 (1) Å. The dihedral angle between the 2-oxindole plane and the N2/C8/C5/C6 plane is 86.9 (3)°. The piperidone ring has the usual chair conformation. In the crystal structure, there is an intermolecular N—H···O hydrogen bond, and also C—H···O and C—H···N interactions (Fig. 2 and Table 2).

Experimental

A mixture of 1-benzyl-3,5-dibenzylidene-piperidin-4-one (2 mmol), isatin (2 mmol) and sarcosine (2 mmol) was refluxed in methanol

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(80 ml) until the disappearance of the starting material, as evidenced by thin-layer chromatography. After the reaction was complete, the solvent was removed *in vacuo* and the residue was separated by column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give the title compound, (I). 20 mg of (I) was dissolved in 15 ml chloroform, the solution was kept at room temperature for 15 d and natural evaporation gave colorless single crystals of (I), suitable for X-ray analysis. M.p. 487–488 K; IR (KBr, cm^{-1}): 3493 (N—H), 1694, 1682 (C═O); ^{1}H NMR (CDCl$_3$, p.p.m.): 1.86 (1H, *d*), 2.16 (3H, *s*), 2.76–2.81 (1H, *m*), 3.18 (1H, *d*), 3.41 (2H, *m*), 3.42–3.64 (1H, *m*), 3.93 (1H, *d*), 3.94–4.00 (1H, *m*), 4.80–4.86 (1H, *m*), 6.67 (1H, *s*), 6.67–7.37 (19H, *m*); 7.40 (1H, *br*).

Crystal data

$C_{36}H_{33}N_3O_2$
$M_r = 539.65$
Triclinic, $P\overline{1}$
$a = 8.535$ (3) Å
$b = 11.441$ (4) Å
$c = 15.682$ (6) Å
$\alpha = 102.027$ (7)°
$\beta = 92.423$ (7)°
$\gamma = 100.637$ (7)°
$V = 1466.8$ (10) Å^3

$Z = 2$
$D_x = 1.222$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 715 reflections
$\theta = 2.5$–25.5°
$\mu = 0.08$ mm^{-1}
$T = 293$ (2) K
Plate, colorless
0.22 × 0.16 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
7663 measured reflections
5152 independent reflections

2883 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$
$\theta_{\rm max} = 25.0°$
$h = -10 \rightarrow 10$
$k = -13 \rightarrow 8$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.181$
$S = 1.10$
5152 reflections
372 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.084P)^2P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.30$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.25$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C16	1.468 (4)	N3—C10	1.403 (4)
N2—C36	1.467 (4)	O1—C9	1.229 (3)
C1—N1—C2	108.7 (2)	C8—N2—C36	115.4 (3)
C7—N2—C8	107.0 (3)	O1—C9—N3	125.2 (3)
C7—N2—C8—C5	33.3 (3)	C7—C6—C30—C35	−13.2 (5)
N2—C8—C9—N3	−119.4 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···O1$^{\rm i}$	0.86	1.99	2.835 (3)	166
C1—H1*A*···O1	0.97	2.34	2.950 (4)	120
C6—H6···O2	0.98	2.29	2.801 (4)	111
C22—H22···N1	0.93	2.57	2.898 (5)	101
C23—H23···O2	0.93	2.47	2.800 (4)	101

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

All H atoms were included in calculated positions, they were included in the refinement in riding-motion approximation.

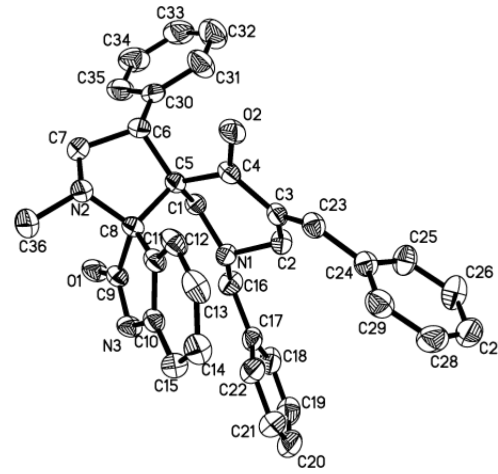

Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids. H atoms have been omitted.

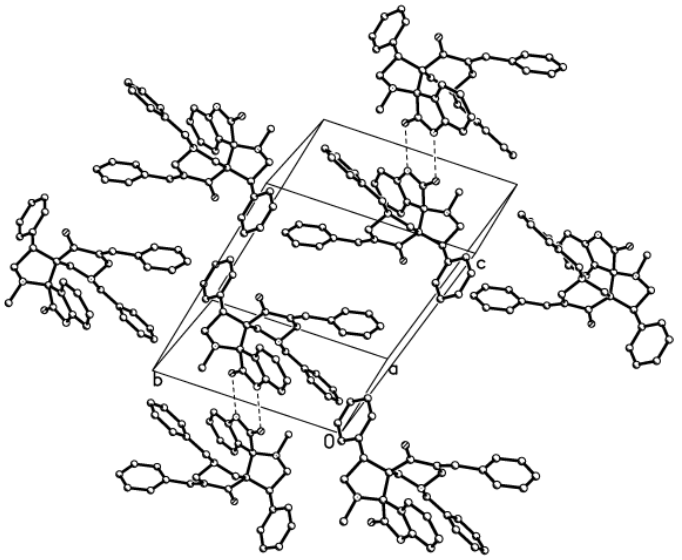

Figure 2
The crystal structure of (I), viewed along the *a* axis. The broken lines indicate hydrogen bonds.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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