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5″-Benzylidene-1′-methyl-4′-phenylindole-3-spiro-2′-pyrrolidine-3′-spiro-3″-piperidine-2(3*H*),4″-dione

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Key indicators

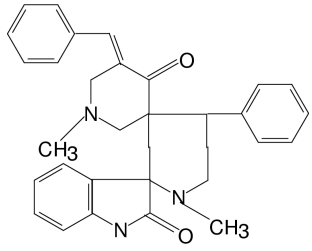
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.053
wR factor = 0.148
Data-to-parameter ratio = 18.2

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

In the title compound, $C_{30}H_{29}N_3O_2$, the dihedral angle between the rings in the indole moiety is 3.3 (1)°. The piperidinone ring adopts a half-chair conformation. The dihedral angle between the pyrrolidine ring and the oxindole moiety is 77.2 (1)°. The packing is stabilized by an N—H···O hydrogen bond and intermolecular C—H···N interactions.

Comment

The spiro ring system containing an indole and a pyrrolidine ring is present in many biologically important and pharmacologically relevant alkaloids (Cordel, 1981). Pyrrolidine compounds are found to be antimicrobial and antifungal (Amal Raj *et al.*, 2003). As a result of the medicinal importance of the compound and also as a continuation of our studies, the X-ray analysis of the title compound, (I), was carried out and the results are presented here.



(I)

Fig. 1 shows a displacement ellipsoid diagram of the molecule with the atomic numbering scheme. Selected geometric

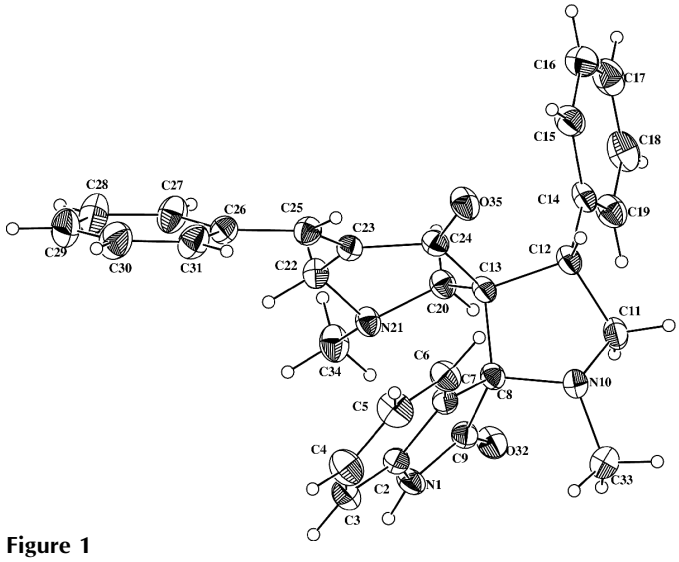


Figure 1
View of (I) (50% probability displacement ellipsoids).

parameters are given in Table 1. The bond lengths in the pyrrolidine moiety are slightly greater than the values reported for similar structures (Jeyabharathi *et al.*, 2001; Seshadri *et al.*, 2003). This may be due to steric forces caused by the bulky substituents on the pyrrolidine moiety. The sum of the angles at atom N10 of the pyrrolidine moiety (339.2°) is in accordance with sp^3 hybridization (Beddoes *et al.*, 1986). The sum of angles at N21 (334.6°) of the piperidinone ring reveal it to be sp^3 hybridized.

Atom O32 is essentially coplanar with the heterocyclic ring to which it is attached, with a deviation of 0.098 (1) Å.

The phenyl ring is attached to the pyrrolidine ring in an equatorial position. The indole moiety (atoms C2–C9/N1) is planar, the dihedral angle between the planes of the heterocyclic and benzene rings being 3.3 (1)°.

The asymmetry parameters (Nardelli, 1995) q_2 = 0.322 (2) Å, φ = −167.5 (4)° and ΔC_2[C13—C8] = 0.0594 (1)° reveal the conformation of the pyrrolidine ring to be half-chair. The piperidinone ring also adopts a half-chair conformation. This is confirmed by the asymmetry parameters q_2 = 0.085 (2) Å, φ = 47.7 (1)°, ΔC_2 [C20] = 0.0494 (7)° and Δ_S[C23] = 0.0459 (5)°.

The molecular structure is influenced by C—H···O intramolecular interactions. In the crystal structure, N1—H1···O32^{i} hydrogen bonds link inversion-related molecules to form dimers (Fig. 2 and Table 2). The crystal structure is also stabilized by C—H···N intermolecular interactions. In addition, symmetry-related molecules are also linked by weak C—H···π intermolecular interactions, such that atom H3 is 2.68 Å from the centroid of the phenyl ring (C14–C19) at ($x-1$, y, z), with a C3—H3···centroid angle of 131° and a C3···centroid distance of 3.359 (2) Å.

Experimental

A mixture of dipolarophile (dibenzylidine-*N*-methylpiperidone), isatin and sarcosine was refluxed in aqueous methanol until the starting materials had disappeared (about 3–4 h), as evidenced by thin-layer chromatography. When the reaction was complete, the solvent was removed *in vacuo* and the residue was chromatographed on silica gel using a hexane–ethyl acetate mixture as eluant and recrystallized from methanol to give (I).

Crystal data

$C_{30}H_{29}N_3O_2$
M_r = 463.56
Monoclinic, $P2_1/n$
a = 10.6889 (7) Å
b = 18.9025 (12) Å
c = 12.5270 (8) Å
β = 95.283 (1)°
V = 2520.3 (3) Å^3
Z = 4
D_x = 1.222 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2146 reflections
θ = 2.4–20.4°
μ = 0.08 mm^{-1}
T = 293 (2) K
Block, colourless
0.21 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
15 727 measured reflections
5738 independent reflections
3798 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 28.0°
$h = -14 \rightarrow 13$
$k = -21 \rightarrow 24$
$l = -13 \rightarrow 16$

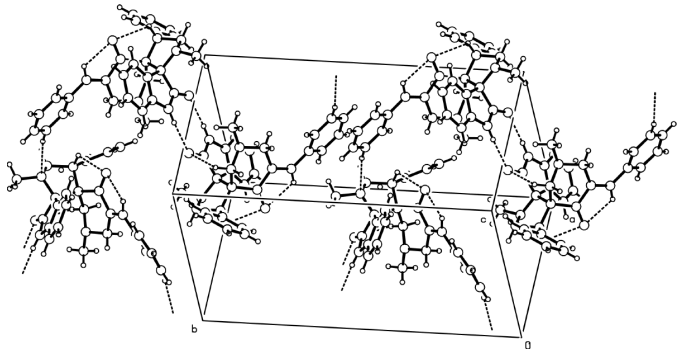


Figure 2
Packing diagram, with hydrogen bonds shown as dashed lines.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.148
S = 0.99
5738 reflections
316 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0826P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N10—C11	1.453 (2)	N10—C33	1.463 (2)
C9—N1—C2	111.7 (1)	C11—N10—C8	107.4 (1)
C11—N10—C33	116.2 (2)	C33—N10—C8	115.6 (1)
C11—C12—C14—C19	27.2 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O35	0.98	2.33	2.804 (2)	109
C20—H20B···O32	0.97	2.35	2.898 (2)	115
C25—H25···O35	0.93	2.41	2.770 (2)	103
N1—H1···O32^{i}	0.86	2.06	2.883 (2)	159
C28—H28···N10^{ii}	0.93	2.61	3.520 (3)	168

Symmetry codes: (i) $1-x, -y, -z$; (ii) $x-\frac{1}{2}, \frac{1}{2}-y, z-\frac{1}{2}$.

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997) and *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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