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3-Benzylidene-1′-methyl-4′-phenylcyclohexanespiro-3′-pyrrolidine-2′-spiro-3″-indoline-2,2″-dione

A. Jeyabharathi,[a] M. N. Ponnuswamy,[a] A. Amal Raj,[b] R. Raghunathan,[b] Ibrahim Abdul Razak,[c] Anwar Usman,[c] Suchada Chantrapromma[c]† and Hoong-Kun Fun[c]*

[a]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, [b]Department of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India, and [c]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia

† Permanent Address, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand.

Correspondence e-mail: hkfun@usm.my

Key indicators

Single-crystal X-ray study
T = 293 K
Mean *σ*(C–C) = 0.005 Å
R factor = 0.057
wR factor = 0.141
Data-to-parameter ratio = 13.4

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

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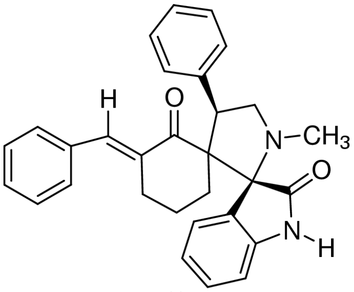
In the title compound, $C_{30}H_{28}N_2O_2$, there are two molecules in the asymmetric unit. In both molecules, the pyrrolidine ring adopts an envelope conformation whereas the conformation of the cyclohexane ring is half-chair. The oxindole moiety is almost perpendicular to the pyrrolidine ring; the benzylidene moiety and the phenyl ring are in equatorial and bisectional configurations with respect to their attached pyrrolidine and cyclohexanone rings, respectively. Intramolecular C—H···O and intermolecular N—H···N and N—H···O interactions stabilize the molecular structure and the packing in the crystal.

Comment

Substituted pyrrolidine compounds have gained much importance since they are the basic structural elements of many alkaloids and pharmacologically active compounds, while molecules with the indole moiety possess anti-inflammatory and antibacterial properties. Our interest in preparing the pharmacologically active pyrrolidine compounds led us to the title compound, (I), and we have undertaken an X-ray crystal structure determination of (I) in order to establish the conformation of the structure.



(I)

In the title structure (Fig. 1), the asymmetric unit contains a pair of molecules (*A* and *B*) with almost identical geometry, except for the rotation of the phenyl rings about the C12—C13 bond. In the two molecules, the bond lengths and angles show normal values and agree with each other (Allen *et al.*, 1987). The values within the oxindole moiety are slightly different compared with those of the indole derivative (Zukerman-Schpector *et al.*, 1992), especially with regard to the longer

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C1—C8 and C7—C8 bond lengths, due to the spiro-atom character and the steric effect of the bulky group substituted at C8. The arguments can also be applied to the longer C—C and C—N bond lengths within the pyrrolidine ring, where the N1 atom is in a pyramidal configuration (Perales *et al.*, 1977), compared with those of the pyrrolidine ring in a previous study (Gzella & Wrzeciono, 1990). The average C—C bond distance in the cyclohexane ring is 1.520 (4) Å in the two molecules.

In both molecules *A* and *B*, the pyrrolidine ring adopts an envelope conformation and the cyclohexane ring is in a half-chair conformation, with a dihedral angle between them of 84.3 (1)° in molecule *A* and 85.3 (1)° in molecule *B*. The conformations were confirmed by their puckering parameters (Cremer & Pople, 1975); for the pyrrolidine ring Q_2 = 0.420 (3) Å and φ_2 = 187.4 (4)° in molecule *A* and Q_2 = 0.405 (3) Å and φ_2 = 0.7 (4)° in molecule *B*, while for the cyclohexane ring, Q_2 = 0.281 (3), Q_3 = 0.403 (3), Q_T = 0.491 (3) Å and φ_2 = 258.6 (6)° in molecule *A*, and Q_2 = 0.311 (3), Q_3 = −0.381 (3), Q_T = 0.492 (3) Å and φ_2 = 93.1 (5)° in molecule *B*. The phenyl ring attached at C23 is in a bisectional configuration with respect to the pyrrolidine ring in both molecules, corresponding to a dihedral angle between the phenyl and pyrrolidine rings of 83.6 (2)° in molecule *A* and 81.4 (2)° in molecule *B*. The phenyl ring attached at C12 makes dihedral angles of 63.2 (2) and 46.2 (2)° with the cyclohexane ring in molecules *A* and *B*, respectively. In the oxindole moiety, the phenyl and pyrrole rings are individually planar and make a dihedral angle of 4.1 (2) and 4.2 (2)° in molecules *A* and *B*, respectively, while the attached O2*A* and O2*B* atoms deviate by −0.204 (2) and 0.171 (2) Å, respectively, from the mean planes of the oxindole moieties in the two molecules. The oxindole moiety makes dihedral angles of 89.9 (1) and 87.8 (1)° with the pyrrolidine ring in molecules *A* and *B*, respectively, corresponding to nearly perpendicular configurations.

The closest distances of C12···O1 and C23···O1 are within 2.764 (3) Å (Table 2), implying intramolecular non-bonded hydrogen interactions and forming closed O1—C10—C11—C12—H12 and O1—C10—C9—C23—H23 rings. These interactions stabilize the molecular structure, while the observed intermolecular N—H···N and N—H···O (Table 2) interactions interconnecting molecules *A* and *B* in different asymmetric units, as well as van der Waals interactions, stabilize the crystal structure.

Experimental

A mixture of 2,6-dibenzylidenecyclohexanone (1 mmol), sarcosine (1 mmol) and isatin (1 mmol) in methanol was refluxed for 4 h. After the reaction, the solvent was evaporated *in vacuo* and the resulting crude product was purified by column chromatography using a hexane–ethyl acetate mixture (9:1). Slow evaporation of the isolated compound from ethanol solution gave light-yellow single crystals suitable for X-ray structure determination.

Crystal data

$C_{30}H_{28}N_2O_2$
M_r = 448.54
Triclinic, $P\overline{1}$
a = 12.3624 (1) Å
b = 13.0640 (2) Å
c = 17.1439 (3) Å
α = 110.066 (1)°
β = 93.200 (1)°
γ = 110.534 (1)°
V = 2386.13 (6) Å^3

Z = 4
D_x = 1.249 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6647 reflections
θ = 1.3–29.5°
μ = 0.08 mm^{-1}
T = 293 (2) K
Slab, light yellow
0.36 × 0.24 × 0.16 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.972, $T_{\max}$ = 0.988
13 509 measured reflections

8228 independent reflections
4402 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.064
$\theta_{\max}$ = 25.0°
$h = -14 \rightarrow 14$
$k = -15 \rightarrow 9$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.141
S = 0.88
8228 reflections
616 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0379P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.23 e Å^{-3}
$\Delta\rho_{\min}$ = −0.26 e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.025 (1)

Table 1
Selected geometric parameters (Å, °).

Molecule *A*		Molecule *B*	
N1—C8	1.460 (3)	N1—C22	1.468 (3)
N1—C22	1.463 (3)	N1—C30	1.472 (3)
N1—C30	1.473 (3)	N1—C8	1.472 (3)
N2—C1	1.359 (3)	N2—C1	1.343 (4)
N2—C2	1.397 (3)	N2—C2	1.397 (3)
C11—C12	1.332 (3)		
C8—N1—C22	106.6 (2)	C22—N1—C30	113.0 (2)
C8—N1—C30	115.8 (2)	C22—N1—C8	106.5 (2)
C22—N1—C30	113.8 (2)	C30—N1—C8	114.4 (2)
C10—C11—C12—C13	−176.8 (2)	C10—C11—C12—C13	−179.5 (2)
C11—C12—C13—C18	51.0 (4)	C11—C12—C13—C18	−39.5 (4)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12*A*—H12*A*···O1*A*	0.93	2.33	2.704 (3)	103
C12*B*—H12*B*···O1*B*	0.93	2.34	2.730 (3)	105
C23*A*—H23*A*···O1*A*	0.98	2.23	2.751 (3)	112
C23*B*—H23*B*···O1*B*	0.98	2.23	2.764 (3)	113
N2*A*—H2*AA*···N1*B*$^{\rm i}$	0.86	2.33	3.029 (3)	139
N2*B*—H2*BA*···O1*A*$^{\rm ii}$	0.86	2.06	2.848 (3)	153

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

After checking their presence in a difference map, all H atoms were fixed geometrically and allowed to ride on the parent C atoms and refined isotropically. Due to the large fraction of weak data at higher angles, the 2θ maximum was limited to 50°.

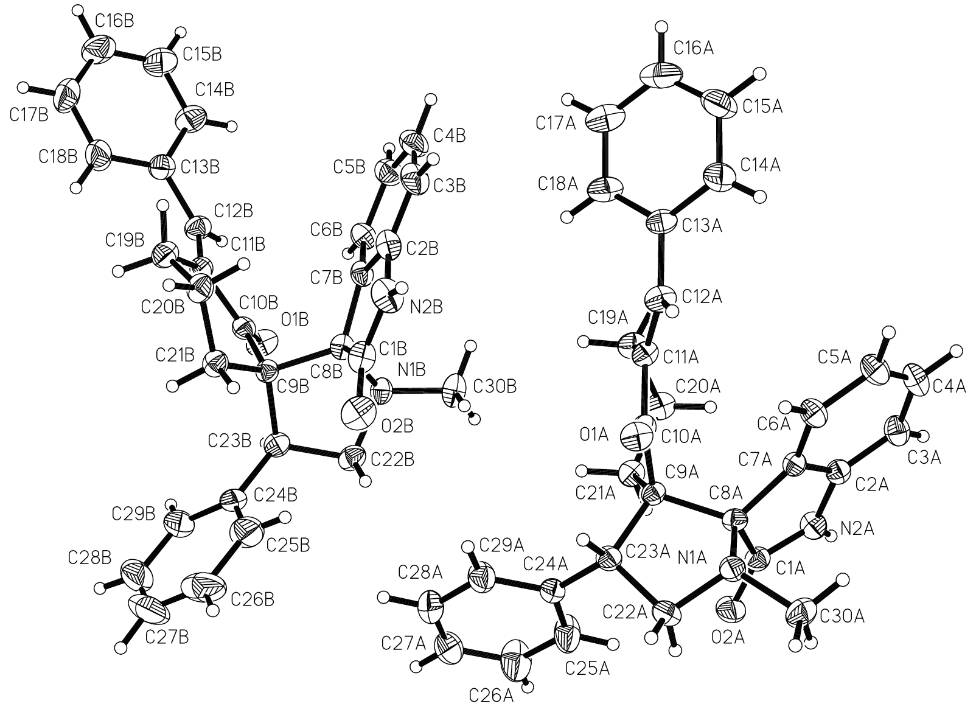

Figure 1
The molecular structure of (I) with 30% probability displacement ellipsoids

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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