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1′-Methyl-4′-phenyl-*H*-fluorene-9-spiro-3′-pyrrolidine-2′-spiro-3″-1*H*-indol-2″(3″*H*)-one

P. G. Aravindan,[a]
S. Selvanayagam,[a]
D. Velmurugan,[a]*
K. Ravikumar,[b]
J. Jayashankaran[c] **and**
R. Raghunathan[c]

[a]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, [b]Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and [c]Department of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India

Correspondence e-mail: d_velu@yahoo.com

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In the title compound, $C_{30}H_{24}N_2O$, the pyrrolidine ring adopts a twist conformation. The dihedral angle between the fluorene ring system and the phenyl ring is 68.3 (1)°. In the solid state, inversion-related molecules form dimers *via* N—H⋯O hydrogen bonds. Weak C—H⋯π interactions also stabilize the molecular packing.

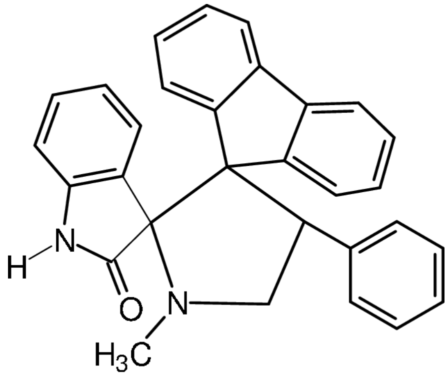
Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.006 Å
R factor = 0.080
wR factor = 0.179
Data-to-parameter ratio = 13.5

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

Comment

Spiro compounds are often encountered in many pharmacologically relevant alkaloids (Cravotto *et al.*, 2001). Pyrrolidine-containing derivatives are used as antibiotics against pathogens of veterinary importance (Fleck *et al.*, 2003). Fluorene derivatives are found to have anticonvulsant activity (Vanvakides *et al.*, 2004). Several unusual amino acids which contain the pyrrolidine system have been investigated by Galeazzi *et al.* (1999). With this background, the crystal structure determination of the title compound, (I), has been carried out and the results are presented here.



(I)

The Csp^3—Nsp^3 bond lengths in the pyrrolidine ring are comparable to those in a structure reported previously (Seshadri *et al.*, 2004). In the fluorene moiety, the C3—C12 and C3—C23 bond distances are almost identical to the values reported in another spiro-linked system (Feng *et al.*, 2004). Atoms O1 and C4 of the oxindole moiety are displaced by 0.240 (2) and −0.111 (2) Å, respectively, from the mean plane through all oxindole ring atoms. This displacement is possibly due to atom O1 being involved in hydrogen bonds with atoms C13 and C1 and to the bulky substituent at C4. The dihedral angle between the fluorene ring system and the phenyl ring is 68.3 (1)°. There is a short contact (2.09 Å) between atoms H1B and H29, which results in the widening of the C2—C24—C29 angle [124.1 (4)°] from the ideal value of 120°. The

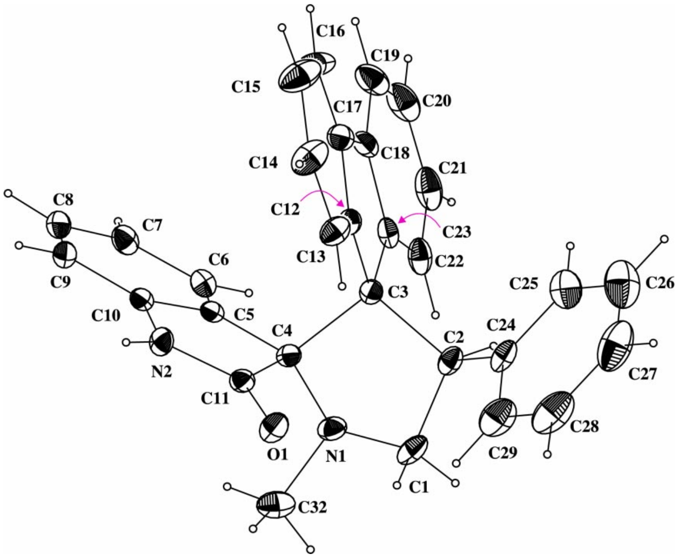


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

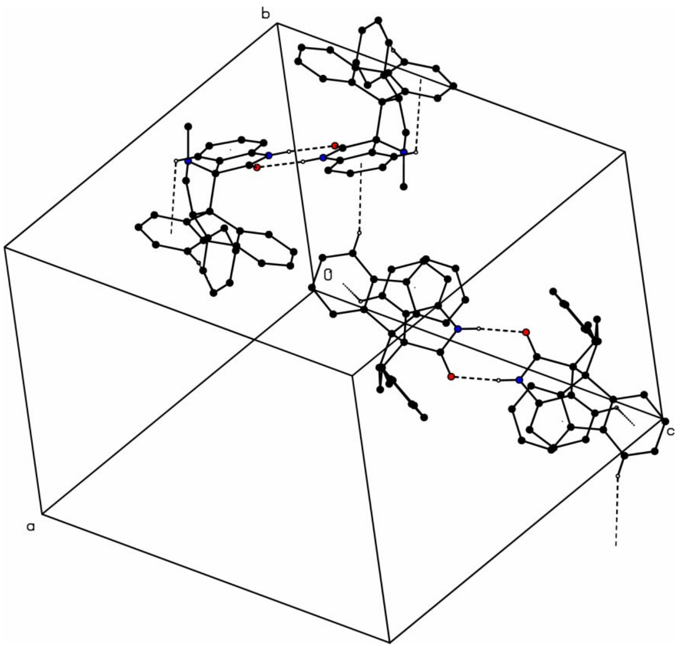


Figure 2
A perspective view of the molecular packing of (I). The N—H$\cdots$O dimer and C—H$\cdots\pi$ interactions are shown as dashed lines. H atoms not involved in C—H$\cdots\pi$ interactions have been omitted.

pyrrolidine ring adopts a twist conformation about N1—C4, with puckering parameters (Cremer & Pople, 1975) q_2 = 0.421 (3) Å and φ = −13.4 (5)° and a small value of the displacement asymmetry parameter (Nardelli, 1983), ΔC_2 = 0.024 (1).

Carbonyl atom O1 acts as a bifurcated acceptor, forming intramolecular interactions with atoms C13 and C1 (Table 2). Inversion-related molecules form dimers *via* strong N—H$\cdots$O hydrogen bonds, the graph-set motif being $R_2^2(8)$. Weak C—H$\cdots\pi$ interactions, arranged in a zigzag fashion, also stabilize the crystal packing.

Experimental

A solution of 9-benzylidene fluorene (6 mmol), sarcosine (6 mmol) and isatin (6 mmol) was refluxed in aqueous methanol until the disappearance of the starting materials, as evidenced by TLC. On completion of the reaction, the crude product was filtered, dried and recrystallized from MeOH/CHCl$_3$ (2:1).

Crystal data

$C_{30}H_{24}N_2O$
M_r = 428.51
Orthorhombic, *Pbca*
a = 18.1752 (1) Å
b = 13.6193 (9) Å
c = 18.4870 (1) Å
V = 4576.2 (5) Å^3
Z = 8
D_x = 1.244 Mg m^{-3}

Mo *Kα* radiation
Cell parameters from 2136 reflections
θ = 2.5–27.9°
μ = 0.08 mm^{-1}
T = 273 (2) K
Block, colourless
0.30 × 0.25 × 0.20 mm

Data collection

Bruker SMART APEX diffractometer
ω scans
Absorption correction: none
22 067 measured reflections
4023 independent reflections

2700 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.063
$\theta_{\rm max}$ = 25.0°
$h = -21 \rightarrow 21$
$k = -14 \rightarrow 16$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.081
$wR(F^2)$ = 0.179
S = 1.12
4023 reflections
298 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 2.8984P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.23 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.19 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C3—C23	1.524 (4)	N1—C4	1.446 (4)
C3—C12	1.527 (4)	N1—C32	1.447 (4)
C1—N1	1.473 (4)		
C4—N1—C32	115.5 (3)	C32—N1—C1	114.1 (3)
C4—N1—C1	106.1 (3)	C11—N2—C10	112.0 (3)

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

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
C1—H1*B*$\cdots$O1	0.97	2.43	2.983 (4)	115
C13—H13$\cdots$O1	0.93	2.53	3.126 (5)	122
N2—H1$\cdots$O1$^{\rm i}$	0.86	1.97	2.823 (3)	171
C6—H6$\cdots$*CgB*	0.93	3.00	3.376 (3)	105
C19—H19$\cdots$*CgA*$^{\rm ii}$	0.93	2.89	3.790 (4)	161

Symmetry codes: (i) $-x, -y, 1-z$; (ii) $-x, \frac{1}{2}+y, \frac{1}{2}-z$. *CgA* is the centroid of ring *A* (C5–C10); *CgB* is the centroid of ring *B* (C18–C23).

The H atoms were positioned geometrically and were treated as riding on their parent atoms, with C—H distances in the range 0.93–0.98 Å and an N—H distance of 0.86 Å. $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for methyl H atoms and 1.2$U_{\rm eq}$(C,N) for all 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

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structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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References

Bruker (2001). *SAINT* (Version 6.28a) and *SMART* (Version 5.625). Bruker AXS Inc., Madison, Wisconsin, USA.

Cravotto, G., Giovenzana, G. B., Pilati, T., Sisti, M. & Palmisano, G. (2001). *J. Org. Chem.* **66**, 8447–8453.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Feng, L., Li, Z.-M., Tan, Y.-S., Chen, M.-Q., Weng, L.-H. & Tao, F.-G. (2004). *Acta Cryst.* C**60**, o473–o474.

Fleck, T. J., McWhorther W. W. Jr, Dekam, R. N. & Pearlman, B. A. (2003). *J. Org. Chem.* **68**, 9612–9617.

Galeazzi, R., Geremia, S., Mobbili, G. & Orena, M. (1999). *Tetrahedron Asymmetry*, **10**, 587–605.

Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

Seshadri, P. R., Selvanayagam, S., Velmurugan, D., Ravilumar, K., Sureshbabu, A. R., Parthasarathy, K. & Raghunathan, R. (2004). *Acta Cryst.* E**60**, o553–o555.

Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Vanvakides, A., Antoniou, K. & Daifoti, Z. (2004). *Ann. Pharm. Fr.* **62**, 49–55.

Zsolnai, L. (1997). *ZORTEP*. University of Heidelberg, Germany.

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