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4′-(4-Methoxyphenyl)-1′-methyl-9*H*-fluorene-9-spiro-3′-pyrrolidine-2′-spiro-2″-indan-1″,3″-dione

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Key indicators

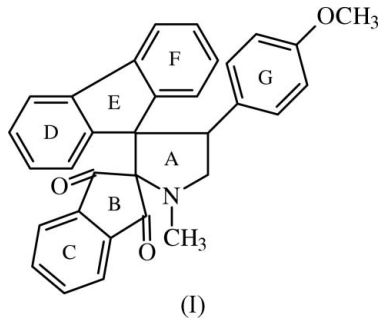
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.063
wR factor = 0.161
Data-to-parameter ratio = 16.9

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

In the title compound, $C_{32}H_{25}NO_3$, the pyrrolidine ring and the five-membered ring of the indan moiety adopt twist and envelope conformations, respectively. The molecules in the crystal structure are linked by an intermolecular C—H···O hydrogen bond, forming chains running along the [$\bar{1}$01] direction.

Comment

Substituted pyrrolidine compounds have been found to have antimicrobial and antifungal activity against various pathogens (Amalraj *et al.*, 2003). Several optically active pyrrolidine compounds have been used as intermediates in controlled asymmetric synthesis (Suzuki *et al.*, 1994). The spiro-indole-pyrrolidine ring system is a frequently encountered structural motif in many biologically important and pharmacologically relevant alkaloids, *e.g.* vincrinstine, vinblastine and spirotypostatins (Cordel, 1981). Pyrrolidine derivatives possess anti-influenza virus (Stylianakis *et al.*, 2003) and anti-convulsant (Obniska *et al.*, 2002) activities. Against this background and in order to obtain detailed information on its molecular conformation, the structure determination 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. (I) consists of a pyrrolidine ring (*A*) connected to an indan moiety (rings *B* and *C*) at C2, a fluorene moiety (rings *D*, *E* and *F*) at C3, and a methoxyphenyl ring (*G*) at C4.

The pyrrolidine ring adopts a twist conformation, with puckering parameters q_2 = 0.416 (2) Å and φ = −121.0 (3)° (Cremer & Pople, 1975); the displacement asymmetry parameter (Nardelli, 1983) are Δ_2(C5) = 0.017 (1) and Δ_s(C3) = 0.078 (1). The five-membered ring (*B*) (atoms C2/C23–C25/C18) of the indandione moiety adopts an envelope conformation, with puckering parameters q_2 = 0.178 (2) Å and φ = −0.1 (6)° and lowest displacement asymmetry parameter Δ_s(C2) = 0.000 (1), with atom C2 deviating by 0.290 (2) Å

from the least-squares plane through the remaining atoms.

In addition to van der Waals interactions, the crystal structure is stabilized by an intermolecular C—H···O hydrogen bond between C21 at (x, y, z) and O3 at ($x-1$, y, $z+1$) (Table 1). This intermolecular hydrogen bond links the molecules into chains running along the [$\bar{1}$01] direction.

Experimental

A mixture of ninhydrin (1 mmol), sarcosine (1 mmol) and *p*-methoxybenzylidenefluorene (1 mmol) was refluxed in aqueous methanol (10 ml). The resulting crude product was purified by column chromatography to obtain the title compound. The compound was recrystallized from petroleum ether–ethyl acetate (9:1).

Crystal data

$C_{32}H_{25}NO_3$	D_x = 1.299 Mg m^{-3}
M_r = 471.53	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2814 reflections
a = 8.6501 (8) Å	θ = 2.4–22.8°
b = 25.738 (2) Å	μ = 0.08 mm^{-1}
c = 10.9358 (10) Å	T = 293 (2) K
β = 97.901 (2)°	Block, colourless
V = 2411.6 (4) Å^3	0.25 × 0.21 × 0.17 mm
Z = 4	

Data collection

Bruker CCD area-detector diffractometer	3677 reflections with $I > 2\sigma(I)$
	R_{int} = 0.031
ω scans	θ_{max} = 28.0°
Absorption correction: None	$h = -11 \rightarrow 11$
14537 measured reflections	$k = -33 \rightarrow 32$
5505 independent reflections	$l = -14 \rightarrow 11$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0749P)^2 + 0.4127P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.063	
$wR(F^2)$ = 0.161	
S = 1.03	$(\Delta/\sigma)_{max} < 0.001$
5505 reflections	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
325 parameters	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
H-atom parameters constrained	

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C21—H21···O3^{i}	0.93	2.55	3.420 (3)	155

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

All H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C—H = 0.93–0.98 Å, and

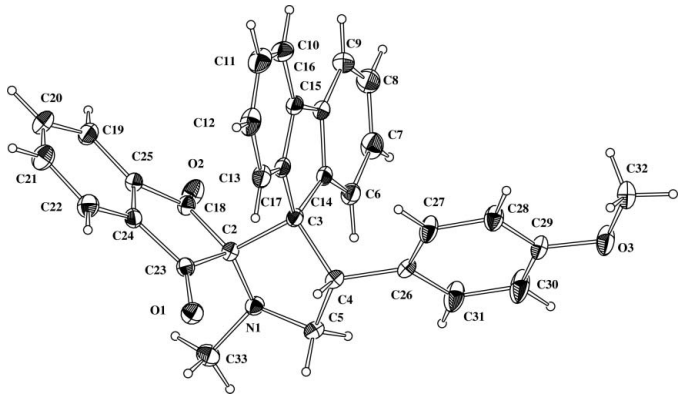


Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

U_{iso}(H) = 1.5U_{eq}(C) for the methyl H atoms and 1.2U_{eq}(C) for the other H atoms.

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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References

Amalraj, A., Raghunathan, R., Sridevi Kumari, M. R. & Raman, N. (2003). *Bioorg. Med. Chem.* **11**, 407–419.
Bruker (2001). *SAINT* (Version 6.28a) and *SMART* (Version 5.625) Bruker AXS Inc., Madison, Wisconsin, USA.
Cordel, G. (1981). *Introduction to Alkaloids, A Biogenetic Approach.* New York: Wiley International.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Obniska, J., Zeic, A. & Zagorska, A. (2002). *Acta Pol. Pharm.* **59**, 209–213.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Stylianakis, I., Kolocousis, A., Kolocousis, N. & Fytas, E. (2003). *Bioorg. Med. Chem. Lett.* **10**, 1699–1703.
Suzuki, H., Soyagi, S. & Kibayashi, C. (1994). *Tetrahedron Lett.* **35**, 6119–6122.
Zsolnai, L. (1997). *ZORTEP*. University Of Heidelberg, Germany.