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4′-(4-Methoxybenzoyl)-1′-methyldispiro-[indole-3(2*H*),2′-pyrrolidine-3′,3″(2″*H*)-indole]-2,2″-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.074
wR factor = 0.130
Data-to-parameter ratio = 12.9

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

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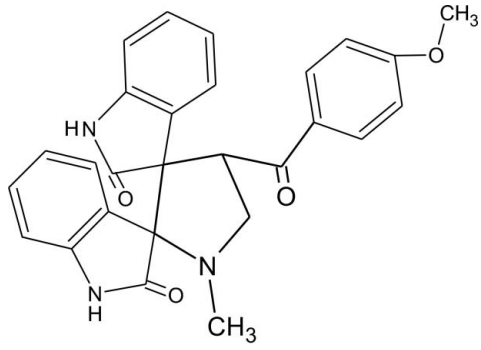
In the title compound, $C_{27}H_{23}N_3O_4$, the pyrrolidine ring adopts a half-chair conformation. Inversion-related molecules are linked *via* N—H···O hydrogen bonds into a zigzag chain. In addition, intermolecular C—H···O and C—H···π hydrogen bonds are observed.

Comment

Spiro-compounds are a particular class of naturally occurring substances characterized by highly pronounced biological properties (Kobayashi *et al.*, 1991; James *et al.*, 1991). In this paper, the crystal structure of the title compound, (I), is reported.



(I)

A *ZORTEP* (Zsolnai, 1997) plot of the molecule is shown in Fig. 1. The pyrrolidine ring adopts a half-chair conformation with puckering parameters q_2 = 0.454 (3) Å and φ_2 = 231.2 (4)° (Cremer & Pople, 1975), and the asymmetry parameter ΔC_2(C26) = 0.0167 (1) (Nardelli, 1995). This puckering causes significant contraction of the N1—C26—C18 angle [105.3 (2)°]. The bond lengths in both oxindole ring systems indicate electron delocalization. The methoxy group is coplanar with the C20—C25 benzene ring [C24—C23—O4—C27 = −176.2 (6)°].

The crystal packing shows that inversion-related molecules are linked through N—H···O hydrogen bonds into a zigzag chain (Fig. 2). In addition, the packing is stabilized by C—H···O and C—H···π hydrogen bonds (Table 2).

Experimental

A mixture of (*E*)-3-(4′-methoxyphenacylidine)oxindole (1 mmol), isatin (indole-2,3-dione) (1 mmol), and sarcosine (*N*-methylglycine) (1 mmol) was refluxed in aqueous methonal for 3 h. On completion of the reaction the solvent was evaporated in a vacuum and the resulting crude product was purified by column chromatography using an *n*-

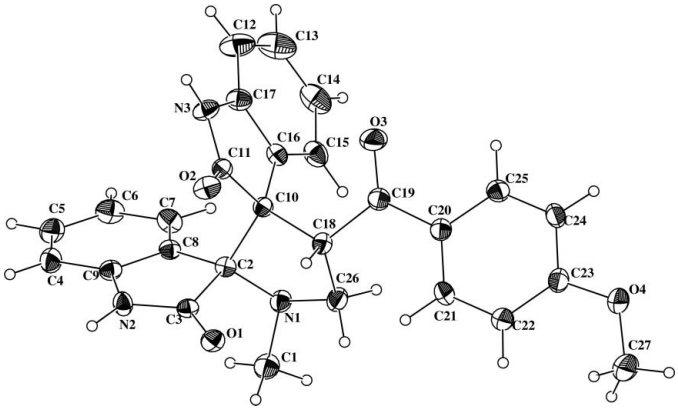


Figure 1
A view of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

hexane–ethyl acetate mixture (7:3) as eluent. The title compound was recrystallized from a methanol–chloroform mixture (2:1 *v*/*v*).

Crystal data

$C_{27}H_{23}N_3O_4$	D_x = 1.328 Mg m^{-3}
M_r = 453.48	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 4532 reflections
a = 8.684 (5) Å	θ = 1.8–26.5°
b = 11.485 (7) Å	μ = 0.09 mm^{-1}
c = 22.751 (13) Å	T = 293 (2) K
β = 90.852 (10)°	Block, colourless
V = 2269 (2) Å^3	0.30 × 0.20 × 0.20 mm
Z = 4	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3371 reflections with $I > 2\sigma(I)$
	R_{int} = 0.040
ω scans	θ_{max} = 27.4°
Absorption correction: none	$h = -11 \rightarrow 11$
17380 measured reflections	$k = -14 \rightarrow 13$
5167 independent reflections	$l = -29 \rightarrow 28$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0254P)^2 + 1.7317P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.074	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.130	$\Delta\rho_{max}$ = 0.27 e Å^{-3}
S = 1.19	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
5167 reflections	
399 parameters	
All H-atom parameters refined	

Table 1
Selected geometric parameters (Å, °).

O2—C11	1.223 (3)	N1—C26	1.470 (3)
O1—C3	1.226 (3)	C2—C10	1.573 (4)
N2—C3	1.346 (3)	C10—C18	1.543 (3)
N3—C11	1.349 (3)	C18—C26	1.531 (4)
N1—C2	1.456 (3)		
C2—N1—C1	116.2 (2)	C7—C8—C9	119.1 (2)
C2—N1—C26	108.7 (2)	C15—C16—C17	119.1 (3)
C1—N1—C26	114.0 (2)	N1—C26—C18	105.3 (2)
N1—C2—C10	99.98 (19)	C12—C17—C16	122.4 (3)
C4—C9—C8	122.4 (3)		
C20—C19—C18—C26	−51.4 (4)	C27—O4—C23—C24	−176.2 (4)
C1—N1—C26—C18	−150.2 (3)		

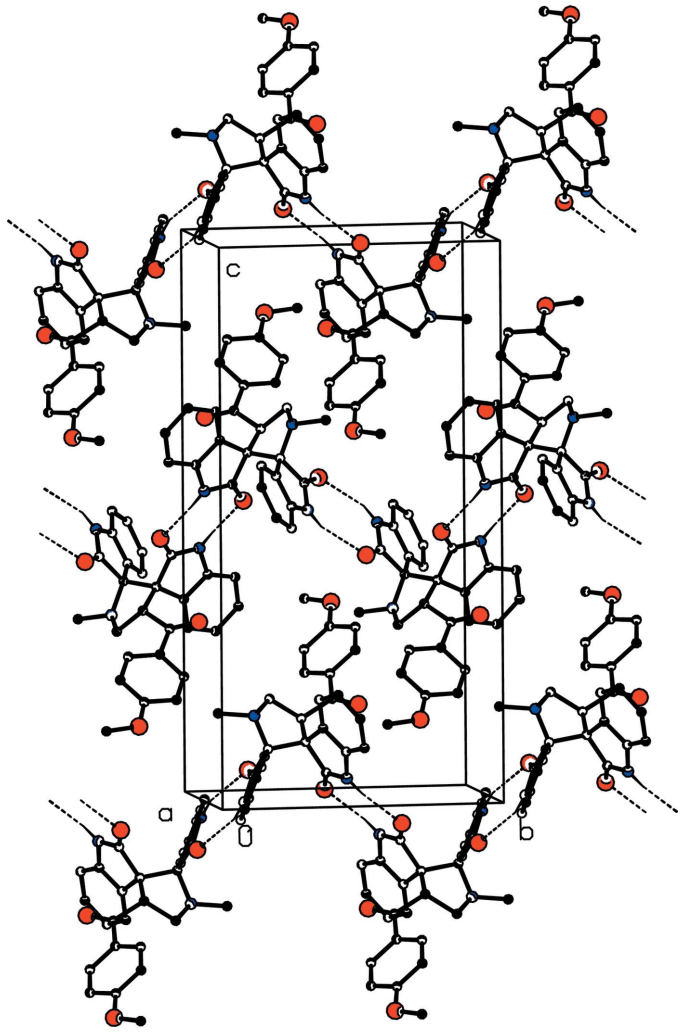


Figure 2
The crystal packing of (I), showing N—H···O hydrogen-bonded (dashed lines) chains. H atoms have been omitted.

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

D—H···A	D—H	H···A	D···A	D—H···A
C18—H18···O1	0.95 (2)	2.47 (2)	3.049 (4)	120 (2)
N2—H2···O1^{i}	0.89 (3)	2.19 (3)	3.039 (3)	162 (3)
N3—H3···O2^{ii}	0.86 (3)	2.11 (3)	2.941 (4)	163 (2)
C6—H6···O2^{iii}	0.93 (3)	2.57 (3)	3.421 (4)	151 (2)
C22—H22···O3^{iv}	0.94 (3)	2.53 (2)	3.464 (4)	169 (2)
C24—H24···O1^{v}	0.92 (3)	2.59 (3)	3.380 (4)	145 (2)
C14—H14···Cg^{iii}	0.97 (3)	2.76 (3)	3.610 (4)	146 (3)

Symmetry codes: (i) $-x, -y, -z$; (ii) $-x, -y+1, -z$; (iii) $x+1, y, z$; (iv) $-x-\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (v) $-x-\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$. Note: Cg is the centroid of the C20–C25 ring.

H atoms were located in a difference Fourier map and refined isotropically. The ranges of C—H and N—H bond lengths are 0.91 (3)–1.03 (4) Å and 0.86 (3)–0.89 (3) Å, respectively.

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: *PLATON* (Spek, 2003).

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