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4′-(4-Methylbenzoyl)-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) = 0.003 Å
R factor = 0.051
wR factor = 0.135
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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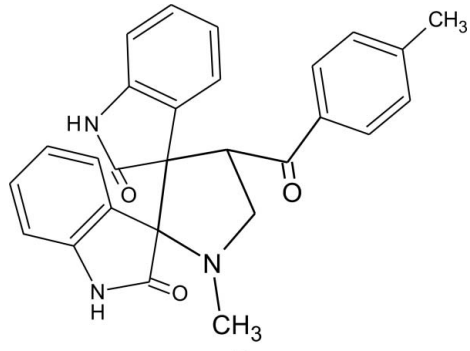
In the title compound, $C_{27}H_{23}N_3O_3$, the central pyrrolidine ring adopts an envelope conformation. In the crystal structure, the molecules exist as centrosymmetric N—H···O hydrogen-bonded dimers. The dimers are linked *via* C—H···O and C—H···π hydrogen bonds, forming a chain along the *b* axis.

Comment

The spiro-pyrrolidine ring system is a frequently encountered structural motif in many pharmacologically relevant alkaloids (Cordel, 1981). It is also found in phermones, antibiotics (Gore *et al.*, 1991) and antitumour agents (Tietze *et al.*, 1988; Araki *et al.*, 2002). Several optically active pyrrolidines have been used as intermediates in controlled asymmetric syntheses (Suzuki *et al.*, 1994). In view of this importance, the crystal structure of the title compound, (I), has been determined and the results are presented here.



(I)

A *ZORTEP* (Zsolnai, 1997) plot of the molecule is shown in Fig.1. The slightly longer N—C and C—C bond lengths (Table 1) in the pyrrolidine ring are due to the bulky substituents and the steric interactions between them (Seshadri *et al.*, 2003; Abdul Ajees *et al.*, 2002). The N2—C3 and C3—O1 bond lengths show electron delocalization over atoms N2, C3 and O2. A similar situation is also observed for atoms N3, C11 and O2. In the oxindole ring systems, the variations in endocyclic angles are due to the fusion of five- and six-membered rings (Govind *et al.*, 2003).

The asymmetry parameters [ΔC_s(C2) = 0.065 (1); Nardelli, 1995] and puckering parameters [q_2 = 0.468 (2) Å and φ_2 = 225.6 (2)°; Cremer & Pople, 1975] reveal that the pyrrolidine ring adopts an envelope conformation. Atom C2 deviates from the N1/C10/C18/C26 plane by 0.698 (2) Å. The methyl group attached at N1 is in the equatorial position, as evidenced by the C1—N1—C26—C18 torsion angle of

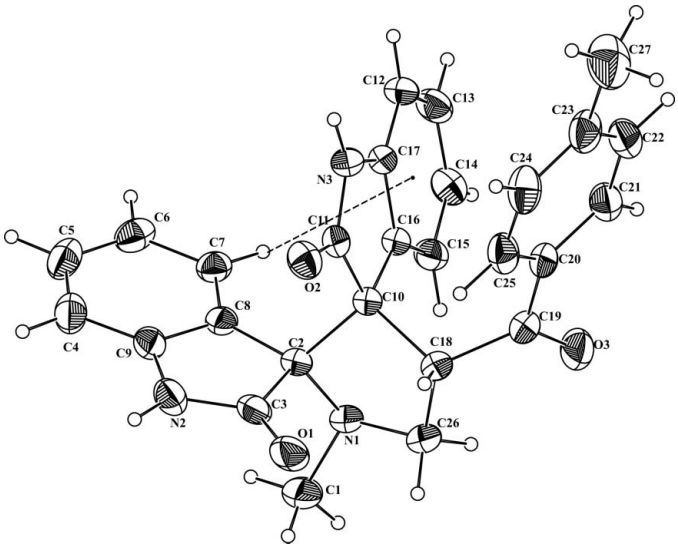


Figure 1
A view of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. The dashed line indicates a C—H···π interaction.

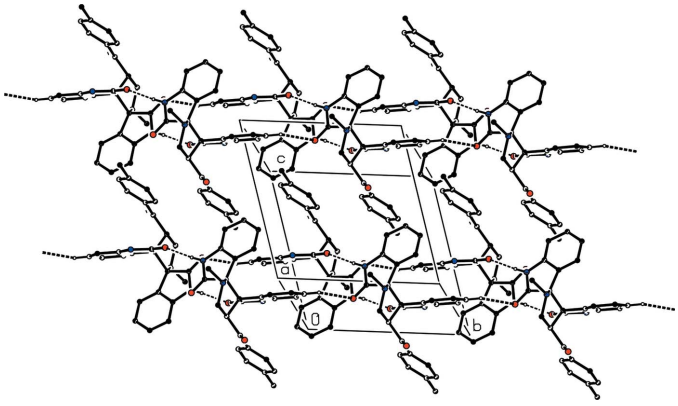


Figure 2
The crystal packing of (I), viewed approximately down the *a* axis. Only H atoms involved in hydrogen bonding (dashed lines) are shown.

−152.44 (19)°. The benzoyl group is bisectionally attached to the pyrrolidine ring, C26—C18—C19—C20 = −175.89 (16)°.

An intramolecular C–H···π interaction involving the C12–C17 benzene ring is observed in the molecular structure. In the crystal structure, symmetry-related molecules form N—H···O hydrogen-bonded dimers, which are linked *via* C—H···O and C—H···π hydrogen bonds (Table 2), forming a chain along the *b* axis (Fig. 2).

Experimental

A mixture of (*E*)-3-(4′-methylphenacylidine)oxindole (1 mmol), isatin (indole-2,3-dione) (1 mmol), and sarcosine (*N*-methylglycine) (1 mmol) was refluxed in aqueous methanol for 3 h. On completion of the reaction the solvent was evaporated in a vaccum and the resulting crude product was purified by coloumn chromatography using an *n*-hexane–ethyl acetate mixture (7:3) as eluent. The title compound was recrystallized from a methanol–chloroform mixture (2:1 *v*/*v*).

Crystal data

$C_{27}H_{23}N_3O_3$
M_r = 437.48
Triclinic, $P\overline{1}$
a = 9.6158 (10) Å
b = 10.3812 (11) Å
c = 11.7099 (12) Å
α = 105.047 (2)°
β = 94.850 (2)°
γ = 100.087 (2)°
V = 1101.0 (2) Å^3
Z = 2
D_x = 1.320 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4387 reflections
θ = 2.5–28.0°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colourless
0.20 × 0.19 × 0.19 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
7012 measured reflections
4904 independent reflections
3781 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 28.0°
$h = -12 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -15 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.135
S = 1.01
4904 reflections
379 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0665P)^2 + 0.1767P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.456 (2)	O1—C3	1.213 (2)
N1—C2	1.464 (2)	O2—C11	1.215 (2)
N1—C26	1.474 (2)	O3—C19	1.207 (2)
N2—C3	1.351 (2)	C2—C10	1.559 (2)
N2—C9	1.389 (2)	C10—C18	1.560 (2)
N3—C11	1.352 (2)	C18—C26	1.531 (3)
N3—C17	1.389 (2)		
C1—N1—C2	115.72 (15)	C8—C2—C10	115.90 (12)
C1—N1—C26	113.69 (15)	C11—C10—C18	116.54 (14)
C2—N1—C26	106.97 (14)	C2—C10—C18	100.56 (12)
N1—C2—C10	99.79 (12)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2^{i}	0.90 (2)	1.99 (2)	2.879 (2)	170 (2)
C13—H13···O1^{ii}	0.97 (2)	2.37 (2)	3.232 (2)	149 (2)
C7—H7···$Cg1$	0.98 (2)	2.87 (2)	3.551 (3)	128 (1)
C12—H12···$Cg2^{iii}$	0.95 (2)	2.75 (2)	3.613 (3)	153 (1)

Symmetry codes: (i) $-x+2, -y+1, -z$; (ii) $x, y-1, z$; (iii) $-x+2, -y, -z$. $Cg1$ and $Cg2$ are the centroids of the C12—C17 and C4—C9 benzene rings, respectively.

Methyl H atoms were placed in calculated positions and constrained to ride on their parent atoms, with C—H = 0.96 Å and $1.5U_{eq}$(C). The remaining H atoms were located in a difference Fourier map and refined isotropically [C—H = 0.93 (2)–1.02 (2) Å].

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT*; data reduction: *SAINT* (Bruker, 2001); 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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