

4'-(4-Chlorobenzoyl)-1'-methyldispiro-[indole-3(2*H*),2'-pyrrolidine-3',3''(2''*H*)-indole]-2,2''-dioneK. Palani,^a M. N. Ponnuswamy,^{a*}
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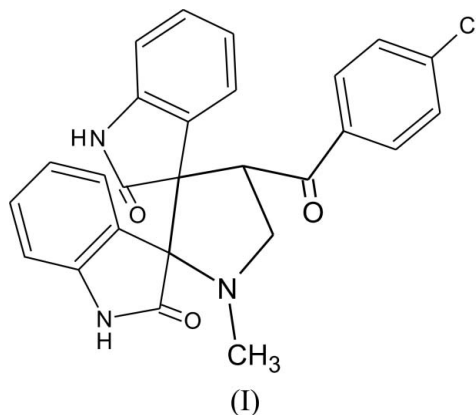
Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.040
wR factor = 0.108
Data-to-parameter ratio = 11.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $\text{C}_{26}\text{H}_{20}\text{ClN}_3\text{O}_3$, the central pyrrolidine ring adopts an envelope conformation. In the crystal structure, the molecules exist as centrosymmetric $\text{N}-\text{H} \cdots \text{O}$ hydrogen-bonded dimers. The dimers are linked *via* $\text{C}-\text{H} \cdots \text{O}$ 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). A new class of spiro-pyrrolidines has been screened for their antibacterial and antifungal activity against ten human pathogenic bacteria and four dermatophytic fungi (Raj *et al.*, 2003). In view of this medicinal importance, the crystal structure of the title compound, (I), has been determined and the results are presented here.



A *ZORTEP* (Zsolnai, 1997) plot of the molecule is shown in Fig. 1. The slightly longer $\text{N}-\text{C}$ and $\text{C}-\text{C}$ 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 $\text{N}2-\text{C}3$ and $\text{C}3-\text{O}1$ bond lengths show electron delocalization over atoms $\text{N}2$, $\text{C}3$ and $\text{O}2$. A similar situation is also observed for atoms $\text{N}3$, $\text{C}11$ and $\text{O}2$. 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 pyrrolidine ring adopts an envelope conformation. The asymmetry parameter $\Delta C_s(\text{C}2)$ is 0.065 (1) (Nardelli, 1995) and the puckering parameters (Cremer & Pople, 1975) q_2 and φ_2 are 0.467 (2) \AA and 46.8 (2)°, respectively. Atom $\text{C}2$ deviates by 0.698 (2) \AA from the $\text{N}1/\text{C}10/\text{C}18/26$ plane. This causes the significant contraction of the $\text{N}1-\text{C}2-\text{C}10$ [99.8 (1)°] angle. The methyl group substituted at $\text{N}1$ is in the equatorial position [$\text{C}1-\text{N}1-\text{C}26-\text{C}18 = 151.80$ (15)°]. The oxindole

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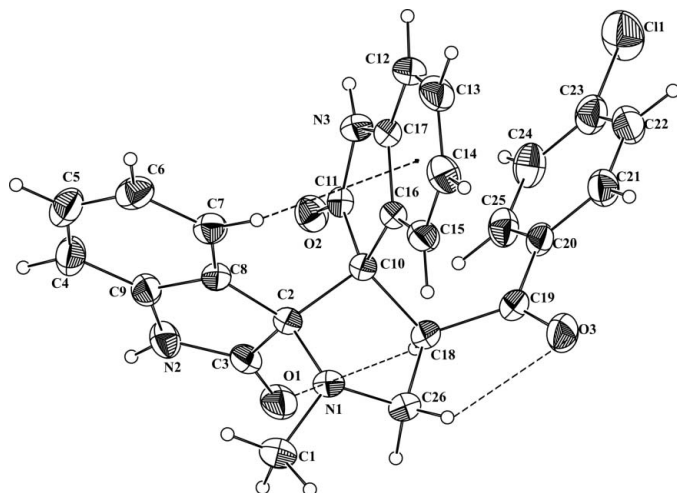


Figure 1
A view of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Dashed lines indicate hydrogen bonds.

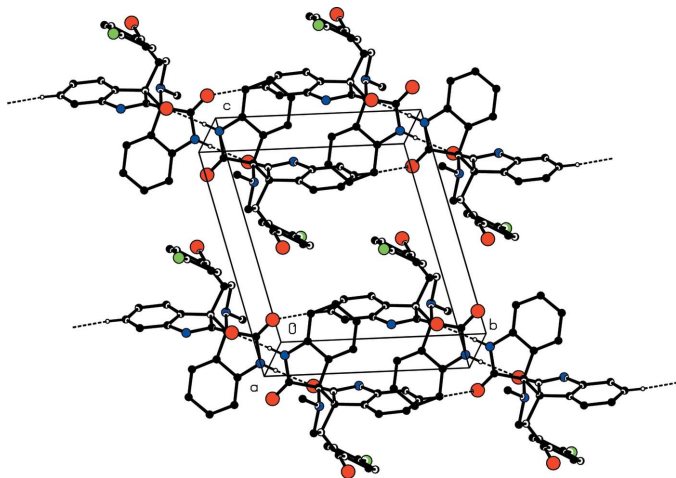


Figure 2
The crystal packing of (I), viewed approximately along the *a* axis. Dashed lines indicate hydrogen bonds. H atoms have been omitted.

planes O1/N2/C2–C9 and O2/N3/C10–C17 form dihedral angles of 89.23 (6) and 74.39 (7)°, respectively, with the N1/C10/C18/26 plane.

The molecular structure is stabilized by intramolecular C–H···O and C–H··· π interactions. In the crystal structure, inversion-related molecules form N–H···O hydrogen-bonded dimers, which are linked *via* C–H···O hydrogen bonds, forming a chain along the *b* axis (Fig. 2). A short C11···O3(*x* – 1, *y*, *z*) contact [3.256 (3) Å] is also observed in the structure.

Experimental

A mixture of (*E*)-3-(4'-chlorophenacylidine)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 vacuo* and the resulting crude product was purified by column chromatography using an *n*-

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

Crystal data

C₂₆H₂₀ClN₃O₃
M_r = 457.90
 Triclinic, *P* $\bar{1}$
a = 9.795 (6) Å
b = 10.316 (6) Å
c = 11.670 (7) Å
 α = 104.275 (9)°
 β = 93.998 (10)°
 γ = 102.305 (9)°
V = 1107.2 (12) Å³

Z = 2
D_x = 1.373 Mg m^{–3}
 Mo *K* α radiation
 Cell parameters from 6234 reflections
 θ = 2.1–27.3°
 μ = 0.21 mm^{–1}
T = 293 (2) K
 Block, colourless
 0.22 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.955, *T_{max}* = 0.959
 11608 measured reflections

4491 independent reflections
 3746 reflections with *I* > 2 σ (*I*)
R_{int} = 0.016
 θ_{max} = 27.3°
h = –12 → 12
k = –13 → 13
l = –14 → 15

Refinement

Refinement on *F*²
R[*F*² > 2 σ (*F*²)] = 0.040
wR(*F*²) = 0.108
S = 1.04
 4491 reflections
 379 parameters
 All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0518P)^2 + 0.2477P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.28 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.30 \text{ e \AA}^{-3}$
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0105 (18)

Table 1

Selected geometric parameters (Å, °).

C11–C23	1.735 (2)	O1–C3	1.218 (2)
N3–C11	1.354 (2)	N1–C1	1.460 (2)
N3–C17	1.400 (2)	N1–C2	1.469 (2)
O2–C11	1.2166 (18)	N1–C26	1.477 (2)
N2–C3	1.358 (2)	O3–C19	1.214 (2)
N2–C9	1.394 (2)		
C15–C16–C17	119.62 (14)	C1–N1–C26	113.85 (14)
C12–C17–C16	121.93 (15)	C2–N1–C26	107.34 (13)
C7–C8–C9	118.93 (15)	C4–C9–C8	122.44 (16)
C1–N1–C2	115.58 (14)		

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
C18–H18···O1	0.97 (2)	2.42 (2)	2.936 (3)	113 (1)
C26–H26B···O3	0.97 (2)	2.43 (2)	2.848 (3)	106 (1)
N2–H2···O2 ⁱ	0.85 (2)	2.05 (2)	2.896 (3)	172 (2)
C13–H13···O1 ⁱⁱ	0.92 (2)	2.40 (2)	3.217 (3)	148 (2)
C7–H7···Cg1	0.96 (2)	2.90 (2)	3.572 (3)	128 (1)
C12–H12···Cg2 ⁱⁱⁱ	0.94 (2)	2.90 (2)	3.730 (3)	149 (2)

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

H atoms were located in a difference Fourier map and refined isotropically. The ranges of C–H and N–H bond lengths are 0.92 (2)–1.03 (2) Å and 0.84 (2)–0.85 (2) 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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