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4′-(4-Methoxybenzoyl)-1′-methyl acenaphthene-1-spiro-2′-pyrrolidine-3′-spiro-3″-1*H*-indole-2,2″(1*H*,3″*H*)-dione hemihydrate

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Key indicators

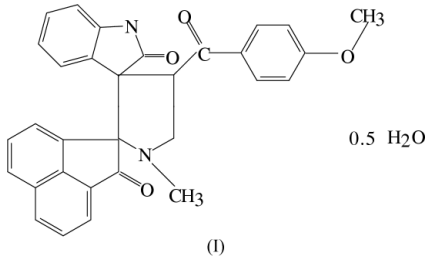
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.083
wR factor = 0.219
Data-to-parameter ratio = 16.8

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

The acenaphthene keto O atom of the title compound, $C_{31}H_{24}N_2O_4\cdot0.5H_2O$, is displaced by 0.132 (2) Å from the acenaphthene plane, while the indole keto O atom is displaced by only 0.096 (2) Å from the oxindole plane. The molecular structure is stabilized by intramolecular C—H···O interactions. The crystal packing is stabilized by C—H···O, N—H···O and N—H···N intermolecular interactions.

Comment

The spiro-indole-pyrrolidine ring system is a frequently encountered structural motif in many pharmacologically relevant alkaloids (Cravotto *et al.*, 2001). 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). They were found to have antimicrobial and antifungal activity against various pathogens, except *Bacillus subtilis* (Raj *et al.*, 2003). Against this background and in order to obtain detailed information on its molecular conformation, X-ray studies on the title compound, (I), have been carried out.



(I)

Fig. 1 shows a displacement ellipsoid diagram of the molecule, with the atomic numbering scheme. Selected geometric parameters are given in Table 1.

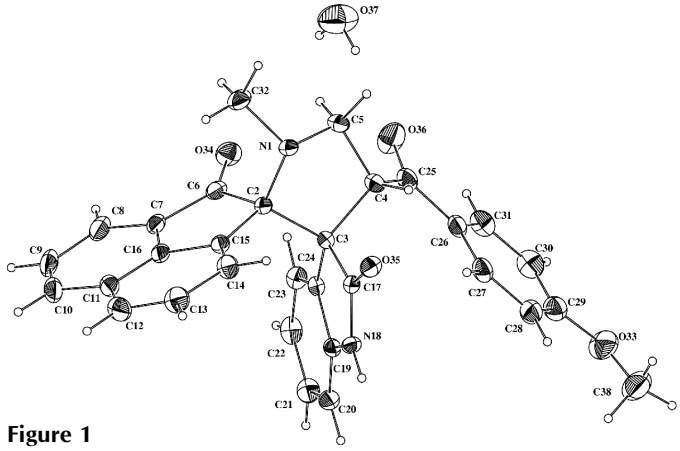


Figure 1
View of (I) (50% probability displacement ellipsoids).

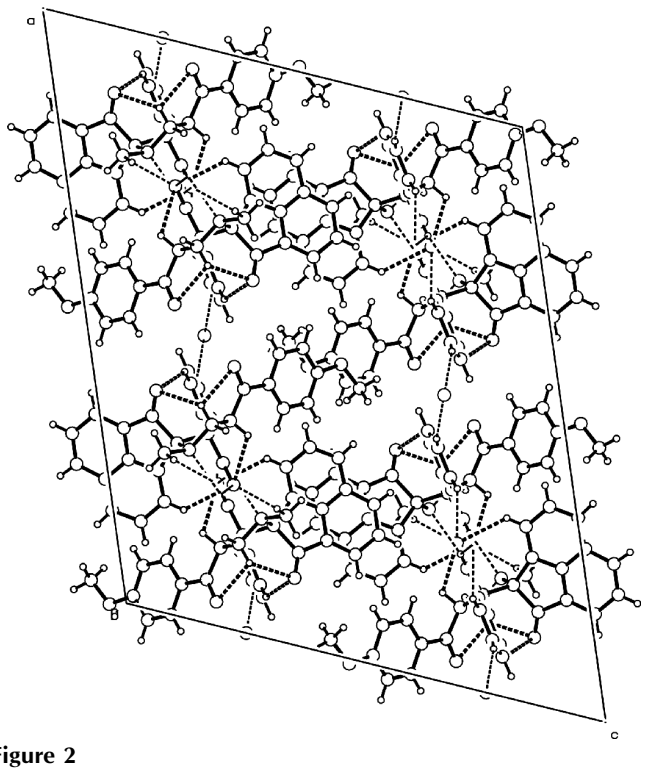


Figure 2
A view of the packing of the molecules, viewed down the *b* axis.

The N—C and C—C bond lengths in the pyrrolidine moiety are slightly longer than the normal values reported for similar structures (Seshadri *et al.*, 2003; Abdul Ajees *et al.*, 2002). This may be due to steric forces caused by the bulky substituents on the pyrrolidine moiety. The sum of angles around N1 (333.1°) is in accordance with sp^3 hybridization.

The keto O atom O34 is 0.132 (2) Å from the acenaphthene plane. It is an acceptor of weak intramolecular hydrogen bonds from C5—H5*B* and C23—H23. The keto O atom O36 is 0.190 (3) Å out of the plane through C4/C25–C31, with a C25═O36 bond direction defined by the O36—C25—C26—C31 [−4.0 (5)°] and O36—C25—C26—C27 [173.3 (4)°] torsion angles. Atom O36 is an acceptor of a weak intramolecular interaction from C5—H5*B*. Atom C38 of the methoxy group is 0.216 (5) Å out of the plane through C26–C31/O33, with O33 at 0.038 (3) Å from the C26–C31 benzene plane.

The pyrrolidine ring adopts an envelope conformation with Cremer & Pople (1975) puckering parameters q_2 = 0.775 (6) Å and φ = −45.1 (3)°, and the smallest Nardelli (1983) displacement asymmetry parameter is Δ_2(C4) = 0.068 (2).

The molecular structure is influenced by C—H···O intramolecular interactions. The crystal packing is stabilized by C—H···O, N—H···O and N—H···N intermolecular interactions (Table 2).

Experimental

A mixture of (*E*)-3-(*p*-methoxyphenacilidine)oxindole (1 mmol), acenaphthenequinone (1 mmol) and sarcosine (1 mmol) was stirred in aqueous methanol at room temperature. The resulting crude product was purified by column chromatography to obtain the title compound. The compound was recrystallized from chloroform–methanol (1:1) to yield the title compound.

Crystal data

$C_{31}H_{24}N_2O_4{\cdot}0.5H_2O$	D_x = 1.310 Mg m^{-3}
M_r = 497.53	Mo $K\alpha$ radiation
Monoclinic, *C*2/*c*	Cell parameters from 1300 reflections
a = 25.647 (2) Å	θ = 2.2–20.7°
b = 10.0538 (9) Å	μ = 0.09 mm^{-1}
c = 21.0859 (18) Å	*T* = 293 (2) K
β = 111.933 (2)°	Block, colorless
V = 5043.5 (7) Å^3	0.21 × 0.20 × 0.20 mm
Z = 8	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3290 reflections with $I > 2\sigma(I)$
	R_{int} = 0.045
ω scans	θ_{max} = 28.0°
Absorption correction: none	*h* = −32 → 26
15002 measured reflections	*k* = −13 → 12
5795 independent reflections	*l* = −26 → 27

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0971P)^2 + 2.3543P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.084	
$wR(F^2)$ = 0.219	
S = 1.04	$(\Delta/\sigma)_{max} < 0.001$
5795 reflections	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
345 parameters	$\Delta\rho_{min}$ = −0.40 e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

N1—C5	1.467 (4)	C2—C3	1.578 (4)
N1—C2	1.468 (4)	C3—C4	1.578 (4)
N1—C32	1.470 (4)		
C5—N1—C2	105.9 (2)	C2—N1—C32	113.8 (2)
C5—N1—C32	113.4 (3)		
O36—C25—C26—C27	173.3 (4)	O36—C25—C26—C31	−4.0 (5)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O35	0.98	2.48	2.947 (4)	109
C5—H5*B*···O34	0.97	2.45	3.017 (4)	116
C5—H5*B*···O36	0.97	2.46	2.804 (5)	100
C14—H14···O35	0.93	2.40	3.038 (4)	125
C23—H23···O34	0.93	2.43	3.079 (4)	126
O37—H37···O36	0.87 (5)	2.02 (4)	2.848 (5)	159 (4)
N18—H18···N1^{i}	0.86	2.38	3.146 (3)	148
C20—H20···O35^{i}	0.93	2.41	3.143 (4)	135
C21—H21···O37^{ii}	0.93	2.58	3.445 (5)	154
C32—H32*B*···O35^{iii}	0.96	2.37	3.222 (4)	147
N18—H18···O35^{i}	0.86	2.59	3.225 (3)	131

Symmetry codes: (i) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (ii) $x, y-1, z$; (iii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.

H atoms bonded to C and N atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å, N—H = 0.86 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C,N) for other H atoms. The coordinates of the H atom bonded to O37 were refined with a restraint of 0.87 (1) Å for the O—H bond length.

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 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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