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5″-Benzylidene-1′,1″-dimethyl-4′-phenylacenapthene-2-spiro-2′-pyrrolidine-3′-spiro-3″-piperidine-1,4″-dione

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
R factor = 0.070
wR factor = 0.167
Data-to-parameter ratio = 17.8

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

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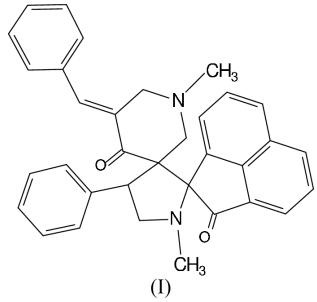
In the title compound, $C_{34}H_{30}N_2O_2$, the pyrrolidine and piperidinone rings each adopt a half-chair conformation. The dihedral angle between the two phenyl-ring substituents is 70.4 (1)°. Intramolecular C—H···O hydrogen bonds are observed in the crystal structure. In addition to these interactions, C—H···π interactions also play a role in stabilizing the crystal structure.

Comment

Spiro-pyrrolidine compounds find applications in the synthesis of biologically active compounds. Spiro compounds are often encountered in pharmacologically relevant alkaloids (Cravotto *et al.*, 2001). Synthetic spiro-pyrrolidine derivatives exhibit activity against the aldose reductase enzyme, which controls influenza (Stylianakis *et al.*, 2003). The fungal metabolism of acenaphthene resembles bacterial and mammalian metabolism, since two C atoms of the five-membered ring are involved in enzymatic attack (Pothuluri *et al.*, 1992). Piperidinone derivatives possess anticonvulsant activity (Brouillelte & Grunwald, 1984). Several unusual amino acids containing pyrrolidine moieties have been investigated by Galeazzi *et al.* (1999). With this background and in continuation of our structural analysis of spiro-pyrrolidine derivatives, the X-ray crystal structure determination of the title compound, (I), was undertaken.



(I)

The bond lengths and angles in the pyrrolidine ring are slightly larger than normal values, but are comparable to values in previously reported structures (Gzella & Wrzeciono, 1990; Jeyabharathi *et al.*, 2001). The distortion is due to the bulky substituents on the pyrrolidine moiety. The sum of the angles at atom N2 (334.3°) of the piperidinone ring indicates sp^3-hybridization. A short contact between atoms H32 and H1*B* (2.121 Å) results in the slight widening of the C32—C27—C2 angle [123.4 (2)°] from the ideal value of 120°. Keto atom O2 is displaced by 0.308 (2) Å from the acenapthene plane, probably as a result of the intramolecular hydrogen bond it forms with the C16—H16*A* group.

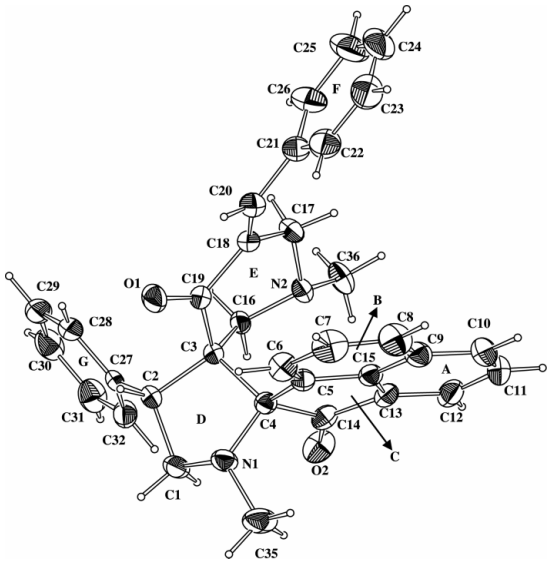


Figure 1
The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

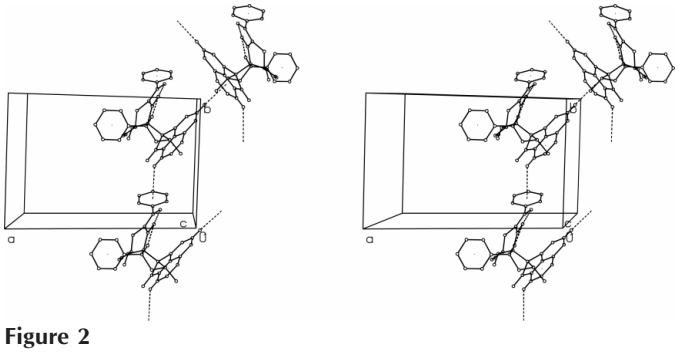


Figure 2
A stereoview down the *c* axis of the packing of the title compound. C—H···π interactions are indicated by dashed lines.

The dihedral angles between the acenaphthene ring system and rings *F* and *G* (Fig. 1) are 60.3 (1) and 56.0 (1)°, respectively, while that between rings *F* and *G* is 70.5 (1)°. The pyrrolidine ring adopts a half-chair conformation, which is confirmed by the puckering parameters (Cremer & Pople, 1975) q_2 = 0.416 (2) Å and φ = 19.7 (3)°, and the small value of the displacement asymmetry parameter (Nardelli, 1983), *viz*. ΔC_2(C3) = 0.008 (1)°. The piperidinone ring adopts a half-chair conformation [q_2 = 0.319 (2) Å, φ = 48.5 (4)°, ΔC_s(C16) = 0.507 (1) and ΔC_2(C16—N2) = 0.057 (1)°].

Atom O1 acts as an acceptor for two intramolecular hydrogen bonds, the angle H20···O1···H2 being 160°. These two hydrogen bonds result in two five-membered rings, graph-set $R_1^1(5)$ (Bernstein *et al.*, 1995). The packing of the molecules in the solid state is also stabilized by C—H···π intermolecular interactions.

Experimental

A mixture of a dipolarophile (dibenzylidene-*N*-methylpiperidone, 1 mmol), acenaphthenequinone (1 mmol) and sarcosine (1 mmol) was refluxed in aqueous methanol until the starting materials had disappeared (about 3–4 h), as evidenced by thin-layer chromatography. When the reaction was complete, the solvent was removed *in vacuo* and the residue was chromatographed on silica gel, using a hexane–ethyl acetate mixture as eluant and recrystallizing from methanol to give the title compound (I).

Crystal data

$C_{34}H_{30}N_2O_2$	D_x = 1.219 Mg m^{-3}
M_r = 498.60	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2836 reflections
a = 16.6609 (1) Å	θ = 2.3–25.4°
b = 10.4558 (9) Å	μ = 0.08 mm^{-1}
c = 17.4457 (2) Å	T = 273 (2) K
β = 116.589 (1)°	Block, colourless
V = 2717.7 (2) Å^3	0.21 × 0.20 × 0.20 mm
Z = 4	

Data collection

Bruker SMART APEX diffractometer	R_{int} = 0.035
ω scan	θ_{max} = 28.0°
16 066 measured reflections	$h = -21 \rightarrow 21$
6130 independent reflections	$k = -13 \rightarrow 12$
4068 reflections with $I > 2\sigma(I)$	$l = -22 \rightarrow 22$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0702P)^2 + 0.4469P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.070	$(\Delta/\sigma)_{max} < 0.0001$
$wR(F^2)$ = 0.167	$\Delta\rho_{max}$ = 0.28 e Å^{-1}
S = 1.08	$\Delta\rho_{min}$ = −0.17 e Å^{-1}
6130 reflections	
345 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.453 (3)	C35—N1	1.453 (3)
C4—N1	1.467 (3)	C36—N2	1.458 (3)
C16—N2	1.445 (2)		
C1—N1—C35	114.2 (2)	C16—N2—C17	110.6 (2)
C1—N1—C4	107.4 (2)	C16—N2—C36	112.3 (2)
C35—N1—C4	115.8 (2)	C17—N2—C36	111.4 (2)
C1—C2—C27—C32	-26.0 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O1	0.98	2.33	2.834 (3)	111
C16—H16A···O2	0.97	2.42	2.985 (3)	117
C20—H20···O1	0.93	2.41	2.770 (3)	103
C7—H7···CgA^{i}	0.93	2.65	3.544 (3)	162
C12—H12···CgF^{ii}	0.93	3.09	3.824 (2)	137
C36—H36B···CgG^{iii}	0.96	2.58	3.426 (6)	147

Symmetry codes: (i) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x, y-1, z$; (iii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$. *CgA*, *CgF* and *Cg*G are the centroids of rings *A*, *F* and *G*.

The H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H distances in the range 0.93–0.98 Å; U_{iso}(H) values were set equal to 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for all 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) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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