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1′-Methylacenapthylene-1(2*H*)-spiro-2′-pyrrolidine-3′-spiro-2″(3″*H*)-carbazole-2,1″(4″*H*)-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.051
wR factor = 0.146
Data-to-parameter ratio = 17.5

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

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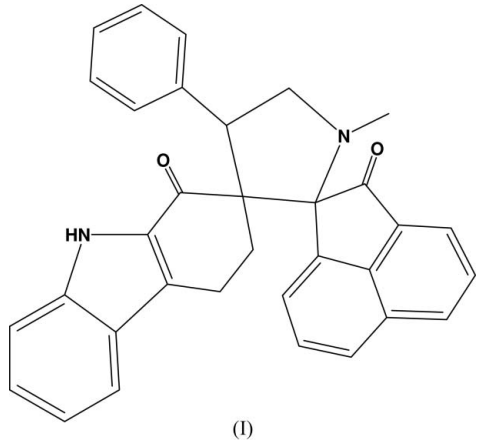
In the title compound, $C_{33}H_{26}N_2O_2$, the pyrrolidine ring adopts an envelope conformation with the N atom deviating by 0.600 (1) Å from the plane of the other four atoms. The molecule is stabilized by weak intramolecular C—H···O interactions and the crystal packing is stabilized by N—H···O and C—H···O hydrogen bonds, generating centrosymmetric dimers of $R_2^2(10)$ and $R_2^2(14)$ rings.

Comment

Spiro-indole-pyrrolidine ring systems have acquired a special place in the heterocyclic field as this is a frequently encountered structural motif in many pharmacologically relevant alkaloids (Amal Raj *et al.*, 2003). It has been shown that functionalized pyrrolidines inhibit γ-mannosidase activity and growth of human glioblastoma and melanoma cells (Fiaux *et al.*, 2005). Pyrrolidine dithiocarbamate exerts anti-proliferative and pro-apoptotic effects in renal cell carcinoma cell lines (Morais *et al.*, 2006).



(I)

The bond lengths and bond angles in the title compound, (I) (Table 1), are comparable to similar structures determined previously (Satis Kumar *et al.*, 2006). The sum of the bond angles around N1 atom (336.9°) indicates sp^3 hybridization. Atom O1 deviates by 0.069 (1) Å from the C6/C7/C16/C15/C1 plane. The pyrrolidine ring (N1/C1–C4) adopts an envelope conformation, with atom N1 deviating by 0.600 (1) Å from the other atoms in the ring. The acenapthene unit is planar with a dihedral angle between the two benzene rings of 1.6 (1)°; the dihedral angles between the benzene rings (C7–C11/C16) and (C11–C16) and the five-membered ring are 0.5 (1) and 1.8 (1)°, respectively. The dihedral angle between the planar pyrrole (C19/C20/C25/N2/C26) and the C20–C25 benzene ring is 2.6 (1)°, while that between the two C20–C25 and C28–C33 benzene rings is 89.6 (1)°. The puckering parameters (Cremer

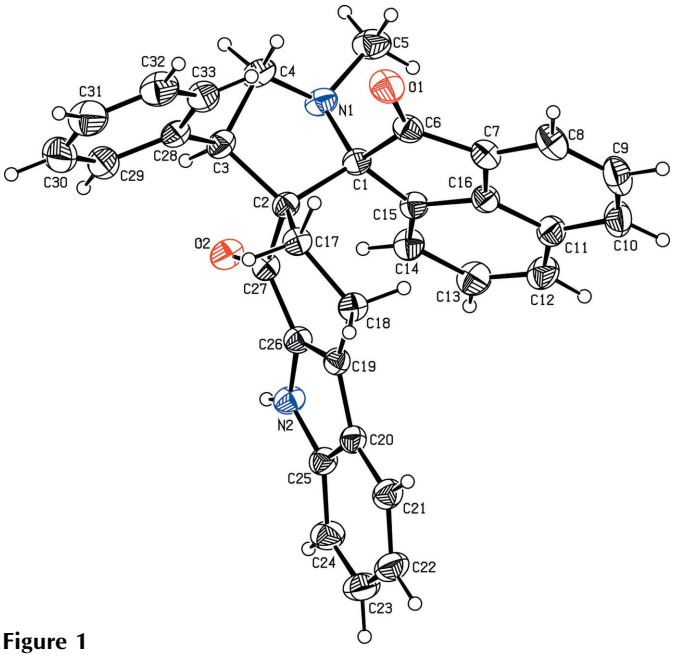


Figure 1
The molecular structure of the title compound, (I), showing 30% probability displacement ellipsoids.

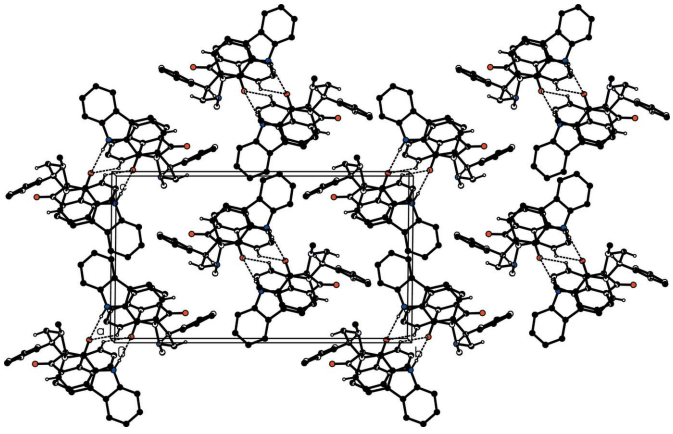


Figure 2
The molecular packing of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines. For the sake of clarity, H atoms not involved in hydrogen bonding have been omitted.

& Pople, 1975) and the smallest displacement asymmetry parameters (Nardelli, 1983) for the pyrrolidine ring are q_2 = 0.411 (2), φ = 178.9 (2)° and $\Delta_s(N_1)$ = 2.1 (1).

The molecule is stabilized by weak intramolecular C—H···O interactions and the crystal packing is stabilized by intermolecular N—H···O and C—H···O interactions. In the N—H···O and C—H···O hydrogen bonds, atoms N2 and C14 act as donors to O2($-x$, 1 − y, 1 − z), generating centrosymmetric dimers of $R_2^2(10)$ and $R_2^2(14)$ rings.

Experimental

A mixture of acenaphthaquinone (0.18 g, 1 mmol), sarcosine (0.89 g, 1 mmol) and 2-benzylidene-3,4-tetrahydro-1-ketocarbazole (0.31 g, 1 mmol) was refluxed in 1,4-dioxane–methanol (1:1) for 6 h. The reaction was monitered by thin-layer chromatography and after completion of the reaction, the solvent was removed under reduced pressure. The crude product was subjected to column chromatography (silica gel, 100–200 mesh) using hexane–EtOAc (8:2) as eluent. The pure compound was recrystallized from EtOAc.

Crystal data

$C_{33}H_{26}N_2O_2$
M_r = 482.56
Monoclinic, $P2_1/c$
a = 9.2279 (6) Å
b = 21.9358 (13) Å
c = 12.7618 (8) Å
β = 103.163 (1)°
V = 2515.4 (3) Å^3
Z = 4
D_x = 1.274 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 (2) K
Block, yellow
0.24 × 0.22 × 0.21 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
28499 measured reflections
5873 independent reflections
4870 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 28.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.146
S = 1.00
5873 reflections
335 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0882P)^2 + 0.4529P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.471 (2)	C3—C4	1.531 (2)
C1—C15	1.526 (2)	C4—N1	1.453 (2)
C1—C6	1.582 (2)	C5—N1	1.458 (2)
C1—C2	1.585 (2)	C6—O1	1.216 (2)
C2—C27	1.543 (2)	C25—N2	1.363 (2)
C2—C17	1.545 (2)	C26—N2	1.381 (2)
C2—C3	1.583 (2)	C27—O2	1.224 (2)
C3—C28	1.517 (2)		
C4—N1—C5	114.3 (1)	C5—N1—C1	115.6 (1)
C4—N1—C1	107.0 (1)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2^{i}	0.86	2.27	2.929 (2)	133
C3—H3···O2	0.98	2.23	2.779 (2)	115
C4—H4B···O1	0.97	2.48	3.058 (2)	118
C14—H14···O2^{i}	0.93	2.60	3.325 (2)	135
C17—H17A···O1	0.97	2.51	3.187 (2)	127
C33—H33···O1	0.93	2.57	3.391 (2)	148

Symmetry code: (i) $-x, -y+1, -z+1$.

All H atoms were refined using a riding model, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic, C—H = 0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) for CH, C—H = 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) for CH$_2$, C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for CH$_3$, and N—H = 0.86 Å and U_{iso}(H) = 1.2U_{eq}(N) for NH hydrogens.

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:

PLATON (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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