1,2,3,4-Tetrahydro-4′-furfuryl-1′-methyl-naphthalene-2-spiro-3′-pyrrolidine-2′-spiro-1″-acenaphthene-1,2″-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.055
wR factor = 0.187
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.

In the title compound, $C_{29}H_{23}NO_3$, the dihedral angle between the fused rings in the tetrahydronaphthalene moiety is 8.0 (7)°. The pyrrolidine ring adopts an envelope conformation and is orthogonal to the tetrahydronaphthalene and acenaphthene rings, the dihedral angles being 89.0 (4) and 85.5 (5)°, respectively. The molecular structure and the packing are stabilized by intramolecular C—H···O interactions.

Comment

Pyrrolidine compounds are reported to exhibit antimicrobial and antifungal activity (Amal Raj *et al.*, 2003). They are also found to occur in many biologically important alkaloids, as reported in the literature (Cordel, 1981). The importance of this type of compound prompted us to undertake the X-ray analysis of the title compound, (I), and the results are presented here.
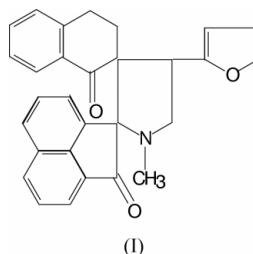

(I)

Fig. 1 shows a displacement ellipsoid diagram of the molecule with the atomic numbering scheme. Selected geometric parameters are given in Table 1. The bond lengths in the pyrrolidine moiety are slightly greater than the values reported for similar structures (Jeyabharathi *et al.*, 2001; Govind *et al.*, 2003). This may be due to steric forces caused by the bulky substituents on the pyrrolidine moiety. The sum of the angles at N1 of the pyrrolidine ring [336.8°] is in accordance with sp^3 hybridization (Beddoes *et al.*, 1986).

The atom O32 attached at C16 deviates by 0.176 (2) Å from the plane of the acenaphthene moiety. The deviation of atom O33 from the tetrahydronaphthalene plane is 0.227 (2) Å. Atoms N1 and C31 are displaced by −0.227 (2) and 0.097 (3) Å, respectively, from the C2/C3/C4/C5 plane.

The furfuryl ring is attached in an equatorial position to the pyrrolidine ring. The pyrrolidine ring is perpendicular to both the acenaphthene and tetrahydronaphthalene ring systems, the dihedral angles being 89.0 (4) and 85.5 (5)°, respectively.

The asymmetry parameters (Nardelli, 1995) q_2 = 0.396 (2) Å, φ = 29.9 (4)° and ΔC_2[C3—C2] = 0.0642 (2)°
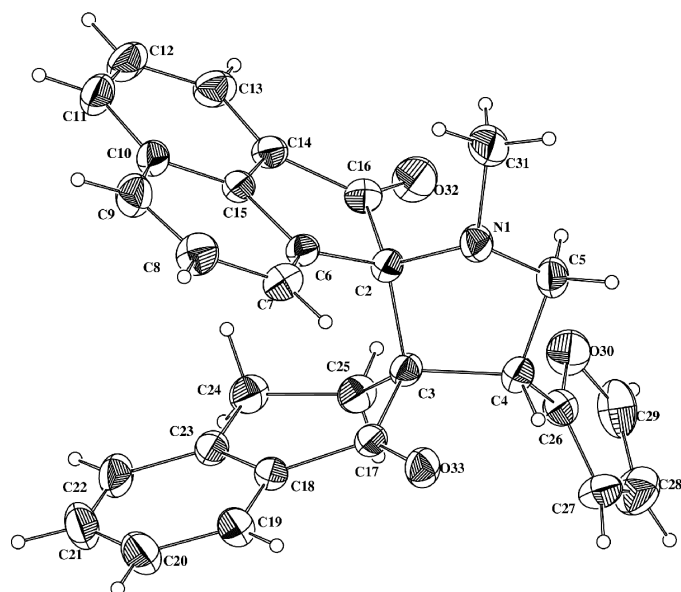
Figure 1
View of (I) (50% probability displacement ellipsoids).

Figure 2
Packing diagram, with hydrogen bonds shown as dashed lines.

reveal the conformation of the pyrrolidine ring to be that of an envelope. The cyclohexanone fused to the benzene ring adopts a half-chair conformation, which is confirmed by the asymmetry parameters $q_2 = 0.233$ (2) Å, $\varphi = -138.1$ (5)°, $\Delta C_2[\text{C}18] = 0.038$ (8)° and $\Delta_S[\text{C}3] = 0.090$ (8)°.

The molecular structure is influenced by C—H···O intramolecular interactions. In the crystal structure, C4—H4···O33^i hydrogen bonds link inversion-related molecules to form dimers (Fig. 2 and Table 2). The crystal packing is stabilized by C—H···O intermolecular interactions.

Experimental

To a solution of sarcosine (1 mmol) and acenaphthoquinone (1 mmol) in an aqueous solution of CH_3OH was added the dipolarophile 2-furfurylidene-1-tetralone. The solution was refluxed for 3–4 h. The crude mixture obtained after the removal of solvent was subjected to column chromatography with petroleum ether and ethyl acetate (9:1) as eluant, to obtain (I).

Crystal data

$C_{29}H_{23}NO_3$	$D_x = 1.267$ Mg m^{-3}
$M_r = 433.48$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 4019
$a = 11.9109$ (7) Å	reflections
$b = 11.3292$ (7) Å	$\theta = 2.2$–25.8°
$c = 17.3573$ (10) Å	$\mu = 0.08$ mm^{-1}
$\beta = 103.993$ (1)°	$T = 293$ (2) K
$V = 2272.7$ (2) Å^3	Needle, yellow
$Z = 4$	$0.18 \times 0.17 \times 0.13$ mm

Data collection

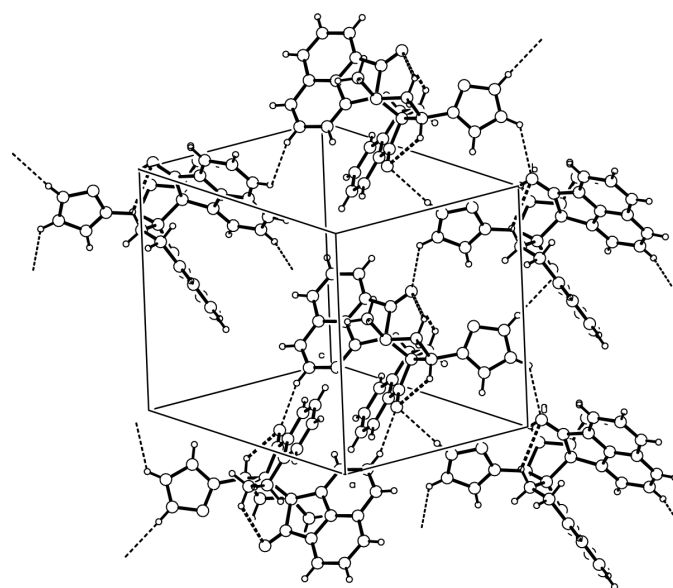|	
Bruker SMART APEX CCD area-detector diffractometer	3529 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.022$
Absorption correction: none	$\theta_{max} = 28.0°$
14010 measured reflections	$h = -15 \rightarrow 8$
5225 independent reflections	$k = -14 \rightarrow 14$
	$l = -21 \rightarrow 22$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1086P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.055$	$+ 0.3011P]$
$wR(F^2) = 0.187$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} < 0.001$
5225 reflections	$\Delta\rho_{max} = 0.33$ e Å^{-3}
298 parameters	$\Delta\rho_{min} = -0.25$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

N1—C5	1.459 (2)	C2—C3	1.591 (2)
N1—C2	1.464 (2)		
C31—N1—C5	113.8 (2)	C5—N1—C2	106.7 (1)
C31—N1—C2	116.2 (2)		
C5—C4—C26—O30	−45.9 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O33	0.98	2.21	2.762 (2)	114
C5—H5B···O32	0.97	2.51	3.071 (3)	117
C25—H25A···O32	0.97	2.48	3.141 (3)	126
C29—H29···O33^i	0.93	2.52	3.417 (3)	163
C28—H28···O32^{ii}	0.93	2.58	3.217 (4)	126
C8—H8···O33^{iii}	0.93	2.52	3.392 (3)	156

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

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å and $U_{iso}(\text{H}) = 1.5U_{eq}(\text{C})$ for methyl H atoms and $1.2U_{eq}(\text{C})$ for other H atoms.

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, 1990); software used

References

Amal Raj, A., Ragunathan, R., Sridevi Kumari, M. R. & Raman, N. (2003). *Bioorg. Med. Chem.* **11**, 407–409.

Beddoes, R. L., Dalton, L., Joule, T. A., Mills, O. S., Street, J. D. & Watt, C. I.-F. (1986). *J. Chem. Soc. Perkin Trans.* 2, pp. 787–797.

Bruker (2001). *SAINT* (Version 6.28a) and *SMART* (Version 5.625). Bruker AXS Inc., Madison, Wisconsin, USA.

Cordel, G. (1981). *Oxindole Alkaloids, Alkaloid Chemistry and Physiology*, edited by R. H. F. Manke. New York: Academic Press.

Govind, M. M., Selvanayagam, S., Velmurugan, D., Ravikumar, K., Sridhar, G. & Raghunathan, R. (2003). *Acta Cryst.* E**59**, o1438–o1440.

Jeyabharathi, A., Ponnusamy, M. N., Amalraj, R., Ragunathan, R., Razak, I. A., Usman, A., Chandrapromma, S. & Fun, H.-K. (2001). *Acta Cryst.* E**57**, o901–o903.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.

Zsolnai, L. (1997). *ZORTEP*. University of Heidelberg, Germany.