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1′-Methyl-2″-phenylcyclohexane-1-spiro-4′-[acenaphthene-1-spiro-2′-pyrrolidine-3′-spiro-4″(5″*H*)-[1,3]oxazole]-2,5″-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.044
wR factor = 0.134
Data-to-parameter ratio = 14.8

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

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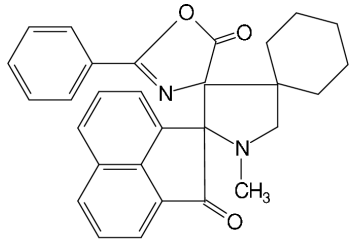
In the title compound, $C_{29}H_{26}N_2O_3$, the pyrrolidine ring adopts an envelope conformation and the cyclohexane ring adopts a chair conformation. The structure is stabilized by intramolecular C—H···O and π–π interactions.

Comment

The pyrrolidine skeleton occurs in many families of biologically important compounds. The resulting functionality, due to ease of substitution and, therefore, modification at several positions (Baldwin *et al.*, 1994*a*,*b*), has been utilized to synthesize compounds with varying properties. Such derivatives are found to have antimicrobial and antifungal activity against various pathogens, except *Bacillus subtilis* (Amal Raj *et al.*, 2003). An acenaphthene derivative was found to have high κ-opioid receptor affinity and selectivity (Halfpenny *et al.*, 1991). These derivatives are used as new conformationally restricted ligands for melatonin receptors (Jellimann *et al.*, 2000), liver regeneration (Gershbein, 1975) and antitumoral agents (Boido *et al.*, 1994).



(I)

The C—C bond lengths in the pyrrolidine moiety are somewhat longer and the C—N bond lengths are somewhat shorter than normal values (Table 1). A similar effect has been observed in related reported structures (Abdul Ajees *et al.*, 2002; Usha *et al.*, 2003. The geometry of the acenaphthene moiety compares well with that reported in other compounds, for example, by Edwards *et al.* (1980) and Govind *et al.* (2004). The C—C bond lengths in the phenyl and cyclohexane rings are comparable to the reported mean values of 1.384 (13) and 1.535 (16) Å, respectively (Allen *et al.*, 1987).

The sum of the angles (337.2°) at atom N1 is in accordance with sp^3 hybridization. The torsion angle N19—C20—C24—C29 of 4.5 (2)° indicates that the conformation of the attachment of the phenyl ring with respect to the oxazolone ring is +*syn*-periplanar; the dihedral angle between these two rings is 5.8 (1)°. The acenaphthene moiety is planar, with a maximum deviation of 0.109 (2) Å for atom C7; the attached atom O18 deviates by 0.386 (1) Å from this plane.

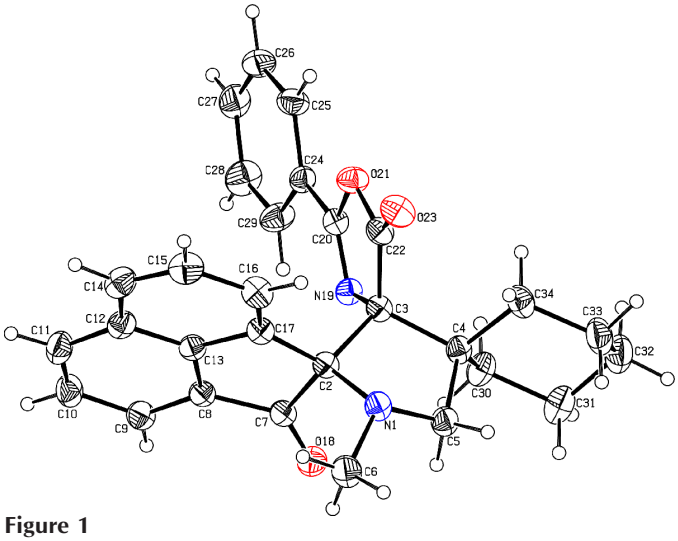


Figure 1
The molecular structure and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

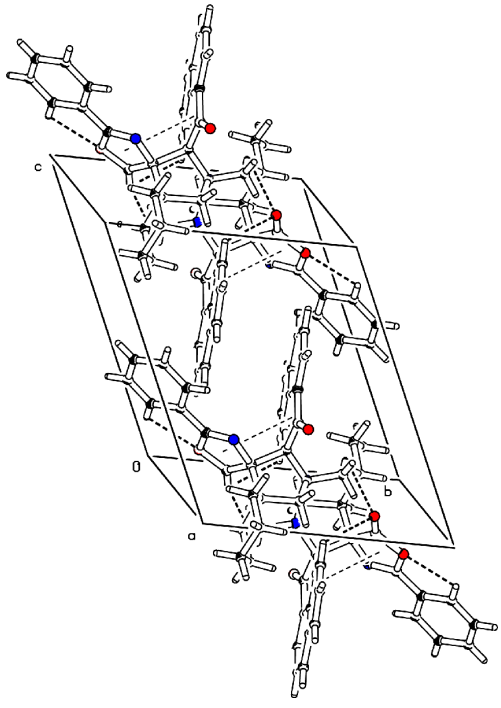


Figure 2
The molecular packing of (I), viewed approximately down the *a* axis. Dashed lines indicate intramolecular hydrogen bonds and π–π interactions.

The pyrrolidine ring adopts an envelope conformation with puckering parameters q_2 = 0.382 (1) Å and φ = 7.5 (2)° (Cremer & Pople, 1975). Atom N1 deviates by 0.562 (1) Å from the least-squares plane through the remaining four atoms C2–C5 of the ring. The cyclohexane ring adopts a chair conformation, confirmed by the puckering parameters q_2 = 0.032 (2) Å, q_3 = −0.555 (2) Å, Q_T = 0.556 (2) Å and θ = 176.8 (2)°. Atoms C4, C30, C32 and C33 lie in a plane, whereas C31 and C34 deviate by −0.668 (2) and 0.624 (2) Å from the plane.

In addition to van der Waals interactions, the structure is stabilized by intramolecular C—H···O hydrogen bonds (Table 2 and Fig. 2), also by π–π interactions between the oxazolone ring (C3/N19/C20/O21/C22) and the five-membered ring of the acenaphthene moiety (C2/C7/C8/C13/C17), with a centroid–centroid separation of 3.048 (1) Å (Fig. 2).

Experimental

To a refluxing solution of 2-cyclohexylideneoxazol-1-one (1 mmol) in methanol was added sarcosine (1 mmol) and acenapthenequinone (1 mmol); the mixture was refluxed for 2 h. The solvent was evaporated under reduced pressure and the residue was subjected to column chromatography to afford the title trispiro compound; this had not been reported earlier in the literature, using 1,3-dipolar cycloaddition methodology in a one-pot synthesis. The compound was recrystallized from a methanol solution, yielding crystals of good diffraction quality.

Crystal data

$C_{29}H_{26}N_2O_3$
M_r = 450.52
Triclinic, $P\overline{1}$
a = 9.8626 (8) Å
b = 10.4697 (9) Å
c = 12.5629 (11) Å
α = 114.049 (1)°
β = 103.946 (1)°
γ = 92.633 (2)°
V = 1134.39 (17) Å^3
Z = 2
D_x = 1.319 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2364 reflections
θ = 2.5–22.9°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colourless
0.24 × 0.22 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
6911 measured reflections
4539 independent reflections
3881 reflections with $I > 2\sigma(I)$
R_{int} = 0.013
θ_{max} = 28.0°
$h = -11 \rightarrow 12$
$k = -11 \rightarrow 13$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.134
S = 1.00
4539 reflections
307 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0865P)^2 + 0.1476P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C5	1.449 (2)	C4—C30	1.517 (2)
N1—C2	1.451 (2)	C4—C34	1.539 (2)
N1—C6	1.456 (2)	C4—C5	1.544 (2)
C2—C17	1.517 (2)	C7—O18	1.206 (2)
C2—C3	1.574 (2)	N19—C20	1.265 (2)
C2—C7	1.586 (2)	C20—O21	1.386 (2)
C3—N19	1.462 (2)	O21—C22	1.389 (2)
C3—C22	1.531 (2)	C22—O23	1.185 (2)
C3—C4	1.595 (2)		
C5—N1—C2	107.5 (1)	C2—N1—C6	115.1 (1)
C5—N1—C6	114.6 (1)		
N19—C20—C24—C29	4.5 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5A···O18	0.97	2.54	3.067 (2)	114
C16—H16···O23	0.93	2.57	3.029 (2)	111
C25—H25···O21	0.93	2.45	2.781 (2)	101
C34—H34B···O23	0.97	2.49	2.944 (2)	109

The H atoms were positioned geometrically and treated as riding on their parent C atoms, with aromatic C—H = 0.93 Å, methyl C—H = 0.96 Å and other Csp^3—H = 0.97 Å; U_{iso}(H) = 1.5U_{eq}(C) for methyl H and 1.2U_{eq}(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: *ORTEP*-3 (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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