organic papers

1N-Methyl-spiro[2-3']indan-1,3-dione-spiro[3-3'']-5''-benzylidene-N-methyl-piperidinone-4-phenylpyrrolidine

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.066
wR factor = 0.186
Data-to-parameter ratio = 17.4

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

The conformation of the pyrrolidine ring of the title compound, $C_{31}H_{28}N_2O_3$, lies between half-chair and envelope. The dihedral angle between the fused rings is 3.4 (1)°.

Comment

Compounds containing the pyrrolidine moiety are biologically important as they occur in many pharmacologically significant alkaloids (Bindra, 1973). Several optically active pyrrolidine compounds have been used as intermediates in controlled asymmetric synthesis (Suzuki *et al.*, 1994). They are also found to be antimicrobial and antifungal (Govind *et al.*, 2003). Owing to the medicinal importance of the title compound, (I), its structure has been characterized and the results are presented here.
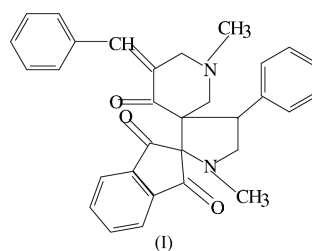

(I)

Fig. 1 shows the molecule with the atomic numbering scheme. Selected geometric parameters are given in Table 1.

The bond lengths in the pyrrolidine moiety (Table 1) are slightly longer than normal values, but they are comparable with those in reported structures (Jeyabharathi *et al.*, 2001; Gzella & Wrzeciono, 1990). This may be due to steric forces caused by the bulky substituents at pyrolidine moiety. The sum of the angles at atom N1 of the pyrolidine ring (337.8°) and at N15 (334.9°) are in accordance with sp^3 hybridization (Beddoes *et al.*, 1986). The phenyl ring is attached equatorially to the pyrrolidine ring..

The asymmetry parameters [q_2 = 0.443 Å, φ = 117.5 (3)°, ΔC_2(C5) = 0.0618 (1)° and Δ_S(C3) = 0.0370 (1)°; Nardelli, 1995] show the conformation of pyrrolidine ring to be between a half-chair and an envelope. The piperidinone ring adopts a half-chair conformation, as revealed by the asymmetry parameters [q_2 = 0.299 (2), φ = 107.3 (4)°, ΔC_2(C17–C18) = 0.0493 (1)° and Δ_S(C17) = 0.0517 (1)°].

In addition to van der Waals interactions, the molecular structure is stabilized by weak C—H···O intramolecular interactions. The crystal structure is stabilized by C—H···O intermolecular interactions (Fig. 2). Furthermore, symmetry-related molecules are linked by weak C—H···π interactions, so that H33B is 2.81 Å from the centroid of the phenyl ring
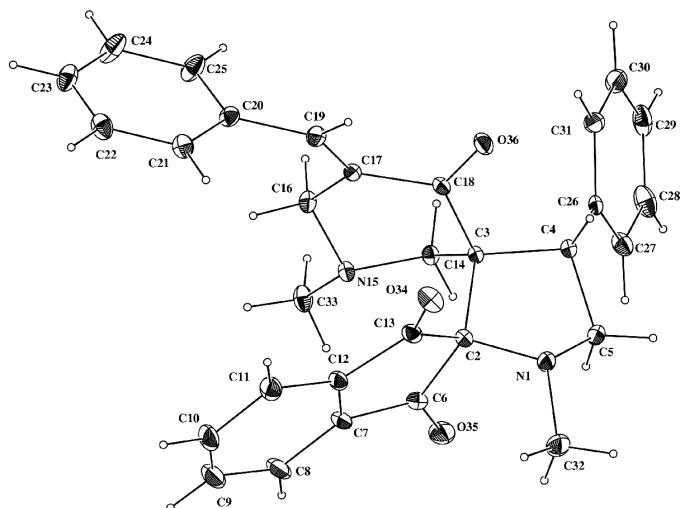
Figure 1
View of the title compound (50% probability displacement ellipsoids).
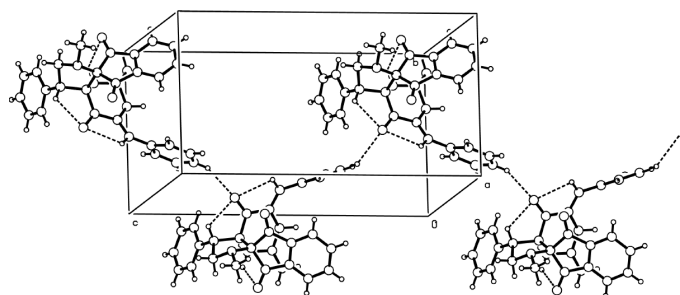

Figure 2
Packing diagram of the title compound, viewed approximately along the a axis.

(C26–C31) at $(x, \frac{3}{2} - y, z - \frac{1}{2})$, with a C33—H33B···centroid angle of 146° and a C33···centroid distance of 3.654 (3) Å.

Experimental

A mixture of a dipolarophile (dibenzylidine-*N*-methylpiperidone), ninhydrin and sacrosine was refluxed for about 5–6 h in aqueous methanol until the starting material had disappeared, as evidenced by TLC. After completion of the reaction, the solvent was removed *in vacuo* and the residue was chromatographed on silica gel, using a hexane–ethyl acetate mixture as eluent. Recrystallization from methanol yielded the title compound.

Crystal data

$C_{31}H_{28}N_2O_3$	$D_x = 1.272$ Mg m^{-3}
$M_r = 476.55$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 5701
$a = 14.3186$ (11) Å	reflections
$b = 9.6801$ (7) Å	$\theta = 2.3$–28.0°
$c = 17.9616$ (13) Å	$\mu = 0.08$ mm^{-1}
$\beta = 91.190$ (1)°	$T = 293$ K
$V = 2489.0$ (3) Å^3	Block, colourless
$Z = 4$	$0.30 \times 0.25 \times 0.20$ mm

Data collection

Bruker SMART APEX CCD-	$R_{int} = 0.036$
detector diffractometer	$\theta_{max} = 28.0°$
ω scans	$h = -18 \rightarrow 18$
14 792 measured reflections	$k = -12 \rightarrow 11$
5701 independent reflections	$l = -23 \rightarrow 17$
3121 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0886P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.066$	$+ 0.1571P]$
$wR(F^2) = 0.186$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} < 0.001$
5701 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}
327 parameters	$\Delta\rho_{min} = -0.16$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

N1—C5	1.454 (3)	C2—C3	1.601 (3)
N1—C2	1.455 (3)		
C5—N1—C2	106.5 (2)	C14—N15—C16	110.5 (2)
C5—N1—C32	115.3 (2)	C14—N15—C33	113.1 (2)
C2—N1—C32	116.0 (2)	C16—N15—C33	111.3 (2)
C5—C4—C26—C31	−160.7 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O36	0.98	2.36	2.825 (3)	109
C14—H14B···O35	0.97	2.42	2.970 (3)	115
C19—H19···O36	0.93	2.47	2.794 (3)	100
C23—H23···O36^i	0.93	2.52	3.252 (3)	136

Symmetry code: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$.

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}(H) = 1.5 U_{eq}(C) for methyl H atoms and 1.2 U_{eq}(C) for other H atoms. The methyl groups were allowed to rotate but not to tip.

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); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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