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1′-Methyl-4′-phenylindan-2-spiro-2′-pyrrolidine-3′-spiro-1″-cyclooctane-1,3,2″-trione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.061
wR factor = 0.167
Data-to-parameter ratio = 18.0

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

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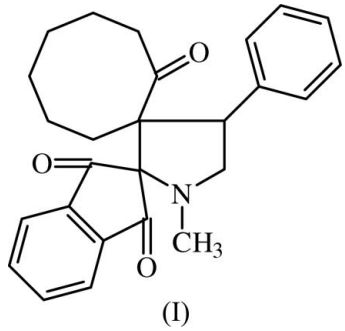
In the title compound, $C_{26}H_{27}NO_3$, the pyrrolidine ring adopts an envelope conformation. The indanedione group is planar, the dihedral angle between the fused five- and six-membered rings of this group being 1.1 (2)°. There are intramolecular C—H···O and C—H···π interactions.

Comment

Pyrrolidine derivatives possess anti-influenza virus (Stylianakis *et al.*, 2003), anticonvulsant (Obniska & Zagorska, 2003; Obniska *et al.*, 2005), and other antiviral (Kolocouris *et al.*, 1994) activities. They are found to have antimicrobial and antifungal activities against various pathogens except *Bacillus subtilis* (Amal Raj *et al.*, 2003). In view of its importance and to obtain more detailed information of the structure and conformation of the molecule, the crystal structure of the title compound, (I), was determined.



(I)

The molecular structure of (I) is illustrated in Fig. 1. Selected geometric parameters are presented in Table 1. All the C—C and C—N bond lengths in the pyrrolidine ring are comparable with the values in related dispiro-pyrrolidine derivatives (Abdul Ajees *et al.*, 2002; Gzella & Wrzeciono, 1990; Seshadri *et al.*, 2003).

The indanedione group is planar, with a maximum deviation of 0.069 (2) Å for atom C1. The keto atoms O1 and O2 deviate from the mean plane through the fused-ring system by 0.241 (2) and 0.236 (2) Å, respectively. The dihedral angle between the fused six-membered and five-membered rings is 1.1 (2)°.

The sum of the angles at N1 of the pyrrolidine ring (338.3°) is in accordance with sp^3-hybridization. The methyl group is attached equatorially to the pyrrolidine ring. The pyrrolidine ring adopts an envelope conformation with puckering parameters q_2 = 0.394 (2) Å and φ = 171.7 (3)° (Cremer & Pople, 1975). Atom N1 deviates by 0.560 (2) Å from the least-squares plane through the remaining four atoms (C1/C2/C3/C4) of the ring. This plane makes a dihedral angle of 79.8 (1)° with the

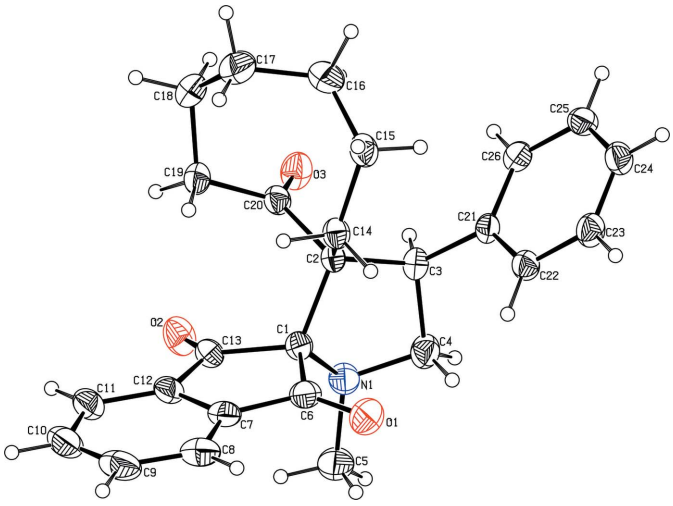


Figure 1
The molecular structure and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

indanedione group and 78.2 (1)° with the phenyl ring. The indanedione group makes a dihedral angle of 39.5 (1)° with the phenyl ring. The eight-membered ring adopts a boat–chair conformation.

The molecular structure is stabilized by weak C—H···O interactions (Table 2), and further influenced by a C—H···π interaction, such that atom H15*B* is 2.66 Å from the centroid of the C21–C26 benzene ring, with a C15—H15*B*···centroid angle of 154° and a C15···centroid distance of 3.557 (2) Å.

Experimental

A mixture of benzylidene octanone (1 mmol), ninhydrin (1.2 mmol) and sarcosine (1.2 mmol) in 20 ml of methanol was refluxed until the disappearance of the starting materials. The solvent was then evaporated *in vacuo* and the residue was chromatographed on a column (silica gel, 100–200 mesh) eluted with a hexane–ethyl acetate (9:1 *v*/*v*) mixture to obtain the title compound, which was recrystallized from methanol.

Crystal data

$C_{26}H_{27}NO_3$
M_r = 401.49
Monoclinic, $P2_1/c$
a = 14.3552 (8) Å
b = 9.9061 (6) Å
c = 15.9692 (9) Å
β = 113.700 (1)°
V = 2079.4 (2) Å^3
Z = 4
D_x = 1.282 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 (2) K
Block, colourless
0.24 × 0.22 × 0.19 mm

Data collection

Bruker SMART APEX diffractometer
ω scans
Absorption correction: none
23284 measured reflections
4895 independent reflections
3862 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 28.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.167
S = 1.02
4895 reflections
272 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0889P)^2 + 0.6378P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C4	1.449 (2)	O3—C20	1.205 (2)
N1—C1	1.449 (2)	C1—C2	1.598 (2)
N1—C5	1.460 (3)	C2—C3	1.584 (2)
O1—C6	1.208 (2)	C3—C4	1.529 (3)
O2—C13	1.205 (2)		
C4—N1—C1	107.1 (1)	C1—N1—C5	116.4 (2)
C4—N1—C5	114.8 (2)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O3	0.98	2.20	2.759 (2)	115
C4—H4*B*···O1	0.97	2.60	3.149 (3)	116
C14—H14*B*···O1	0.97	2.47	3.133 (2)	126
C19—H19*B*···O2	0.97	2.58	3.011 (2)	107

The H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C—H = 0.93–0.98 Å, and with U_{iso}(H) = 1.5U_{eq}(C) for methyl 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: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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