

## 1'-Methyl-4'-phenylacenaphthene-1-spiro-2'-pyrrolidine-3'-spiro-2''-indan-2,1'',3''-trione

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## Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$

$R$  factor = 0.052

w $R$  factor = 0.151

Data-to-parameter ratio = 14.6

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

In the title compound,  $\text{C}_{30}\text{H}_{21}\text{NO}_3$ , the pyrrolidine ring adopts a twist conformation. The dihedral angle between the indan moiety and the phenyl ring is  $75.4(1)^\circ$ . The structure is stabilized by intramolecular  $\text{C}-\text{H}\cdots\text{O}$  interactions.

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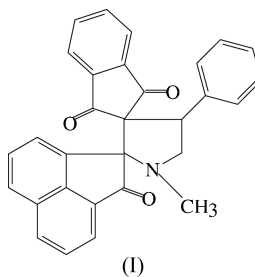
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## Comment

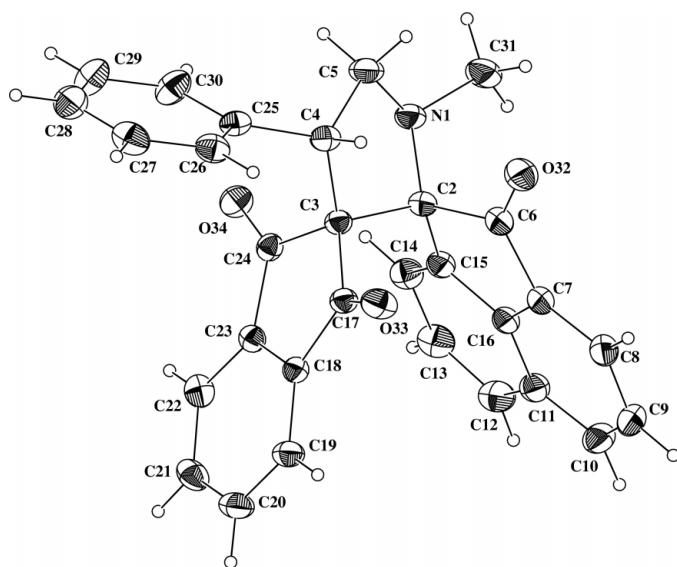
Spiro compounds are frequently of structural significance in many pharmacologically relevant alkaloids (Cravotto *et al.*, 2001) and the present study is part of an ongoing investigation of these.

In the title compound, (I), the C—C bond lengths in the pyrrolidine moiety are somewhat longer and the C—N bond lengths are somewhat shorter than normal. A similar effect has been observed in a related structure (Abdul Ajees *et al.*, 2002). This effect may be due to steric forces caused by the bulky substituents on the pyrrolidine ring. The sum of angles ( $340.8^\circ$ ) at atom N1 (Fig. 1) is in accordance with  $sp^3$  hybridization (Beddoes *et al.*, 1986). The N1/C2—C5 ring adopts a twist conformation, with puckering parameters  $q_2 = 0.497(3)\text{ \AA}$  and  $\varphi = 174.2(4)^\circ$  (Cremer & Pople, 1975); the smallest displacement asymmetry parameter (Nardelli, 1983) is  $\Delta C_2(\text{C}2) = 0.058(1)$ .



The geometry of the indandione moiety is perfectly normal, as evidenced by 41 examples in the Cambridge Structural Database (November 2003 Release; Allen, 2002). Keto atom O33 lies  $0.256(2)\text{ \AA}$  from the indan plane, while keto atom O34 is displaced from the plane by  $0.170(1)\text{ \AA}$ . This difference may be due to the different environments of the two O atoms, O33 being involved in hydrogen bonding (Table 2) while O34 is not.

The geometry of the acenaphthylene moiety compares well with that reported in other compounds, for example, by Guirado *et al.* (1989), Edwards *et al.* (1980) and Suzuki *et al.* (2003). The five-membered C2/C6/C7/C16/C15 ring adopts a twist conformation, with puckering parameters (Cremer & Pople, 1975)  $q_2 = 0.148(3)\text{ \AA}$  and  $\varphi = -150.4(1)^\circ$ , and displacement asymmetry parameter  $\Delta C_2(\text{C}16) = 0.018(1)$ .



**Figure 1**  
View of (I), with the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

In addition to van der Waals interactions, the molecular structure is stabilized by a C—H···O intramolecular interaction (Table 2).

## Experimental

A mixture of benzylidene-1,3-indandione (1 mmol), acenaphthenequinone (1 mmol) and sarcosine (1 mmol) was refluxed in aqueous methanol until the completion of the reaction, as evidenced by thin-layer chromatography. The title compound was isolated by column chromatography.

### Crystal data

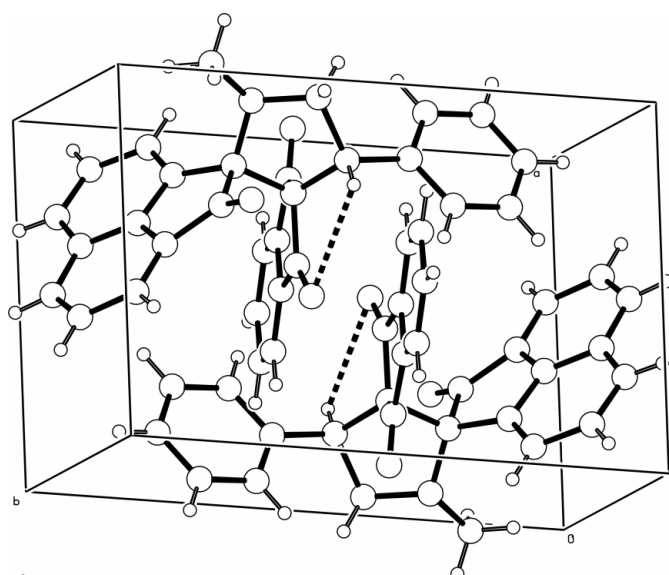
$C_{30}H_{21}NO_3$	$Z = 2$
$M_r = 443.48$	$D_x = 1.316 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.9022 (7) \text{ \AA}$	Cell parameters from 2474 reflections
$b = 11.9197 (10) \text{ \AA}$	$\theta = 2.5\text{--}26.9^\circ$
$c = 12.0382 (10) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$\alpha = 84.444 (2)^\circ$	$T = 293 (2) \text{ K}$
$\beta = 84.649 (2)^\circ$	Block, colourless
$\gamma = 84.176 (2)^\circ$	$0.21 \times 0.20 \times 0.20 \text{ mm}$
$V = 1118.76 (17) \text{ \AA}^3$	

### Data collection

Bruker SMART APEX CCD area-detector diffractometer	$R_{\text{int}} = 0.012$
$\omega$ scans	$\theta_{\text{max}} = 28.0^\circ$
6877 measured reflections	$h = -10 \rightarrow 10$
4493 independent reflections	$k = -14 \rightarrow 15$
3487 reflections with $I > 2\sigma(I)$	$l = -15 \rightarrow 11$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0889P)^2 + 0.1654P]$
$R[F^2 > 2\sigma(F^2)] = 0.052$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.151$	$(\Delta/\sigma)_{\text{max}} < 0.000$
$S = 1.00$	$\Delta\rho_{\text{max}} = 0.28 \text{ e \AA}^{-3}$
4493 reflections	$\Delta\rho_{\text{min}} = -0.15 \text{ e \AA}^{-3}$
308 parameters	
H-atom parameters constrained	



**Figure 2**  
Packing of the molecules, showing the intramolecular C—H···O interactions as dashed lines.

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

N1—C2	1.449 (2)	C2—C3	1.570 (2)
N1—C31	1.452 (3)	C3—C4	1.551 (2)
N1—C5	1.460 (2)	C4—C5	1.540 (2)
C2—N1—C31	116.5 (2)	C4—C3—C2	101.60 (13)
C2—N1—C5	109.6 (1)	C5—C4—C3	104.18 (13)
C31—N1—C5	114.7 (2)	N1—C5—C4	106.17 (13)
N1—C2—C3	101.54 (13)		
C5—C4—C25—C30	38.4 (3)		

**Table 2**

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
C4—H4···O33	0.98	2.60	2.997 (2)	104

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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