

4'-(*p*-Methoxyphenyl)-1'-methyl-1*H*-indole-3-spiro-2'-pyrrolidine-3'-spiro-1''-cyclopentane-2(3*H*),2''-dione

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Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$

R factor = 0.048

wR factor = 0.141

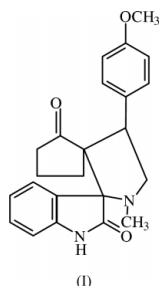
Data-to-parameter ratio = 16.6

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

The indole moiety of the title compound, $\text{C}_{23}\text{H}_{24}\text{N}_2\text{O}_3$, is planar; but the pyrrolidine ring adopts an envelope conformation. The conformation is stabilized by intramolecular $\text{C}-\text{H}\cdots\text{O}$ interactions and the packing of the molecules is stabilized by strong $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds.

Comment

Pyrrolidine derivatives occur in many biologically important compounds (Badwin *et al.*, 1994). The spiro(indole pyrrolidine) ring system is a frequently encountered structural motif in many pharmacologically important alkaloids (Cordel, 1981). Pyrrolidine compounds are capable of exhibiting antimicrobial and antifungal activity as reported in the literature (Amal Raj *et al.*, 2003). The above-mentioned uses of the compounds in medicine prompted us to undertake the X-ray analysis of the title compound and the results are presented here.



The bond lengths in the pyrrolidine moiety are slightly longer than normal values, but are comparable with values reported in similar structures (Gzella & Wrzeciono, 1990; Jeyabharathi *et al.*, 2001). This may be due to steric forces caused by the bulky substituents at various positions of the pyrrolidine ring. The sum of angles at N1 of the heterocyclic ring, 360° , is in accordance with sp^2 hybridization (Beddoes *et al.*, 1986) and the sum of the angles at N11 of the pyrrolidine moiety, 333° , is in accordance with sp^3 hybridization.

Atom O10 is essentially coplanar with the heterocyclic ring to which it is attached, with a deviation of $0.0598(1)\text{ \AA}$ and oxo atom O20 attached to C19 of cyclopentanone ring deviates by $-0.299(2)\text{ \AA}$.

The indole moiety (C2–C9/N1) is planar, with a dihedral angle between the planes of the heterocyclic and benzene rings of $2.4(6)^\circ$.

The asymmetry parameters (Nardelli, 1995) $q_2 = 0.427(1)\text{ \AA}$, $\varphi = 140.8(2)^\circ$, $\Delta_s(\text{N11}) = 0.0206(8)\text{ \AA}$ reveal that the pyrrolidine moiety adopts an envelope conformation. The cyclopentanone ring also adopts an envelope conformation.

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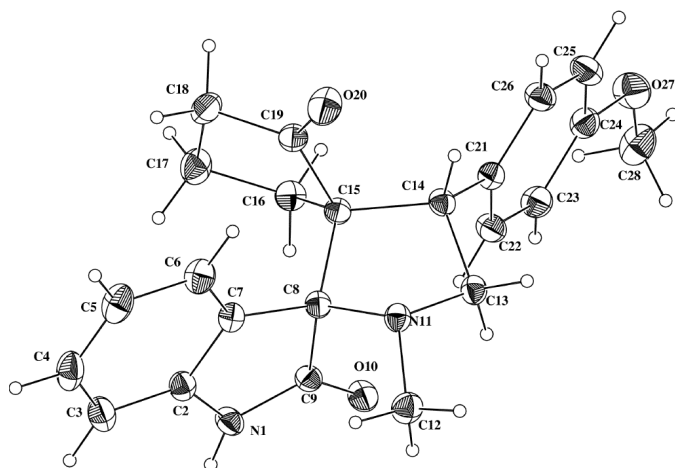


Figure 1
The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

This is confirmed by the puckering parameters $q_2 = 0.364$ (2) Å, $\varphi = 38.9$ (3)° and $\Delta_s(\text{C16}) = 0.0158$ (1) Å.

Methoxy atoms O27 and C28 tend to be coplanar with the phenyl moiety, as observed in anisoles (Domiano *et al.*, 1979), but the orientation of the methyl group is opposite, as shown by the torsion angles C28–O27–C24–C25 = 175.7 (2)° and C28–O27–C24–C23 = –4.8 (3)°. Similar results have been observed in related molecules reported in the literature (Usman *et al.*, 2002).

In addition to van der Waals interactions, the crystal structure is stabilized by C–H···O intramolecular interactions. The packing of the molecules is stabilized by strong N–H···N intermolecular hydrogen bonds which run along the *ac* plane (Fig. 2).

Experimental

A mixture of benzylidenecyclopentanone (0.5 mmol), isatin (0.5 mmol) and sarcosine (0.5 mmol) was refluxed. After the completion of the reaction, the solvent was removed *in vacuo* and the residue was chromatographed on silica gel and recrystallized from methanol, giving the title compound, (I).

Crystal data

$\text{C}_{23}\text{H}_{24}\text{N}_2\text{O}_3$	$Z = 2$
$M_r = 376.44$	$D_x = 1.317 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 6.3317$ (5) Å	Cell parameters from 2626 reflections
$b = 11.5470$ (8) Å	$\theta = 3.1\text{--}27.9^\circ$
$c = 14.1402$ (10) Å	$\mu = 0.09 \text{ mm}^{-1}$
$\alpha = 72.240$ (1)°	$T = 293$ (2) K
$\beta = 77.366$ (1)°	Block, colourless
$\gamma = 77.773$ (1)°	$0.24 \times 0.20 \times 0.18 \text{ mm}$
$V = 948.93$ (12) Å ³	

Data collection

Bruker SMART APEX area-detector diffractometer	4209 independent reflections
ω scans	3390 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	$R_{\text{int}} = 0.012$
$T_{\text{min}} = 0.979$, $T_{\text{max}} = 0.984$	$\theta_{\text{max}} = 28.0^\circ$
6064 measured reflections	$h = -8 \rightarrow 7$
	$k = -15 \rightarrow 15$
	$l = -18 \rightarrow 9$

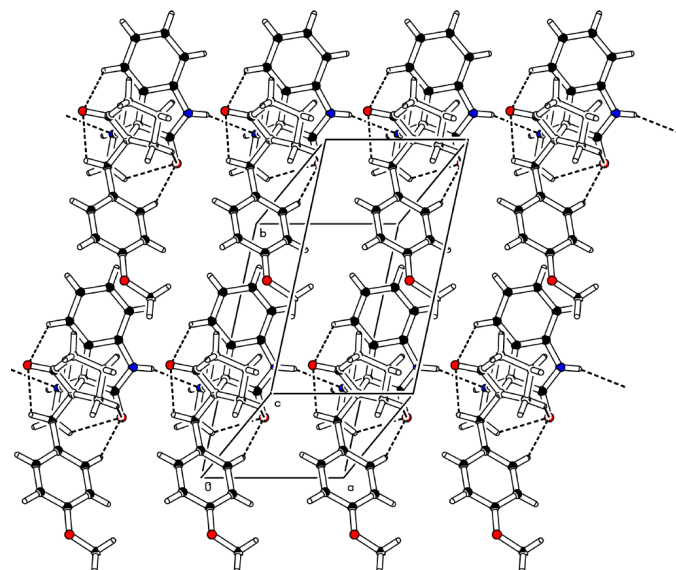


Figure 2
Packing of the molecules, viewed down the *a* axis.

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.082P)^2 + 0.136P]$
$R[F^2 > 2\sigma(F^2)] = 0.048$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.141$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.29 \text{ e \AA}^{-3}$
4209 reflections	$\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$
253 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, °).

N11–C12	1.463 (2)	N11–C8	1.478 (2)
N11–C13	1.465 (2)		
C9–N1–C2	111.8 (1)	C12–N11–C13	113.4 (2)
C9–N1–H1	124.1	C12–N11–C8	114.5 (1)
C2–N1–H1	124.1	C13–N11–C8	105.1 (1)
C28–O27–C24–C25	175.7 (2)	C28–O27–C24–C23	–4.8 (3)

Table 2

Hydrogen-bonding geometry (Å, °).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
C6–H6···O20	0.93	2.59	3.238 (2)	128
C13–H13A···O10	0.97	2.45	3.040 (2)	119
C14–H14···O20	0.98	2.35	2.876 (2)	113
C16–H16B···O10	0.97	2.50	3.183 (2)	127
C22–H22···O10	0.93	2.47	3.327 (2)	153
N1–H1···N11 ⁱ	0.86	2.38	3.194 (2)	157

Symmetry code: (i) $1 + x, y, z$.

The H atoms were positioned geometrically and were treated as riding on their parent atoms, with aromatic C–H distances of 0.93 Å, methylene C–H distances of 0.98 Å, ethylene C–H distances of 0.97 Å and N–H distances of 0.86 Å. $U_{\text{iso}} = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2U_{\text{eq}}(\text{C})$ for all other H atoms.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve

structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997) and *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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