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4′-(4-Methylphenyl)-3′-nitrospiro[1*H*-indole-3,2′-pyrrolidin]-2-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.063
wR factor = 0.151
Data-to-parameter ratio = 17.9

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

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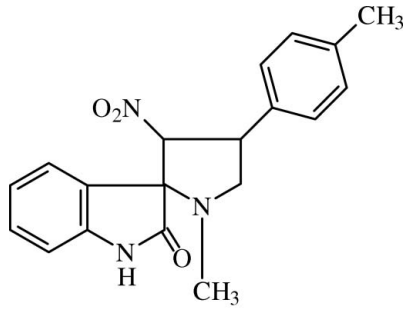
In the title compound, $C_{19}H_{19}N_3O_3$, the oxindole residue and the 4-methylphenyl ring are almost perpendicular to the pyrrolidine ring. The pyrrolidine ring adopts an envelope conformation. The molecular packing is stabilized by N—H···O hydrogen bonds and C—H···O interactions, in addition to van der Waals forces.

Comment

Heterocyclic compounds, particularly compounds containing five- and six-membered rings, have occupied a prominent place among various classes of organic compounds because of their diverse biological activities. Spiro[indole-pyrrolidine] ring systems have acquired a special place in the field of heterocyclic chemistry because they are a frequently encountered structural motif in many pharmacologically relevant alkaloids (Amal Raj *et al.*, 2003). These derivatives also possess anti-influenza virus (Stylianakis *et al.*, 2003) and anticonvulsant (Obniska *et al.*, 2002) activities. In order to determine the conformation of the title compound, (I), the present X-ray crystal structure determination was undertaken.



(I)

The molecular structure of (I) is illustrated in Fig. 1. Selected geometric parameters are presented in Table 1. The geometry of the pyrrolidine ring in (I) compares well with that reported in other related structures (see, for example, Selvanayagam *et al.*, 2004; Gzella & Wrzeciono, 1990).

The sum of the angles at N1 of the pyrrolidine ring [339.9°] is in accordance with sp^3 hybridization. The methyl group is attached equatorially to the pyrrolidine ring.

The dihedral angle between the oxindole and pyrrolidine rings (mean plane calculated through atoms N1/C1/C2/C3) is 87.1 (1)°, and that between the *p*-tolyl and pyrrolidine rings is 88.9 (1)°. This indicates that the oxindole residue and the *p*-tolyl ring are almost perpendicular to the pyrrolidine ring. The pyrrolidine ring adopts an envelope conformation, with

puckering parameters (Cremer & Pople, 1975) q_2 = 0.414 (2) Å and φ = −43.8 (3)°. Atom C4 deviates by 0.632 (2) Å from the least-squares plane through the remaining four atoms.

In the molecular packing, N—H···O hydrogen bonds link inversion-related molecules. The molecules are further linked by C—H···O interactions (Table 2).

Experimental

A mixture of 4-methylnitrostyrene (1 mmol), isatin (1 mmol) and sarcosine (1 mmol) was refluxed in methanol (20 ml) for 6 h. After completion of the reaction, the solvent was evaporated *in vacuo*. The residue was subjected to column chromatography using a hexane and ethyl acetate mixture (9:1) to yield the title compound. Single crystals of (I) suitable for X-ray diffraction were obtained from methanol.

Crystal data

$C_{19}H_{19}N_3O_3$
M_r = 337.37
Orthorhombic, *Pbca*
a = 9.8697 (8) Å
b = 16.4460 (14) Å
c = 21.4643 (18) Å
V = 3484.0 (5) Å^3
Z = 8
D_x = 1.286 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9788 reflections
θ = 2.7–26.6°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colourless
0.26 × 0.24 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
19862 measured reflections
4074 independent reflections
2927 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 28.0°
$h = -12 \rightarrow 12$
$k = -21 \rightarrow 21$
$l = -28 \rightarrow 28$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.151
S = 1.05
4074 reflections
228 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0679P)^2 + 1.113P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C5	1.449 (3)	C1—C2	1.538 (3)
N1—C4	1.452 (2)	C2—C3	1.526 (2)
N1—C1	1.465 (2)	C3—C4	1.540 (2)
C5—N1—C4	116.2 (2)	O1—N2—O2	124.0 (2)
C5—N1—C1	114.7 (2)	O1—N2—C3	119.7 (2)
C4—N1—C1	109.0 (2)		
C5—N1—C1—C2	−154.9 (2)	C7—C8—C9—C12	179.7 (2)
C5—N1—C4—C3	171.1 (2)	C12—C9—C10—C11	179.9 (2)

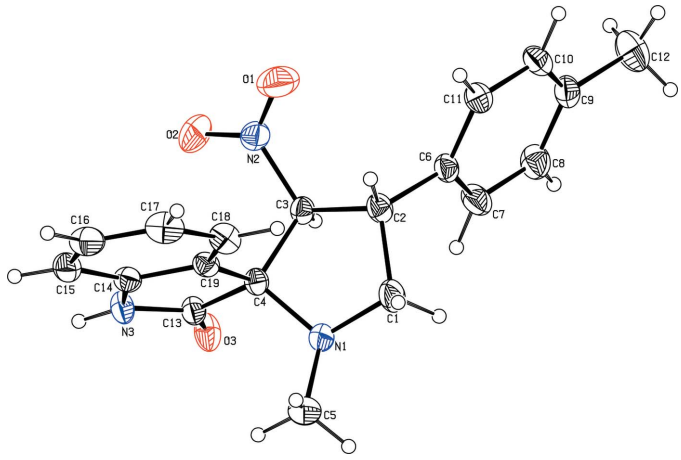


Figure 1
The molecular structure and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O3^{i}	0.86	2.00	2.849 (2)	171
C16—H16···O3^{ii}	0.93	2.60	3.525 (3)	174
C18—H18···O1^{iii}	0.93	2.58	3.430 (3)	153

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $-x+\frac{3}{2}, y-\frac{1}{2}, z$; (iii) $x+\frac{1}{2}, y, -z+\frac{3}{2}$.

The H atoms were positioned geometrically and treated as riding on their parent C atoms, with C—H distances of 0.93–0.98 Å and an N—H distance of 0.86 Å, and with U_{iso}(H) = 1.5U_{eq}(C) for methyl H and 1.2U_{eq}(C,N) for other H. In addition, 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: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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