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4′-(2-Chlorophenyl)-2,3-dihydro-1′-methyl-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) = 0.004 Å
R factor = 0.057
wR factor = 0.166
Data-to-parameter ratio = 22.5

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

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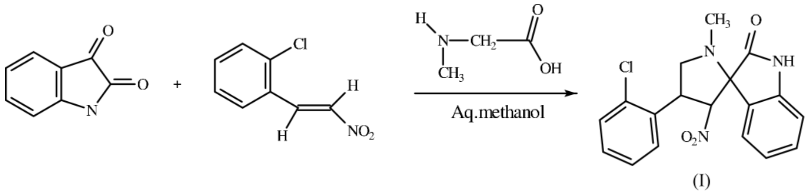
The crystal structure of the title compound, $C_{18}H_{16}ClN_3O_3$, was determined because of its biological and pharmaceutical properties. In the molecule, the spiro junction links a planar oxindole ring and a pyrrolidine ring. The pyrrolodine ring is in an envelope conformation. The methyl, nitro and chlorophenyl groups are equatorial with respect to the pyrrolidine ring. A centrosymmetric dimer is formed *via* N—H···O intermolecular hydrogen bonds. C—H···O and C—H···Cl intermolecular hydrogen bonds stabilize the molecules in the crystal structure. A couple of weak C—H···*π* intermolecular interactions are also observed.

Comment

Pyrrolidine derivatives possess many biologically important properties (Baldwin *et al.*, 1994). The spiro[indole-3,2′-pyrrolidine] ring system containing chlorine is a frequently encountered structural motif in many of the pharmacologically important alkaloids (Cordel, 1981). Oxindole alkaloids are important medicinal ingredients (Jones, 1995). Spiro compounds represent an important class of naturally occurring substances, which in many cases exhibit important biological properties (Kobayashi *et al.*, 1991; James *et al.*, 1991). Several amino acids containing the pyrrolidine moiety have been investigated by many groups (Galeazzi *et al.*, 1999). 1,3-Dipolar cycloaddition reactions are important for the construction of spiro compounds (Caramella & Grunanger, 1984). The title compound, (I), was synthesized by the intermolecular [3 + 2]-cycloaddition of the azomethine ylide, derived from isatin and sarcosine by a decarboxylative route, and 2-chloronitrostyrene.



(I)

A *ZORTEP* plot (Zsolnai, 1998) of the molecule of (I) is shown in Fig. 1. In the molecule, the 2-oxindole and pyrrolidine moieties are connected *via* a spiro junction. The pyrrolidine ring has an envelope conformation, with atom C5 deviating by 0.632 (3) Å from the mean plane defined by atoms N1, C2, C3 and C4. The methyl, nitro and 2-chlorophenyl groups are substituted equatorially on the pyrrolidine ring (Table 1). The N atom of the pyrrolidine ring shows sp^3 hybridization, a fact confirmed by the angles around this atom (Table 1).

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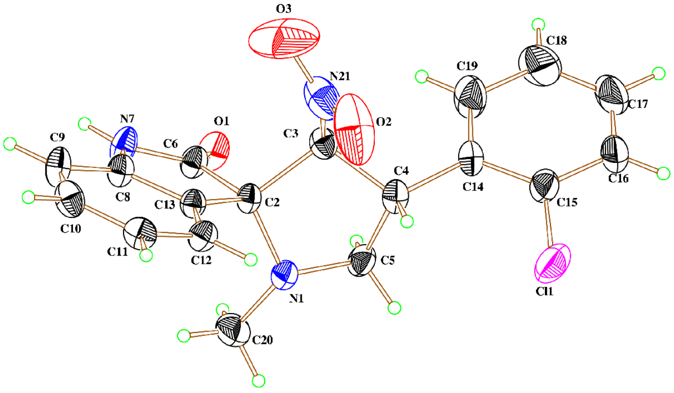

Figure 1
ZORTEP plot (Zsolnai, 1998) of the title molecule, showing 50% probability displacement ellipsoids.

The planar nitro group subtends an angle of 75.3 (1)° to the mean plane of the pyrrolidine ring. In the planar oxindole moiety, bond lengths C8—N7 and N7—C6 are similar to values reported in the literature (Jeyabharathi *et al.*, 2001).

The packing of the molecules viewed down the *a* axis is shown in Fig. 2. A dimer is formed *via* N—H···O intermolecular hydrogen bonds between O1 and N7($-x-1, 2-y, -z$). Intramolecular C—H···O, and intermolecular C—H···O, C—H···Cl and C—H···π contactsare involved in the crystal packing (Desiraju, 1989).

Experimental

A solution of sarcosine (1 mmol) and isatin (1 mmol) was added to a solution of 2-chloronitrostyrene (1 mmol) in aqueous methanol (20 ml) and the resulting solution refluxed for 8 h. After completion of the reaction, the solvent was evaporated in a vacuum. The brown residue was subjected to column chromatography to obtain the pure cycloadduct. The resulting compound was crystallized from methanol by slow evaporation and colorless single crystals were chosen for the crystallographic study.

Crystal data

$C_{18}H_{16}ClN_3O_3$
$M_r = 357.79$
Monoclinic, $P2_1/c$
$a = 13.1473$ (3) Å
$b = 7.8193$ (12) Å
$c = 17.4268$ (6) Å
$\beta = 102.086$ (2)°
$V = 1751.8$ (3) Å^3
$Z = 4$

$D_x = 1.357$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 1.6$–30.0°
$\mu = 0.24$ mm^{-1}
$T = 293$ (2) K
Block, colorless
0.38 × 0.34 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
5298 measured reflections
5099 independent reflections
2375 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.022$
$\theta_{\rm max} = 30.0°$
$h = 0 \rightarrow 18$
$k = 0 \rightarrow 10$
$l = -24 \rightarrow 23$

Refinement

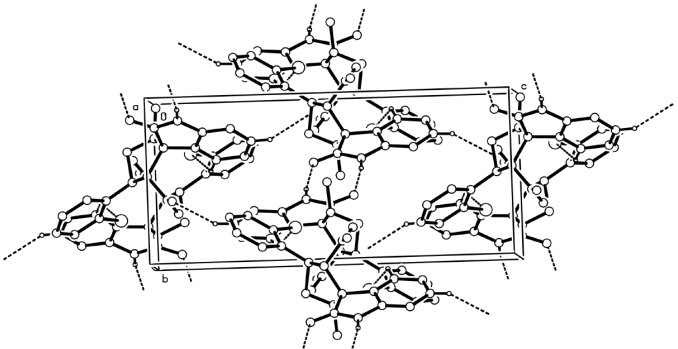

Figure 2
Packing of the molecules, viewed down the *a* axis. Dashed lines represent hydrogen bonds.

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.166$
$S = 1.01$
5099 reflections
227 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0667P)^2 + 0.4302P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.35$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.39$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0091 (15)

Table 1
Selected geometric parameters (Å, °).

Cl1—C15	1.736 (3)	N7—C8	1.403 (3)
O1—C6	1.222 (3)	C3—N21	1.508 (4)
N1—C20	1.452 (3)	N21—O3	1.196 (4)
N7—C6	1.341 (3)	N21—O2	1.214 (4)
C20—N1—C5	115.9 (2)	N1—C2—C3	102.63 (16)
C20—N1—C2	116.01 (19)	C13—C2—C3	117.64 (19)
C5—N1—C2	108.38 (18)	C6—C2—C3	107.71 (18)
N1—C2—C13	113.23 (18)	O3—N21—O2	126.3 (4)
N1—C2—C6	114.71 (19)	O3—N21—C3	115.9 (4)
C13—C2—C6	101.33 (16)	O2—N21—C3	117.5 (3)
C5—N1—C2—C3	−23.7 (2)	C5—C4—C3—C2	29.3 (2)
C20—N1—C5—C4	175.4 (2)	N1—C2—C3—N21	−129.4 (2)
C3—C4—C5—N1	−43.2 (2)	N1—C2—C3—C4	−4.8 (2)
C5—C4—C3—N21	152.3 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···Cl1	0.98	2.55	3.056 (2)	112
C3—H3···O1	0.98	2.59	3.024 (3)	107
N7—H7···O1$^{\rm i}$	0.86	2.04	2.882 (3)	166
C9—H9···$Cg1^{\rm ii}$	0.93	3.12	3.884 (3)	141

Symmetry codes: (i) $-1-x, 2-y, -z$; (ii) $1-x, \frac{1}{2}+y, \frac{3}{2}-z$. Note: *Cg*1 is the centroid of ring C8–C13.

All H atoms were positioned geometrically (NH = 0.86, C—H = 0.93–0.98 Å) and allowed to ride on their parent atoms, with $U_{\rm iso}$(H) = 1.2 or 1.5 times $U_{\rm eq}$(parent atom).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1998) and

PLATON (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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