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3′-(2-Chlorophenyl)-1′-methyl-4′nitrospiro[indan-2,2′-pyrrolidine]-1,3-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.102
Data-to-parameter ratio = 13.6

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

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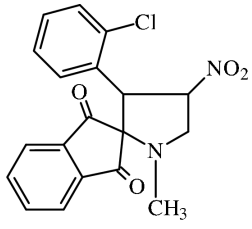
In the title compound, $C_{19}H_{15}ClN_2O_4$, the pyrrolidine ring adopts an envelope conformation. The five-membered ring in the indanedione moiety also adopts an envelope conformation. The dihedral angle between the chlorophenyl ring and the benzene ring of the indanedione moiety is 38.2 (1)°. The molecular structure is stabilized by intramolecular C—H···Cl and C—H···O interactions and the packing is stabilized by intermolecular C—H···O interactions.

Comment

The pyrrolidine skeleton occurs in many families of biologically important compounds. The resulting functionality, due to ease of substitution and therefore modification at several positions (Baldwin *et al.*, 1994*a*,*b*), has been utilized to synthesize compounds with varying properties. For example, several unusual amino acids, which contain the pyrrolidine motif, have been investigated (Galeazzi *et al.*, 1999). These derivatives possess anti-influenza virus (Stylianakis *et al.*, 2003) and anticonvulsant (Obniska *et al.*, 2002) 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 medicinal importance, the crystal and molecular structure determination of the title compound, (I), was carried out by X-ray diffraction.



(I)

A displacement ellipsoid plot of (I) is shown in Fig. 1. The C—Cl bond length is comparable to the reported mean value of 1.739 (10) Å (Allen *et al.*, 1987). The bond lengths in the pyrrolidine ring are comparable to the values reported in related structures (Abdul Ajees *et al.*, 2002; Gzella & Wrzeciono, 1990; Usha *et al.*, 2003). All the bond lengths in the indanedione moiety are comparable to the values in related reported structures (Kendi *et al.*, 1995; Seshadri *et al.*, 2003).

The sum of the angles at N1 of the pyrrolidine moiety [342.1°] is in accordance with sp^3-hybridization and the sum of the angles at N23 [359.9°] is in accordance with sp^2-hybridization.

The methyl group is attached equatorially to the pyrrolidine ring. The chlorophenyl ring and the benzene ring of the indanedione moiety are oriented at an angle of 38.2 (1)° with

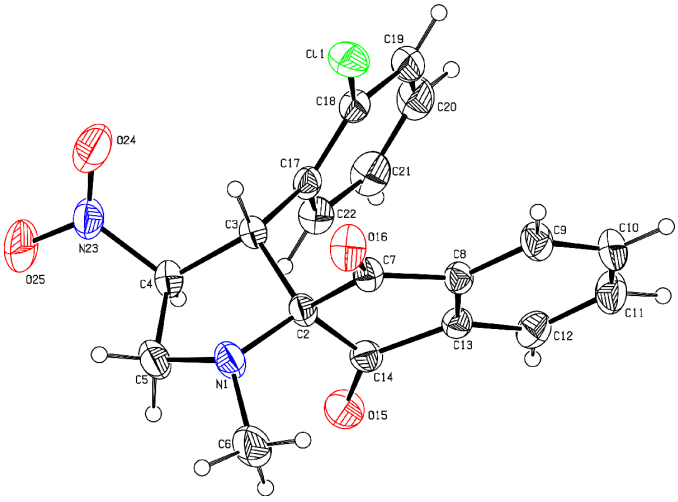


Figure 1
The molecular structure and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

respect to each other. The nitro group makes a dihedral angle of 71.8 (3)° with the chlorophenyl ring and 43.6 (2)° with the benzene ring of the indanedione moiety.

The pyrrolidine ring adopts an envelope conformation, with asymmetry parameters of Δ_s(C2) = 0.022 (1), ΔC_2(C5) = 0.052 (1) and ΔC_2(C4) = 0.080 (1) (Nardelli, 1983). The five-membered ring of the indanedione moiety adopts an envelope conformation, with puckering parameters of q_2 = 0.172 (2) Å and φ = 172.3 (7)° (Cremer & Pople, 1975). Atom C2 deviates by 0.276 (2) Å from the least-squares plane through the remaining four atoms. The keto O atoms O15 and O16 deviate from the mean plane through the ring by 0.562 (2) and 0.133 (2) Å, respectively.

The crystal structure is stabilized by intramolecular C—H···Cl and C—H···O interactions. In addition to the van der Waals interactions, the molecular packing in the crystal is stabilized by intermolecular C—H···O interactions (Fig. 2).

Experimental

A solution of ninhydrin (1 mmol), sarcosine (1 mmol) and 2-chlorophenylnitrostyrene (1 mmol) in aqueous methanol was refluxed on a water bath until the disappearance of the starting materials. The reaction mixture was then concentrated *in vacuo* and column chromatographed over silica gel with a hexane–ethyl acetate mixture (9:1) to obtain the title compound. The compound was recrystallized from methanol by slow evaporation.

Crystal data

$C_{19}H_{15}ClN_2O_4$
M_r = 370.78
Monoclinic, *Cc*
a = 13.0966 (12) Å
b = 8.6225 (8) Å
c = 15.7306 (14) Å
β = 100.045 (1)°
V = 1749.2 (3) Å^3
Z = 4
D_x = 1.408 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 2598 reflections
θ = 2.6–26.1°
μ = 0.25 mm^{-1}
T = 293 (2) K
Block, colourless
0.24 × 0.20 × 0.16 mm

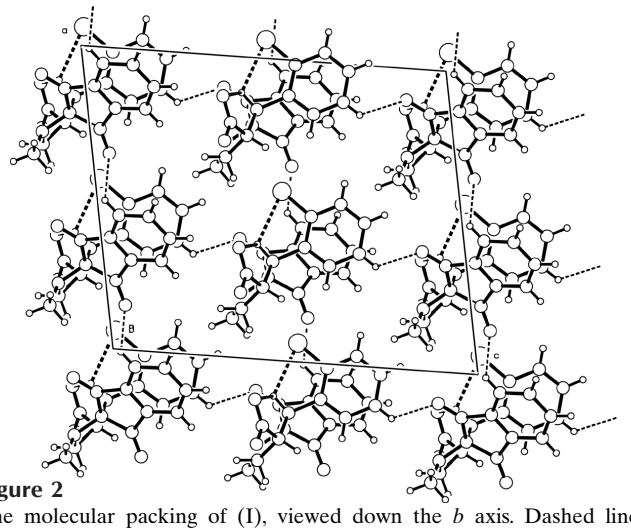


Figure 2
The molecular packing of (I), viewed down the *b* axis. Dashed lines indicate hydrogen bonds.

Data collection

Bruker SMART APEX diffractometer
ω scans
Absorption correction: none
5115 measured reflections
3199 independent reflections
3013 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 27.9°
h = −16 → 16
k = −10 → 9
l = −20 → 20

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.102
S = 1.02
3199 reflections
235 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0607P)^2 + 0.5297P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Absolute structure: Flack (1983), 1228 Friedel pairs
Flack parameter = 0.04 (6)

Table 1
Selected geometric parameters (Å, °).

Cl1—C18	1.742 (3)	C4—C5	1.510 (4)
N1—C5	1.441 (4)	C7—O16	1.194 (3)
N1—C2	1.446 (3)	C14—O15	1.197 (3)
N1—C6	1.454 (4)	N23—O24	1.192 (3)
C3—C4	1.530 (3)	N23—O25	1.195 (3)
C5—N1—C2	109.9 (2)	C18—C17—C22	116.7 (2)
C5—N1—C6	115.5 (2)	O24—N23—O25	122.3 (3)
C2—N1—C6	116.7 (2)	O24—N23—C4	120.8 (2)
C17—C3—C4	116.0 (2)	O25—N23—C4	116.8 (2)
C17—C3—C2	114.9 (2)		
C6—N1—C2—C3	171.2 (2)	C6—N1—C5—C4	−156.7 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···Cl1	0.98	2.59	3.081 (2)	111
C3—H3···O24	0.98	2.30	2.719 (3)	105
C9—H9···O15^{i}	0.93	2.38	3.109 (3)	135
C21—H21···O16^{ii}	0.93	2.60	3.248 (3)	127

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

The H atoms were positioned geometrically and were treated as riding on their parent C atoms, with aromatic C—H distances of 0.93 Å, methyl C—H distances of 0.96 Å, methylene C—H distances of 0.97 Å and methine C—H distances of 0.98 Å, and with U_{iso} = $1.5U_{eq}$(C) for methyl H 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: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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