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2′-(2-Chlorophenyl)-1′-nitro-2′,3′,3a′,4′,5′,6′-hexahydroindan-2-spiro-3′-1′*H*-pyrrolizine-1,3-dione

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In the title compound, $C_{21}H_{17}ClN_2O_4$, both pyrrolidine rings adopt twist conformations. The molecular structure is stabilized by weak intramolecular C—H···Cl, C—H···N and C—H···O interactions. The crystal structure is stabilized by intermolecular C—H···O interactions.

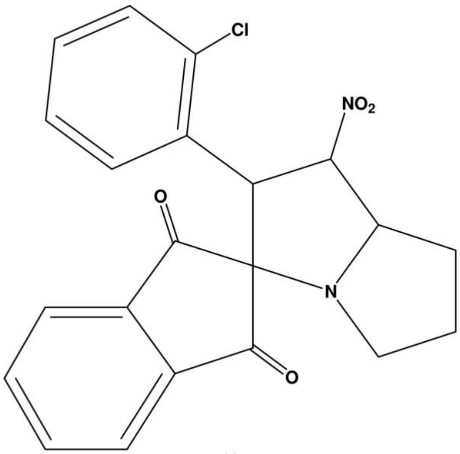
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.060
wR factor = 0.188
Data-to-parameter ratio = 16.7

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

Comment

Pyrrolidine occurs widely in nature and is a structural component of porphyrin heme, chlorophyll and vitamin B12. Pyrrolidine compounds have antifungal and antimicrobial activities (Amal Raj *et al.*, 2003). It has been shown that functionalized pyrrolidines inhibit γ-mannosidase activity and growth of human glioblastoma and melanoma cells (Fiaux *et al.*, 2005). As pyrrolidine compounds are of great pharmaceutical importance, we have determined the crystal structure of the title compound, (I) (Fig. 1).



(I)

Except at the spiro junctions (Table 1), the bond lengths and angles in the structure of (I) are comparable to literature values (Allen *et al.*, 1987); the deviations are due to bulky substituents. The sums of the bond angles around atoms N1 (334.9°) and N2 (359.6°) indicate sp^3- and sp^2-hybridization, respectively.

The dihedral angle between the fused five- and six-membered rings in the indandione group is 1.8 (1)°. Atoms O1 and O2 deviate by 0.074 (2) and 0.065 (2) Å, respectively, from the plane of the five-membered ring. The dihedral angle between the indandione group and the chlorophenyl ring is 64.7 (1)°. Atom Cl1 deviates by 0.011 (1) Å from the benzene ring to which it is attached.

Both the pyrrolidine rings (N1/C2/C1/C7/C6 and N1/C3–C6) adopt twist conformations. The puckering parameters (Cremer & Pople, 1975) and the smallest displacement

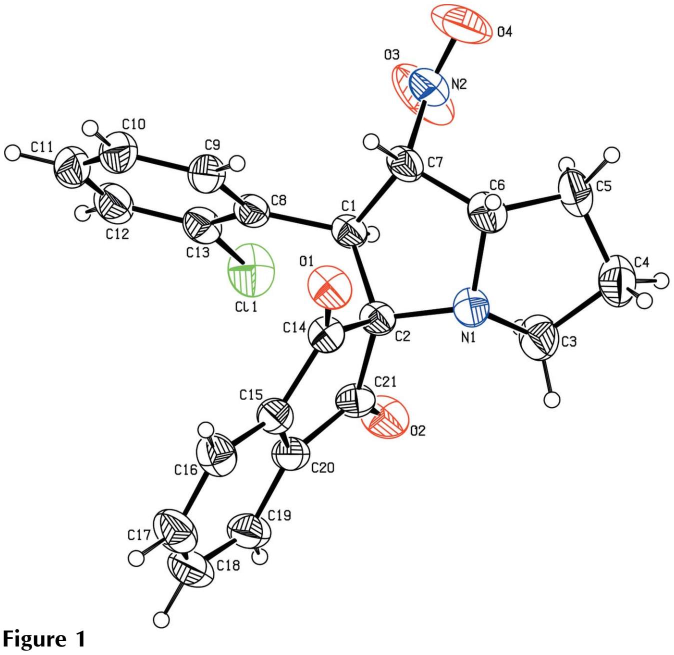


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids.

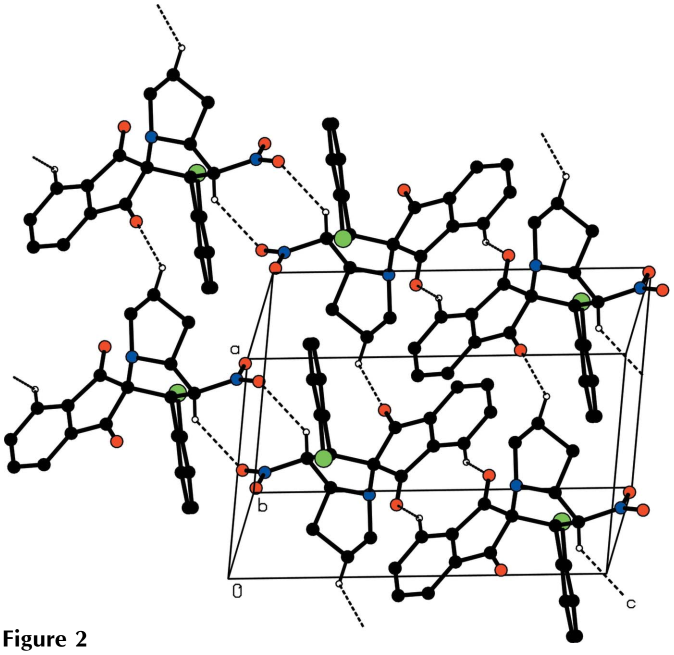


Figure 2
The molecular packing of (I), viewed approximately down the *b* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

asymmetry parameters (Nardelli, 1983) are q_2 = 0.343 (2) Å, φ = 230.9 (3)° and $\Delta C_2(C6)$ = 4.8 (2)° for the N1/C2/C1/C7/C6 ring, and q_2 = 0.425 (3) Å, φ = 241.3 (4)° and $\Delta C_2(C6)$ = 8.2 (2)° for the N1/C3–C6 ring.

C—H···O, C—H···Cl and C—H···N hydrogen-bonding interactions are observed in the molecular structure. The crystal packing is stabilized by C—H···O intermolecular interactions. The C4—H4*B*···O1^{i} interaction generates a *C*(7) chain along the *a* axis, and the C7—H7···O4^{ii} and C19—H19···O2^{iii} hydrogen bonds generate centrosymmetric dimers with $R_2^2(8)$ and $R_2^2(10)$ rings, respectively (Fig. 2). In addition, C—H···π interactions involving the C8–C13 benzene ring (centroid *Cg*1) also form a dimer. Symmetry codes (i)–(iii) are given in Table 2.

Experimental

A mixture of ninhydrin (0.178 g, 1 mmol), L-proline (0.115 g, 1 mmol) and 2-chloronitrostyrene (1 mmol) in methanol (20 ml) was refluxed until the disappearance of starting materials as shown by thin-layer chromatography analysis. The reaction mixture was then concentrated *in vacuo* and extracted with water (50 ml) and dichloromethane (50 ml). The organic fraction was washed with brine, dried and concentrated *in vacuo*. The residue was then purified by column chromatography (silica gel, 100–200 mesh) with a hexane–ethyl acetate (8:2) mixture to give the title compound, which was recrystallized from methanol by slow evaporation.

Crystal data

$C_{21}H_{17}ClN_2O_4$
M_r = 396.82
Triclinic, $P\bar{1}$
a = 7.6022 (5) Å
b = 9.7346 (7) Å
c = 12.6386 (9) Å
α = 85.296 (1)°
β = 84.200 (1)°
γ = 86.527 (1)°
V = 926.10 (11) Å^3
Z = 2
Mo *K*α radiation
μ = 0.24 mm^{-1}
T = 293 (2) K
0.27 × 0.24 × 0.22 mm

Data collection

Bruker SMART APEX area-detector diffractometer
Absorption correction: none
10652 measured reflections
4224 independent reflections
3542 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.188
S = 1.00
4224 reflections
253 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max}$ = 0.54 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.46 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—N1	1.462 (2)	C21—O2	1.204 (2)
C3—N1	1.481 (3)	N2—O3	1.212 (4)
C6—N1	1.482 (3)	N2—O4	1.225 (3)
C7—N2	1.511 (3)		
C2—N1—C3	119.9 (2)	O3—N2—O4	124.9 (3)
C2—N1—C6	107.9 (2)	O3—N2—C7	119.9 (2)
C3—N1—C6	107.1 (2)	O4—N2—C7	114.8 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···Cl1	0.98	2.57	3.090 (2)	113
C1—H1···O3	0.98	2.30	2.720 (4)	105
C3—H3*A*···O2	0.97	2.49	2.925 (3)	107
C5—H5*A*···N2	0.97	2.40	2.839 (4)	107
C4—H4*B*···O1^{i}	0.97	2.57	3.263 (3)	129
C7—H7···O4^{ii}	0.98	2.53	3.357 (3)	143
C19—H19···O2^{iii}	0.93	2.55	3.367 (3)	147
C18—H18···*Cg*1^{iv}	0.93	2.84	3.675 (3)	150

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, -y+1, -z$; (iii) $-x, -y+2, -z+1$; (iv) $-x+1, -y+2, -z+1$.

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H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). Large $U_{\rm eq}$ values for the O atoms of the nitro group suggest disorder, but no suitable disorder model was found.

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: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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