Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

1′-Nitro-2′-(3-nitrophenyl)-2′,3′,5′,6′,7′,7a′-hexahydroindan-2-spiro-3′-1′*H*-pyrrolizine-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 = 0.060
wR factor = 0.194
Data-to-parameter ratio = 16.7

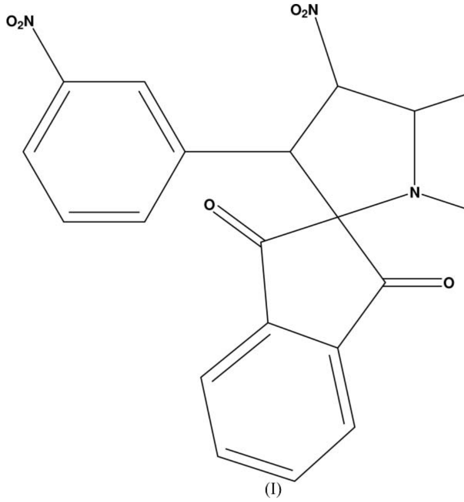
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 15 March 2007
Accepted 25 March 2007

In the title compound, $C_{21}H_{17}N_3O_6$, one of the pyrrolidine ring adopts a twist conformation, while the other is in an envelope conformation. The molecular structure is stabilized by C—H⋯O and C—H⋯N intramolecular interactions, and the crystal packing is stabilized by C—H⋯O intermolecular interactions, generating *C*(7), *C*(8) and *C*(10) chains and an $R_2^2(10)$ ring.

Comment

Substituted pyrrolidine compounds have gained much importance since they are the basic structural elements of many alkaloids and pharmacologically active compounds (Waldmann, 1995). Derivatives of pyrrolidine exhibit a range of activities such as hypotensive, local anesthetic, ganglion blocking, neuromuscular blocking and antispasmodic (Atal, 1978). Nitro compounds are excellent precursors for aromatic amines and medicinally important compounds, and are also well known for their uses as explosives, dye intermediates and battery cathodes (Sivasamy *et al.*, 1988, Renuka *et al.*, 2001). In view of the above wide range of applications, we have undertaken the X-ray crystal structure determination of the title compound.



(I)

The sums of the bond angles around N1 (332.3°), N2 (359.9°) and N3 (360.0°) indicate that N1 is sp^3-hybridized, and N2 and N3 are sp^2-hybridized. The dihedral angle between the five- and six-membered rings in the indanedione unit is 0.5 (1)°, indicating the planarity of this group. Atoms O5 and O6 deviate by 0.100 (2) and 0.160 (2) Å, respectively, from the plane of the five-membered ring (C7/C14/C15/C20/C21) in the indanedione group.

doi:10.1107/S1600536807014286

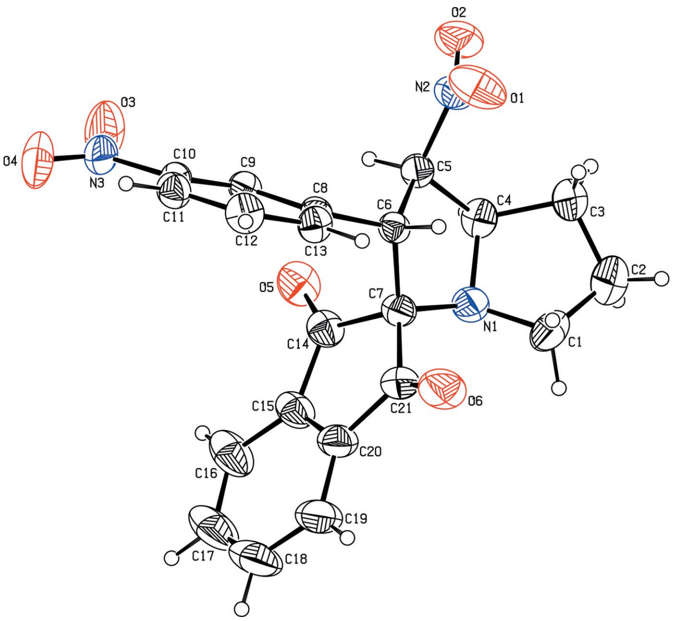


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids.

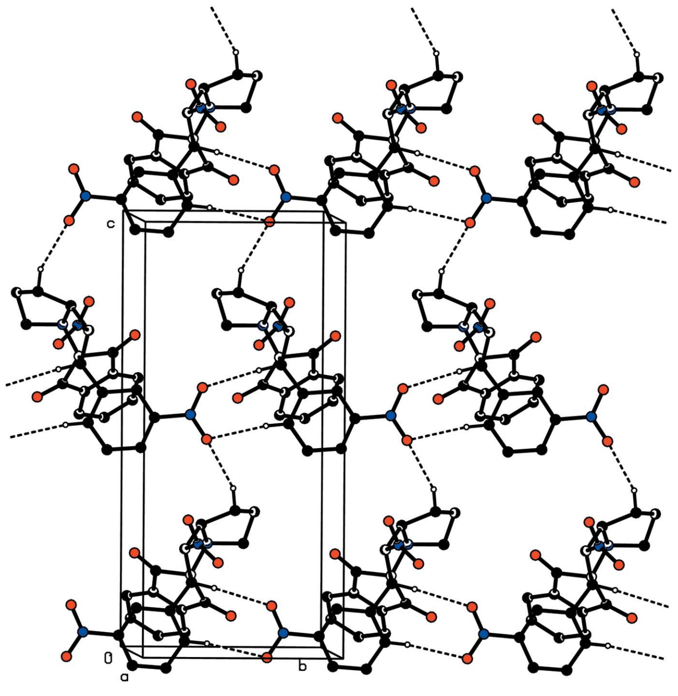


Figure 2
The crystal packing of (I), viewed approximately down the *a* axis, showing selected C—H···O intermolecular interactions (dashed lines). For clarity, H atoms not involved in the hydrogen bonds have been omitted.

The N1/C1–C4 pyrrolidine ring adopts an envelope conformation, with atom C2 deviating by 0.606 (4) Å from the plane of the other atoms in the ring. The N1/C4—C7 pyrrolidine ring adopts a twist conformation, with a pseudo-twofold axis passing through C4 and and the mid-point of the C6—C7 bond. The puckering parameters (Cremer & Pople, 1975) and the smallest displacement asymmetry parameters (Nardelli, 1983) are q_2 = 0.410 (3) Å, φ = 64.2 (4)° and $\Delta_s(C_2)$ = 7.8 (3)° for the N1/C1—C4 pyrrolidine ring, and q_2 = 0.386 (2) Å, φ = 120.1 (3) ° and $\Delta_2(C_4)$ = 5.3 (2)° for the N1/C4–C7 pyrrolidine ring.

The molecular structure is stabilized by weak C—H···O and C—H···N interactions. In the solid state, the molecules are linked into a three-dimensional framework by C—H···O intermolecular hydrogen bonds (Table 1). Atoms C6 and C13 act as donors to O3 and O4 at (x, y + 1, z), generating a $C(7)$ chain along the b axis. Atom C3 interacts with O4(x, $-y$, $\frac{1}{2} + z$) and generates a $C(10)$ chain running along the c axis (Fig. 2). Atom C19 acts as a donor to O6 at ($-x$ + 1, $-y$ + 1, $-z$), generating a centrosymmetric dimer with an $R_2^2(10)$ ring (Fig. 3). Atom C12 acts as a donor to O2 at ($-x + \frac{3}{2}$, $-y + \frac{1}{2}$, $z - \frac{1}{2}$) generating a $C(8)$ chain.

Experimental

A mixture of ninhydrin (0.178 g, 1 mmol), L-proline (0.115 g, 1 mmol) and *m*-nitronitrostyrene (1 mmol) in methanol (20 ml) was refluxed until the disappearance of the starting materials. The reaction mixture was then concentrated *in vacuo* and diluted with water (50 ml). It was extracted with 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 obtain the title compound. The cycloadduct was recrystallized from methanol by slow evaporation.

Crystal data

$C_{21}H_{17}N_3O_6$
M_r = 407.38
Orthorhombic, *Pbcn*
a = 26.1766 (15) Å
b = 8.1726 (5) Å
c = 17.7838 (10) Å
V = 3804.5 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 (2) K
0.26 × 0.24 × 0.21 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: none
30526 measured reflections
4520 independent reflections
3036 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.195
S = 1.02
4520 reflections
271 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max}$ = 0.41 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.19 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*B*···O6	0.97	2.50	3.014 (4)	113
C3—H3*B*···O1	0.97	2.59	3.291 (4)	129
C3—H3*B*···N2	0.97	2.45	2.831 (4)	103
C5—H5···O5	0.98	2.51	3.029 (3)	113
C3—H3*A*···O4$^{\rm i}$	0.97	2.43	3.342 (4)	158
C6—H6···O3$^{\rm ii}$	0.98	2.54	3.514 (2)	176
C12—H12···O2$^{\rm iii}$	0.93	2.57	3.408 (3)	150
C13—H13···O4$^{\rm ii}$	0.93	2.54	3.437 (2)	163
C19—H19···O6$^{\rm iv}$	0.93	2.51	3.333 (4)	147

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

H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

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).

DG thanks CSIR, India, for the award of a senior research fellowship. DV thanks DST, India, for a major research project.

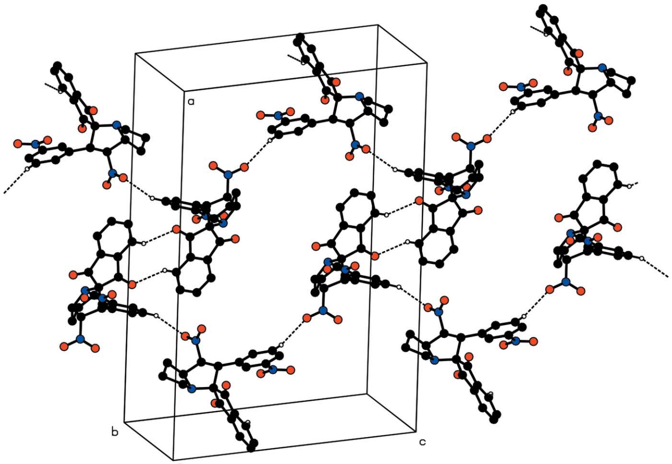


Figure 3

The molecular packing of (I), showing selected C—H···O intermolecular interactions (dashed lines). For clarity, H atoms not involved in the hydrogen bonds have been omitted.

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