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Key indicators

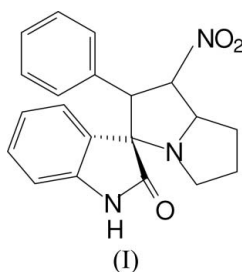
Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.053
wR factor = 0.164
Data-to-parameter ratio = 16.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Hydrogen bonding in 1'-nitro-2'-phenyl-
2',3',4',5',6',7'-hexahydro-1*H*-indole-3-
spiro-3'-1'*H*-pyrrolizidin-2(3*H*)-one: molecular
chains built from alternating $R_2^2(18)$ and $R_2^2(8)$ ringsIn the title cycloadduct, $\text{C}_{20}\text{H}_{19}\text{N}_3\text{O}_3$, the molecules form centrosymmetric dimers linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming an $R_2^2(8)$ ring. The overall conformation of the pyrrolizidine nucleus is folded about the bridging bond.

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Comment

The chemistry of indole compounds has been extensively studied, partly due to their use as pharmaceutical and industrial products. Some indole derivatives are used as neuroprotectants (Stolc, 1999). Spiro-indoles have been reported to show fungicidal activity (Ali *et al.*, 1989). 5-Chloro-3-(phenylsulfonyl)indole-2-carboxamide is reported to be a highly potent non-nucleoside inhibitor of HIV-1 reverse transcriptase (Williams *et al.*, 1993). The pyrrolizidine alkaloids are well documented for their mutagenic, anti-neoplastic, carcinogenic, hepatotoxic and many pharmacological activities. In view of the wide spectrum of biological activity of indole and pyrrolizidine derivatives, the X-ray analysis of a pyrrolizidine alkaloid, (I), has been undertaken and the structural details are presented in this communication.The bond lengths and angles of the indole and benzene ring systems are normal. The pyrrolizidine ring-fusion distance [$\text{N}1-\text{C}5 = 1.481(2) \text{ \AA}$] is in the same range as the other two $\text{N}-\text{C}$ distances and compares quite well with those observed in related structures (Hay *et al.*, 1982; Sussman & Wodak, 1973; Usha *et al.*, 2005). Bond distances and angles around atom C2 are somewhat distorted, which is due to the spiro-atom character.The $\text{C}=\text{O}$ double bond is slightly elongated [$\text{C}9=\text{O}1 = 1.222(2) \text{ \AA}$] due to the hydrogen bonding. This is similar to what was observed for the analogous bond in 1-naphthaleneacetic acid (Rajan, 1984), which forms hydrogen-bonded dimers.In the title adduct, each molecule is linked to a centrosymmetrically related molecule by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds forming $R_2^2(8)$ rings. The nitro group does not play any role in hydrogen bonding, but participates in fairly weak intramolecular contacts (Table 2).

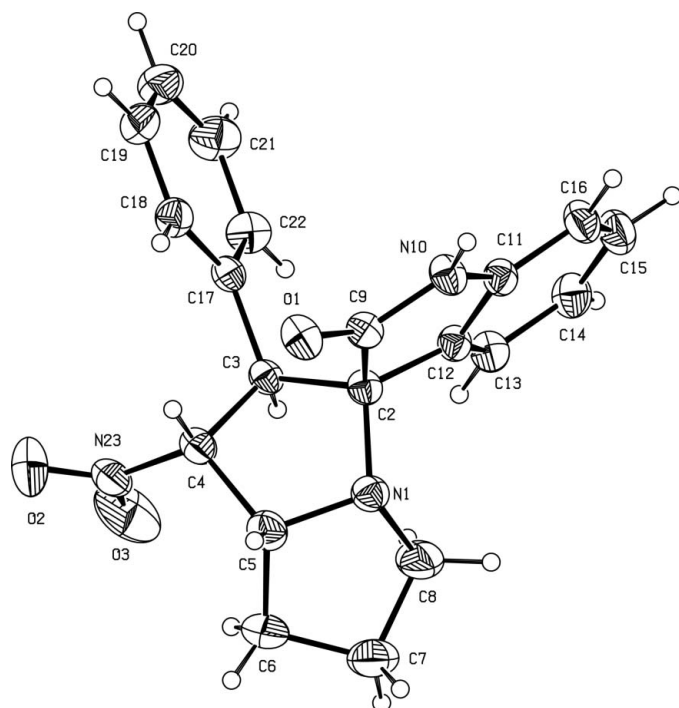


Figure 1
The molecular structure of the title adduct, showing 30% probability displacement ellipsoids.

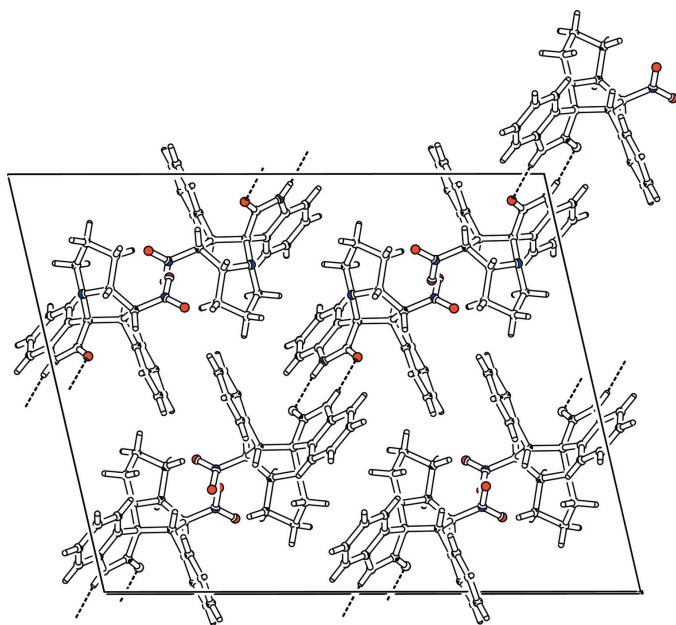


Figure 2
Packing diagram of the title compound, viewed on to the *ac* plane. Hydrogen bonds are shown as dashed lines.

In the pyrrolizidine nucleus, both five-membered rings adopt twist conformations. The smallest displacement asymmetry parameters (Nardelli, 1983) are $\Delta_{C_2}(C_5) = 0.018$ (1) and $\Delta_{C_2}(C_5) = 0.015$ (1) $^\circ$. The overall conformation of the pyrrolizidine nucleus is folded about the bridging bond, *viz.* N1—C5. This observation is consistent with the structure reported by Usha *et al.* (2005).

Experimental

A mixture of nitrostyrene (1 mmol), isatin (1 mmol) and proline (1 mmol) in methanol (20 ml) was refluxed until the disappearance of starting materials. After completion of the reaction, the reaction mixture was concentrated *in vacuo* and the residue was subjected to column chromatography with a hexane–ethyl acetate mixture (8:2) in order to obtain the pure cycloadduct. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of a methanol solution.

Crystal data

$C_{20}H_{19}N_3O_3$
 $M_r = 349.38$
Monoclinic, $C2/c$
 $a = 23.8981$ (15) Å
 $b = 7.5886$ (4) Å
 $c = 19.1370$ (10) Å
 $\beta = 102.941$ (2) $^\circ$
 $V = 3382.4$ (3) Å 3
 $Z = 8$

$D_x = 1.372$ Mg m $^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 7483 reflections
 $\theta = 2.5$ – 26.5 $^\circ$
 $\mu = 0.09$ mm $^{-1}$
 $T = 293$ (2) K
Block, colourless
0.23 × 0.21 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 ω scans
Absorption correction: none
13982 measured reflections
3921 independent reflections

3373 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.018$
 $\theta_{max} = 28.0$ $^\circ$
 $h = -30 \rightarrow 31$
 $k = -9 \rightarrow 9$
 $l = -25 \rightarrow 25$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.164$
 $S = 1.03$
3921 reflections
235 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0985P)^2 + 1.7117P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.29$ e Å $^{-3}$
 $\Delta\rho_{min} = -0.29$ e Å $^{-3}$

Table 1

Selected geometric parameters (Å, $^\circ$).

O1—C9	1.222 (2)	N1—C8	1.478 (2)
O2—N23	1.191 (3)	N10—C9	1.346 (2)
O3—N23	1.213 (3)	N10—C11	1.403 (2)
N1—C2	1.473 (2)	N23—C4	1.493 (2)
N1—C5	1.481 (2)		
C2—N1—C5	107.9 (1)	C5—N1—C8	107.2 (1)
C2—N1—C8	117.0 (1)		
C5—N1—C2—C3	−31.5 (1)	C3—C4—C5—N1	15.6 (2)
N1—C2—C3—C4	40.0 (1)	N1—C5—C6—C7	15.2 (3)
C2—C3—C4—C5	−33.4 (1)	C5—C6—C7—C8	−35.0 (3)
C8—N1—C5—C6	10.7 (2)	C5—N1—C8—C7	−32.8 (2)
C2—N1—C5—C4	10.1 (2)	C6—C7—C8—N1	41.6 (3)

Table 2

Hydrogen-bond geometry (Å, $^\circ$).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C6—H6B...O3	0.97	2.26	3.001 (4)	133
N10—H10...O1 ⁱ	0.86	2.08	2.929 (2)	169
C20—H20...O2 ⁱⁱ	0.93	2.58	3.285 (3)	133

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

H atoms were positioned geometrically and treated as riding on their parent atoms with C—H distances in the range 0.93–0.97 Å and

an N–H distance of 0.86 Å, and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H and $1.2U_{\text{eq}}(\text{N,C})$ for other H atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; 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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