

1-Methyl-4'-(3-nitrophenyl)-1*H*-indole-3-spiro-2'-pyrrolidine-3'-spiro-2''-indan-2,1'',3''-trione

P. R. Seshadri,^{a,b}
 S. Selvanayagam,^b
 D. Velmurugan,^{b*}
 K. Ravikumar,^c
 A. R. Sureshbabu^d and
 R. Raghunathan^d

^aDepartment of Physics, Agurchand Manmull Jain College, Chennai 600 114, India,

^bDepartment of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, ^cLaboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and

^dDepartment of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India

Correspondence e-mail: d_velu@yahoo.com

Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$

R factor = 0.052

wR factor = 0.163

Data-to-parameter ratio = 15.5

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

The pyrrolidine ring of the title compound, $\text{C}_{26}\text{H}_{19}\text{N}_3\text{O}_5$, adopts an envelope conformation. The molecular structure is stabilized by $\text{C}-\text{H}\cdots\text{O}$ interactions and the packing of the molecules is stabilized by an $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond and $\text{C}-\text{H}\cdots\text{O}$ intermolecular interactions. A dimer is formed between symmetry-related molecules through $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Received 3 November 2003

Accepted 17 November 2003

Online 29 November 2003

Comment

Substituted pyrrolidine compounds have become important because they are the basic structural elements of many alkaloids and pharmacologically active compounds. Pyrrolidine derivatives are found to have anticonvulsant properties (Obniska *et al.*, 2002). Structural classification divides this alkaloid family into several subgroups, among which oxindoles deserve to be mentioned (Bindra, 1973). In view of this medicinal importance, the crystal structure of the title compound, (I), has been determined and the results are presented here.

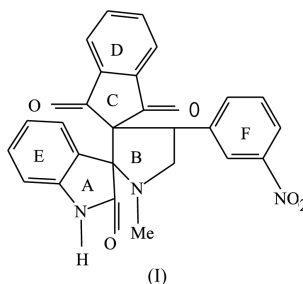


Fig. 1 shows a displacement ellipsoid diagram of the molecule with the atomic numbering scheme. Selected geometric parameters are given in Table 1.

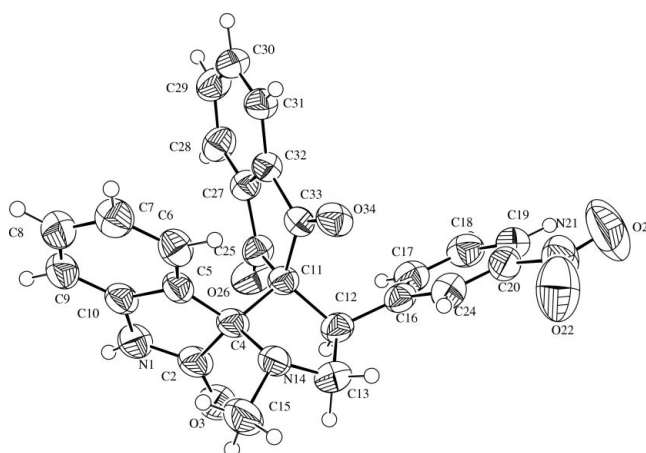


Figure 1
View of (I), with 50% probability displacement ellipsoids.

The sum of the angles around atoms N14 and N21 are 339.8 and 359.9°, respectively, showing that these atoms are in sp^3 and sp^2 configurations.

The bond lengths in the pyrrolidine moiety (Table 1) are slightly longer than normal values but are comparable to those in previously reported structures (Abdul Ajees *et al.*, 2002). The lengthening of these bonds may be due to steric forces of the bulky substituents at atoms C4 and C11 of the pyrrolidine moiety.

The nitro group slightly distorts the ring to which it is attached. The endo angle at atom C20 bearing the O22/N21/O23 nitro group is 123.4 (1)°, while the range is 117.6 (1)–121.4 (2)° for the other ring atoms. The O atoms of the nitro group have somewhat higher displacement parameters than the other atoms, indicating greater thermal motion of these terminal atoms.

The orientation of the N21 nitro group is influenced by weak C19–H19···O23 and C24–H24···O22 interactions, thus causing the mean N21–O22–O23–C20 plane to orient at an angle of 10.0 (1)° to the attached ring. Keto-group atoms O26 and O34 deviate from the mean plane containing the ring to which they are attached by 0.287 (1) and 0.299 (1) Å, respectively.

The total puckering amplitudes (Cremer & Pople, 1975) of rings *A*, *B* and *C* give a quantitative evaluation of the puckering and asymmetry parameters. Ring *A* is in a half-chair conformation, with the lowest asymmetry parameter (Nardelli, 1983) ΔC_2 [C10 = 0.0039 (7)]. Pyrrolidine ring *B* is in an envelope conformation, with puckering parameters $q_2 = 0.4078$ (7) Å and $\varphi = 44.12$ (9)°, and with atom C4 deviating 0.267 (2) Å from the least-squares plane passing through the remaining four atoms of that ring. Indandione ring *C* is in an envelope conformation, as evidenced by the puckering parameters $q_2 = 0.1483$ (7) Å and $\varphi = -8.61$ (3)°, and the asymmetry parameter ΔC_5 [C11 = 0.0179 (1)], with atom C11 deviating 0.090 (2) Å from the least-squares plane of the remaining atoms.

A dimer is formed between symmetry-related molecules through N–H···O hydrogen bonds (Fig. 2). In addition to the van der Waals interactions, the crystal structure is stabilized by intramolecular C–H···O interactions.

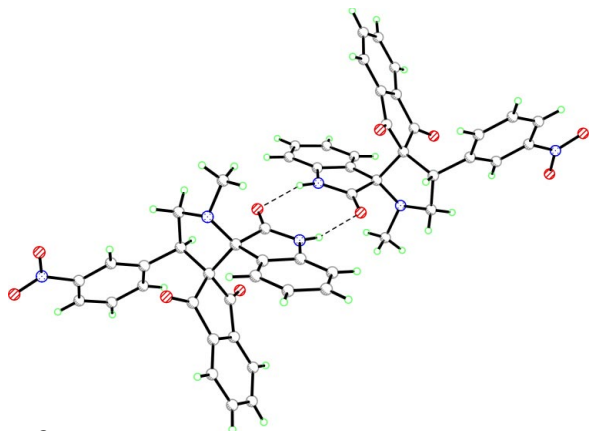


Figure 2
View of a centrosymmetric N–H···O hydrogen-bonded dimer.

Experimental

A mixture of *O*-nitrobenzylidene 1,3-indandione, isatin and sarcosine was refluxed in aqueous methanol. The resulting crude product was filtered and recrystallized from methanol.

Crystal data

$C_{26}H_{19}N_3O_5$	$Z = 2$
$M_r = 453.44$	$D_x = 1.392 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.7610$ (6) Å	Cell parameters from 2386 reflections
$b = 11.6696$ (10) Å	$\theta = 2.4\text{--}27.2^\circ$
$c = 11.9952$ (9) Å	$\mu = 0.10 \text{ mm}^{-1}$
$\alpha = 93.392$ (2)°	$T = 293$ (2) K
$\beta = 91.092$ (1)°	Needle, yellow
$\gamma = 93.660$ (1)°	$0.21 \times 0.16 \times 0.14 \text{ mm}$
$V = 1081.98$ (15) Å ³	

Data collection

Bruker SMART APEX diffractometer	4747 independent reflections
ω scans	3472 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	$R_{\text{int}} = 0.014$
$T_{\text{min}} = 0.980$, $T_{\text{max}} = 0.986$	$\theta_{\text{max}} = 28.0^\circ$
6822 measured reflections	$h = -10 \rightarrow 10$
	$k = -13 \rightarrow 14$
	$l = -10 \rightarrow 15$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0932P)^2 + 0.165P]$
$R[F^2 > 2\sigma(F^2)] = 0.052$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.163$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$
4747 reflections	$\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$
307 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, °).

N1–C2	1.355 (3)	C20–N21	1.471 (3)
N1–C10	1.399 (3)	N21–O23	1.220 (3)
C2–O3	1.221 (2)	N21–O22	1.226 (3)
C4–N14	1.451 (2)	C25–O26	1.206 (2)
C13–N14	1.456 (3)	C33–O34	1.206 (2)
N14–C15	1.454 (3)		
C2–N1–C10	112.19 (15)	C15–N14–C13	114.71 (17)
O3–C2–N1	126.58 (17)	C19–C20–C24	123.37 (19)
O3–C2–C4	126.19 (18)	C19–C20–N21	118.3 (2)
N1–C2–C4	107.21 (16)	C24–C20–N21	118.3 (2)
N14–C4–C5	114.48 (15)	O23–N21–O22	123.4 (2)
N14–C4–C2	115.57 (15)	O23–N21–C20	118.1 (3)
N14–C4–C11	100.67 (13)	O22–N21–C20	118.4 (2)
C5–C10–N1	109.41 (17)	O26–C25–C27	126.87 (17)
N14–C13–C12	106.28 (15)	O26–C25–C11	125.03 (17)
C4–N14–C15	116.02 (16)	O34–C33–C32	126.16 (16)
C4–N14–C13	109.10 (16)	O34–C33–C11	126.14 (16)

Table 2

Hydrogen-bonding geometry (Å, °).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
N1–H1···O3 ⁱ	0.86	2.12	2.949 (2)	162
C28–H28···O34 ⁱⁱ	0.93	2.54	3.150 (2)	124
C31–H31···O22 ⁱⁱⁱ	0.93	2.60	3.296 (3)	133
C19–H19···O23	0.93	2.42	2.703 (4)	98
C24–H24···O22	0.93	2.43	2.717 (3)	98

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

All H atoms were positioned geometrically and allowed to ride on their parent atoms [$C-H = 0.93-0.98 \text{ \AA}$, $N-H = 0.86 \text{ \AA}$, and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $1.2U_{eq}(C,N)$ 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, 1990); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

SSN and DV thank the University Grants Commission (UGC), New Delhi, for financial support under the University With Potential For Excellence Programme.

References

- Abdul Ajees, A., Manikandan, S. & Raghunathan, R. (2002). *Acta Cryst.* **E58**, o802–o804.
- Bindra, J. S. (1973). *Oxindole Alkaloids, Alkaloid Chemistry and Physiology*, edited by R. H. F. Manke. New York: Academic Press.
- Bruker (2001). *SAINTE* (Version 6.28a) and *SMART* (Version 5.625). Bruker AXS Inc., Madison, Wisconsin, USA.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Nardelli, M. (1983). *Acta Cryst.* **C39**, 1141–1142.
- Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
- Obniska, J., Zeic, A. & Zagorska, A. (2002). *Acta Pol. Pharm.* **59**, 209–213.
- Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
- Sheldrick, G. M. (2001). *SADABS*. Version 2.03. University of Göttingen, Germany.
- Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.