Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Spiro-[2-3′]oxindole-spiro-[3-3″]oxindole-4[*p*-methylbenzyl]pyrrolidizine

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

[a]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, and Department of Physics, Agurchand Manmull Jain college, Chennai 600 114, India, [b]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, [c]Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and [d]Department 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 K
Mean σ(C–C) = 0.002 Å
R factor = 0.050
wR factor = 0.155
Data-to-parameter ratio = 16.0

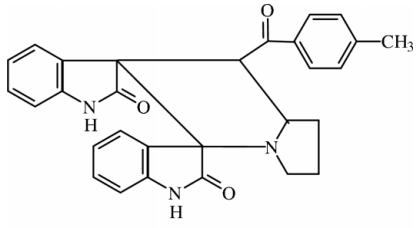
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 26 August 2003
Accepted 1 September 2003
Online 11 September 2003

The oxindole moieties of the title compound, $C_{29}H_{25}N_3O_3$, are planar. The pyrrolidine ring adopts a half-chair conformation. The structure is stabilized by C—H···O intramolecular interactions. It is remarkable that only one of the two NH groups forms a hydrogen bond.

Comment

Pyrrolidine compounds are capable of exhibiting antimicrobial and antifungal activity, as reported by Amal Raj *et al.* (2003). The pyrrolidine moiety occurs in biologically important compounds (Baldwin *et al.*, 1994). Several unusual amino acids which contain pyrrolidine moieties were investigated by Gallazzi *et al.* (1999). The spiro ring system is a frequently encountered structural motif in many pharmacologically relevant alkaloids (Cordel, 1981). Several optically active pyrrolidines have been used as intermediates in controlled asymmetric synthesis (Suzuki *et al.*, 1994). In view of these important aspects, the crystal structure of the title compound, (I), has been determined.



(I)

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

In the benzene rings of the oxindole systems, the endocyclic angles at C9 and C6 are 120.7 (2)°, 122.1 (2)° and at C24 and C27 are 122.4 (2)°, 120.9 (2)°. The endocyclic angles at C10 and C7 are 119.2 (2)°, 117.7 (2) and at C25 and C28 117.4 (2)° and 118.7 (2)°. Similar values are observed in related structures (Seshadri *et al.*, 2002).

The bond geometries correlate with a variety of *N*-phenyl-substituted pyrrolidine-2-one systems (Billing *et al.*, 1991). The bond lengths of the pyrrolidine moiety (Table 1) differ slightly from normal values, but are comparable with those of reported structures (Jeyabharathi *et al.*, 2001; Gzella & Wrzeciono, 1990). This may be due to steric forces of bulky substituents at the pyrrolidine moiety.

The oxindole moieties are planar. The asymmetry parameters (Nardelli, 1995; $q_2 = 0.4621$, $\varphi = 62.27°$, Δ_s[C4 = 0.0648°]

and ΔC_2[C34 = 0.0343]) reveal a half-chair conformation for the pyrolidine ring (*B*). The unsubstituted ring (*C*) of the pyrrolidizine ring system adopts a half-chair conformation. This is confirmed by the puckering parameters $q_2 = 0.3643$, $\varphi = -89.66°$, Δ_s[C33 = 0.0878)°] and ΔC_2[N30 = 0.0019] (Cremer & Pople, 1975).

In addition to van der Waals interactions, the crystal structure is stabilized by C—H···O intramolecular hydrogen bonds. It is remarkable that only one of the two NH groups forms a hydrogen bond.

Experimental

A mixture of (*E*)-3-*p*-(methylphenacylidine)oxindole, isatin and pyrroline was refluxed in aqueous methanol for 2–3 h. On completion of the reaction the solvent was evaporated *in vacuo* and the resulting crude product was purified by column chromatography, using *n*-hexane-ethyl acetate (7:3) as eluent.

Crystal data

$C_{29}H_{25}N_3O_3$
$M_r = 463.52$
Triclinic, $P\overline{1}$
$a = 9.7632$ (6) Å
$b = 10.3841$ (7) Å
$c = 11.6874$ (8) Å
$\alpha = 99.735$ (1)°
$\beta = 96.480$ (1)°
$\gamma = 98.892$ (1)°
$V = 1141.98$ (13) Å^3
$Z = 2$
$D_x = 1.348$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3128 reflections
$\theta = 2.6$–27.5°
$\mu = 0.09$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.40 × 0.30 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{min} = 0.966$, $T_{max} = 0.983$
7283 measured reflections
5057 independent reflections
4178 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 28.0°$
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 5$
$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.155$
$S = 0.95$
5057 reflections
316 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.106P)^2 + 0.1974P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.355 (2)	C21—O22	1.213 (2)
N1—C6	1.396 (2)	C21—N23	1.351 (2)
C2—O3	1.206 (2)	N23—C24	1.391 (2)
C12—O13	1.211 (2)	N30—C31	1.477 (2)
C20—N30	1.460 (2)	N30—C34	1.486 (2)
C2—N1—C6	112.9 (1)	C26—C25—C24	117.4 (2)
C7—C6—C5	122.1 (1)	C26—C27—C28	121.0 (2)
C6—C7—C8	117.7 (2)	C29—C28—C27	118.7 (2)
C8—C9—C10	120.7 (2)	C20—N30—C31	117.2 (1)
C5—C10—C9	119.2 (2)	C20—N30—C34	109.1 (1)
C21—N23—C24	112.3 (2)	C31—N30—C34	109.2 (1)
C25—C24—C29	122.4 (2)		
C4—C11—C12—C14	−62.5 (2)		

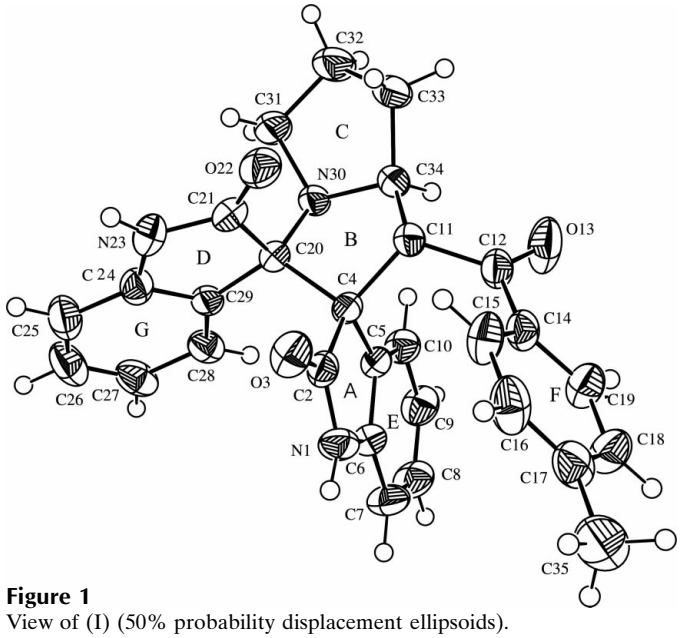


Figure 1
View of (I) (50% probability displacement ellipsoids).

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11···O22	0.98	2.31	2.949 (2)	122
C34—H34···O13	0.98	2.38	2.787 (2)	104
N23—H23···O3^{i}	0.86	2.05	2.892 (2)	168

Symmetry code: (i) $1-x, 1-y, 1-z$.

All 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) = 1.5 times U_{eq}(C) for methyl H atoms and 1.2 times U_{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, 1990); software used to prepare material for publication: *SHELXL*97 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

Amal Raj, A., Raghunathan, R., Sridevikumari, M. R. & Raman, N. (2003). *Bioorg. Med. Chem.* **11**, 407–419.
Baldwin, J. E., Turner, M. S. C. & Molony, M. G. (1994). *Tetrahedron*, 35, 9411–9424.
Billing, D. G., Boeyens, J. C. A., Levendis, D. C. & Micheal, J. P. (1991). *S. Afr. Chem.* **44**, 75–79.
Bruker (2001). *SMART* (Version 5.625) and *SAINT* (Version 6.28*a*). Bruker AXS Inc., Madison, Wisconsin, USA.
Cordel, G. (1981). *Introduction to Alkaloids, A Biogenetic Approach*. New York: Wiley International.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Gallazzi, R., Gremia, S., Mobbili, G. & Orena, M. (1999). *Tetrahedron Asymmetry*, **10**, 587–605.
Gzella, A. & Wrzeciono, U. (1990). *Acta Cryst.* C**46**, 2107–2109.

Jeyabharathi, A., Ponnusamy, M. N., Amal Raj, R., Ragunathan, R., Razak, I. A., Usman, A., Chandrapromma, S. & Fun, H. K. (2001). *Acta Cryst.* E**57**, o901–o903.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Seshadri, P. R., Velmurugan, D., Govindaraj, J., Kannadasan, S., Srinivasan, P. C., Shanmuga Sundara Raj, S., Fun, H. K. & *Kim*, M. J. (2002). *Acta Cryst.* C**58**, o700–o703.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Sheldrick, G. M. (2001). *SADABS*. Version 2.03. University of Göttingen, Germany.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Suzuki,H., Aoyagi, S. & Kibayashi, C. (1994). *Tetrahedron Lett.* **35**, 6119–6122.