Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

1-Benzoylacenaphthylene-1-spiro-3′-pyrrolizidine-2′-spiro-3″-1*H*-indole-2,2″(1*H*,3″*H*)-dione

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Received 12 November 2003
Accepted 27 November 2003
Online 12 December 2003

The title compound, $C_{32}H_{23}ClN_2O_3$, crystallizes in the centrosymmetric space group $P2_1/n$ with one molecule in the asymmetric unit. The pyrrolidine rings of the pyrrolizidine moiety adopt envelope conformations. The structure is stabilized by intramolecular C—H···O interactions. The packing is stabilized by N—H···O intermolecular interactions.

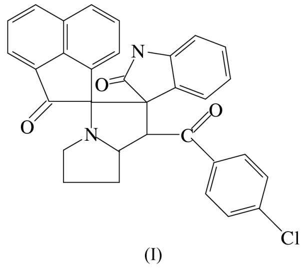
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.051
wR factor = 0.160
Data-to-parameter ratio = 17.0

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

Comment

Pyrrolidine compounds are frequently encountered structural motifs in many pharmacologically relevant alkaloids, as reported in the literature (Bindra, 1973). Several optically active pyrrolidines have been used as intermediates in controlled synthesis (Suzuki *et al.*, 1994). In view of the medicinal importance of the title compound, (I), and as a continuation of our studies, an X-ray structural analysis of the compound was carried out and the results are reported here.



(I)

Fig. 1 shows a displacement ellipsoid diagram of the molecule with the atomic numbering scheme. Selected geometric parameters are given in Table 1. The bond lengths in the pyrrolizidine moiety are slightly longer than the values reported for similar structures (Seshadri *et al.*, 2003; Govind *et

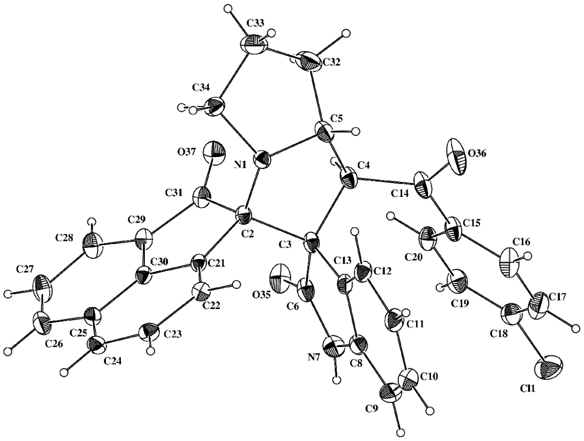


Figure 1
View of (I) (50% probability displacement ellipsoids).

DOI: 10.1107/S1600536803027296

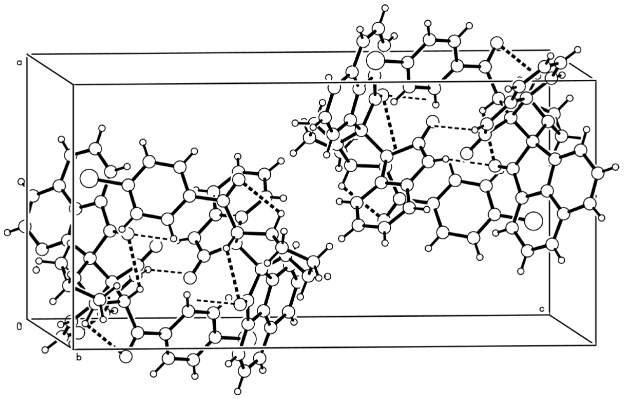


Figure 2
Packing of the molecule, viewed approximately down the *b* axis. Hydrogen bonds are shown as dashed lines.

al., 2003). This may be due to steric forces caused by the bulky substituents on the pyrrolizidine moiety. The sum of angles at N1 of the pyrrolizidine ring system (338.0°) is in accordance with sp^3 hybridization (Beddoes *et al.*, 1986).

In the benzene ring of the indole system, the endocyclic angles at C9 and C12 are contracted to 117.4 (2) and 119.1 (2)°, respectively, while those at C8 and C10 are expanded to 122.1 (2) and 121.5 (2)°, respectively. This would appear to be a real effect caused by the fusion of the smaller pyrrole ring to the six-membered benzene ring, the strain being taken up by angular distortion rather than by bond-length distortions (Sethu Sankar *et al.*, 2002).

Keto atom O37 is displaced by 0.331 (1) Å from the acenaphthylene plane, while keto atom O35 is displaced only 0.045 (2) Å from the oxindole plane; this is probably due to the different environment of the two O atoms, O37 being a hydrogen-bond donor for N7—H7, while there is no hydrogen bond involving O35 (Table 2). Keto atom O36 is 0.521 (3) Å out of the plane through C4/C14/C16/C20, with the C14═O36 bond orientation defined by the torsion angles O36—C14—C15—C20 = −148.6 (3)° and O36—C14—C15—C16 = 32.1 (3)° . Atom O36 is an acceptor of a weak intramolecular interaction from C5—H5.

The two five-membered rings of the pyrrolizidine moiety have envelope conformations, with puckering parameters (q_2 and φ; Cremer & Pople, 1975) and smallest displacement asymmetry parameters (Δ; Nardelli, 1983) as follows: for ring N1/C2–C5: q_2 = 0.424 (2) Å, φ = −114.2 (2)°, Δ_S(C3) = 0.038 (1) and Δ_2(C5) = 0.047 (1); for ring N1/C34/C33/C32/C5: q_2 = 0.377 (2) Å, φ = 89.8 (3)°, Δ_2(N1) = 0.002 (1), Δ_S(C33) = 0.091 (1) and Δ_S(C32) = 0.094 (1).

The molecular structure is influenced by C—H···O intramolecular interactions. The crystal packing is stabilized by N—H···O intermolecular interaction (Fig. 2 and Table 2).

Experimental

A mixture of (*E*)-3-(*p*-Chlorophenacylidine)oxindole, acenaphthenequinone and L-proline was stirred at room temperature in aqueous methanol. The resulting crude product was purified by column chromatography to obtain the title compound.

Crystal data

$C_{32}H_{23}ClN_2O_3$
M_r = 506.96
Monoclinic, $P2_1/n$
a = 11.5085 (9) Å
b = 9.5986 (8) Å
c = 23.0306 (18) Å
β = 91.232 (1)°
V = 2543.5 (4) Å^3
Z = 4
D_x = 1.324 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5762 reflections
θ = 2.3–27.8°
μ = 0.19 mm^{-1}
T = 293 (2) K
Block, colourless
0.21 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
15033 measured reflections
5829 independent reflections
4365 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.017
$\theta_{\rm max}$ = 28.0°
$h = -14 \rightarrow 15$
$k = -12 \rightarrow 9$
$l = -29 \rightarrow 30$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.160
S = 1.01
5829 reflections
343 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0961P)^2 + 0.4518P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.54 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.49 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.453 (2)	N1—C5	1.479 (2)
N1—C34	1.473 (2)	C2—C3	1.567 (2)
C2—N1—C34	118.3 (1)	C9—C8—C13	122.1 (2)
C2—N1—C5	110.4 (1)	C8—C9—C10	117.4 (2)
C34—N1—C5	109.3 (1)	C10—C11—C12	120.5 (2)
C6—N7—C8	112.9 (1)	C13—C12—C11	119.1 (2)
O36—C14—C15—C16	32.1 (3)	O36—C14—C15—C20	−148.6 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O37	0.98	2.51	3.093 (2)	118
C5—H5···O36	0.98	2.37	2.777 (3)	104
N7—H7···O37$^{\rm i}$	0.86	2.15	2.893 (2)	144

Symmetry code: (i) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$.

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å, $U_{\rm iso}$(H) = 1.5 $U_{\rm eq}$(C) for methyl H atoms and 1.2$U_{\rm eq}$(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: *ZORTEP* (Zsolnai, 1997) and *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

DV thanks the University Grants Commission (UGC), New Delhi, for financial support.

References

Beddoes, R. L., Dalton, L., Joule, T. A., Mills, O. S., Street, J. D. & Watt, C. I.-F. (1986). *J. Chem. Soc. Perkin Trans.* pp. 787–797.

Bindra, J. S. (1973). *Oxindole Alkaloids, Alkaloid Chemistry and Physiology*, edited by R. H. F. Manke. New York: Academic Press.

Bruker (2001). *SAINT* (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.

Govind, M. M., Selvanayagam, S., Velmurugan, D., Ravikumar, K., Gowri Sridhar & Raghunathan, R. (2003). *Acta Cryst.* E**59**, o1438–o1440.

Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

Seshadri, P. R., Selvanayagam, S., Velmurugan, D., Ravikumar, K., Sureshbabu, A. R. & Raghunathan, R. (2003). *Acta Cryst.* E**59**, o1458–o1460.

Sethu Sankar, S., Kannadasan, S., Velmurugan, D., Srinivasan, P. C., Shanmuga Sundara Raj., S. & Fun, H.-K. (2002). *Acta Cryst.* C**58**, o277–o279.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. 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.

Zsolnai, L. (1997). *ZORTEP*. University of Heidelberg, Germany.