Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1′-(4-Methoxybenzoyl)acenaphthene-1-spiro-3′-pyrrolizidine-2′-spiro-3″-1*H*-indole-2,2″(1*H*,3″*H*)-dione

M. M. Govind,[a] S. Selvanayagam,[a] D. Velmurugan,[a]* K. Ravikumar,[b] A. R. Sureshbabu[c] and R. Raghunathan[c]

[a]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, [b]Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and [c]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) = 0.005 Å
R factor = 0.085
wR factor = 0.196
Data-to-parameter ratio = 14.6

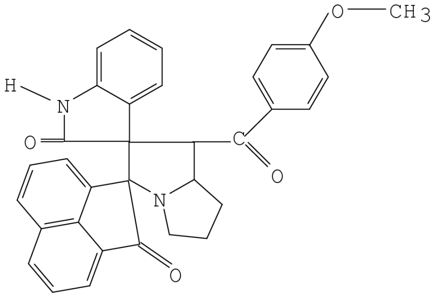
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 25 March 2004
Accepted 15 April 2004
Online 30 April 2004

The N—C and C—C bond distances in the pyrrolizidine moiety of the title compound, $C_{33}H_{26}N_2O_4$, are slightly longer than the normal values. This is due to the steric forces caused by bulky substituents on the pyrrolizidine ring system. The acenaphthene ketone O atom is −0.214 (3) Å from the acenaphthene plane, while the indole ketone O atom is displaced by only 0.004 (3) Å from the indole plane. The molecular structure is stabilized by intramolecular C—H···O interactions. The crystal packing is stabilized by C—H···O intermolecular interactions.

Comment

Among a variety of heterocycles that have been explored for the development of pharmaceutically important molecules, indoles have played an important role. Some of them have received considerable attention as potential antimicrobial agents (Mogilaiah *et al.*, 2001). Spiro-indoles have also been reported to show fungicidal activity (Ali *et al.*, 1989). In view of the pharmacological 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 of (I) with the atomic numbering scheme. Selected geometric parameters are given in Table 1.

The N—C and C—C bond lengths in the pyrrolizidine moiety are slightly longer than the normal values reported for similar structures (Seshadri *et al.*, 2003; Abdul Ajees *et al.*, 2002). This may be due to steric forces caused by the bulky substituents on the pyrrolizidine moiety.

The ketone atom O36 is −0.214 (3) Å from the acenaphthene plane, while the ketone atom O35 is displaced by only 0.004 (3) Å from the oxindole plane, probably due to the different environment of the two O atoms, O36 being a hydrogen-bond acceptor from C10—H10, while there is no hydrogen bond involving O35 (Table 2). The ketone atom O37 is 0.731 (3) Å out of the C4/C14–C20 plane, with a C14═O37

DOI: 10.1107/S1600536804009006

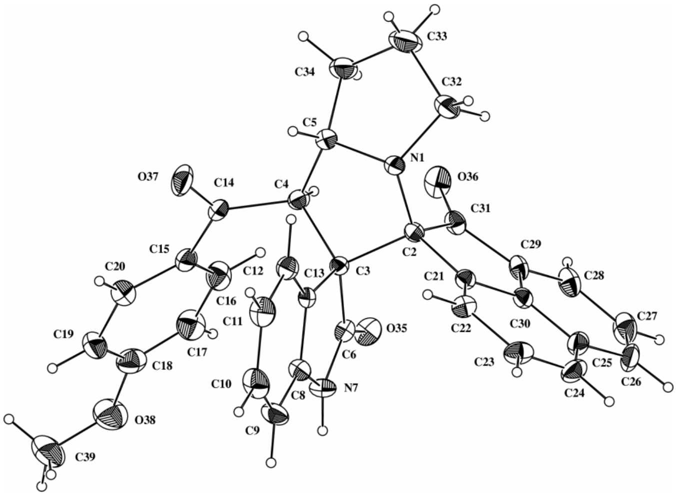


Figure 1
View of (I) (50% probability displacement ellipsoids).

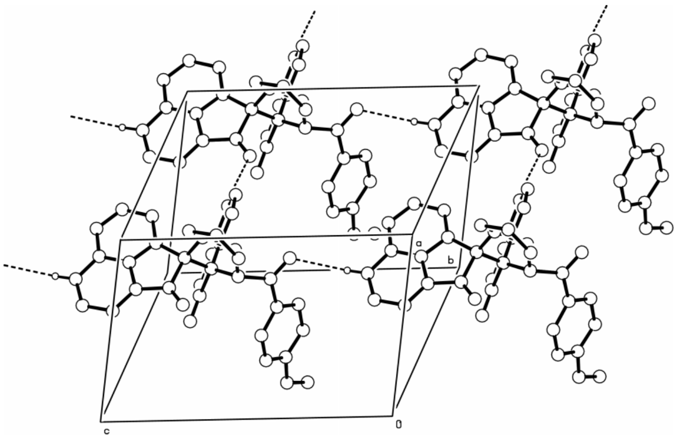


Figure 2
Packing of the structure of (I).

bond direction defined by the torsion angles O37—C14—C15—C20 = −24.1 (5)° and O37—C14—C15—C16 = 157.3 (4)°. Atom O37 is an acceptor of a weak intramolecular interaction from C5—H5.

The orientation of the methoxy group with respect to the benzene ring is defined by torsion angles C19—C18—O38—C39 = 3.5 (6)° and C17—C18—O38—C39 = −176.5 (4)°.

The C2–C5/N1 ring of the pyrrolizidine moiety has an envelope conformation, with Cremer & Pople (1975) puckering parameters q_2 = 1.992 (3) Å and φ = 71.5 (1)°, and the smallest displacement asymmetry parameter (Nardelli, 1983) is Δ_S(C2) = 0.024 (3). The five-membered N1/C5/C32–C34 ring of the pyrrolizidine moiety adopts a twist conformation, with puckering parameters q_2 = 0.588 (9) Å and φ = −141.6 (4)°, the displacement asymmetry parameters being Δ_S(C33) = 0.023 (3) and Δ_2(C34) = 0.090 (3).

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

Experimental

A mixture of (*E*)-3-(*p*-methoxyphenacylidine)oxindole (1 mmol), acenaphthenequinone (1 mmol) and L-proline (1 mmol) was stirred in aqueous methanol at room temperature. The resulting crude product was purified by column chromatography to obtain the title compound, which was recrystallized from chloroform–methanol (1:1).

Crystal data

$C_{33}H_{26}N_2O_4$
M_r = 514.56
Triclinic, $P\overline{1}$
a = 10.2215 (11) Å
b = 10.6228 (12) Å
c = 12.3584 (13) Å
α = 104.178 (2)°
β = 97.238 (2)°
γ = 91.499 (2)°
V = 1288.4 (2) Å^3
Z = 2
D_x = 1.326 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1062 reflections
θ = 2.7–21.0°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colorless
0.21 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX area-detector diffractometer
ω scans
7866 measured reflections
5149 independent reflections
3011 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.029
$\theta_{\rm max}$ = 28.0°
$h = -12 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = -16 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.085$
$wR(F^2) = 0.196$
S = 1.09
5149 reflections
352 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0752P)^2 + 0.2551P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.44 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.461 (4)	C2—C3	1.568 (4)
N1—C32	1.482 (4)	C3—C4	1.563 (4)
N1—C5	1.487 (4)	C5—C34	1.525 (4)
O37—C14—C15—C20	−24.1 (5)	C17—C18—O38—C39	−176.5 (4)
O37—C14—C15—C16	157.3 (4)	C19—C18—O38—C39	3.5 (6)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O36	0.98	2.40	3.009 (4)	120
C5—H5···O37	0.98	2.40	2.824 (4)	105
C10—H10···O36$^{\rm i}$	0.93	2.39	3.301 (5)	165
C26—H26···O37$^{\rm ii}$	0.93	2.49	3.375 (5)	159

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

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å and $U_{\rm iso}$(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, 2003); 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

Abdul Ajees, A., Manikandan, S. & Raghunathan, R. (2002). *Acta Cryst.* E**58**, o802–o804.
Ali, R., Bharati, M. & Nizamuddin (1989). *Indian J. Chem. Sect. B*, **28**, 526–528.
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.
Mogilaiah, K., Ramesh Babu, H. & Babu Rao, R. (2001). *Indian J. Chem. Sect. B*, **40**, 1270–1274.
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.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Zsolnai, L. (1997). *ZORTEP*. University of Heidelberg, Germany.