Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5,7-Dibromo-4′-(4-bromobenzoyl)-1′-methyl-1*H*-indole-3-spiro-2′-pyrrolidine-3′-spiro-3″-1*H*-indole-2,2″(3*H*,3″*H*)-dione methanol solvate

P. R. Seshadri,[a,b] S. Selvanayagam,[a] D. Velmurugan,[a]* 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, [b]Department of Physics, Agurchand Manmull Jain College, Chennai 600 114, 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) = 0.007 Å
R factor = 0.055
wR factor = 0.185
Data-to-parameter ratio = 18.7

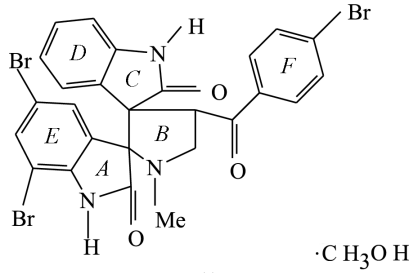
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 26 March 2004
Accepted 19 April 2004
Online 30 April 2004

The pyrrolidine ring of the title compound, $C_{26}H_{18}Br_3N_3O_3\cdot CH_4O$, adopts an envelope conformation. The molecular structure is stabilized by C—H···O interactions and the packing is stabilized by N—H···O and O—H···N intermolecular interactions, which also include the methanol solvent molecules.

Comment

Heterocyclic compounds, especially five- and six-membered rings, have occupied an important place among organic compounds for their biological activities. Some of them have received attention as anti-microbial agents. Substituted pyrrolidine compounds have gained much importance because they are the structural elements of many alkaloids. It has been found that they have antifungal activity against various pathogens (Amal Raj *et al.*, 2003). Structural classification divides this alkaloid family into several subgroups, among which oxindoles deserve to be mentioned (Bindra, 1973). Several unusual amino acids which contain the pyrrolidine motif have been investigated by Galeazzi *et al.* (1999). The spiro ring system is a frequently encountered structural motif in many pharmacologically relevant alkaloids (Cordel, 1981). In view of this biological importance and as a part of studies of spiro pyrrolidines, the crystal structure of the title compound, (I), has been determined and the results are presented here.



(I)

Fig. 1 shows a displacement ellipsoid diagram of the asymmetric unit with the atomic numbering scheme. Selected geometric parameters are given in Table 1. The methyl group at N14 is in an equatorial position [C12—C13—N14—C15 = 170.0 (4)°]. Atom Br3 lies almost in the plane of the benzoyl ring [Br3—C30—C31—C32 = −178.6 (4)°].

The dihedral angle formed by the pyrrole and benzene planes of the two oxindole moieties are 2.8 (1) (for rings *A* and *E*) and 3.6 (1)° (for rings *C* and *D*). The fusion of the pyrrole ring with the benzene group has caused some minor angular distortions in rings *D* and *E*; similar effects have been reported by Govind *et al.* (2003).

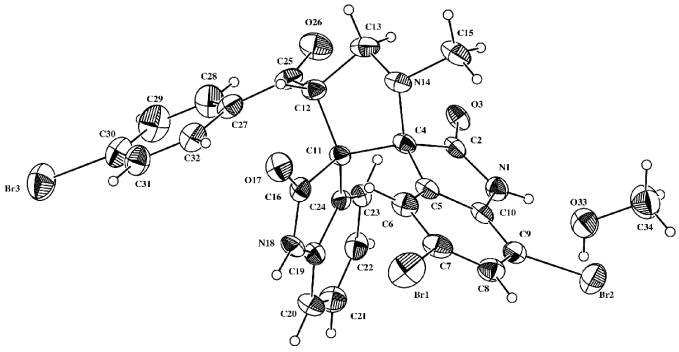


Figure 1
View of (I), with 50% probability displacement ellipsoids.

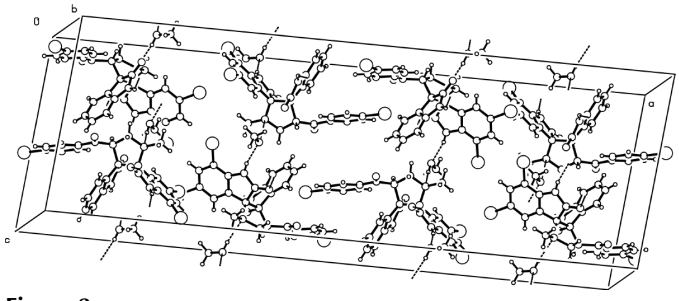


Figure 2
The packing of (I), viewed approximately along the *b* axis. Hydrogen bonds are shown as thin dashed lines.

Ring *A* is almost planar and ring *C* shows a slight envelope conformation, with atoms C11, C24, C19 and N18 in a common plane. The pyrrolidine ring *B* makes dihedral angles of 88.3 (1) and 82.1 (2)° with the oxindole ring systems *A*/*E* and *C*/*D*, respectively, showing they are in nearly perpendicular configurations.

The total puckering amplitudes (Cremer & Pople, 1975) of the rings give a quantitative evaluation of puckering and asymmetry parameters. The pyrrolidine ring (*B*) is in an envelope conformation with lowest asymmetry parameters (Nardelli, 1983) ΔC_S(N14) = 0.028 (2), with N14 deviating by 0.600 (3) Å from the least-squares plane passing through the remaining four atoms, and with puckering parameters q_2 = 0.413 (4) Å and φ = 139.3 (5)°.

The methanol solvent molecule participates in hydrogen-bonding interactions, and further N—H···O and C—H···O intermolecular interactions stabilize the packing. The molecular structure is also stabilized by C—H···O interactions (Table 2).

Experimental

A mixture of (*E*)-3-(*p*-bromophenacylidine)oxindole (1 mmol), 5,7-dibromoisatin (1 mmol) and sarcosine (1 mmol) was stirred at room temperature in aqueous methanol. The resulting crude product was purified by column chromatography. The product was recrystallized from methanol to yield good quality crystals of (I) suitable for data collection.

Crystal data

$C_{26}H_{18}Br_3N_3O_3{\cdot}CH_4O$
M_r = 692.21
Monoclinic, $C2/c$
a = 46.242 (4) Å
b = 7.7993 (6) Å
c = 15.3204 (12) Å
β = 98.958 (2)°
V = 5458.0 (7) Å^3
Z = 8
D_x = 1.685 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2677 reflections
θ = 2.7–21.5°
μ = 4.47 mm^{-1}
T = 293 (2) K
Block, colourless
0.23 × 0.20 × 0.18 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.381, T_{max} = 0.447
16092 measured reflections
6293 independent reflections
3493 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 28.0°
$h = -60 \rightarrow 51$
$k = -10 \rightarrow 9$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.185
S = 0.91
6293 reflections
337 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1114P)^2 + 0.9332P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.79 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C4—N14	1.467 (5)	C12—C13	1.521 (7)
C4—C11	1.584 (6)	C13—N14	1.474 (6)
C11—C12	1.575 (6)		
N14—C4—C11	102.8 (3)	C4—N14—C13	105.9 (3)
C13—C12—C11	105.1 (4)	C4—N14—C15	114.1 (4)
N14—C13—C12	103.4 (3)	C13—N14—C15	112.7 (4)
C12—C13—N14—C15	170.0 (4)	Br3—C30—C31—C32	−178.6 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
N18—H18···O3^{i}	0.86	2.07	2.786 (4)	141
O33—H33···N14^{ii}	0.82	2.33	3.069 (5)	151
C31—H31···O26^{i}	0.93	2.50	3.420 (7)	173
N1—H1···O33	0.86	1.99	2.823 (5)	164
C6—H6···O17	0.93	2.37	3.025 (6)	128
C12—H12···O17	0.98	2.46	2.943 (5)	110
C13—H13*A*···O3	0.97	2.47	3.051 (6)	118
C23—H23···O3	0.93	2.41	2.927 (5)	115

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

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å [U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms] and N—H = 0.86 Å [U_{iso}(H) = 1.2U_{eq}(N)]. In addition, the torsion angles of the methyl and hydroxyl groups were refined.

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

Amal Raj, A., Raghunathan, R., Sridevikumari, M. R. & Raman, N. (2003). *Bioorg. Med. Chem.* **11**, 407–419.
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.
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.
Galeazzi, R., Geremia, S., Mobbilli, G. & Orena, M. (1999). *Tetrahedron Asymm.* **10**, 587–605.
Govind, M. M., Govindaraj, J., Rajakannan, V., Velmurugan, D., Kim, M. J., Srinivasan, P. C. & Kannadasan, S. (2003). *Acta Cryst.* E**59**, o177–o179.
Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
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. (2003). *J. Appl. Cryst.* **36**, 7–13.
Zsolnai. L. (1997). *ZORTEP*. University of Heidelberg, Germany.