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

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.048
wR factor = 0.141
Data-to-parameter ratio = 18.1

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

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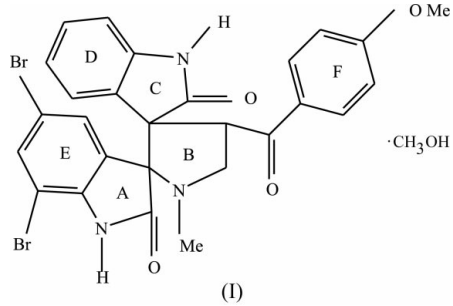
The pyrrolidine ring of the title compound, $C_{27}H_{21}Br_2N_3O_4\cdot CH_4O$, adopts an envelope conformation. The methanol solvent molecule is involved in intermolecular hydrogen bonds. The molecular structure is stabilized by C—H···O interactions, and the packing is stabilized by N—H···O, O—H···O and O—H···N intermolecular interactions.

Comment

Heterocyclic compounds, especially five- and six-membered rings, have occupied an important place among organic compounds because of their biological activities. These compounds have been explored for the development of pharmaceutically important molecules, such as indoles, because of their important role in medicinal chemistry. Some of these compounds have received attention as antimicrobial agents. The biological activities, such as fungicidal activity, of novel heterocycles were reported by Ali *et al.* (1989). Substituted pyrrolidine compounds have gained importance because they are the basic structural elements of many alkaloids and pharmacologically active compounds. Structural classification divides this alkaloid family into several subgroups, among which oxindoles deserve to be mentioned (Bindra, 1973). In view of this biological importance, 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 molecule, with the atomic numbering scheme. Selected geometric parameters are given in Table 1.

The sum of the angles around N14 is 334.4°, showing that it is in sp^3 hybridization. The dihedral angles formed by the pyrrole and benzene planes of the two oxindole moieties are 3.1 (1)° (for rings *C* and *D*) and 5.3 (1)° (for rings *A* and *E*). The fusion of the smaller pyrrole ring with the benzene group causes some minor angular distortions in rings *D* and *E*; similar effects have been reported by Sivaraman *et al.* (1994).

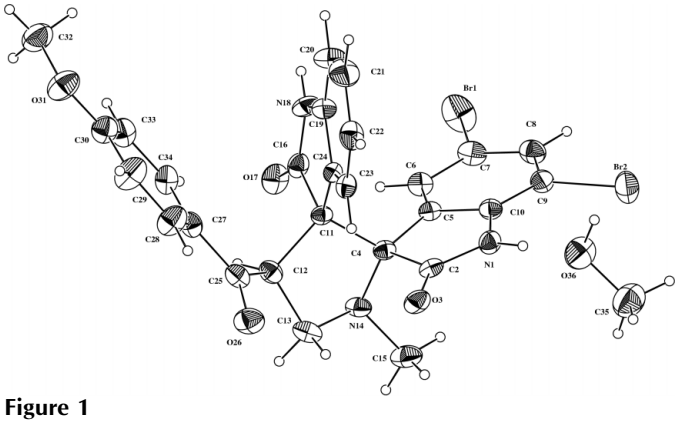


Figure 1
View of (I), with 50% probability displacement ellipsoids.

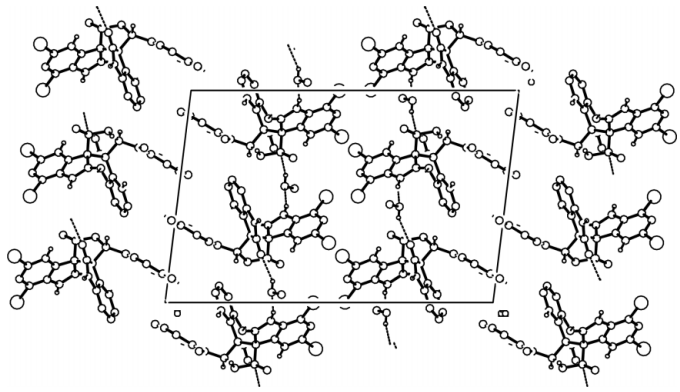


Figure 2
Packing of (I), viewed along the *b* axis. Hydrogen bonds are shown as thin lines.

The methyl group at atom N14 is in an equatorial position [C12—C13—N14—C15 = 170.4 (3)°]. The methoxy group is almost coplanar with the benzoyl ring [C28—C29—C30—O31 = 179.1 (4)°].

Ring *A* is almost planar and ring *C* shows a slight envelope conformation, with atoms C11, C24, C19 and N18 coplanar. The pyrrolidine moiety makes dihedral angles of 86.6 (1) and 80.2 (1)° with the oxindole ring systems *A*/*E* and *C*/*D*, respectively.

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 = 0.009 (2)], with N14 deviating by 0.592 (2) Å from the least-squares plane passing through the remaining four atoms and with puckering parameters q_2 = 0.402 (3) Å and φ = 142.7 (4)°.

The methanol solvent molecule participates in hydrogen-bond interactions. Methanol atom O36 is hydrogen bonded to atoms O17 and N14 of an adjacent symmetry-related molecule.

The molecular structure is stabilized by C—H···O and N—H···O interactions, and the packing is stabilized by N—H···O, O—H···O and O—H···N intermolecular interactions (Table 2.).

Experimental

A mixture of (*E*)-3-(*p*-methoxyphenacylidine)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 using methanol, yielding good quality crystals for data collection.

Crystal data

$C_{27}H_{21}Br_2N_3O_4{\cdot}CH_4O$
M_r = 643.33
Monoclinic, $P2_1/c$
a = 23.285 (2) Å
b = 7.8966 (7) Å
c = 15.1669 (14) Å
β = 96.979 (2)°
V = 2768.1 (4) Å^3
Z = 4
D_x = 1.544 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3283 reflections
θ = 2.7–21.4°
μ = 2.97 mm^{-1}
T = 293 (2) K
Block, colourless
0.23 × 0.21 × 0.17 mm

Data collection

Bruker Smart APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.548, T_{max} = 0.632
16 394 measured reflections
6201 independent reflections
3759 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 28.0°
$h = -25 \rightarrow 29$
$k = -10 \rightarrow 9$
$l = -19 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.141
S = 0.99
6201 reflections
343 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0726P)^2 + 0.703P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.88 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C4—N14	1.456 (4)	C12—C13	1.513 (5)
C4—C11	1.581 (4)	C13—N14	1.458 (5)
C11—C12	1.576 (4)		
N14—C4—C11	102.8 (2)	C13—C12—C11	105.1 (3)
C5—C6—C7	118.0 (3)	N14—C13—C12	104.1 (3)
C8—C7—C6	122.3 (3)	C4—N14—C13	106.3 (3)
C10—C9—C8	119.8 (3)	C4—N14—C15	115.1 (3)
C9—C10—C5	120.6 (3)	C13—N14—C15	113.0 (3)
C12—C11—C4	103.2 (2)		
C12—C13—N14—C15	170.4 (3)	C28—C29—C30—O31	179.1 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O36	0.86	1.96	2.802 (4)	165
C6—H6···O17	0.93	2.37	3.046 (5)	129
C12—H12···O17	0.98	2.47	2.943 (4)	110
C13—H13*A*···O3	0.97	2.47	3.036 (5)	117
C13—H13*A*···O26	0.97	2.47	2.820 (5)	101
C23—H23···O3	0.93	2.38	2.966 (4)	121
N18—H18···O3^{i}	0.86	2.03	2.806 (3)	150
O36—H36···O17^{ii}	0.82	2.54	3.053 (3)	122
O36—H36···N14^{ii}	0.82	2.46	3.183 (4)	147

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

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å, N—H = 0.86 Å, and U_{iso}(H) set at 1.5U_{eq}(C) for methyl H atoms, 1.2U_{eq}(C) for other C-bound H atoms and 1.2U_{eq}(N) for N-bound 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).

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