

2'-(*p*-Methoxybenzoyl)-1',2,2',3,5',6',7',7a'-octahydro-1*H*-indan-2-spiro-3'-(3'*H*-pyrrolizine)-1'-spiro-3''-1*H*-indoline-1,2'',3-trione

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Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$

R factor = 0.049

wR factor = 0.139

Data-to-parameter ratio = 16.6

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

The pyrrolidine ring of the title compound, $\text{C}_{30}\text{H}_{24}\text{N}_2\text{O}_5$, adopts a half-chair conformation. The structure is stabilized by $\text{C}-\text{H}\cdots\text{O}$ intramolecular interactions and the packing of the molecules is stabilized by $\text{C}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ intermolecular interactions.

Comment

Several optically active pyrrolidines have been used as intermediates in controlled asymmetric synthesis (Suzuki *et al.*, 1994). The spiro-indole-pyrrolidine ring system is a frequently encountered structural motif in many pharmacologically relevant alkaloids as typified by vincristine, vinblastine and spirotopostatins. In view of this medicinal importance, the crystal structure of the title compound, (I), has been carried out and the results are presented here.

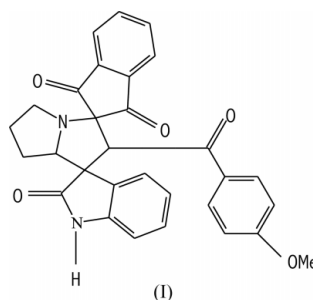


Fig. 1 shows a displacement ellipsoid plot of (I) with the atomic numbering scheme. Selected geometric parameters are given in Table 1. The bond lengths in the pyrrolidine ring (Table 1) deviate from normal values due to steric forces of the bulky substituents at the pyrrolidine ring, as reported in related structures (Jeyabharathi *et al.*, 2001; Gzella & Wrzecziono, 1990). Keto atoms O22 and O30 deviate from the mean plane of the ring to which they are attached by 0.087 (1) and 0.293 (1) Å, respectively.

In the benzene ring of the oxindole system, the endocyclic angles at C10 and C7 are 122.08 (18) and 120.91 (19)°, respectively. At C9 and C6, the angles are 117.91 (19) and 118.89 (18)°, respectively. The deviation of these angles from the normal value of 120° may be due to the fusion of the small pyrrole ring to the six-membered benzene ring. A similar effect is observed in related structures (Seshadri *et al.*, 2003; Govind *et al.*, 2003).

The methoxybenzoyl ring is attached in an equatorial position to the pyrrolidine ring. This is confirmed by the torsion angle C14–C12–C11–C4 of 73.0 (2)°. In the methoxy group, methyl atom C37 is turned towards C18. This is confirmed by the torsion angle C18–C17–O36–C37 of –1.5 (3)°.

Received 6 October 2003

Accepted 13 October 2003

Online 23 October 2003

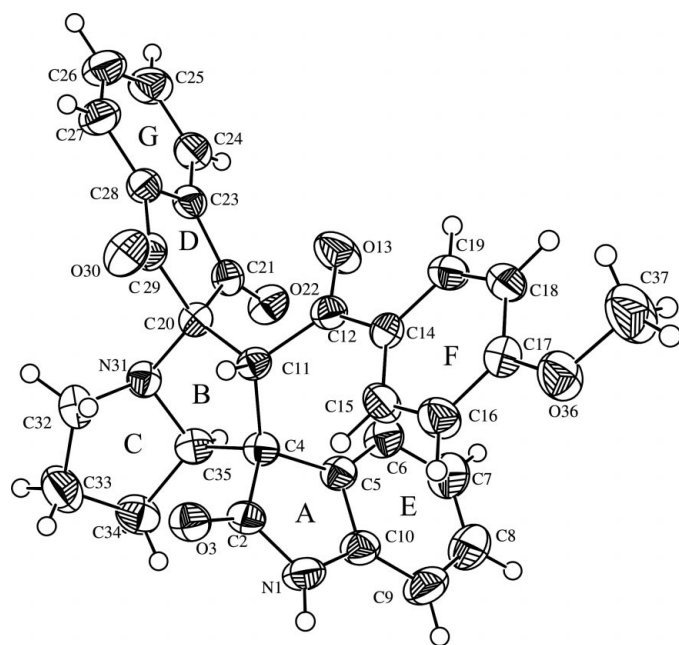


Figure 1
View of the title compound (50% probability displacement ellipsoids).

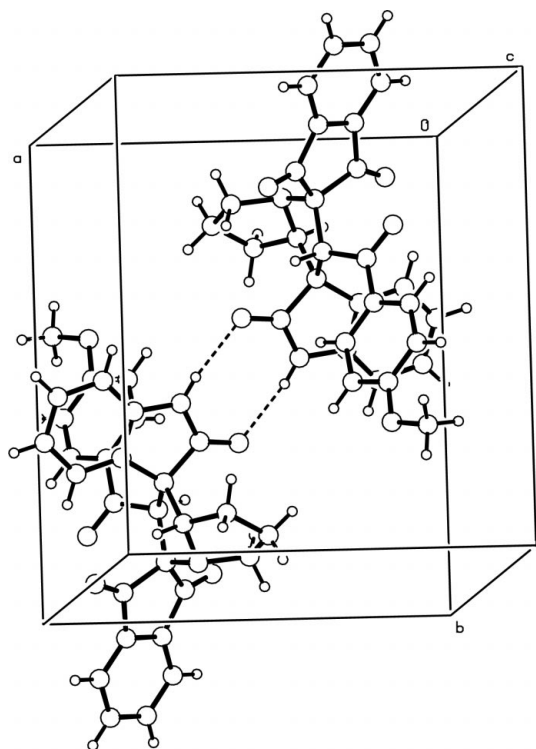


Figure 2
Packing diagram, showing a dimer formed by N—H...O hydrogen bonds, viewed along *c*.

The total puckering amplitudes (Cremer & Pople, 1975) of the rings give a quantitative evaluation of puckering and asymmetry parameters. Ring *A* is planar. The asymmetry parameters (Nardelli, 1995) $q_2 = 0.3501$ (5), $\varphi = 86.19$ (8) $^\circ$ and $\Delta C_2[C4] = 0.0143$ (7) $^\circ$ reveal a half-chair conformation for ring *B*. Ring *C* adopts an envelope conformation. This is

confirmed by the asymmetry parameters $q_2 = 0.3326$ (7), $\varphi = 2.10$ (9) $^\circ$, $\Delta C_s[N31] = 0.0097$ (1) $^\circ$. The asymmetry parameters $q_2 = 0.1267$ (7), $\varphi = -5.07$ (2) $^\circ$ and $\Delta C_s[C20] = 0.0091$ (8) $^\circ$ reveal an envelope conformation for ring *D*.

In addition to van der Waals interactions, the crystal structure is stabilized by C—H...O intramolecular hydrogen bonds. In the crystal structure, symmetry-related molecules are linked by C—H...O and N—H...O intermolecular interactions. Details of these interactions are given in Table 2.

Experimental

A mixture of (*E*)-3-(*p*-methoxyphenacylidine)oxindole–ninhydrin and sacrosine was stirred in aqueous methanol at room temperature. The resulting crude product was filtered and recrystallized from methanol.

Crystal data

$C_{30}H_{24}N_2O_5$
 $M_r = 492.51$
Monoclinic, $P2_1/c$
 $a = 11.4931$ (8) Å
 $b = 13.6374$ (9) Å
 $c = 15.5377$ (10) Å
 $\beta = 96.351$ (1) $^\circ$
 $V = 2420.4$ (3) Å 3
 $Z = 4$

$D_x = 1.352$ Mg m $^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 3592 reflections
 $\theta = 2.3$ – 26 $^\circ$
 $\mu = 0.09$ mm $^{-1}$
 $T = 293$ (2) K
Prism, yellow
0.23 × 0.20 × 0.17 mm

Data collection

Bruker SMART APEX area-detector diffractometer
 ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)
 $T_{\min} = 0.979$, $T_{\max} = 0.984$
14548 measured reflections

5528 independent reflections
3764 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$
 $\theta_{\max} = 28.0$ $^\circ$
 $h = -14 \rightarrow 14$
 $k = -17 \rightarrow 17$
 $l = -20 \rightarrow 13$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.139$
 $S = 1.00$
5528 reflections
334 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0798P)^2 + 0.041P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.27$ e Å $^{-3}$
 $\Delta\rho_{\min} = -0.16$ e Å $^{-3}$

Table 1

Selected geometric parameters (Å, $^\circ$).

N1—C2	1.342 (2)	C20—N31	1.462 (2)
N1—C10	1.403 (2)	C21—O22	1.211 (2)
C2—O3	1.228 (2)	C29—O30	1.208 (2)
C4—C11	1.564 (2)	N31—C35	1.463 (2)
C12—O13	1.217 (2)	N31—C32	1.479 (2)
C17—O36	1.356 (2)	O36—C37	1.413 (2)
C2—N1—C10	112.0 (1)	O22—C21—C23	125.4 (2)
O3—C2—C4	126.4 (2)	O22—C21—C20	126.7 (2)
N1—C2—C4	108.3 (1)	O30—C29—C28	126.9 (2)
C9—C10—N1	128.9 (2)	O30—C29—C20	124.7 (2)
O13—C12—C14	120.4 (2)	C20—N31—C35	105.0 (1)
O13—C12—C11	118.1 (2)	C20—N31—C32	117.2 (1)
O36—C17—C16	115.8 (2)	N31—C32—C33	103.5 (2)
O36—C17—C18	124.4 (2)	N31—C35—C34	104.3 (1)
N31—C20—C29	113.2 (1)	N31—C35—C4	107.5 (1)
N31—C20—C21	104.5 (1)	C17—O36—C37	118.7 (2)
C4—C11—C12—C14	−73.0 (2)	C18—C17—O36—C37	−1.5 (3)

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

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O3 ⁱ	0.86	2.12	2.960 (2)	165
C6—H6...O22	0.93	2.41	3.215 (2)	145
C11—H11...O3	0.98	2.54	3.033 (2)	111
C11—H11...O30	0.98	2.50	2.924 (2)	106
C16—H16...O30 ⁱⁱ	0.93	2.52	3.190 (2)	130
C26—H26...O36 ⁱⁱⁱ	0.93	2.59	3.241 (2)	127
C27—H27...O3 ^{iv}	0.93	2.49	3.211 (2)	134
C35—H35...O22	0.98	2.56	3.084 (2)	113

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

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

Financial support from the Department of Science and Technology (DST) and the University Grants Commission (UGC), India, is gratefully acknowledged.

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