

2'-Benzoyl-1',2,2',3,4',5',6',6a'-octahydro-1H-indan-2-spiro-3'-(3'H-pyrrolizine)-1'-spiro-3''-1H-indoline-1,2'',3-trione

P. R. Seshadri,^a
S. Selvanayagam,^b
D. Velmurugan,^{b*}
K. Ravikumar,^c
A. R. Sureshbabu^d and
R. Raghunathan^d

^aDepartment of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, Department of Physics, Agurchand Manmull Jain college, Chennai 600 114, India, ^bDepartment of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, ^cLaboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and ^dDepartment 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 $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.060
wR factor = 0.171
Data-to-parameter ratio = 16.7

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, $\text{C}_{29}\text{H}_{22}\text{N}_2\text{O}_4$, adopts a half-chair conformation. The molecular structure is stabilized by $\text{C}-\text{H}\cdots\text{O}$ interactions and the packing is stabilized by $\text{C}-\text{H}\cdots\pi$ and $\text{N}-\text{H}\cdots\text{O}$ interactions. A dimer is formed between symmetry-related molecules through $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Comment

The spiro indole-pyrrolidine ring system is a frequently encountered structural motif in many pharmacologically relevant alkaloids, e.g. vincristine, vinblastine and spiro-topostatin. Further structural classification divides this alkaloid family into several subgroups, among which oxindoles deserve mention (Cordel, 1981; Bindra, 1973). Several optically active pyrrolidines have been used as intermediates in controlled asymmetric synthesis (Suzuki *et al.*, 1994). Substituted pyrrolidine compounds have gained much importance in the past, since they are the basic structural elements of many alkaloids and pharmacologically active compounds. They are found to have antimicrobial and antifungal activity against various pathogens except *Bacillus subtilis* (Amal Raj *et al.*, 2003). In view of this medicinal importance, the crystal structure of the title compound has been carried out and the results are presented here.

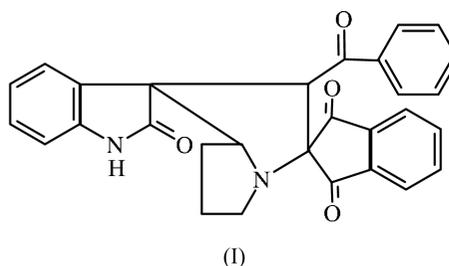


Fig. 1 shows a displacement ellipsoid diagram of the title molecule, (I), with the atomic numbering scheme. Selected geometric parameters are given in Table 1.

The bond lengths in the pyrrolidine moiety (Table 1) are slightly longer than normal values, but are comparable with those in previously reported structures (Jeyabharathi *et al.*, 2001; Gzella & Wrzeczono, 1990). This may be due to steric forces of bulky substituents on the pyrrolidine moiety. The keto O atoms O22 and O30 deviate from the mean plane containing the ring to which they are attached by -0.236 (1) and 0.059 (1) \AA , respectively.

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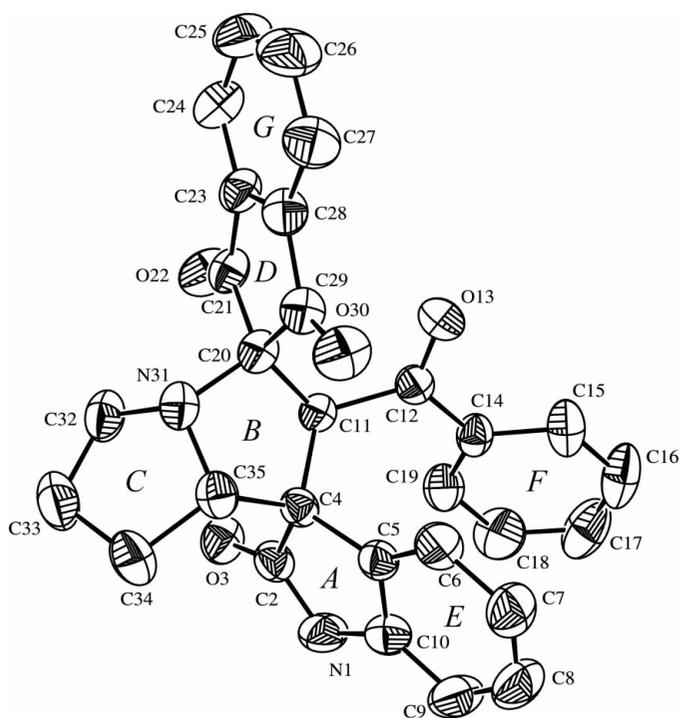


Figure 1
View of (I) (50% probability displacement ellipsoids). H atoms have been omitted.

In the benzene ring of the oxindole system, the endocyclic angles at C10 and C7 are 122.1 (2) and 121.2 (2)°, respectively, and at C9 and C6, the angles are 117.6 (2) and 118.1 (2)°, respectively. They differ from 120°. This may be caused by the fusion of the small pyrrole ring to the six-membered benzene ring. A similar effect is observed in related structures (Sethu Sankar *et al.*, 2002; Seshadri *et al.*, 2002; Govind *et al.*, 2003).

The benzoyl ring is attached to the pyrrolidine ring in a +synclinal conformation, confirmed by the torsion angle C4–C11–C12–C14 of 56.2 (2)°.

The total puckering amplitudes (Cremer & Pople, 1975) of rings A, B, C and D give a quantitative evaluation of puckering and asymmetry parameters. Ring A is planar. The asymmetry parameters (Nardelli, 1995), $q_2 = 0.3331$ (6), $\varphi = -168.15$ (1)°, $\Delta C_2[C4] = 0.0216$ (7)° reveal a half-chair conformation for ring B. For ring C, $q_2 = 0.3545$ (3), $\varphi = -163.19$ (1)°, $\Delta C_2[C34] = 0.006$ (1)° confirm its half-chair conformation. Ring D adopts a half-chair conformation, which is confirmed by the asymmetry parameters $q_2 = 0.0854$ (7), $\varphi = 10.86$ (3)° and $\Delta C_2[C28] = 0.0058$ (7).

It is interesting to note that a dimer is formed between symmetry-related molecules through N–H···O hydrogen bonds (Fig. 2).

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

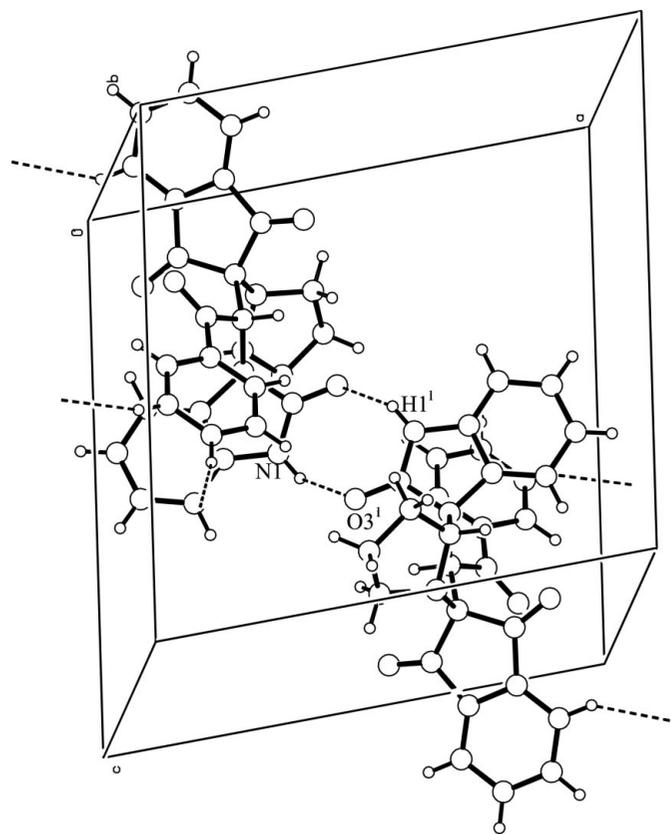


Figure 2
Packing diagram illustrating the N–H···O hydrogen-bonded dimers.

Experimental

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

Crystal data

$C_{29}H_{22}N_2O_4$
 $M_r = 462.49$
Monoclinic, $P2_1/c$
 $a = 13.1419$ (10) Å
 $b = 12.5844$ (9) Å
 $c = 14.0110$ (11) Å
 $\beta = 100.601$ (2)°
 $V = 2277.6$ (3) Å³
 $Z = 4$

$D_x = 1.349$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 2873 reflections
 $\theta = 2.5$ – 22.9 °
 $\mu = 0.09$ mm⁻¹
 $T = 293$ (2) K
Rod, colourless
 $0.25 \times 0.20 \times 0.18$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)
 $T_{\min} = 0.978$, $T_{\max} = 0.984$
14121 measured reflections

5281 independent reflections
3561 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$
 $\theta_{\text{max}} = 28.0$ °
 $h = -17 \rightarrow 13$
 $k = -16 \rightarrow 16$
 $l = -17 \rightarrow 16$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.060$
 $wR(F^2) = 0.171$
 $S = 1.01$
5281 reflections
316 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.096P)^2 + 0.132P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.28$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.343 (3)	C21—O22	1.207 (2)
N1—C10	1.402 (3)	C29—O30	1.207 (2)
C2—O3	1.228 (2)	N31—C35	1.464 (3)
C12—O13	1.215 (2)	N31—C32	1.471 (2)
C20—N31	1.470 (2)		
C2—N1—C10	111.6 (2)	N31—C20—C11	105.6 (2)
O3—C2—N1	125.6 (2)	O22—C21—C23	126.6 (2)
O3—C2—C4	125.7 (2)	O22—C21—C20	125.0 (2)
N1—C2—C4	108.6 (2)	O30—C29—C28	126.3 (2)
C5—C6—C7	118.1 (2)	O30—C29—C20	125.7 (2)
C8—C7—C6	121.2 (2)	C35—N31—C20	105.7 (1)
C9—C10—N1	128.4 (2)	C35—N31—C32	106.9 (2)
C5—C10—N1	109.5 (2)	C20—N31—C32	117.4 (2)
O13—C12—C14	120.8 (2)	N31—C32—C33	102.8 (2)
O13—C12—C11	119.2 (2)	N31—C35—C34	104.7 (2)
N31—C20—C21	113.0 (2)	N31—C35—C4	107.7 (2)
N31—C20—C29	106.1 (1)		
C4—C11—C12—C14	56.2 (2)		

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>
C6—H6...O30	0.93	2.44	3.250 (3)	146
C11—H11...O3	0.98	2.44	2.958 (2)	112
C11—H11...O22	0.98	2.55	2.976 (2)	106
C32—H32A...O3	0.97	2.58	3.260 (3)	127
N1—H1...O3 ⁱ	0.86	2.21	2.897 (2)	137
C16—H16...O30 ⁱⁱ	0.93	2.53	3.431 (3)	163
C17—H17...O13 ⁱⁱⁱ	0.93	2.37	3.268 (3)	163
C27—H27...O13 ^{iv}	0.93	2.59	3.357 (3)	139
C17—H17...Cg1 ⁱⁱⁱ	0.93	3.39	3.863 (3)	114
C19—H19...Cg2	0.93	2.78	3.056 (2)	98

Symmetry codes: (i) $1-x, -y, 1-z$; (ii) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iii) $x, \frac{1}{2}-y, z+\frac{1}{2}$; (iv) $-x, -y, -z$. Note: Cg1 is the centroid of the ring *D* and Cg2 is the centroid of ring *A*.

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

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