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2′-(*p*-Chlorobenzoyl)-2,2′,3,3′,4′,5′,6′,6a′-octahydro-1*H*-indan-2-spiro-3′-(1*H*-pyrrolizine)-1′-spiro-3″-indoline-1,2″,3-trione

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The pyrrolidine ring of the title compound, $C_{29}H_{21}ClN_2O_4$, adopts an envelope conformation. The structure is stabilized by C—H⋯O intramolecular interactions. A dimer is formed between symmetry-related molecules through C—H⋯O hydrogen bonds.

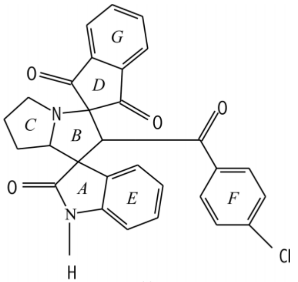
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.051
wR factor = 0.144
Data-to-parameter ratio = 16.1

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

Comment

The pyrrolidine skeleton occurs in many biologically important compounds (Baldwin *et al.*, 1994). Several optically active pyrrolidines have been used as intermediates in controlled asymmetric synthesis (Suzuki *et al.*, 1994). 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, (I), has been carried out and the results are presented here.



(I)

Fig. 1 shows a displacement ellipsoid diagram of (I) with the atomic numbering scheme. Selected geometric parameters are given in Table 1. The bond lengths in the pyrrolidine ring are slightly longer than normal values but are comparable with those in previously reported structures (Seshadri *et al.*, 2003; Gzella & Wrzeciono, 1990). This may be due to steric forces of bulky substituents on the pyrrolizidine moiety.

The keto group atoms O22 and O30 deviate from the mean plane containing the ring to which they are attached by 0.207 (1) and 0.260 (1) Å, respectively. The chlorobenzoyl ring is attached to the pyrrolizidine moiety in a synclinal conformation, confirmed by the torsion angle C14—C12—C11—C4 of −49.2 (2)°.

The two five-membered rings of the pyrrolizidine moiety have envelope conformations, with Cremer & Pople (1975) q_2 and φ puckering parameters, and the smallest of Nardelli's (1983) displacement asymmetry parameters, Δ, as follows: for ring (C4/C35/N31/C20/C11): $q_2 = 0.320$ (2) Å, $\varphi = 76.8$ (3)° and ΔC_S(N31) = 0.021 (1); for ring (N31/C32–C35): q_2 = 0.339 (2) Å, φ = 11.4 (4)° and ΔC_2(C34) = 0.024 (1). Ring *D* (C20–C23/C28/C29) adopts an envelope conformation, which

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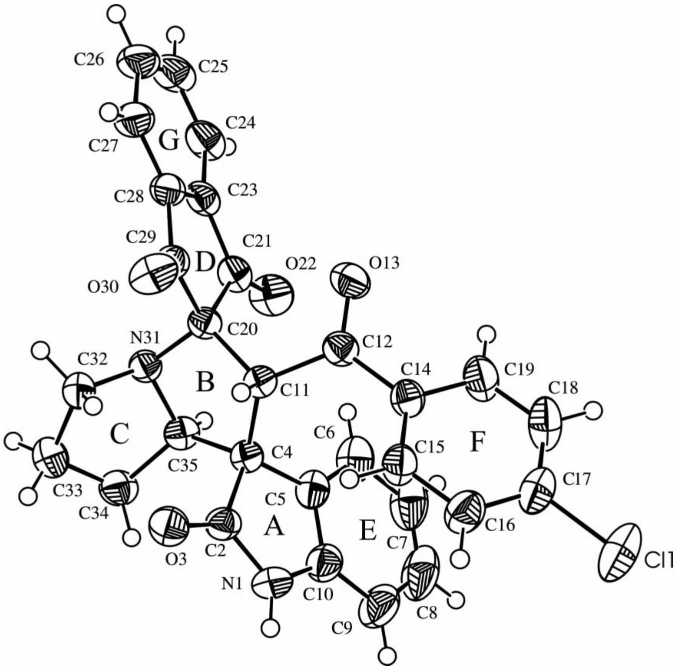

Figure 1
View of (I) (50% probability displacement ellipsoids).

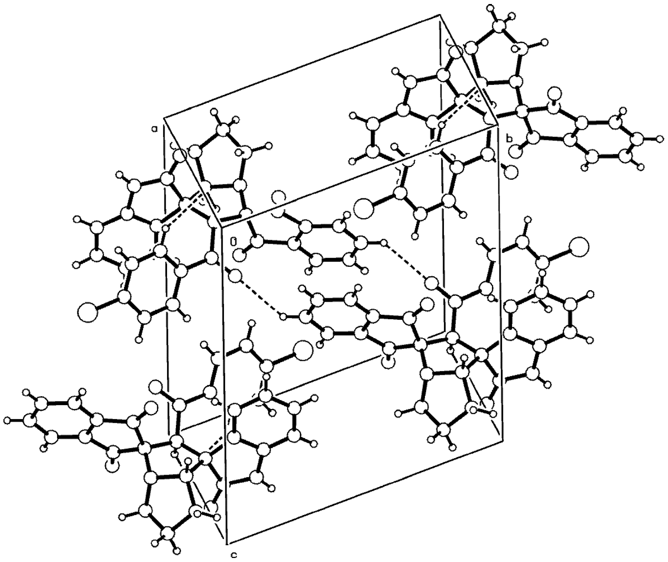

Figure 2
Packing diagram showing the C—H$\cdots$O hydrogen-bonded dimers.

is evident from the puckering parameters $q_2 = 0.145$ (2) Å, $\varphi = 5.7$ (8)° and asymmetry parameter $\Delta C_S[\mathrm{C}20] = 0.012$ (1).

In addition to van der Waals interactions, the crystal structure is stabilized by C—H$\cdots$O intra- and intermolecular hydrogen bonds. A dimer is formed between symmetry-related molecules through C—H$\cdots$O hydrogen bonds. Details of these interactions are given in Table 2.

Experimental

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

Crystal data

$C_{29}H_{21}ClN_2O_4$
$M_r = 496.93$
Triclinic, $P\overline{1}$
$a = 8.0057$ (6) Å
$b = 11.7864$ (9) Å
$c = 13.6991$ (11) Å
$\alpha = 109.164$ (1)°
$\beta = 102.190$ (1)°
$\gamma = 96.484$ (1)°
$V = 1170.0$ (2) Å^3
$Z = 2$
$D_x = 1.411$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2484 reflections
$\theta = 2.7$–27.4°
$\mu = 0.20$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.40 × 0.30 × 0.20 mm

Data collection

Bruker SMART APEX area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{\min} = 0.923$, $T_{\max} = 0.960$
7458 measured reflections
5223 independent reflections
3895 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.013$
$\theta_{\max} = 28.2°$
$h = -10 \rightarrow 7$
$k = -15 \rightarrow 15$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.144$
$S = 1.01$
5223 reflections
325 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0778P)^2 + 0.2186P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.33$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C17	1.731 (2)	C20—N31	1.458 (2)
N1—C2	1.353 (2)	C21—O22	1.195 (2)
N1—C10	1.390 (3)	C29—O30	1.205 (2)
C2—O3	1.203 (2)	N31—C35	1.458 (2)
C4—C11	1.560 (2)	N31—C32	1.463 (2)
C4—C35	1.572 (2)	C33—C34	1.525 (3)
C12—O13	1.208 (2)	C34—C35	1.525 (3)
C2—N1—C10	112.0 (2)	C16—C17—Cl1	118.9 (2)
O3—C2—N1	125.1 (2)	N31—C20—C29	112.0 (1)
O3—C2—C4	127.0 (2)	N31—C20—C11	106.8 (1)
N1—C2—C4	107.9 (2)	N31—C20—C21	102.7 (1)
C5—C6—C7	118.0 (2)	O22—C21—C23	126.7 (2)
C8—C7—C6	120.9 (2)	O22—C21—C20	126.3 (2)
C10—C9—C8	117.4 (3)	C20—N31—C35	105.7 (1)
C9—C10—C5	121.8 (2)	C20—N31—C32	117.7 (1)
C9—C10—N1	128.8 (2)	C35—N31—C32	107.4 (1)
C5—C10—N1	109.3 (2)	N31—C32—C33	103.2 (2)
O13—C12—C14	120.0 (2)	N31—C35—C34	104.8 (2)
O13—C12—C11	118.9 (2)	N31—C35—C4	107.5 (1)
C18—C17—Cl1	119.6 (2)		
C4—C11—C12—C14	−49.2 (2)		

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

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
C6—H6$\cdots$O22	0.93	2.56	3.371 (3)	145
C11—H11$\cdots$O3	0.98	2.58	3.042 (2)	109
C11—H11$\cdots$O30	0.98	2.55	2.977 (2)	106
C32—H32*B*$\cdots$O3	0.97	2.47	3.107 (3)	123
N1—H1$\cdots$O30$^{\rm i}$	0.86	2.37	3.104 (2)	144
C11—H11$\cdots$O3$^{\rm i}$	0.98	2.35	3.288 (2)	160
C15—H15$\cdots$O3$^{\rm i}$	0.93	2.46	3.252 (3)	144
C26—H26$\cdots$O13$^{\rm ii}$	0.93	2.52	3.260 (3)	136

Symmetry codes: (i) $1 - x, 2 - y, -z$; (ii) $1 - x, 3 - y, 1 - z$.

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All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and 1.2 $U_{eq}(C)$ 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: *ORTEP*-3 (Farrugia, 1997) and *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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