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1-(2-Chlorophenyl)-1*H*-indan-2-spiro-2′-pyrrolizidine-3′-spiro-3″-1*H*-indole-1,3,2″(2*H*,3″*H*)-trione

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The title compound, $C_{28}H_{21}ClN_2O_3$, crystallizes in the centrosymmetric space group $P2_1/n$ with one molecule in the asymmetric unit. The pyrrolidine rings of pyrrolizidine adopt envelope conformations. The structure is stabilized by intramolecular C—H···O and C—H···Cl interactions. The packing is stabilized by C—H···O and N—H···O intermolecular interactions.

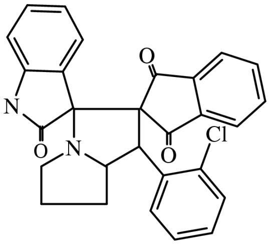
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.058
wR factor = 0.178
Data-to-parameter ratio = 17.5

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

Comment

The spiro (indole–pyrrolidine) ring system is encountered in many pharmacologically important alkaloids (Cordel, 1981; Bindra, 1973). They exhibit antimicrobial and antifungal activities, as reported in the literature (Amal Raj *et al.*, 2003). Owing to the medicinal importance of these compounds and as a continuation of our studies, an X-ray analysis of the title compound, (I), was carried out and the results are reported 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 bond lengths in the pyrrolizidine moiety are slightly longer than the values reported for similar structures (Seshadri *et al.*, 2003; Jeyabharathi *et al.*, 2001). This may be due to steric forces caused by the bulky substituents on the pyrrolizidine moiety. The sum of angles at N10 of the pyrrolizidine ring (336.8°) is in accordance with sp^3 hybridization (Beddoes *et al.*, 1986), and the sum of angles at N1 of the oxindole moiety (359.7°) is in accordance with sp^2 hybridization.

In the benzene ring of the indole system, the endocyclic angles at C3 and C7 are contracted to 118.1 (2) and 119.5 (2)°, respectively, while those at C2 and C5 are expanded to 122.3 (2) and 121.4 (2)°, respectively. This would appear to be a real effect caused by the fusion of the smaller pyrrole ring to the six-membered benzene ring, and the strain is taken up by angular distortion rather than by bond-length distortions (Sethu Sankar *et al.*, 2002).

Ketone atom O31 deviates by 0.128 (2) Å from the pyrrole ring of the oxindole moiety. Atom O32 is less displaced

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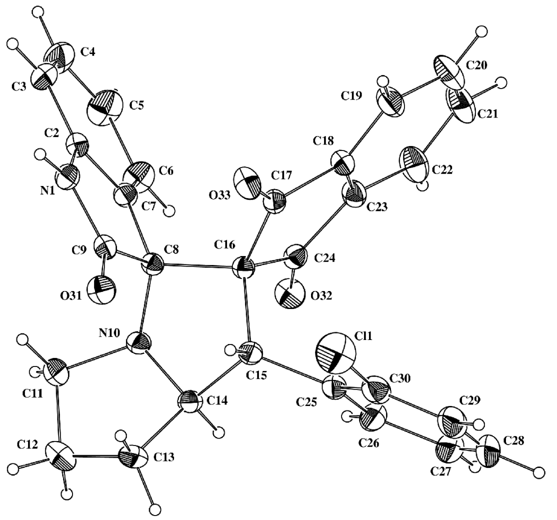

Figure 1
View of (I) (50% probability displacement ellipsoids).

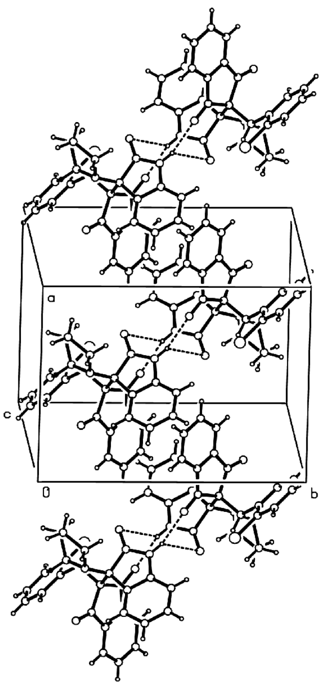

Figure 2
Packing of the molecules. Hydrogen bonds are shown as dashed lines.

[0.051 (2) Å] than O33 [0.125 (2) Å] out of the indan plane because of the different interactions in which these two keto O atoms are involved (Table 2).

The two five-membered rings of the pyrrolizidine moiety have envelope conformations, with the Cremer & Pople (1975) q_2 and φ puckering parameters, and the smallest Nardelli (1983) displacement asymmetry parameters, Δ, as follows: for ring N10/C8/C16/C15/C14: q_2 = 0.426 (2) Å, φ = −121.0 (3)°, Δ_2(C14) = 0.018 (1) and Δ_S(C16) = 0.079 (1); for ring N10/C11–C14: q_2 = 0.356 (3) Å, φ = 97.2 (3)°, Δ_2(N10) = 0.026 (1) and Δ_S(C13) = 0.053 (1).

The molecular structure is influenced by C—H···O and C—H···Cl intramolecular interactions. In the crystal structure, N1—H1···O31^{i} hydrogen bonds link inversion-related molecules to form dimers (Fig. 2 and Table 2). In addition, symmetry-related molecules are linked by weak C—H···π intermolecular interactions, as shown in Table 2.

Experimental

A mixture of *o*-chlorobenzylidene-1,3-indandione, isatin and proline was refluxed in aqueous methanol. The resulting crude product was filtered off and recrystallized from methanol to obtain the title compound.

Crystal data

$C_{28}H_{21}ClN_2O_3$
M_r = 468.92
Monoclinic, $P2_1/n$
a = 10.8456 (6) Å
b = 14.1502 (8) Å
c = 15.2068 (9) Å
β = 92.053 (1)°
V = 2332.3 (2) Å^3
Z = 4

D_x = 1.335 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4187 reflections
θ = 2.3–24.3°
μ = 0.20 mm^{-1}
T = 293 (2) K
Block, pale yellow
0.18 × 0.17 × 0.11 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
14407 measured reflections
5387 independent reflections

3882 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.023
$\theta_{\rm max}$ = 28.0°
$h = -8 \rightarrow 14$
$k = -18 \rightarrow 18$
$l = -20 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.178
S = 1.03
5387 reflections
307 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1051P)^2 + 0.4748P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.48 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.44 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C8—N10	1.451 (2)	N10—C14	1.476 (2)
N10—C11	1.472 (3)		
C9—N1—C2	112.5 (2)	C6—C7—C2	119.5 (2)
C3—C2—C7	122.3 (2)	C8—N10—C11	117.5 (2)
C4—C3—C2	118.1 (2)	C8—N10—C14	109.9 (1)
C4—C5—C6	121.4 (2)	C11—N10—C14	109.4 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O32	0.93	2.57	3.208 (3)	127
C13—H13B···O31	0.97	2.53	3.190 (3)	125
C15—H15···Cl1	0.98	2.56	3.115 (2)	116
C15—H15···O31	0.98	2.43	3.045 (2)	121
C26—H26···O32	0.93	2.50	3.123 (3)	124
N1—H1···O31^{i}	0.86	2.59	3.152 (2)	124
N1—H1···O33^{i}	0.86	2.11	2.926 (2)	157
C21—H21···O31^{ii}	0.93	2.43	3.357 (3)	173
C20—H20···$C1g^{iii}$	0.93	2.59	3.508 (4)	170

Symmetry codes: (i) $2-x, -y, -z$; (ii) $x-1, y, z$; (iii) $1-x, -y, -z$. $C1g$ is the centroid of the C2–C7 ring

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

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