Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

4′-(3-Chlorophenyl)-1-methyl-1*H*-indole-3-spiro-2′-pyrrolidine-3′-spiro-2″-indan-2,1″,3″-trione

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Received 13 November 2003
Accepted 27 November 2003
Online 12 December 2003

The pyrrolidine ring of the title compound, $C_{26}H_{19}ClN_2O_3$, adopts a half-chair conformation. The molecular structure is stabilized by C—H···O interactions and the packing of the molecule is stabilized by C—H···O and N—H···O intermolecular interactions. A dimer is formed between symmetry-related molecules through N—H···O hydrogen bonds.

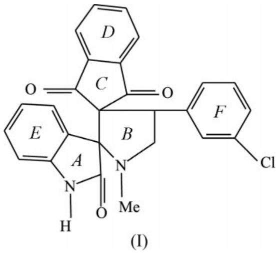
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.056
wR factor = 0.171
Data-to-parameter ratio = 16.4

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

Comment

The spiro ring system is a frequently encountered structural motif in many pharmacologically relevant alkaloids (Cordel, 1981). Several optically active pyrrolidines have been used as intermediates in controlled asymmetric synthesis (Suzuki *et al.*, 1994). In view of this 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 the molecule with the atomic numbering scheme. Selected geometric parameters are given in Table 1. The bond lengths correlate with a variety of *N*-phenyl-substituted pyrrolidin-2-one systems (Billing *et al.*, 1991). The bond lengths of the pyrrolidine moiety (Table 1) differ slightly from normal values but are comparable with those of previously reported structures (Gzella & Wrzeciono, 1990). This may be due to steric forces of bulky substituents on the pyrrolidine ring.

The total puckering amplitudes (Cremer & Pople, 1975) of the rings A, B, C give a quantitative evaluation of puckering and asymmetry parameters. Ring A is in a half-chair conformation, with lowest asymmetry parameter (Nardelli, 1983) ΔC_2[C10] = 0.0047 (8) and puckering parameters q_2 = 0.0987 (9) Å and $\varphi = -120.7$ (1)°. The pyrrolidine ring B is in a half-chair conformation, with puckering parameters q_2 = 0.407 (2) Å and φ = 16.1 (3)° and asymmetry parameter ΔC_2[C13] = 0.0116 (9). The indandione ring C is in an envelope conformation, as evidenced from puckering parameters q_2 = 0.1291 (9) Å and φ = 7.29 (8)°, and asymmetry parameter Δ_S[C11] = 0.013 (1), with atom C11 deviating by 0.082 (2) Å from the least-squares plane through the remaining atoms.

DOI: 10.1107/S1600536803027302

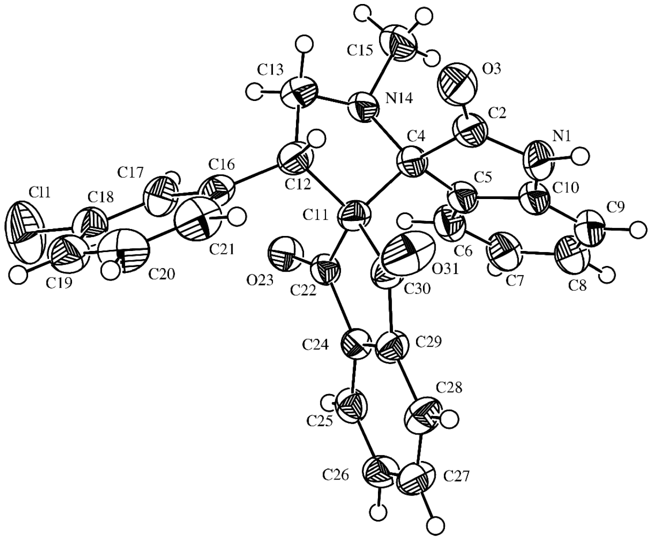


Figure 1
View of (I) (50% probability displacement ellipsoids).

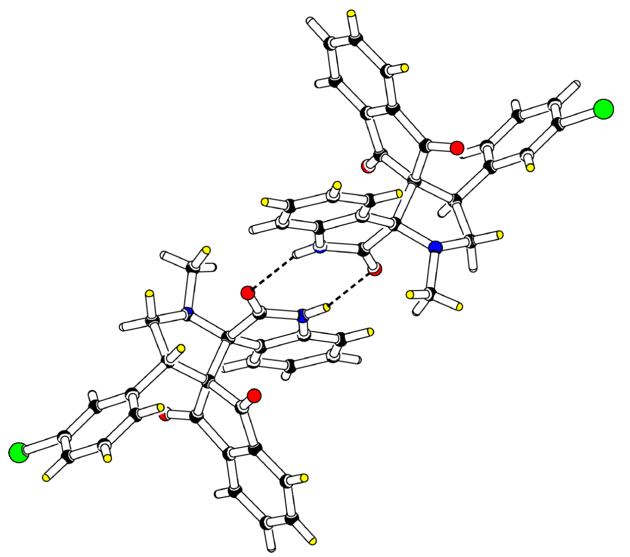


Figure 2
View of (I) illustrating the N—H$\cdots$O hydrogen-bonded dimers.

In the benzene ring of the oxindole moiety, the endocyclic angles at C9 and C6 are 118.1 (2) and 118.9 (2)°, respectively. At C10 and C7, the angles are 121.3 (2) and 120.7 (2)°, respectively. The deviations 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 reported structures (Sethusankar *et al.*, 2002). The chlorophenyl ring is attached in an equatorial position to the pyrrolidine ring.

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

Experimental

A mixture of *O*-chlorobenzylidene-1,3-indandione, isatin and sarcosine was refluxed in aqueous methanol. The resulting crude product was filtered and recrystallized from methanol.

Crystal data

$C_{26}H_{19}ClN_2O_3$
$M_r = 442.88$
Triclinic, $P\overline{1}$
$a = 7.8394$ (6) Å
$b = 11.6760$ (9) Å
$c = 11.8628$ (10) Å
$\alpha = 85.657$ (1)°
$\beta = 89.752$ (1)°
$\gamma = 83.726$ (2)°
$V = 1076.22$ (15) Å^3
$Z = 2$
$D_x = 1.367$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2195 reflections
$\theta = 2.4$–26.9°
$\mu = 0.21$ mm^{-1}
$T = 293$ (2) K
Needle, yellow
0.18 × 0.16 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{min} = 0.954$, $T_{max} = 0.970$
6811 measured reflections
4731 independent reflections
3499 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 28.0$°
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 15$
$l = -15 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.172$
$S = 1.09$
4731 reflections
289 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C18	1.735 (2)	C4—N14	1.452 (2)
N1—C2	1.356 (3)	N14—C15	1.450 (3)
N1—C10	1.402 (3)	C22—O23	1.202 (2)
C2—O3	1.221 (3)	C30—O31	1.205 (2)
C2—N1—C10	111.9 (2)	C9—C10—N1	128.7 (2)
O3—C2—N1	126.0 (2)	N14—C13—C12	106.1 (2)
O3—C2—C4	126.7 (2)	C4—N14—C15	116.1 (2)
N1—C2—C4	107.3 (2)	C4—N14—C13	109.9 (2)
N14—C4—C5	113.9 (2)	C15—N14—C13	115.2 (2)
N14—C4—C2	115.6 (2)	C17—C18—Cl1	119.5 (2)
N14—C4—C11	101.4 (2)	C19—C18—Cl1	118.5 (2)
C5—C6—C7	118.9 (2)	O23—C22—C24	126.1 (2)
C8—C7—C6	120.7 (2)	O23—C22—C11	126.3 (2)
C10—C9—C8	118.1 (2)	O31—C30—C11	125.5 (2)
C5—C10—C9	121.3 (2)	C29—C30—C11	108.0 (2)
C5—C10—N1	109.9 (2)		
C12—C16—C17—C18	−179.5 (2)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C12—H12$\cdots$O3	0.98	2.59	3.154 (3)	117
N1—H1$\cdots$O3^{i}	0.86	2.09	2.920 (2)	163
C15—H15$A\cdots$O31^{ii}	0.96	2.57	3.452 (3)	152

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

All H atoms were geometrically positioned 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 $1.2U_{eq}(C)$ for other H atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); 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).

SSN and DV thank the University Grants Commission (UGC), New Delhi, for financial support under the University With Potential For Excellence Programme.

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