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4′-(4-Chlorophenyl)-1′-methylcycloheptane-1-spiro-3′-pyrrolidine-2′-spiro-2″-indan-2,1″,3″-trione

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In the title compound, $C_{25}H_{24}ClNO_3$, the indandione group is planar and the pyrrolidine ring adopts a twist conformation. The molecules associate *via* C—H···O interactions to form $R_2^2(10)$ and $R_2^2(14)$ dimers.

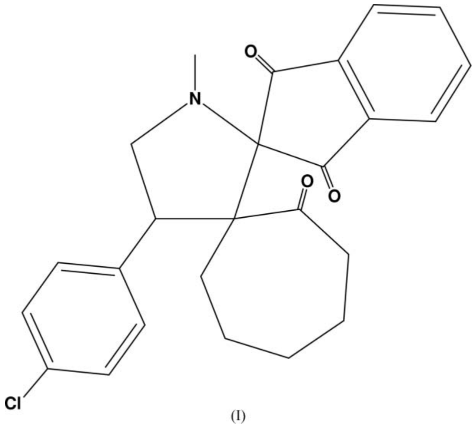
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.061
wR factor = 0.160
Data-to-parameter ratio = 18.6

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

Comment

Highly substituted pyrrolidines have attracted much interest in the past few years, since they constitute the main structural element of many alkaloids and pharmacologically active compounds (Waldmann, 1995). 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)

Bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987). The sum of the angles at N1 of the pyrrolidine ring (339.3°) is in accordance with sp^3-hybridization.

The indandione group is planar, with a maximum deviation of 0.032 (2) Å for atom C18. The keto atoms O2 and O3 deviate from the plane of the five-membered ring by 0.214 (1) and 0.172 (1) Å, respectively.

The pyrrolidine ring adopts a twist conformation (Fig. 1), with a pseudo-twofold axis passing through atom C1 and the C3—C4 bond; the puckering parameters (Cremer & Pople, 1975) and the smallest displacement asymmetry parameters (Nardelli, 1983) are $q_2 = 0.452$ (2) Å, $\varphi = 306.8$ (2)° and Δ_s(C1) = 4.6 (2)°. The seven-membered ring adopts a twist-boat conformation. The indandione group makes a dihedral angle of 37.68 (8)° with the chlorophenyl ring.

The molecular structure is stabilized by a weak C2—H2···O2 interaction (Table 1). In the crystal structure, the

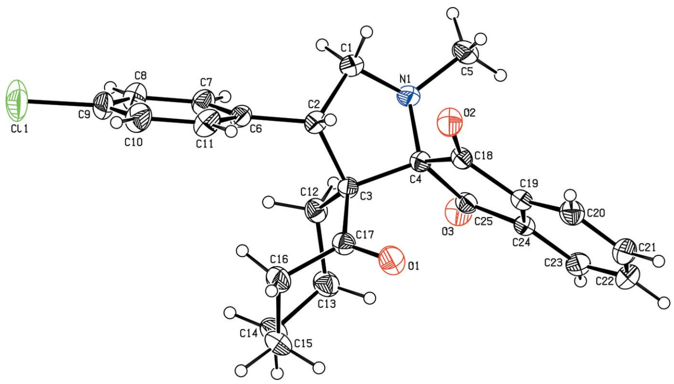


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids.

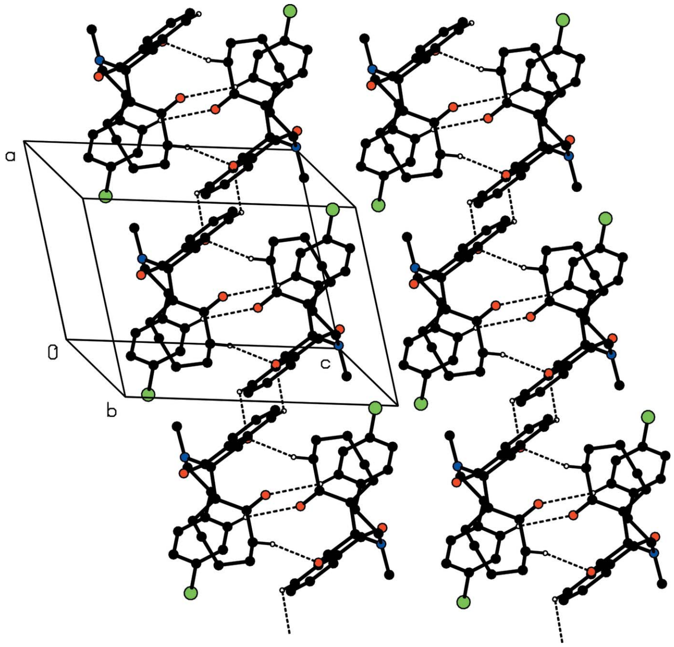


Figure 2
The molecular packing of (I), viewed approximately down the *b* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

C11—H11···O1^{i}, C16—H16*B*···O2^{i} and C20—H20···O2^{ii} hydrogen-bonding interactions (symmetry codes are given in Table 2) form $R_2^2(14)$, $R_2^2(14)$ and $R_2^2(10)$ centrosymmetric dimers, respectively. The molecules are linked into a chain along the *a* axis by C—H···π interactions involving the C19–C24 benzene ring (centroid *Cg*1).

Experimental

A mixture of ninhydrin (0.178 g, 1 mmol), sarcosine (0.089 g, 1 mmol) and *p*-chlorobenzylidenecycloheptanone (1 mmol) in methanol (20 ml) was refluxed until the disappearance of the starting materials as shown by thin-layer chromatography. The reaction mixture was then concentrated *in vacuo* and extracted with water (50 ml) and dichloromethane (50 ml). The organic layer was washed with brine, dried with anhydrous sodium sulfate and concentrated *in vacuo*. The residue was purified by column chromatography (silica gel, 100–200 mesh) eluted with a hexane–ethyl acetate (8:2) mixture to give the title compound, which was recrystallized from methanol by slow evaporation.

Crystal data

$C_{25}H_{24}ClNO_3$
$M_r = 421.90$
Monoclinic, $P2_1/n$
$a = 9.3282$ (10) Å
$b = 19.025$ (2) Å
$c = 12.5249$ (14) Å
$\beta = 105.101$ (2)°
$V = 2146.0$ (4) Å^3
$Z = 4$
Mo *Kα* radiation
$\mu = 0.21$ mm^{-1}
$T = 293$ (2) K
0.26 × 0.23 × 0.22 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
24440 measured reflections
5070 independent reflections
4196 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.029$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.160$
$S = 1.09$
5070 reflections
272 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max} = 0.38$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.24$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of the C19–C24 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O2	0.98	2.45	3.049 (2)	119
C11—H11···O1^{i}	0.93	2.57	3.471 (2)	162
C16—H16*B*···O2^{i}	0.97	2.49	3.436 (3)	165
C20—H20···O2^{ii}	0.93	2.53	3.152 (2)	124
C15—H15*B*···*Cg*1^{iii}	0.97	2.70	3.596 (2)	155

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

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$U_{\rm eq}$(methyl C) or 1.2$U_{\rm eq}$(C).

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: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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