organic papers

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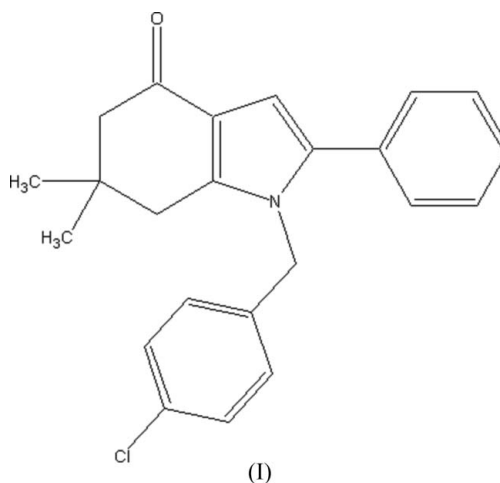
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1-(4-Chlorobenzyl)-6,6-dimethyl-2-phenyl-1,5,6,7-tetrahydro-4H-indol-4-one

In the title compound, $C_{23}H_{22}ClNO$, there are two independent molecules showing similar conformations, the tetrahydroindole ring system being approximately planar except for the dimethyl-substituted C atom. Molecules are linked *via* C—H···O, C—H···π and C—H···Cl interactions, forming a sheet-like structure.

Comment

The previous paper (Chopra *et al.*, 2005) describes the background to this study. Unlike the compound reported in that paper, the presence of chlorine in the title compound, (I), generates C—H···Cl interactions contributing to a change in the packing mode.

(I)

In compound (I), there are two independent molecules (*A* and *B*) in the asymmetric unit (Fig. 1). In the tetrahydroindole ring system in molecule *A*, atom C5 deviates by 0.621 (4) Å from the C6–C8/C3/C4 mean plane, whereas in molecule *B*, atom C28 deviates 0.601 (4) Å from the C29–C31/C26/C27 mean plane. Cremer & Pople (1975) analysis for the six-membered ring of molecule *A* reveals the puckering parameters as $Q(2) = 0.350$ (4) Å, $\varphi(2) = 116.1$ (8)°, $Q(3) = 0.273$ (5) Å, $Q = 0.444$ (5) Å and $\theta = 52.1$ (5)°, and in molecule *B* these values are $Q(2) = 0.341$ (4) Å, $\varphi(2) = 118.3$ (7)°, $Q(3) = 0.265$ (4) Å, $Q = 0.431$ (5) Å and $\theta = 52.1$ (5)°. The conformation of molecule *B* is stabilized by an intramolecular C—H···π interaction (Fig. 1 and Table 2). A C—H···O intermolecular hydrogen bond involving H9*A* holds the two molecules in the asymmetric unit together. Intermolecular C—H···π interactions involving atoms H4*B* and H27*B* form molecular dimers and such dimers are held together by C—H···O interactions involving atom H34, forming molecular
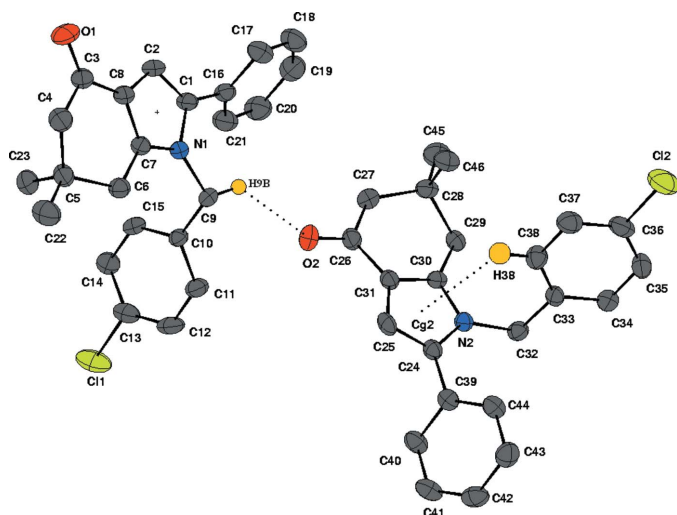
Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids. Dotted lines indicate C—H···O and C—H···π interactions. H atoms have been omitted unless they are involved in hydrogen bonding.
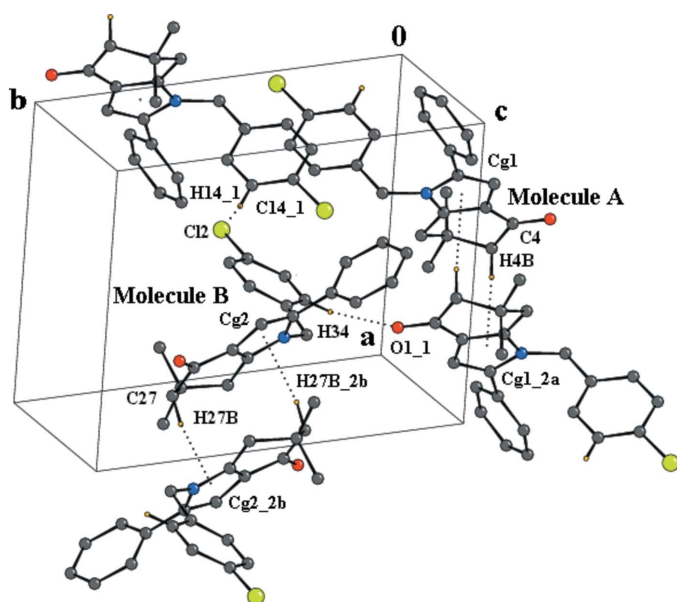

Figure 2
Packing of molecules in (I). Dotted lines indicate C—H···O, C—H···π and C—H···Cl interactions. H atoms have been omitted unless they are involved in hydrogen bonding. [Symmetry codes: ($_1$) $x + 1, y, z - 1$; ($_{2a}$) $-x + 1, -y + 2, -z$; ($_2b$) -x, -y+1, -z+1.]

chains along the b axis; $Cg1$ and $Cg2$ in Table 2 are the centroids of the indole rings N1/C1/C2/C8/C7 and N2/C24/C25/C31/C30, respectively. In addition, intermolecular C—H···Cl interactions (Table 2) involving atom H14 (molecule A) and the chlorine atom Cl2 (molecule B) further hold the molecules together, forming chains along the c axis, leading to the formation of a sheet-like structure (Fig. 2).

Experimental

Compound (I) was synthesized according to the procedure reported in the literature (Nagarajan *et al.*, 1985). Crystals were obtained from a solution of dichloromethane and hexane (1:2) by slow evaporation at 278 K.

Crystal data

$C_{23}H_{22}ClNO$	$Z = 4$
$M_r = 363.87$	$D_x = 1.251$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 9.7761$ (14) Å	Cell parameters from 636
$b = 13.0082$ (19) Å	reflections
$c = 15.832$ (2) Å	$\theta = 1.4$–$26.4°$
$\alpha = 75.900$ (3)°	$\mu = 0.21$ mm^{-1}
$\beta = 87.872$ (3)°	$T = 290$ (2) K
$\gamma = 81.639$ (3)°	Plate, colourless
$V = 1931.9$ (5) Å^3	$0.21 \times 0.06 \times 0.02$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	7488 independent reflections
	3904 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.042$
Absorption correction: multi-scan	$\theta_{max} = 26.4°$
($SADABS$; Sheldrick, 1996)	$h = -12 \rightarrow 11$
$T_{min} = 0.946$, $T_{max} = 0.996$	$k = -16 \rightarrow 15$
15035 measured reflections	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.088$	$w = 1/[\sigma^2(F_o^2) + (0.0861P)^2]$
$wR(F^2) = 0.200$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{max} < 0.001$
7488 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
473 parameters	$\Delta\rho_{min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C13	1.741 (4)	N2—C24	1.400 (4)
Cl2—C36	1.743 (4)	N2—C30	1.365 (4)
N1—C1	1.398 (4)	N2—C32	1.450 (4)
N1—C7	1.360 (4)	O1—C3	1.221 (4)
N1—C9	1.455 (4)	O2—C26	1.224 (4)
C10—C9—N1—C7	74.5 (4)	C29—C30—C31—C26	−1.7 (6)
N1—C9—C10—C15	17.3 (5)	C44—C39—C24—N2	54.7 (5)
C8—C7—C6—C5	25.2 (5)	C31—C30—C29—C28	25.1 (5)
C6—C7—C8—C3	−1.5 (6)	C30—N2—C32—C33	73.9 (4)
C7—C6—C5—C4	−48.4 (4)	C38—C33—C32—N2	−3.6 (5)
C7—C8—C3—C4	3.4 (6)	C30—C31—C26—C27	2.3 (5)
C8—C3—C4—C5	−30.4 (6)	C28—C27—C26—C31	−27.9 (5)
C6—C5—C4—C3	53.6 (5)	C26—C27—C28—C29	50.7 (5)
C21—C16—C1—N1	55.1 (5)	C30—C29—C28—C27	−46.6 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9B···O2^i	0.97	2.51	3.276 (4)	136
C34—H34···O1^{ii}	0.93	2.45	3.245 (5)	143
C4—H4B···Cg1^{iii}	0.97	2.86	3.804 (5)	163
C27—H27B···Cg2^{iv}	0.97	2.73	3.653 (4)	159
C38—H38···Cg2^i	0.93	2.75	3.409 (5)	128
C14—H14···Cl2^v	0.93	2.86	3.637 (5)	143

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

All H atoms were positioned geometrically and allowed to ride on the parent C atoms, with C—H = 0.93–0.97 Å and with U_{iso}(H) =

$1.2U_{eq}(C)$ $[1.5U_{eq}(C_{Me})]$. The methyl groups were allowed to rotate freely about the C—C bond.

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 2003).

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