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1-(4-Chlorophenacyl)-4-methyl-6,7,8,9-tetrahydro-5*H*-cyclohepta[*b*]pyridin-2(1*H*)-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.041
wR factor = 0.106
Data-to-parameter ratio = 14.5

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

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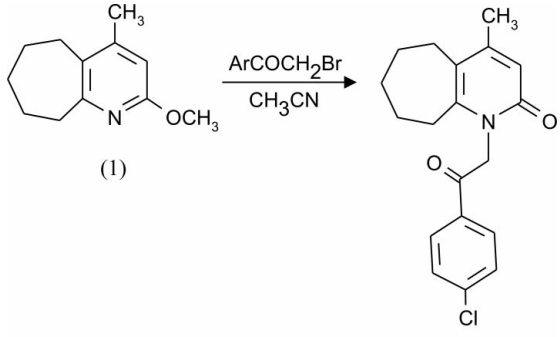
In the pyridone ring of the title compound, $C_{19}H_{20}ClNO_2$, single and double bonds alternate, though allowing some degree of conjugation. One C atom in the cycloheptene ring is disordered over two positions, which form boat and chair conformations of cycloheptene, respectively.

Comment

In the course of our systematic study of the size effect of cycloalkane fragments on the reactivity of pyridine-based heterocycles (Albov, Rybakov, Babaev & Aslanov, 2004), we have described earlier the crystal structure of 2-methoxy-4-methyl-6,7,8,9-tetrahydro-5*H*-cyclohepta[*b*]pyridine, (1) (Albov, Rybakov, Babaev, Fedyanin & Aslanov, 2004). We report here the crystal structure of the title compound, (2) (Fig. 1).



(1) (2)

In the pyridone ring (N1/C11) of (2), the single and double bonds alternate (Table 1), though allowing some degree of conjugation. Atoms C7 and C9 are displaced from the plane of the pyridone ring by 1.412 (4) and 1.322 (3) Å, respectively. Atom C8 of the cycloheptene ring is disordered over two sites, with occupancies of 0.69 (1) and 0.31 (1), forming the boat and chair conformations of cycloheptene, respectively. The torsion angle C20—C15—C14—O14 is 24.4 (2)° and the dihedral angle between the benzene and pyridone rings is 49.88 (6)°.

Experimental

Compound (1) (2.50 g) and 4-chlorophenacyl bromide (3.06 g) were boiled in acetonitrile for 6 h. When thin-layer chromatography showed only traces of the source compounds in the solution, the solvent was evaporated and the product was washed with acetone (yield 2.51 g, 58%). The product was recrystallized from acetone

(m.p. 481–483 K). ^{1}H NMR (DMSO-d_6, 400 MHz, p.p.m.): 1.55 (*m*, 4H, 7-CH_2 + 8-CH_2), 1.78 (*m*, 2H, 9-CH_2), 2.65 (*m*, 4H, 6-CH_2 + 10-CH_2), 5.66 (*s*, 2H, 13-CH_2), 6.10 (*s*, 1H, 3-CH), 7.56, 8.08 (*dd*, 4-H, Ar).

Crystal data

$C_{19}H_{20}ClNO_2$	$Z = 2$
$M_r = 329.81$	$D_x = 1.356$ Mg m^{-3}
Triclinic, $P\overline{1}$	Cu $K\alpha$ radiation
$a = 7.9540$ (7) Å	Cell parameters from 25 reflections
$b = 8.6902$ (7) Å	$\theta = 30$–35°
$c = 12.4984$ (8) Å	$\mu = 2.16$ mm^{-1}
$\alpha = 108.342$ (6)°	$T = 293$ (2) K
$\beta = 94.660$ (6)°	Prism, colourless
$\gamma = 96.760$ (7)°	0.30 × 0.30 × 0.30 mm
$V = 807.88$ (11) Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	$\theta_{max} = 74.7^\circ$
Non-profiled ω scans	$h = -9 \rightarrow 9$
Absorption correction: none	$k = -10 \rightarrow 10$
3173 measured reflections	$l = 0 \rightarrow 15$
3173 independent reflections	1 standard reflection
2843 reflections with $I > 2\sigma(I)$	every 200 reflections
	intensity decay: 1%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 0.3555P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.041$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.106$	$\Delta\rho_{max} = 0.17$ e Å^{-3}
$S = 1.06$	$\Delta\rho_{min} = -0.20$ e Å^{-3}
3173 reflections	
219 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Cl1—C18	1.7414 (16)	C6—C7	1.538 (3)
N1—C11	1.3747 (19)	C7—C8*B*	1.485 (9)
N1—C2	1.399 (2)	C7—C8*A*	1.511 (5)
N1—C13	1.4641 (17)	C8*A*—C9	1.434 (4)
C2—O2	1.241 (2)	C8*B*—C9	1.461 (8)
C2—C3	1.431 (2)	C9—C10	1.542 (3)
C3—C4	1.357 (2)	C10—C11	1.509 (2)
C4—C5	1.426 (2)	C13—C14	1.5150 (19)
C4—C12	1.509 (2)	C14—O14	1.2122 (18)
C5—C11	1.3730 (19)	C14—C15	1.490 (2)
C5—C6	1.507 (2)		
C11—N1—C2	122.99 (12)	C11—C5—C6	118.81 (15)
C11—N1—C13	121.37 (12)	C4—C5—C6	122.58 (14)
C2—N1—C13	115.56 (13)	C8B—C9—C10	120.8 (3)
O2—C2—N1	120.49 (14)	C5—C11—N1	120.98 (13)
O2—C2—C3	124.82 (15)	C5—C11—C10	119.17 (14)
N1—C2—C3	114.69 (14)	N1—C11—C10	119.85 (13)
C4—C3—C2	123.28 (15)	N1—C13—C14	111.69 (12)
C3—C4—C5	119.42 (13)	O14—C14—C15	121.28 (13)
C3—C4—C12	119.76 (16)	O14—C14—C13	120.76 (13)
C5—C4—C12	120.82 (16)	C15—C14—C13	117.95 (12)
C11—C5—C4	118.58 (14)		

All H atoms were positioned geometrically and refined as riding (C—H = 0.93–0.97 Å), with U_{iso}(H) = 1.2 or 1.5U_{eq}(C).

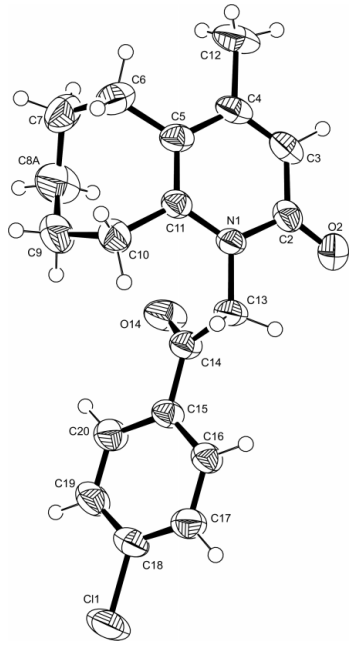


Figure 1
ORTEP-3 (Farrugia, 1997) view of (2), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Only the major component of disordered atom C8, namely C8*A*, is shown.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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