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1-(4-Chlorophenacyl)-4-methyl-5,6,7,8,9,10-hexahydrocycloocta[*b*]pyridin-2(1*H*)-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.050
wR factor = 0.137
Data-to-parameter ratio = 17.4

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

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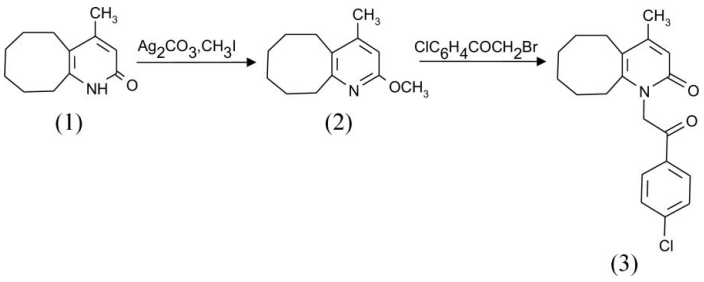
In the pyridone ring of the title compound, $C_{20}H_{22}ClNO_2$, single and double bonds alternate, though allowing some degree of delocalization. Two C atoms in the cyclooctene ring are disordered, indicating some flexibility of the large ring.

Comment

In the course of our systematic study of the size effect of cycloalkane fragments on the reactivity of pyridine-based heterocycles, we have previously described the crystal structure of 4-methyl-5,6,7,8,9,10-hexahydrocycloocta[*b*]pyridin-2(1*H*)-one, (1), (Albov, Mazina *et al.*, 2004). Following our investigations of cycloheptene derivatives (Albov, Rybakov, Babaev, Fedyanin & Aslanov, 2004; Albov, Rybakov, Babaev & Aslanov, 2004), we have now synthesized the title compound, (3) (Fig. 1).



(1) (2) (3)

In the pyridone ring of (3), the single and double bonds alternate (Table 1), though allowing some degree of delocalization. Atoms C8 and C9 of the cyclooctene ring are disordered over two sites each, forming two conformations with occupancies of 0.719 (7) and 0.281 (7), respectively. The torsion angle C21—C16—C15—O15 is 25.6 (3)°, the same as in a related cycloheptene derivative (Albov, Rybakov, Babaev & Aslanov, 2004). The dihedral angle between the benzene and pyridone rings is 57.96 (8)°.

Experimental

For the preparation of 2-methoxy-4-methyl-5,6,7,8,9,10-hexahydrocycloocta[*b*]pyridine, (2), compound (1) (8.30 g), methyl iodide (7.78 g) and silver carbonate (6.00 g) were boiled in 70 ml of benzene for 50 h. The reaction flask was protected against light. The mixture was then filtered and the solvent was evaporated (yield 5.04 g, 57%). The product was recrystallized from chloroform (m.p. 313–314 K). ^{1}H NMR (DMSO-d_6, 400 MHz, p.p.m.): 1.33 (*m*, 2H, 8-CH_2), 1.41 (*m*, 2H, 9-CH_2), 1.60 (*m*, 2H, 7-CH_2), 1.70 (*m*, 2H, 6-CH_2), 2.25 (*s*, 3H, 13-CH_3), 2.71 (*t*, 2H, 6-CH_2), 2.80 (*t*, 2H, 11-CH_2), 3.60 (*s*, 3H, O-CH_3), 6.31 (*s*, 1H, 3-CH). Atoms are numbered as in the title compound (Fig. 1).

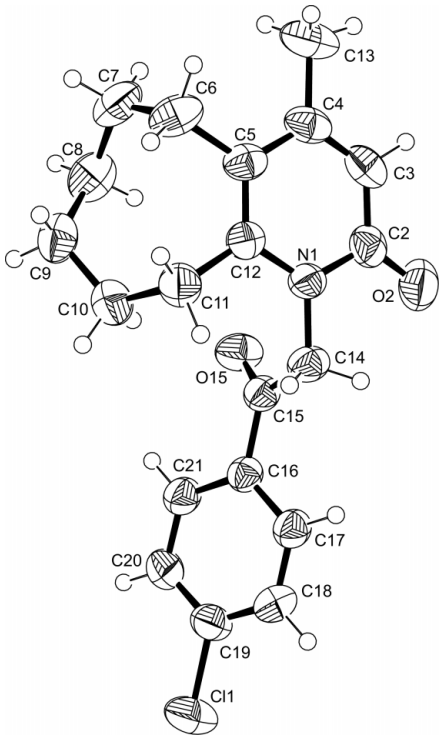


Figure 1
ORTEP-3 view (Farrugia, 1997) of (3), with the atom-numbering scheme. Atomic displacement ellipsoids are drawn at the 50% probability level. Only the major components of disordered atoms C8 and C9, namely C8*A* and C9*A*, are shown.

For the preparation of 1-(4-chlorophenacyl)-4-methyl-5,6,7,8,9,10-hexahydrocycloocta[*b*]pyridin-2(1*H*)- one, (3), compound (2) (5.00 g) and 4-chlorophenacyl bromide (5.70 g) were boiled in acetonitrile for 6 h. When thin-layer chromatography showed only traces of source compounds in the solution, the solvent was evaporated and the product was washed with acetone (yield 3.54 g, 42%). The product was recrystallized from acetone (m.p. 453–455 K).[1]H NMR (DMSO-d_6, 400 MHz, p.p.m.): 1.45 (*m*, 4H, 8-CH_2 + 9-CH_2), 1.62 (*m*, 4H, 7-CH_2 + 10-CH_2), 1.70 (*m*, 2H, 6-CH_2), 2.20 (*s*, 3H, 13-CH_3), 2.63 (*t*, 2H, 6-CH_2), 2.70 (*t*, 2H, 11-CH_2), 5.45 (*s*, 2H, 14-CH_2), 6.13 (*s*, 1H, 3-CH), 7.53, 8.08 (*dd*, 4H, Ar). Atom numbering as in Fig. 1.

Crystal data

$C_{20}H_{22}ClNO_2$	$Z = 2$
$M_r = 343.84$	$D_x = 1.332$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 8.239$ (3) Å	Cell parameters from 25 reflections
$b = 9.115$ (3) Å	$\theta = 13$–$15°$
$c = 12.42$ (1) Å	$\mu = 0.24$ mm^{-1}
$\alpha = 111.20$ (4)°	$T = 293$ (2) K
$\beta = 93.80$ (4)°	Prism, colourless
$\gamma = 96.76$ (3)°	0.22 × 0.20 × 0.18 mm
$V = 857.6$ (9) Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	$\theta_{max} = 28.0°$
Non-profiled ω scans	$h = -10 \rightarrow 10$
Absorption correction: none	$k = -12 \rightarrow 11$
4125 measured reflections	$l = 0 \rightarrow 16$
4125 independent reflections	1 standard reflection
2645 reflections with $I > 2\sigma(I)$	every 200 reflections
	intensity decay: 3%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0608P)^2 + 0.1283P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.050$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.137$	$\Delta\rho_{max} = 0.29$ e Å^{-3}
$S = 1.05$	$\Delta\rho_{min} = -0.42$ e Å^{-3}
4125 reflections	
237 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Cl1—C19	1.737 (2)	C9*A*—C10	1.510 (4)
N1—C12	1.392 (2)	C8*B*—C9*B*	1.48 (2)
N1—C2	1.396 (3)	C9*B*—C10	1.641 (10)
N1—C14	1.462 (2)	C10—C11	1.527 (3)
C2—O2	1.242 (2)	C11—C12	1.507 (3)
C2—C3	1.418 (3)	C14—C15	1.508 (3)
C3—C4	1.352 (3)	C15—O15	1.217 (2)
C4—C5	1.425 (3)	C15—C16	1.490 (3)
C4—C13	1.509 (3)	C16—C21	1.386 (2)
C5—C12	1.368 (3)	C16—C17	1.386 (2)
C5—C6	1.514 (3)	C17—C18	1.383 (2)
C6—C7	1.532 (4)	C18—C19	1.379 (3)
C7—C8*B*	1.403 (15)	C19—C20	1.376 (2)
C7—C8*A*	1.560 (6)	C20—C21	1.380 (2)
C8*A*—C9*A*	1.536 (8)		
C12—N1—C2	122.83 (16)	C9*A*—C8*A*—C7	113.6 (4)
C12—N1—C14	122.10 (16)	C10—C9*A*—C8*A*	114.3 (3)
C2—N1—C14	114.93 (16)	C7—C8*B*—C9*B*	118.7 (10)
O2—C2—N1	119.92 (19)	C8*B*—C9*B*—C10	111.3 (8)
O2—C2—C3	124.9 (2)	C9*A*—C10—C11	117.0 (2)
N1—C2—C3	115.15 (18)	C11—C10—C9*B*	111.8 (3)
C4—C3—C2	123.5 (2)	C12—C11—C10	117.23 (18)
C3—C4—C5	119.16 (18)	C5—C12—N1	119.85 (17)
C3—C4—C13	119.9 (2)	C5—C12—C11	122.01 (18)
C5—C4—C13	120.9 (2)	N1—C12—C11	118.14 (16)
C12—C5—C4	119.43 (18)	N1—C14—C15	112.31 (15)
C4—C5—C6	119.96 (18)	O15—C15—C16	121.25 (16)
C5—C6—C7	113.53 (18)	O15—C15—C14	120.99 (16)
C8*B*—C7—C6	111.3 (5)	C16—C15—C14	117.76 (15)
C6—C7—C8*A*	115.6 (2)		

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} of the parent C atom.

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* publication routines (Farrugia, 1999).

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