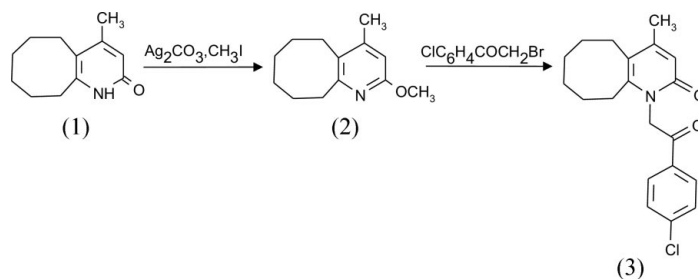


Dmitry V. Albov,* Victor B.
Rybakov, Eugene V. Babaev and
Leonid A. AslanovDepartment of Chemistry, Moscow State
University, 119992 Moscow, Russian
FederationCorrespondence e-mail:
albov@biocryst.phys.msu.su

Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
Disorder in main residue
 R factor = 0.050
 wR factor = 0.137
Data-to-parameter ratio = 17.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.1-(4-Chlorophenacyl)-4-methyl-5,6,7,8,9,10-
hexahydrocycloocta[*b*]pyridin-2(1*H*)-oneIn the pyridone ring of the title compound, $\text{C}_{20}\text{H}_{22}\text{ClNO}_2$, 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.Received 8 June 2004
Accepted 11 June 2004
Online 26 June 2004

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).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) $^\circ$, 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) $^\circ$.

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). ^1H 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).

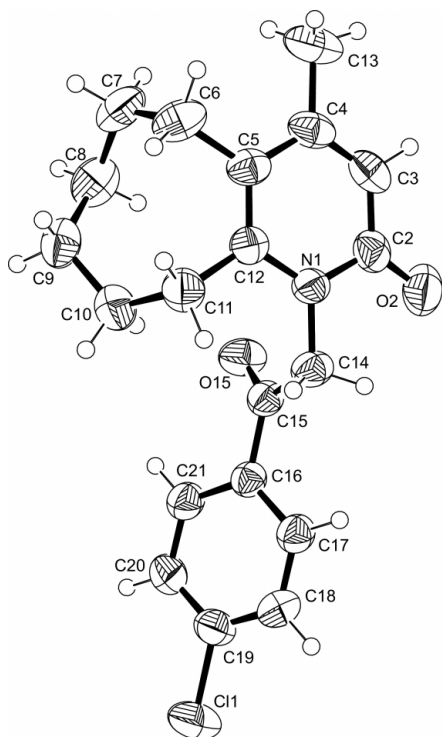


Figure 1
ORTEP-3 view (Farrugia, 1997) of (3), with the atom-numbering scheme. Atomic displacement ellipsoids are drawn at the 50% probability level. Only the major components of disordered atoms C8 and C9, namely C8A and C9A, are shown.

For the preparation of 1-(4-chlorophenacyl)-4-methyl-5,6,7,8,9,10-hexahydrocycloocta[b]pyridin-2(1H)-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). $^1\text{H NMR}$ (DMSO- d_6 , 400 MHz, p.p.m.): 1.45 (*m*, 4H, 8-CH₂ + 9-CH₂), 1.62 (*m*, 4H, 7-CH₂ + 10-CH₂), 1.70 (*m*, 2H, 6-CH₂), 2.20 (*s*, 3H, 13-CH₃), 2.63 (*t*, 2H, 6-CH₂), 2.70 (*t*, 2H, 11-CH₂), 5.45 (*s*, 2H, 14-CH₂), 6.13 (*s*, 1H, 3-CH), 7.53, 8.08 (*dd*, 4H, Ar). Atom numbering as in Fig. 1.

Crystal data

C₂₀H₂₂ClNO₂
 $M_r = 343.84$
 Triclinic, $P\bar{1}$
 $a = 8.239$ (3) Å
 $b = 9.115$ (3) Å
 $c = 12.42$ (1) Å
 $\alpha = 111.20$ (4)°
 $\beta = 93.80$ (4)°
 $\gamma = 96.76$ (3)°
 $V = 857.6$ (9) Å³

$Z = 2$
 $D_x = 1.332$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25
 reflections
 $\theta = 13\text{--}15^\circ$
 $\mu = 0.24$ mm⁻¹
 $T = 293$ (2) K
 Prism, colourless
 0.22 × 0.20 × 0.18 mm

Data collection

Enraf–Nonius CAD-4
 diffractometer
 Non-profiled ω scans
 Absorption correction: none
 4125 measured reflections
 4125 independent reflections
 2645 reflections with $I > 2\sigma(I)$

$\theta_{\text{max}} = 28.0^\circ$
 $h = -10 \rightarrow 10$
 $k = -12 \rightarrow 11$
 $l = 0 \rightarrow 16$
 1 standard reflection
 every 200 reflections
 intensity decay: 3%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.137$
 $S = 1.05$
 4125 reflections
 237 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0608P)^2 + 0.1283P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.29$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.42$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

C11–C19	1.737 (2)	C9A–C10	1.510 (4)
N1–C12	1.392 (2)	C8B–C9B	1.48 (2)
N1–C2	1.396 (3)	C9B–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–C8B	1.403 (15)	C19–C20	1.376 (2)
C7–C8A	1.560 (6)	C20–C21	1.380 (2)
C8A–C9A	1.536 (8)		
C12–N1–C2	122.83 (16)	C9A–C8A–C7	113.6 (4)
C12–N1–C14	122.10 (16)	C10–C9A–C8A	114.3 (3)
C2–N1–C14	114.93 (16)	C7–C8B–C9B	118.7 (10)
O2–C2–N1	119.92 (19)	C8B–C9B–C10	111.3 (8)
O2–C2–C3	124.9 (2)	C9A–C10–C11	117.0 (2)
N1–C2–C3	115.15 (18)	C11–C10–C9B	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)
C8B–C7–C6	111.3 (5)	C16–C15–C14	117.76 (15)
C6–C7–C8A	115.6 (2)		

All H atoms were positioned geometrically and refined as riding (C–H = 0.93–0.97 Å), with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5U_{\text{eq}}$ of the parent C atom.

Data collection: CAD-4 EXPRESS (Enraf–Nonius, 1994); cell refinement: CAD-4 EXPRESS; data reduction: XCAD4 (Harms & Wocadlo, 1995); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX publication routines (Farrugia, 1999).

The authors are indebted to the Russian Foundation for Basic Research for covering the licence fee for use of the Cambridge Structural Database (Allen, 2002) (project No. 02-07-90322).

References

- Albov, D. V., Mazina, O. S., Rybakov, V. B., Babaev, E. V., Chernyshev, V. V. & Aslanov, L. A. (2004). *Crystallogr. Rep.* **49**, 158–168.
 Albov, D. V., Rybakov, V. B., Babaev, E. V. & Aslanov, L. A. (2004). *Acta Cryst.* **E60**, o894–o895.
 Albov, D. V., Rybakov, V. B., Babaev, E. V., Fedyanin, I. V. & Aslanov, L. A. (2004). *Acta Cryst.* **E60**, o892–o893.
 Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.

Enraf–Nonius (1994). *CAD-4 EXPRESS*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.