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2-Methoxy-4-methyl-6,7,8,9-tetrahydro-5*H*-cyclohepta[*b*]pyridine

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The title compound, $C_{12}H_{17}NO$, was synthesized and characterized by ^{1}H NMR and X-ray diffraction techniques.

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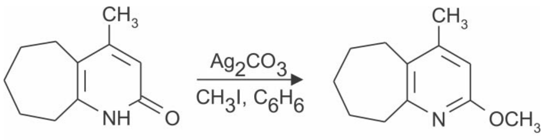
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.067
wR factor = 0.165
Data-to-parameter ratio = 12.6

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

Comment

In the course of systematic investigations of the size effect of cycloalkane fragments on the reactivity of corresponding heterocycles based on pyridine, we have described earlier the crystal structure of 4-methyl-1,5,6,7,8,9-hexahydro-2*H*-cyclohepta[*b*]pyridin-2-one, (1) (Albov, Mazina *et al.*, 2004). According to the procedure of Albov, Rybakov *et al.* (2004), we have synthesized and characterized the title compound, (2).



(1) (2)

In (2), the pyridine ring (N1/C11) is planar within 0.0072 (12) Å. The attached atoms C6, C10, O2, C12 and C13 lie in that plane. Atoms C7, C8 and C9 are displaced from the plane by 1.324 (5), 1.367 (5) and 1.324 (5) Å, respectively. Bond lengths in the pyridine ring confirm its aromatic character. The cycloheptene ring is in a chair conformation (Fig. 1).

The above results should be compared with the crystal structures of five- and eight-membered cycloalkane derivatives, which are in progress, as well as with published (Albov, Rybakov *et al.*, 2004) six-membered cycloalkane derivatives.

Experimental

For the preparation of the title compound, (2), 4-methyl-1,5,6,7,8,9-hexahydro-2*H*-cyclohepta[*b*]pyridin-2-one [(1); 7.69 g], methyl iodide (7.78 g) and silver carbonate (6.00 g) were boiled in benzene (70 ml) for 50 h. The reaction flask was protected against light. The mixture was then filtered and the solvent was evaporated by rotary evaporation (yield: 3.14 g, 38%). The product was recrystallized from chloroform (m.p. 310–314 K). ^{1}H NMR (DMSO-d_6, 400 MHz, p.p.m.): 1.55 (*m*, 2H, 8-CH_2), 1.62 (*m*, 2H, 7-CH_2), 1.84 (*m*, 2H, 9-CH_2), 2.21 (*s*, 3H, 41-CH_3), 2.67 (*t*, 2H, 6-CH_2), 2.86 (*t*, 2H, 10-CH_2), 3.77 (*s*, 3H, 21-CH_3), 6.27 (*s*, 1H, 3-CH).

Crystal data

$C_{12}H_{17}NO$
$M_r = 191.27$
Monoclinic, $P2_1/c$
$a = 8.762\ (2)$ Å
$b = 14.937\ (4)$ Å
$c = 8.542\ (7)$ Å
$\beta = 109.659\ (6)^\circ$
$V = 1052.8\ (9)$ Å^3
$Z = 4$

$D_x = 1.207$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 450 reflections
$\theta = 3\text{–}26^\circ$
$\mu = 0.08$ mm^{-1}
$T = 120\ (2)$ K
Needle, colourless
0.3 × 0.1 × 0.1 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1998)
$T_{\min} = 0.991$, $T_{\max} = 0.992$
7211 measured reflections
2498 independent reflections
1435 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.007$
$\theta_{\max} = 28.0°$
$h = -10 \rightarrow 11$
$k = -19 \rightarrow 19$
$l = -9 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.165$
$S = 0.90$
2498 reflections
129 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1049P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.43$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.325 (2)	C5—C11	1.383 (3)
N1—C11	1.358 (2)	C5—C6	1.512 (3)
O2—C2	1.359 (2)	C6—C7	1.548 (3)
O2—C13	1.428 (3)	C7—C8	1.520 (3)
C2—C3	1.383 (3)	C8—C9	1.513 (3)
C3—C4	1.381 (3)	C9—C10	1.537 (3)
C4—C5	1.412 (3)	C10—C11	1.498 (2)
C4—C12	1.495 (3)		
C2—N1—C11	117.40 (16)	C11—C5—C6	119.94 (17)
C2—O2—C13	116.29 (16)	C4—C5—C6	121.70 (17)
N1—C2—O2	118.95 (17)	C5—C6—C7	114.66 (16)
N1—C2—C3	123.75 (17)	C8—C7—C6	114.11 (17)
O2—C2—C3	117.31 (17)	C9—C8—C7	115.69 (17)
C4—C3—C2	119.32 (17)	C8—C9—C10	114.26 (17)
C3—C4—C5	118.05 (17)	C11—C10—C9	114.20 (16)
C3—C4—C12	119.29 (18)	N1—C11—C5	123.11 (17)
C5—C4—C12	122.65 (17)	N1—C11—C10	114.34 (16)
C11—C5—C4	118.35 (17)	C5—C11—C10	122.52 (17)

H atoms were included in calculated positions and refined as riding atoms, with C—H lengths in the range 0.95–0.99 Å. For methyl H atoms, $U_{\rm iso}$ values were set equal to $1.5U_{\rm eq}$ of the carrier atoms; for other H atoms, $U_{\rm iso}$ values were set to $1.2U_{\rm eq}$ of the carrier atoms.

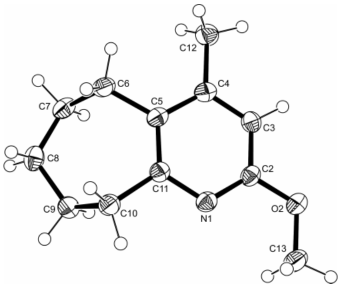


Figure 1
ORTEP-3 (Farrugia, 1997) plot of the molecule of (2), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-Plus*; 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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