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6-Ethoxy-1,2,3,4-tetrahydro-2,2,4-trimethylquinoline

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.053
wR factor = 0.142
Data-to-parameter ratio = 17.8

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

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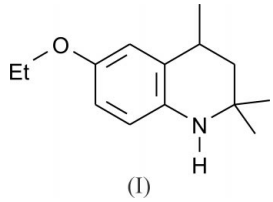
As part of structural studies of 6-ethoxy-2,3,4-tetrahydro-2,2,4-trimethylquinoline derivatives, the crystal structure of the title compound, $C_{14}H_{21}NO$, has been investigated. The conformation of the tetrahydropyridine ring differs markedly from that of similar compounds.

Comment

The crystal and molecular structures of hydrogenated quinolines have been studied previously (Shchegoleva *et al.*, 1980; Obodovskaya *et al.*, 1985, 1990; Davydov *et al.*, 1994). Here, we report the crystal structure of the title compound, (I) (Fig. 1).



(I)

The bicyclic system in the molecule of (I) consists of a planar benzene ring and a non-planar tetrahydropyridine ring. The r.m.s. deviation of the benzene atoms from their mean plane is about 0.006 Å. Atoms N1, C4 and O7, bonded to C atoms of the benzene ring, lie in the same plane. Atoms C71 and C72 are also close to that plane, deviating from it by 0.063 (2) and 0.086 (3) Å, respectively.

The C—C bond lengths in the benzene ring (Table 1) are the same as in other substituted benzene molecules (Cambridge Structural Database, version of November 2003; Allen, 2002). The benzene ring is linked *via* the common C5—C10 bond to the non-planar tetrahydropyridine ring, which has a distorted half-chair conformation. Atoms C2 and C3 are located on opposite sides of the benzene ring plane. The torsion angles C3—C4—C5—C10 and C2—N1—C10—C5 are 15.4 (2) and 21.5 (2)°, respectively, as a result of $A^{1,2}$–allyl tension, which exists in cyclohexene rings (Johanson, 1968).

The bond lengths and angles in (I) are normal and agree well with those in 6-acetoxy-1,2,3,4-tetrahydro-2,2,4-trimethylquinoline (Obodovskaya *et al.*, 1985).

Experimental

The title compound was prepared from crude 6-ethoxy-2,2,4-trimethyl-1,2-dihydroquinoline by hydrogenation in an autoclave in the presence of a Raney catalyst. At the end of the hydrogenation stage, the reaction mixture was cooled, filtered and distilled *in vacuo*. Rose-coloured crystals were obtained after storing for 1 d in the cold [273 (5) K]. After recrystallization, firstly from propan-2-ol and then from hexane, colourless crystals of (I) were obtained (m.p. 311–

312 K). Analysis found: C 76.62, H 9.61, N 6.32, O 7.30%; calculated: C 76.67, H 9.65, N 6.39, O 7.29%. The composition of (I) was confirmed by ^{1}H NMR spectroscopy, measured on a Bruker AM-360 spectrometer (360 MHz, DMSO-d_6, δ, p.p.m): 1.09 (*s*, 3H, 2-CH_3), 1.14 (*s*, 3H, 2-CH_3), 1.21 (*m*, 4H, H3+ 4-CH_3), 1.25 (*t*, 3H, CH_3), 1.66 (*dd*, 1H, H_3), 2.77 (*m*, 1H, H_1), 3.86 (*q*, 2H, OCH_2), 4.9 (*br*, 1H, H_1), 6.36 (*d*, 1H, H_8), 6.46 (*dd*, 1H, H_7), 6.64 (*d*, 1H, H_5).

Crystal data

$C_{14}H_{21}NO$
M_r = 219.32
Monoclinic, $P2_1/c$
a = 13.196 (4) Å
b = 8.865 (6) Å
c = 11.330 (11) Å
β = 90.77 (6)°
V = 1325.3 (16) Å^3
Z = 4
D_x = 1.099 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 30–35°
μ = 0.53 mm^{-1}
T = 293 (2) K
Prism, colourless
0.3 × 0.3 × 0.3 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Non-profiled $\omega/2\theta$ scans
Absorption correction: none
5533 measured reflections
2717 independent reflections
1805 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 74.9°
$h = -16 \rightarrow 16$
$k = -11 \rightarrow 11$
$l = 0 \rightarrow 14$
1 standard reflection
frequency: 60 min
intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.143
S = 0.99
2717 reflections
153 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0647P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C10	1.399 (2)	C5—C6	1.388 (2)
N1—C2	1.469 (2)	C5—C10	1.399 (2)
N1—H1	0.80 (2)	C6—C7	1.390 (2)
C2—C3	1.514 (2)	C7—C8	1.375 (2)
C2—C22	1.521 (3)	C7—O7	1.377 (2)
C2—C21	1.537 (2)	O7—C71	1.413 (2)
C3—C4	1.522 (3)	C71—C72	1.489 (3)
C4—C41	1.514 (3)	C8—C9	1.386 (2)
C4—C5	1.523 (2)	C9—C10	1.398 (2)
C10—N1—C2	118.34 (14)	C6—C5—C4	120.72 (14)
C10—N1—H1	121.7 (15)	C10—C5—C4	120.78 (14)
C2—N1—H1	116.8 (16)	C5—C6—C7	122.27 (14)
N1—C2—C3	106.40 (14)	C8—C7—O7	125.28 (14)
N1—C2—C22	107.72 (15)	C8—C7—C6	119.27 (14)
C3—C2—C22	111.22 (16)	O7—C7—C6	115.44 (14)
N1—C2—C21	111.04 (15)	C7—O7—C71	118.20 (14)
C3—C2—C21	110.84 (14)	O7—C71—C72	108.27 (17)
C22—C2—C21	109.55 (16)	C7—C8—C9	119.32 (14)
C2—C3—C4	114.33 (14)	C8—C9—C10	121.93 (14)
C41—C4—C3	111.30 (17)	C9—C10—N1	119.30 (14)
C41—C4—C5	113.67 (15)	C9—C10—C5	118.71 (14)
C3—C4—C5	109.93 (14)	N1—C10—C5	121.89 (14)
C6—C5—C10	118.49 (14)		

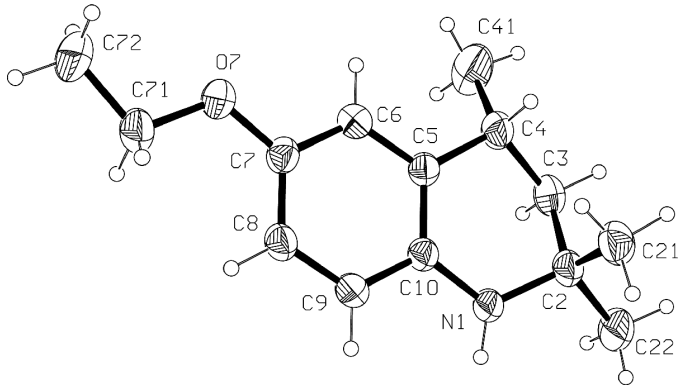


Figure 1
The molecular structure of (I), with 50% probability displacement ellipsoids. H atoms are shown as spheres of arbitrary radii.

The H atom bonded to N was located in a difference Fourier map and refined isotropically. H atoms bonded to C atoms were placed in calculated positions and refined as riding atoms, with C—H distances in the range 0.93–0.96 Å and with U_{iso}(H) = 1.2–1.5U_{eq}(C). No hydrogen bonds are found in this structure.

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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