

4-[2-(5-Ethylpyridin-2-yl)ethoxy]benzaldehyde oxime

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Key indicators

Single-crystal X-ray study

$T = 173\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$

R factor = 0.049

wR factor = 0.129

Data-to-parameter ratio = 15.4

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

The title compound, $\text{C}_{16}\text{H}_{18}\text{N}_2\text{O}_2$, is a key intermediate for the synthesis of biologically active novel heterocycles, *e.g.* isoxazolines and isoxazoles. Geometric parameters of the molecule are in the usual ranges. The crystal packing is stabilized by an $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond.

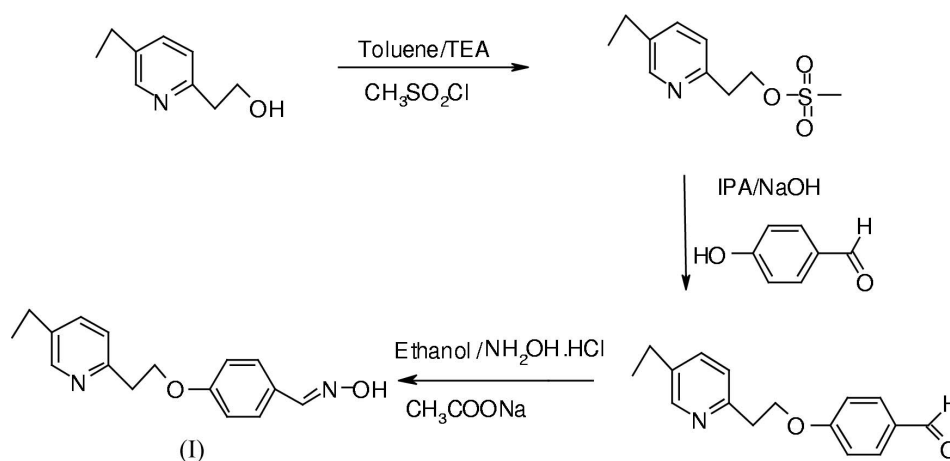
Received 15 February 2005

Accepted 16 February 2005

Online 26 February 2005

Comment

The title compound, (I), is a key intermediate for the synthesis of biologically active novel heterocycles, *e.g.* isoxazolines, isoxazoles, *etc.* (Hassner & Rai, 1989; Ajay Kumar *et al.*, 2001). A crystal structure determination was carried out in order to elucidate the molecular conformation.



A perspective view of (I) is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database Version 5.26; *ConQuest* Version 1.7; *MOGUL* Version 1.0.1; Allen, 2002). The oxime group is almost coplanar with the benzene ring to which it is attached [$\text{O1}-\text{N1}-\text{C1}-\text{C11} = 179.02(14)^\circ$]. The ethyl group is far from perpendicular to the pyridyl ring [$\text{C5}-\text{C4}-\text{C24}-\text{C23} = 74.1(2)^\circ$]. The angle between the benzene and pyridyl rings is $74.80(5)^\circ$. The $\text{O2}-\text{C2}$ and $\text{C2}-\text{C3}$ bonds are in an anti-periplanar conformation. The $\text{O2}-\text{C2}$ bond is nearly coplanar with the benzene ring, whereas the $\text{C2}-\text{C3}$ bond deviates markedly from the plane of the pyridyl ring (Table 1). The crystal packing is stabilized by an $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond (Table 2).

Experimental

2-(5-Ethylpyridin-2-yl)ethanol (1.51 g, 10 mmol) in toluene (10 ml) and triethylamine (2.0 g, 20 mmol) were treated with methane-

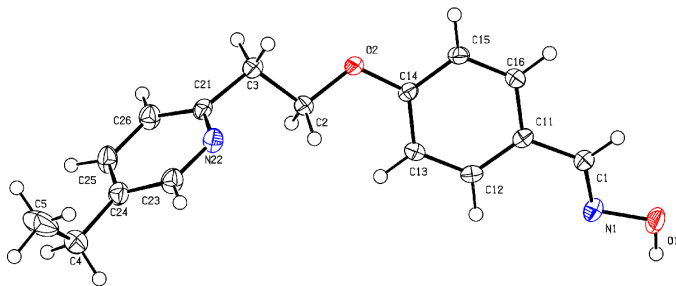


Figure 1
Perspective view of the title compound with the atom numbering; displacement ellipsoids are drawn at the 50% probability level.

sulfonyl chloride (1.14 g, 10 mmol) at 273–278 K. The resulting product was refluxed with NaOH flakes (0.50 g, 12 mmol) and *p*-hydroxybenzaldehyde (1.22 g, 10 mmol) in isopropyl alcohol (10 ml) to yield 4-[2-(5-ethylpyridin-2-yl)ethoxy]benzaldehyde, which was warmed with $\text{NH}_2\text{OH}\cdot\text{HCl}$ (0.7 g, 10 mmol) and sodium acetate (0.82 g, 10 mmol) in ethanol (10 ml) to produce the title compound, which was crystallized from ethyl acetate (m.p. 391 K) (see scheme). IR (KBr, ν cm^{-1}): 3212 (*br*), 3022 (*m*), 2930 (*m*), 2910 (*w*), 1980 (*w*), 1658 (*m*), 1628 (*w*), 1390 (*m*), 990 (*m*), 721 (*m*). ^1H NMR (CDCl_3 , p.p.m.): 1.32 (*t*, 3H, CH_3), 2.64 (*q*, 2H, CH_2), 3.21 (*t*, 2H, CH_2), 4.42 (*t*, 2H, CH_2), 6.92 (*d*, 2H, ArH), 7.19 (*d*, 1H, ArH), 7.42 (*d*, 2H, ArH), 7.59 (*d*, 1H, ArH), 8.12 (*s*, 1H, ArH), 8.54 (*s*, 1H, CH), 9.88 (*s*, 1H, OH). ^{13}C NMR (CDCl_3 , p.p.m.): 15.8 (*q*, CH_3), 28.1 (*t*, CH_2), 35.7 (*t*, CH_2), 73.2 (*t*, CH_2), 112.2 (*d*, ArC), 119.2 (*d*, ArC), 121.0 (*s*, ArC), 128.4 (*d*, ArC), 134.3 (*d*, ArC), 135.6 (*s*, ArC), 147.8 (*d*, ArC), 152.2 (*d*, CH), 161.0 (*s*, ArC), 162.2 (*s*, ArC). Analysis calculated for $\text{C}_{16}\text{H}_{18}\text{N}_2\text{O}_2$: C 71.09, H 6.71, N 10.36%; found: C 71.15, H 6.75, N 10.31%.

Crystal data

$\text{C}_{16}\text{H}_{18}\text{N}_2\text{O}_2$
 $M_r = 270.32$
Monoclinic, $P2_1/c$
 $a = 16.4538$ (19) Å
 $b = 5.3346$ (5) Å
 $c = 17.952$ (2) Å
 $\beta = 114.709$ (9)°
 $V = 1431.4$ (3) Å³
 $Z = 4$

$D_x = 1.254$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 6268 reflections
 $\theta = 3.7$ – 26.3 °
 $\mu = 0.08$ mm⁻¹
 $T = 173$ (2) K
Needle, colourless
 $0.42 \times 0.13 \times 0.08$ mm

Data collection

Stoe IPDS-II two-circle diffractometer
 ω scans
Absorption correction: none
7184 measured reflections
2848 independent reflections

2222 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.047$
 $\theta_{\text{max}} = 26.4$ °
 $h = -19 \rightarrow 20$
 $k = -6 \rightarrow 6$
 $l = -22 \rightarrow 14$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.129$
 $S = 1.04$
2848 reflections
185 parameters
H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0771P)^2 + 0.1912P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

O1–N1	1.4077 (18)	N1–C1	1.284 (2)
C1–N1–O1	110.52 (13)	N1–C1–C11	121.91 (14)
C14–O2–C2–C3	170.10 (13)	C2–O2–C14–C15	−164.93 (14)
O2–C2–C3–C21	−169.22 (13)	C2–C3–C21–N22	73.96 (18)

Table 2

Hydrogen-bonding geometry (Å, °).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
O1–H1O \cdots N22 ⁱ	0.95 (3)	1.86 (3)	2.7948 (19)	169 (3)

Symmetry code: (i) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$.

All H atoms were located in a difference map. Those bonded to C atoms were positioned geometrically and refined with fixed individual displacement parameters (set to 1.2 times U_{eq} of the parent atom, or 1.5 for methyl groups), using a riding model, with C–H distances ranging from 0.95 to 0.99 Å. The hydroxyl H atom was refined independently with an isotropic displacement parameter.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PLATON*.

One of the authors (SLG) is grateful to the University of Mysore for permitting him to carry out the research work.

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