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

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The title compound, $C_{16}H_{18}N_2O_2$, 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 O—H···N hydrogen bond.

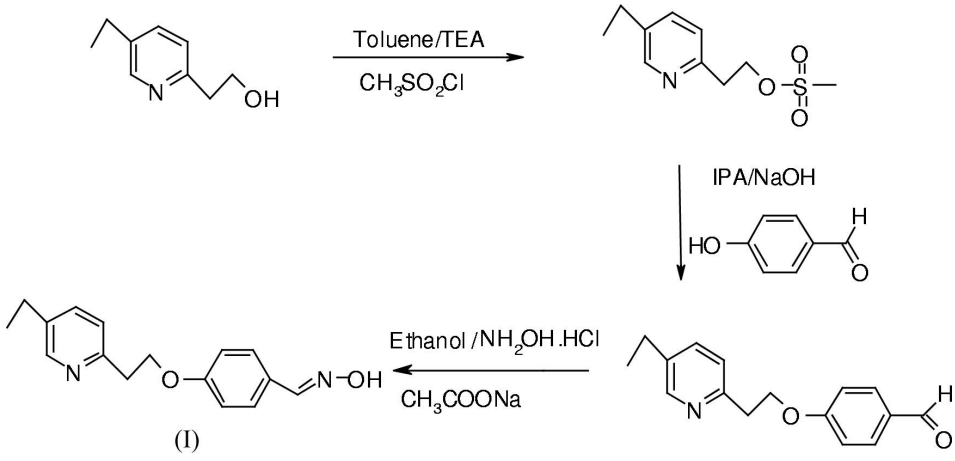
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
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.

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.



(I)

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 [O1—N1—C1—C11 = 179.02 (14)°]. The ethyl group is far from perpendicular to the pyridyl ring [C5—C4—C24—C23 = 74.1 (2)°]. The angle between the benzene and pyridyl rings is 74.80 (5)°. The O2—C2 and C2—C3 bonds are in an anti-periplanar conformation. The O2—C2 bond is nearly coplanar with the benzene ring, whereas the C2—C3 bond deviates markedly from the plane of the pyridyl ring (Table 1). The crystal packing is stabilized by an O—H···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-

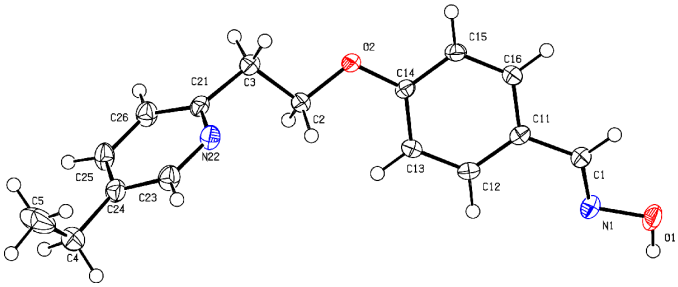


Figure 1
Perspective view of the title compound with the atom numbering; displacement ellipsoids are drawn at the 50% probability level.

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 $NH_2OH{\cdot}HCl$ (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*). ^{1}H 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 $C_{16}H_{18}N_2O_2$: C 71.09, H 6.71, N 10.36%; found: C 71.15, H 6.75, N 10.31%.

Crystal data

$C_{16}H_{18}N_2O_2$
M_r = 270.32
Monoclinic, $P2_1/c$
a = 16.4538 (19) Å
b = 5.3346 (5) Å
c = 17.952 (2) Å
β = 114.709 (9)°
V = 1431.4 (3) Å^3
Z = 4
D_x = 1.254 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6268 reflections
θ = 3.7–26.3°
μ = 0.08 mm^{-1}
T = 173 (2) K
Needle, colourless
0.42 × 0.13 × 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_{int} = 0.047
θ_{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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-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—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···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*.

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