Xian Tang and De-Cai Wang*

Crystal structure of 4-isobutoxybenzaldehyde oxime, $C_{11}H_{15}NO_2$

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Abstract

$C_{11}H_{15}O_2N$, monoclinic, $P2_1/n$ (no. 14), $a = 13.5152(2)$ Å, $b = 6.19120(10)$ Å, $c = 14.3917(2)$ Å, $\beta = 115.467(2)^\circ$, $V = 1087.22(3)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0384$, $wR_{ref}(F^2) = 0.1104$, $T = 170(2)$ K.

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The hydrogen bonded dimer of the title structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of materials

Commercially available *p*-hydroxybenzaldehyde (1.22 g, 10 mmol), and 1-bromo-2-methylpropane (2.72 g, 10 mmol) were dissolved in 40 mL *N,N*-dimethylformamide at 85 °C, and then stirring 4 h to generate 4-isobutoxybenzaldehyde. The title compound was synthesized in a second step by the reaction of 4-isobutoxybenzaldehyde and hydroxylamine (molar ratio 1:2) in ethanol. A yellow powder was obtained by extraction and evaporation. Crystals were obtained by recrystallization from *n*-hexan in the yield of 79%. ESI $m/z = 194.11(M + 1)$, 1H NMR (400 MHz, DMSO-*d*₆) δ [p.p.m.] 10.96 (s, 1H),

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.41 \times 0.33 \times 0.21$ mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ :	0.66 mm^{-1}
Diffractometer, scan mode:	XtaLAB Synergy R, ω
$\theta_{\max}$, completeness:	75.4° , 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6556, 2176, 0.011
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2061
$N(\text{param})_{\text{refined}}$:	129
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3, 4]

8.07 (s, 1H), 7.49 (d, 2H), 6.89 (d, 2H), 3.74 (d, 2H), 2.14–2.03 (m, 1H), 1.03 (d, 6H).

Experimental details

All H atoms attached to carbon were included using a riding-model, with C–H = 0.93–0.97 Å, and their U_{iso} values were set to $1.2U_{\text{eq}}$ or $1.5U_{\text{eq}}$. The H atom at O2 was also included using a riding-mode, with O–H = 0.82 Å and their U_{iso} values were set to $1.5U_{\text{eq}}$.

Comment

The title compound, 4-isobutoxybenzaldehyde oxime is an important drug intermediate [5, 6]. Further surveys of crystal structures containing such oxime were recently carried out [7]. To the best of our knowledge, there is no example of an isobutoxy benzaldehyde oxime that has been reported so far.

The asymmetric unit of the title structure contains one 4-isobutoxybenzaldehyde oxime molecule. The bond lengths and angles within the structure of 4-isobutoxybenzaldehyde oxime are unexceptional and lie within the expected ranges. In details, the C–O distances within 4-isobutoxybenzaldehyde oxime have values between 1.359(1)–1.434(1) Å. The N1–O2 distance is

*Corresponding author: De-Cai Wang, Institut für College of Biotechnology and Pharmaceutical Engineering, Universität Nanjing Tech University, Nanjing 211816, China, e-mail: dcwang@njut.edu.cn

Xian Tang: Institut für College of Biotechnology and Pharmaceutical Engineering, Universität Nanjing Tech University, Nanjing 211816, China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
O1	0.02528(6)	0.22943(12)	0.11522(6)	0.0464(2)
O2	0.55866(7)	0.27237(16)	0.04356(8)	0.0620(3)
H2	0.570882	0.395739	0.012382	0.093*
N1	0.46013(7)	0.32671(16)	0.04898(7)	0.0459(2)
C1	−0.18343(10)	−0.0681(2)	0.17044(10)	0.0563(3)
H1A	−0.142580	−0.095837	0.244338	0.084*
H1B	−0.182691	−0.197906	0.131805	0.084*
H1C	−0.259303	−0.030185	0.155090	0.084*
C2	−0.12766(12)	0.3193(2)	0.19971(12)	0.0666(4)
H2A	−0.090347	0.435133	0.180848	0.100*
H2B	−0.088173	0.289379	0.273541	0.100*
H2C	−0.202803	0.363985	0.183786	0.100*
C3	−0.13022(8)	0.11786(18)	0.13955(8)	0.0435(3)
H3	−0.175696	0.148825	0.064939	0.052*
C4	−0.01664(9)	0.05442(18)	0.15313(8)	0.0428(3)
H4A	0.031596	0.027967	0.226733	0.051*
H4B	−0.020108	−0.079276	0.114105	0.051*
C5	0.12547(8)	0.20846(17)	0.11539(7)	0.0384(2)
C6	0.15825(8)	0.38244(17)	0.07351(8)	0.0428(3)
H6	0.112335	0.505719	0.049565	0.051*
C7	0.25638(8)	0.37628(18)	0.06677(8)	0.0423(3)
H7	0.277705	0.495708	0.038084	0.051*
C8	0.32585(8)	0.19607(17)	0.10163(7)	0.0396(2)
C9	0.29339(9)	0.02768(18)	0.14577(8)	0.0436(3)
H9	0.340337	−0.093646	0.171700	0.052*
C10	0.19410(9)	0.03107(17)	0.15325(8)	0.0419(3)
H10	0.173575	−0.086225	0.183802	0.050*
C11	0.42728(9)	0.17745(19)	0.08921(9)	0.0468(3)
H11	0.469879	0.049666	0.111958	0.056*

1.408(1) Å. The N1–C11 distance is 1.276(1) Å, which suggests a N=C double bond. Two intermolecular H-bonds, O–H⋯N (dO⋯N = 2.776(2) Å; dH⋯N = 1.895(2) Å;

∠OHN = 156.14(1)°), connect neighboring molecules pairwise (*cf.* the figure). In total, the crystal structure of 4-isobutoxybenzaldehyde oxime could be viewed as dimers connected with each other by weak interaction for construction a three dimensional network.

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