

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## 2-(Hydroxymethyl)pyridin-3-ol

Xue-Jun He, Jiang-Kai Qiu, Yu-Chi Jia, De-Cai Wang\* and Ping-Kai Ou-Yang

State Key Laboratory of Materials-Oriented Chemical Engineering, School of Pharmaceutical Sciences, Nanjing University of Technology, Nanjing 210009, People's Republic of China

Correspondence e-mail: dc\_wang@hotmail.com

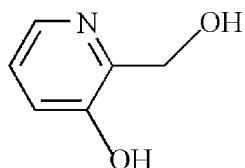
Received 31 December 2011; accepted 22 January 2012

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.036;  $wR$  factor = 0.136; data-to-parameter ratio = 12.8.

In the crystal structure of the title compound,  $\text{C}_6\text{H}_7\text{NO}_2$ , the molecules are linked by intermolecular  $\text{O}-\text{H}\cdots\text{N}$  and  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds;  $\pi-\pi$  stacking is observed between parallel pyridine rings of adjacent molecules [centroid-to-centroid distance = 3.7649 (12) Å].

## Related literature

For the synthesis of the title compound, see: Dabak (2002).



## Experimental

## Crystal data

 $\text{C}_6\text{H}_7\text{NO}_2$  $M_r = 125.13$ Monoclinic,  $P2_1/n$  $a = 7.0430$  (14) Å $b = 7.1280$  (14) Å $c = 12.264$  (3) Å $\beta = 100.30$  (3)° $V = 605.8$  (2) Å<sup>3</sup> $Z = 4$ Mo  $K\alpha$  radiation $\mu = 0.10$  mm<sup>-1</sup> $T = 293$  K $0.30 \times 0.20 \times 0.10$  mm

## Data collection

Enraf–Nonius CAD-4 diffractometer  
Absorption correction:  $\psi$  scan (*XCAD4*; Harms & Wocadlo, 1995) $T_{\min} = 0.969$ ,  $T_{\max} = 0.990$   
2263 measured reflections1089 independent reflections  
932 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.028$   
3 standard reflections every 200 reflections  
intensity decay: 1%

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.036$  $wR(F^2) = 0.136$  $S = 0.99$ 

1089 reflections

85 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.18$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.15$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1A}\cdots\text{O2}^{\text{i}}$	0.82	1.85	2.6502 (17)	166
$\text{O2}-\text{H2A}\cdots\text{N}^{\text{ii}}$	0.82	1.92	2.7216 (17)	167

Symmetry codes: (i)  $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$ ; (ii)  $-x + 1, -y, -z$ .

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

This work was supported by the Center of Testing and Analysis, Nanjing University, China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5437).

## References

- Dabak, K. (2002). *Turk. J. Chem.* **26**, 955–963.  
Enraf–Nonius (1994). *CAD-4 EXPRESS*. Enraf–Nonius, Delft, The Netherlands.  
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

## supporting information

*Acta Cryst.* (2012). E68, o722 [doi:10.1107/S1600536812002759]

**2-(Hydroxymethyl)pyridin-3-ol**

Xue-Jun He, Jiang-Kai Qiu, Yu-Chi Jia, De-Cai Wang and Ping-Kai Ou-Yang

**S1. Comment**

The title compound is an important organic intermediate for the synthesis of 2-pyrimidine-oxy-*N*-aryl benzyl amine derivatives, an important compound for new pesticides. In the process of synthesis, we obtained the crystal of the intermediate and we report its crystal structure.

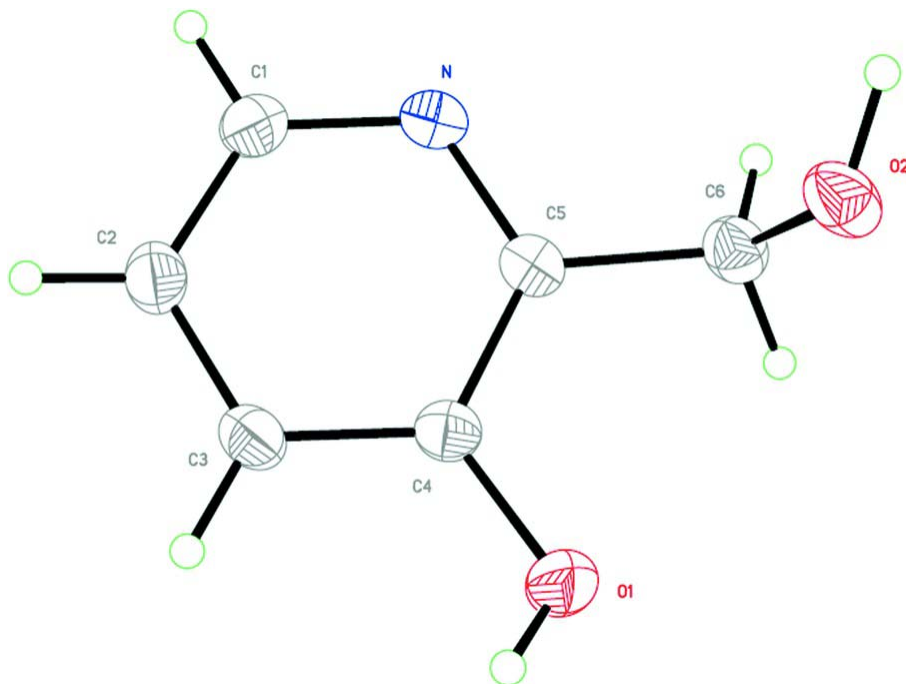
As illustrated in Fig. 1, the hydroxyl oxygen O1 and the hydroxymethyl carbon C6 are approximately coplanar with the pyridine ring (C1—C5/N) with the maximum deviation of  $-0.0227 \text{ \AA}$ . The crystal structure is stabilized by intermolecular N—H $\cdots$ O and O—H $\cdots$ O hydrogen bonds (Table 1), and is further stabilized by  $\pi$ – $\pi$  stacking between pyridine rings [centroid–centroid distance =  $3.7649 (12) \text{ \AA}$ ]

**S2. Experimental**

The synthesis is according to the literature (Dabak, 2002). The formaldehyde solution (12.6 ml, 0.156 mol) and sodium hydroxide (6.3 g, 0.158 mol) was added to a solution of 3-hydroxypyridine (15.0 g, 0.156 mol) in water (63 ml). The reaction mixture was heated at 373 K for 12 h and then allowed to cool to ambient temperature. Acetic acid (9.47 ml, 0.156 mol) was added and water was removed *in vacuo* and the solid obtained was stirred with acetone (200 ml). The extract was purified by silica gel column chromatography and the colourless crystals were obtained in a yield of 20.3%.

**S3. Refinement**

H atoms were placed at calculated positions and were treated in riding mode with C—H = 0.93 (aromatic), 0.97  $\text{\AA}$  (methylene) and O—H = 0.82  $\text{\AA}$ .  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  and  $1.5U_{\text{eq}}(\text{O})$ .

**Figure 1**

The structure of the title compound, showing the atomic numbering scheme. Non-H atoms are shown with 30% probability displacement ellipsoids.

## 2-(Hydroxymethyl)pyridin-3-ol

### Crystal data

$C_6H_7NO_2$

$M_r = 125.13$

Monoclinic,  $P2_1/n$

Hall symbol: -P 2yn

$a = 7.0430$  (14) Å

$b = 7.1280$  (14) Å

$c = 12.264$  (3) Å

$\beta = 100.30$  (3)°

$V = 605.8$  (2) Å<sup>3</sup>

$Z = 4$

$F(000) = 264$

$D_x = 1.372$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 25 reflections

$\theta = 9\text{--}13^\circ$

$\mu = 0.10$  mm<sup>-1</sup>

$T = 293$  K

Block, colourless

$0.30 \times 0.20 \times 0.10$  mm

### Data collection

Enraf-Nonius CAD-4  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega/2\theta$  scans

Absorption correction:  $\psi$  scan

(*XCAD4*; Harms & Wocadlo, 1995)

$T_{\min} = 0.969$ ,  $T_{\max} = 0.990$

2263 measured reflections

1089 independent reflections

932 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\max} = 25.2^\circ$ ,  $\theta_{\min} = 3.1^\circ$

$h = 0 \rightarrow 8$

$k = -8 \rightarrow 8$

$l = -14 \rightarrow 14$

3 standard reflections every 200 reflections

intensity decay: 1%

Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.036$   
 $wR(F^2) = 0.136$   
 $S = 0.99$   
 1089 reflections  
 85 parameters  
 0 restraints  
 Primary atom site location: structure-invariant  
 direct methods  
 Secondary atom site location: difference Fourier  
 map

Hydrogen site location: inferred from  
 neighbouring sites  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.069P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.18 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.15 \text{ e } \text{\AA}^{-3}$   
 Extinction correction: *SHELXTL* (Sheldrick,  
 2008),  $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$   
 Extinction coefficient: 0.67 (5)

Special details

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N	0.59730 (19)	0.28061 (17)	0.05259 (10)	0.0374 (4)
O1	0.21831 (16)	0.3738 (2)	0.20521 (9)	0.0531 (5)
H1A	0.2217	0.4225	0.2661	0.080*
C1	0.7372 (2)	0.3911 (2)	0.10511 (13)	0.0418 (5)
H1B	0.8548	0.3932	0.0807	0.050*
O2	0.27768 (19)	-0.02901 (16)	0.08581 (9)	0.0504 (5)
H2A	0.3277	-0.1099	0.0527	0.076*
C2	0.7152 (2)	0.5019 (2)	0.19373 (13)	0.0436 (5)
H2B	0.8160	0.5772	0.2284	0.052*
C3	0.5409 (2)	0.4995 (2)	0.23031 (13)	0.0410 (5)
H3A	0.5223	0.5731	0.2901	0.049*
C4	0.3939 (2)	0.3855 (2)	0.17644 (12)	0.0358 (5)
C5	0.4270 (2)	0.2764 (2)	0.08702 (11)	0.0340 (5)
C6	0.2761 (2)	0.1456 (2)	0.02825 (13)	0.0421 (5)
H6A	0.1500	0.2034	0.0225	0.050*
H6B	0.2994	0.1228	-0.0462	0.050*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N	0.0432 (8)	0.0343 (7)	0.0360 (7)	0.0046 (5)	0.0108 (5)	0.0008 (5)
O1	0.0435 (8)	0.0704 (9)	0.0487 (8)	-0.0087 (6)	0.0176 (5)	-0.0177 (6)
C1	0.0383 (9)	0.0407 (9)	0.0478 (10)	0.0024 (7)	0.0115 (7)	0.0030 (7)

O2	0.0729 (9)	0.0392 (7)	0.0462 (7)	-0.0084 (6)	0.0299 (6)	-0.0068 (5)
C2	0.0415 (9)	0.0399 (9)	0.0471 (10)	-0.0041 (7)	0.0020 (7)	-0.0038 (6)
C3	0.0462 (9)	0.0392 (8)	0.0377 (9)	0.0008 (7)	0.0076 (7)	-0.0074 (6)
C4	0.0381 (9)	0.0362 (8)	0.0336 (8)	0.0030 (6)	0.0078 (6)	0.0003 (6)
C5	0.0416 (9)	0.0318 (8)	0.0284 (8)	0.0033 (6)	0.0053 (6)	0.0034 (5)
C6	0.0472 (10)	0.0421 (9)	0.0371 (8)	-0.0027 (7)	0.0078 (7)	-0.0049 (6)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

N—C1	1.334 (2)	C2—C3	1.381 (2)
N—C5	1.3412 (19)	C2—H2B	0.9300
O1—C4	1.3478 (19)	C3—C4	1.387 (2)
O1—H1A	0.8200	C3—H3A	0.9300
C1—C2	1.375 (2)	C4—C5	1.397 (2)
C1—H1B	0.9300	C5—C6	1.499 (2)
O2—C6	1.430 (2)	C6—H6A	0.9700
O2—H2A	0.8200	C6—H6B	0.9700
C1—N—C5	119.08 (12)	O1—C4—C3	123.57 (14)
C4—O1—H1A	109.5	O1—C4—C5	117.43 (13)
N—C1—C2	122.91 (15)	C3—C4—C5	118.99 (15)
N—C1—H1B	118.5	N—C5—C4	121.25 (13)
C2—C1—H1B	118.5	N—C5—C6	117.37 (12)
C6—O2—H2A	109.5	C4—C5—C6	121.35 (14)
C1—C2—C3	118.82 (15)	O2—C6—C5	111.22 (13)
C1—C2—H2B	120.6	O2—C6—H6A	109.4
C3—C2—H2B	120.6	C5—C6—H6A	109.4
C2—C3—C4	118.94 (14)	O2—C6—H6B	109.4
C2—C3—H3A	120.5	C5—C6—H6B	109.4
C4—C3—H3A	120.5	H6A—C6—H6B	108.0
C5—N—C1—C2	0.0 (2)	O1—C4—C5—N	179.61 (13)
N—C1—C2—C3	-0.1 (2)	C3—C4—C5—N	-0.1 (2)
C1—C2—C3—C4	0.0 (2)	O1—C4—C5—C6	-2.4 (2)
C2—C3—C4—O1	-179.62 (15)	C3—C4—C5—C6	177.80 (13)
C2—C3—C4—C5	0.1 (2)	N—C5—C6—O2	95.53 (15)
C1—N—C5—C4	0.1 (2)	C4—C5—C6—O2	-82.49 (17)
C1—N—C5—C6	-177.96 (13)		

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1A $\cdots$ O2 <sup>i</sup>	0.82	1.85	2.6502 (17)	166
O2—H2A $\cdots$ N <sup>ii</sup>	0.82	1.92	2.7216 (17)	167

Symmetry codes: (i)  $-x+1/2, y+1/2, -z+1/2$ ; (ii)  $-x+1, -y, -z$ .