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# Crystal structure of 2,6-dimethyl-4-pyridone hemihydrate

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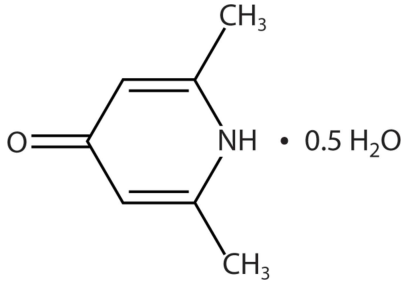
The title compound (systematic name: 2,6-dimethyl-1*H*-pyridin-4-one hemihydrate), $C_7H_9NO{\cdot}0.5H_2O$, has a single planar molecule in the asymmetric unit with the non-H atoms possessing a mean deviation from planarity of 0.021 Å. There is also half of a water molecule present in the asymmetric unit. In the crystal, infinite (001) sheets are formed by N—H···O and O—H···O hydrogen bonds.

**Keywords:** crystal structure; hydrogen bonding; pyridones.

**CCDC reference**: 1409189

## 1. Related literature

For the crystal structure of the parent 4-pyridone, see: Jones (2001); Tyl *et al.* (2008). For the title compound bound to zirconium, see: Castillo *et al.* (1987). For the structure of a chloro-substituted variant of the title compound, see: Boer (1972).




## 2. Experimental

### 2.1. Crystal data

$C_7H_9NO{\cdot}0.5H_2O$ $M_r$ = 132.16

Orthorhombic, *Aba*2
$a$ = 12.4859 (17) Å
$b$ = 14.3697 (19) Å
$c$ = 7.732 (1) Å
$V$ = 1387.3 (3) Å$^3$
$Z$ = 8
Cu $K\alpha$ radiation
$\mu$ = 0.73 mm$^{-1}$
$T$ = 120 K
0.5 × 0.1 × 0.1 mm

### 2.2. Data collection

Bruker Venture D8 CMOS diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)
$T_{min}$ = 0.554, $T_{max}$ = 0.754
11786 measured reflections
1366 independent reflections
1352 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.060

### 2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.078
$S$ = 1.09
1366 reflections
95 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.19 e Å$^{-3}$
Absolute structure: Flack *x* determined using 611 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013).
Absolute structure parameter: 0.05 (12)

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2···O1 | 0.86 (1) | 1.96 (1) | 2.8174 (17) | 173 (2) |
| N1—H1···O1$^{i}$ | 0.87 (1) | 1.86 (1) | 2.7154 (18) | 166 (3) |

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

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* and *publCIF* (Westrip, 2010).

## Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: FF2139).

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# supporting information

*Acta Cryst.* (2015). E**71**, o533 [https://doi.org/10.1107/S2056989015012402]

## Crystal structure of 2,6-dimethyl-4-pyridone hemihydrate

**Dalena M. Nguyen, Vasumathi Desikan, James A. Golen and David R. Manke**

### S1. Comment

The structure of the title compound shows that 2,6-dimethyl-4-hydroxypyridine takes the pyridone form in the solid state. Though the title compound has not been crystallographically characterized, a structure of the molecule bound to zirconium has been reported (Castillo *et al.*, 1987) with similar bond distances and angles observed. The parent 4-pyridone has been structurally characterized (Tyl *et al.*, 2008) and exhibits similar chains linked by head-to-tail N–H···O hydrogen bonds, which are also observed in the close derivative Clopidol (Boer, 1972). While the title compound crystallizes in a 2:1 ratio of pyridone to water, the hydrate of the parent molecule crystallizes in a 5:6 ratio (Jones, 2001).

The molecular structure of the title compound is shown in Figure 1. The molecule is near planar with the non-hydrogen atoms possessing a mean deviation from the plane of 0.021 Å. Head-to-tail N1–H1···O1 hydrogen bonding leads to chains that are further linked by O2–H2···O1 hydrogen bonds with water molecules in the crystal to form two-dimensional (001) sheets. The packing of the title compound indicating hydrogen bonding is shown in Figure 2.

### S2. Experimental

A commercial sample (Oakwood Chemical) of 4-hydroxypyridine was used for the crystallization. Crystals suitable for single crystal X-ray analysis were grown by slow evaporation of a methanol solution.

### S3. Refinement

All non-hydrogen atoms were refined anisotropically (*XL*) by full matrix least squares on $F^2$. Hydrogen atoms H1 and H2 were found from a Fourier difference map. H1 was refined at a fixed distance of 0.87 (0.005) Å and an isotropic displacement parameter 1.20 times $U_{eq}$ of the parent N atom. H2 was refined at a fixed distance of 0.86 (0.005) Å and an isotropic displacement parameter 1.50 times $U_{eq}$ of the parent O atom. The remaining hydrogen atoms were placed in calculated positions and then refined with riding models with C—H lengths of 0.98 Å for ($CH_3$) and 0.95 Å for (CH) with isotropic displacement parameters set to 1.20 times $U_{eq}$ of the parent C atoms.

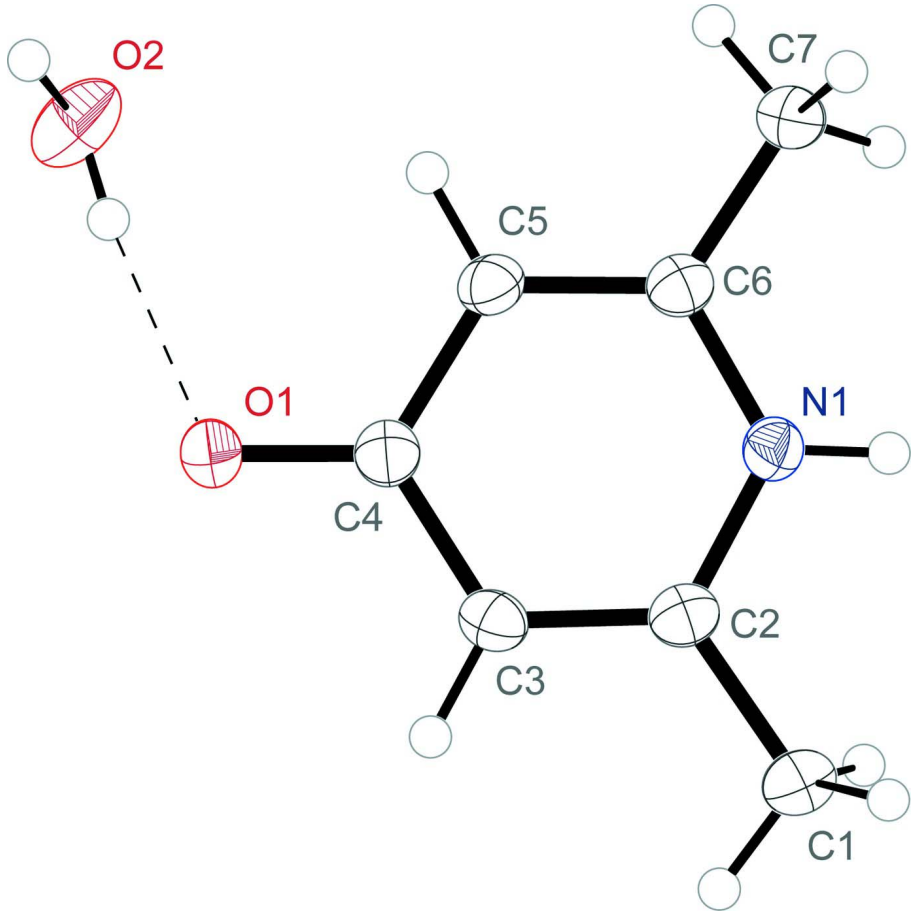


**Figure 1**

Molecular structure of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radius.

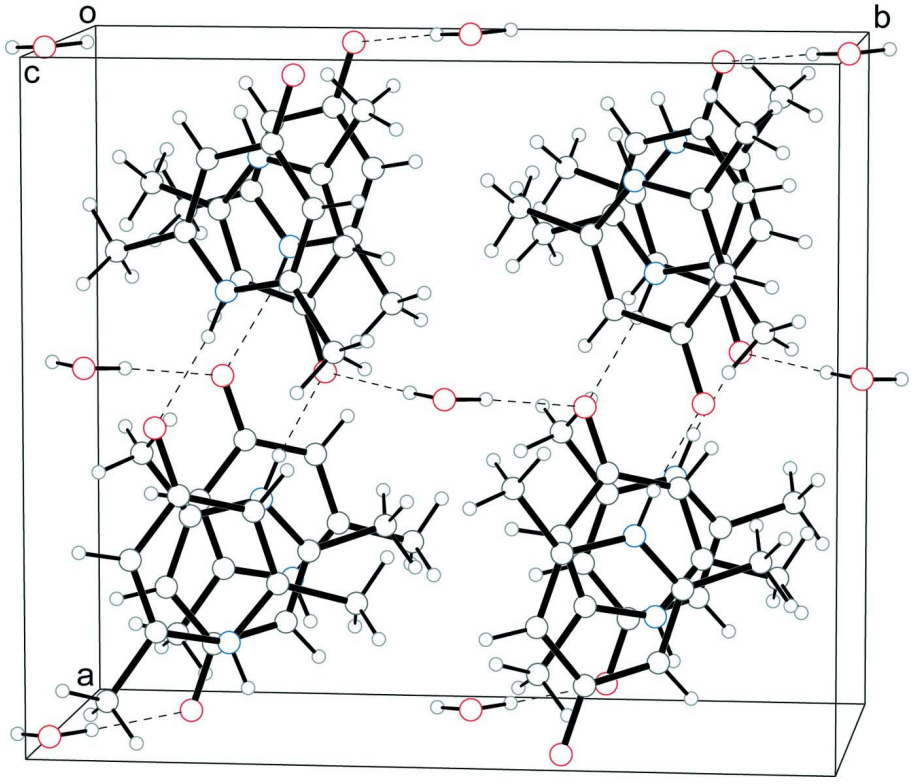


**Figure 2**

Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

### 2,6-Dimethyl-1*H*-pyridin-4-one hemihydrate

*Crystal data*

$C_7H_9NO{\cdot}0.5H_2O$
$M_r = 132.16$
Orthorhombic, *Aba*2
Hall symbol: A 2 -2ac
$a = 12.4859\ (17)$ Å
$b = 14.3697\ (19)$ Å
$c = 7.732\ (1)$ Å
$V = 1387.3\ (3)$ Å$^3$
$Z = 8$
$F(000) = 568$
$D_x = 1.266$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 9829 reflections
$\theta = 7.1–72.4°$
$\mu = 0.73$ mm$^{-1}$
$T = 120$ K
NEEDLE, colourless
0.5 × 0.1 × 0.1 mm

*Data collection*

Bruker Venture D8 CMOS diffractometer
Radiation source: microfocus Cu
HELIOS MX monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)
$T_{min} = 0.554$, $T_{max} = 0.754$
11786 measured reflections
1366 independent reflections
1352 reflections with $I > 2\sigma(I)$
$R_{int} = 0.060$
$\theta_{max} = 72.4°$, $\theta_{min} = 7.1°$
$h = -15\rightarrow15$
$k = -17\rightarrow17$
$l = -9\rightarrow9$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.078$
$S = 1.09$
1366 reflections
95 parameters
3 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 0.3383P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å$^{-3}$
$\Delta\rho_{min} = -0.19$ e Å$^{-3}$
Absolute structure: Flack *x* determined using 611 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013.
Absolute structure parameter: 0.05 (12)

*Special details*

**Experimental**. Absorption correction: SADABS-2014/4 (Bruker,2014) was used for absorption correction. wR2(int) was 0.1440 before and 0.0881 after correction. The Ratio of minimum to maximum transmission is 0.7350. The $\lambda/2$ correction factor is 0.00150.

**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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.47723 (9) | 0.33620 (8) | 0.49203 (19) | 0.0287 (3) |
| O2 | 0.5000 | 0.5000 | 0.6889 (3) | 0.0399 (5) |
| N1 | 0.16883 (11) | 0.24949 (11) | 0.4446 (2) | 0.0215 (3) |
| C3 | 0.35308 (14) | 0.22232 (12) | 0.3962 (2) | 0.0234 (4) |
| H3 | 0.4083 | 0.1841 | 0.3504 | 0.028* |
| C2 | 0.24877 (14) | 0.19343 (12) | 0.3853 (2) | 0.0222 (4) |
| C4 | 0.38007 (12) | 0.30864 (12) | 0.4748 (2) | 0.0226 (4) |
| C7 | 0.09367 (14) | 0.39066 (13) | 0.5690 (3) | 0.0265 (4) |
| H7A | 0.0630 | 0.4210 | 0.4669 | 0.040* |
| H7B | 0.1158 | 0.4381 | 0.6527 | 0.040* |
| H7C | 0.0399 | 0.3499 | 0.6218 | 0.040* |
| C6 | 0.18900 (13) | 0.33399 (11) | 0.5164 (2) | 0.0217 (4) |
| C1 | 0.21576 (15) | 0.10115 (12) | 0.3125 (3) | 0.0270 (4) |
| H1A | 0.1736 | 0.0672 | 0.3988 | 0.040* |
| H1B | 0.2797 | 0.0650 | 0.2828 | 0.040* |
| H1C | 0.1724 | 0.1109 | 0.2085 | 0.040* |
| C5 | 0.29237 (13) | 0.36395 (12) | 0.5339 (2) | 0.0229 (4) |
| H5 | 0.3061 | 0.4226 | 0.5862 | 0.027* |
| H2 | 0.4956 (19) | 0.4524 (12) | 0.621 (3) | 0.034* |
| H1 | 0.1043 (9) | 0.2265 (14) | 0.444 (4) | 0.027* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0205 (5) | 0.0250 (6) | 0.0405 (8) | −0.0007 (5) | −0.0004 (6) | −0.0028 (6) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O2 | 0.0553 (14) | 0.0318 (11) | 0.0324 (11) | −0.0147 (10) | 0.000 | 0.000 |
| N1 | 0.0206 (6) | 0.0206 (7) | 0.0234 (7) | −0.0014 (5) | 0.0004 (6) | 0.0008 (6) |
| C3 | 0.0240 (8) | 0.0215 (8) | 0.0246 (8) | 0.0035 (6) | 0.0014 (7) | 0.0012 (8) |
| C2 | 0.0268 (8) | 0.0193 (8) | 0.0205 (7) | 0.0009 (6) | −0.0014 (7) | 0.0022 (7) |
| C4 | 0.0229 (8) | 0.0208 (8) | 0.0241 (9) | 0.0003 (6) | −0.0011 (7) | 0.0038 (8) |
| C7 | 0.0245 (8) | 0.0243 (8) | 0.0309 (10) | 0.0025 (7) | −0.0008 (7) | −0.0041 (8) |
| C6 | 0.0244 (8) | 0.0196 (8) | 0.0213 (8) | 0.0009 (6) | −0.0012 (7) | 0.0019 (7) |
| C1 | 0.0309 (9) | 0.0216 (8) | 0.0284 (10) | −0.0005 (7) | −0.0007 (8) | −0.0012 (8) |
| C5 | 0.0255 (8) | 0.0174 (7) | 0.0258 (8) | −0.0004 (6) | −0.0006 (7) | 0.0004 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C4 | 1.283 (2) | C7—H7A | 0.9800 |
| O2—H2 | 0.862 (7) | C7—H7B | 0.9800 |
| N1—C2 | 1.362 (2) | C7—H7C | 0.9800 |
| N1—C6 | 1.359 (2) | C7—C6 | 1.498 (2) |
| N1—H1 | 0.871 (7) | C6—C5 | 1.367 (2) |
| C3—H3 | 0.9500 | C1—H1A | 0.9800 |
| C3—C2 | 1.370 (2) | C1—H1B | 0.9800 |
| C3—C4 | 1.422 (3) | C1—H1C | 0.9800 |
| C2—C1 | 1.498 (2) | C5—H5 | 0.9500 |
| C4—C5 | 1.428 (2) | | |
| | | | |
| C2—N1—H1 | 116.8 (15) | C6—C7—H7A | 109.5 |
| C6—N1—C2 | 122.02 (14) | C6—C7—H7B | 109.5 |
| C6—N1—H1 | 120.9 (16) | C6—C7—H7C | 109.5 |
| C2—C3—H3 | 119.5 | N1—C6—C7 | 116.70 (14) |
| C2—C3—C4 | 121.06 (16) | N1—C6—C5 | 119.79 (15) |
| C4—C3—H3 | 119.5 | C5—C6—C7 | 123.49 (16) |
| N1—C2—C3 | 119.80 (16) | C2—C1—H1A | 109.5 |
| N1—C2—C1 | 116.63 (15) | C2—C1—H1B | 109.5 |
| C3—C2—C1 | 123.57 (16) | C2—C1—H1C | 109.5 |
| O1—C4—C3 | 122.52 (15) | H1A—C1—H1B | 109.5 |
| O1—C4—C5 | 121.34 (16) | H1A—C1—H1C | 109.5 |
| C3—C4—C5 | 116.14 (15) | H1B—C1—H1C | 109.5 |
| H7A—C7—H7B | 109.5 | C4—C5—H5 | 119.4 |
| H7A—C7—H7C | 109.5 | C6—C5—C4 | 121.12 (16) |
| H7B—C7—H7C | 109.5 | C6—C5—H5 | 119.4 |
| | | | |
| O1—C4—C5—C6 | −179.53 (17) | C2—C3—C4—C5 | −2.6 (3) |
| N1—C6—C5—C4 | 1.2 (3) | C4—C3—C2—N1 | 2.5 (3) |
| C3—C4—C5—C6 | 0.7 (3) | C4—C3—C2—C1 | −176.66 (16) |
| C2—N1—C6—C7 | 177.28 (17) | C7—C6—C5—C4 | −177.33 (17) |
| C2—N1—C6—C5 | −1.4 (3) | C6—N1—C2—C3 | −0.5 (3) |
| C2—C3—C4—O1 | 177.66 (16) | C6—N1—C2—C1 | 178.74 (17) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2···O1 | 0.86 (1) | 1.96 (1) | 2.8174 (17) | 173 (2) |
| N1—H1···O1$^{i}$ | 0.87 (1) | 1.86 (1) | 2.7154 (18) | 166 (3) |

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