

Fig. 1. ORTEP drawing of the title compound with 50% probability ellipsoids. $R_1 = \text{COCH}_3$, $R_2 = \text{COOCH}_3$.

1970; B. A. Frenz & Associates, Inc., 1989) of the molecule and the atomic labeling scheme.

Related literature. The crystal structure of a 7-azanorbornadiene derivative-Fe complex compound has been reported by Sun, Chow & Liu (1990).

These studies are supported by the National Science Council (NSC80-0208-M031-05) of Taiwan.

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Acta Cryst. (1991). **C47**, 2461-2463

Structure of Potassium 2-Pyridonide Monohydrate

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(Received 14 January 1991; accepted 11 June 1991)

Abstract. $\text{K}^+ \cdot \text{C}_5\text{H}_4\text{NO}^- \cdot \text{H}_2\text{O}$, $M_r = 151.20$, orthorhombic, $Pbcn$, $a = 6.2727$ (21), $b = 7.0732$ (10), $c = 28.502$ (10) Å, $V = 1265$ Å³, $Z = 8$, $D_x = 1.588$ Mg m⁻³, $\bar{\lambda}(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.752$ mm⁻¹, $F(000) = 624$, $T = 120.0$ (1) K, $R = 0.0274$ for 910 unique observed reflections. The structure comprises hydrogen-bonded planes of K^+ ions and water molecules to which the pyridonide anions are hydrogen-bonded orthogonally on each side, resulting in a structure which has alternating hydrophilic and hydrophobic zones.

Experimental. Title compound prepared by reaction of aqueous solutions of KOH and 2-pyridone, crystals obtained by recrystallization from *n*-propanol/diethyl ether. Colourless plate, $0.12 \times 0.35 \times 0.74$ mm, Stoe STADI-4 four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, Oxford Cryosystems low-temperature device (Cosier & Glazer, 1986), cell parameters from 2θ values of 21

reflections measured at $\pm\omega$ ($30 < 2\theta < 32^\circ$). For data collection at $T = 120$ K, ω scans with scan width $(1.32 + 0.35 \tan \theta)^\circ$, $2\theta_{\text{max}} = 50^\circ$, $h 0 \rightarrow 7$, $k 0 \rightarrow 8$, $l 0 \rightarrow 33$, no significant crystal movement or decay, no absorption correction, 1369 unique reflections, giving 910 with $F > 4\sigma(F)$. Structure solution from a Patterson synthesis (K) followed by iterative cycles of least-squares refinement and difference Fourier synthesis, and refinement using full-matrix least-squares on F (*SHELX76*; Sheldrick, 1976). Anisotropic thermal parameters for all non-H atoms, H atoms refined freely with individual isotropic thermal parameters, secondary-extinction parameter refined to $3.7(12) \times 10^{-7}$. At final convergence, $R = 0.0274$, $wR = 0.0475$, $S = 1.339$ for 107 parameters, $(\Delta/\sigma)_{\text{max}}$ in final cycle 0.04, max. and min. $\Delta\rho$ in final ΔF synthesis 0.27, -0.31 e Å⁻³ respectively. The weighting scheme $w^{-1} = \sigma^2(F) + 0.00415F^2$ gave satisfactory agreement analyses. Scattering factors were inlaid (*SHELX76*; Sheldrick, 1976) except for K (Cromer & Mann, 1968). Atomic coordinates and equivalent isotropic thermal parameters are given in

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Table 1. Atomic coordinates with *e.s.d.*'s

	x	y	z	U_{iso} (Å ²)
K	0.62790 (5)	-0.33813 (7)	0.304760 (10)	0.0130 (4)
N(1)	0.58836 (22)	0.31467 (25)	0.37182 (6)	0.0140 (8)
C(2)	0.49324 (25)	0.1324 (3)	0.36174 (6)	0.0115 (9)
O(2)	0.48914 (18)	0.06976 (22)	0.31822 (4)	0.0138 (7)
C(3)	0.4033 (3)	0.0153 (3)	0.39801 (7)	0.0155 (9)
C(4)	0.4145 (3)	0.0849 (3)	0.44350 (7)	0.0173 (10)
C(5)	0.5138 (3)	0.2709 (3)	0.45382 (7)	0.0179 (10)
C(6)	0.5965 (3)	0.3780 (3)	0.41701 (7)	0.0162 (10)
O(1W)	0.30371 (20)	0.40182 (23)	0.27050 (5)	0.0162 (7)
H(3)	0.340 (4)	-0.105 (4)	0.3881 (8)	0.030 (7)
H(4)	0.353 (4)	-0.001 (4)	0.4671 (8)	0.027 (6)
H(5)	0.526 (3)	0.319 (3)	0.4839 (8)	0.018 (6)
H(6)	0.667 (4)	0.504 (4)	0.4220 (9)	0.028 (6)
H(1W)	0.350 (5)	0.297 (6)	0.2822 (11)	0.062 (12)
H(2W)	0.224 (3)	0.448 (4)	0.2922 (8)	0.018 (5)

Table 1, selected bond lengths and angles appear in Table 2 and the environment of the K⁺ ion is described in Table 3.* The atom-numbering scheme is shown in Fig. 1, which was generated using *ORTEP* (Mallinson & Muir, 1985). The packing diagrams in Fig. 2 were produced from *PLUTO* (Motherwell & Clegg, 1978). Molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

Related literature. 2-Pyridonide and its substituted analogues have been used as binucleating bridging ligands in multitudinous metal dimers (Cotton & Walton, 1982). It has also been characterized as a trinucleating ligand (Goodgame, Williams & Winpenny, 1989) and in the protonated form it acts

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54339 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å), angles (°) and torsion angles (°) with *e.s.d.*'s

N(1)—C(2)	1.3576 (24)	C(4)—C(5)	1.394 (3)
N(1)—C(6)	1.349 (3)	C(5)—H(5)	0.913 (22)
C(2)—O(2)	1.3014 (22)	C(5)—C(6)	1.376 (3)
C(2)—C(3)	1.419 (3)	C(6)—H(6)	0.94 (3)
C(3)—H(3)	0.92 (3)	O(1W)—H(1W)	0.81 (4)
C(3)—C(4)	1.370 (3)	O(1W)—H(2W)	0.888 (23)
C(4)—H(4)	0.96 (3)		
C(2)—N(1)—C(6)	118.11 (16)	H(4)—C(4)—C(5)	123.1 (15)
N(1)—C(2)—O(2)	117.83 (16)	C(4)—C(5)—H(5)	121.5 (14)
N(1)—C(2)—C(3)	120.27 (16)	C(4)—C(5)—C(6)	117.52 (18)
O(2)—C(2)—C(3)	121.89 (16)	H(5)—C(5)—C(6)	120.9 (14)
C(2)—C(3)—H(3)	114.6 (16)	N(1)—C(6)—C(5)	124.47 (18)
C(2)—C(3)—C(4)	119.91 (18)	N(1)—C(6)—H(6)	114.3 (17)
H(3)—C(3)—C(4)	125.5 (16)	C(5)—C(6)—H(6)	121.2 (17)
C(3)—C(4)—H(4)	117.2 (15)	H(1W)—O(1W)—H(2W)	103.6 (29)
C(3)—C(4)—C(5)	119.70 (18)		
C(6)—N(1)—C(2)—O(2)	-178.64 (16)	H(3)—C(3)—C(4)—H(4)	1.2 (26)
C(6)—N(1)—C(2)—C(3)	0.9 (3)	H(3)—C(3)—C(4)—C(5)	-179.7 (19)
C(2)—N(1)—C(6)—C(5)	-0.4 (3)	C(3)—C(4)—C(5)—H(5)	-178.2 (16)
C(2)—N(1)—C(6)—H(6)	178.5 (18)	C(3)—C(4)—C(5)—C(6)	0.4 (3)
N(1)—C(2)—C(3)—H(3)	179.0 (17)	H(4)—C(4)—C(5)—H(5)	0.9 (24)
N(1)—C(2)—C(3)—C(4)	-0.8 (3)	H(4)—C(4)—C(5)—C(6)	179.5 (18)
O(2)—C(2)—C(3)—H(3)	-1.5 (18)	C(4)—C(5)—C(6)—N(1)	-0.3 (3)
O(2)—C(2)—C(3)—C(4)	178.68 (18)	C(4)—C(5)—C(6)—H(6)	-179.1 (19)
C(2)—C(3)—C(4)—H(4)	-179.0 (17)	H(5)—C(5)—C(6)—N(1)	178.3 (16)
C(2)—C(3)—C(4)—C(5)	0.2 (3)	H(5)—C(5)—C(6)—H(6)	-0.5 (25)

Table 3. Coordination environment of the potassium ion

Errors are 0.003 Å on distances and 0.05° on angles.

(1)	N(1)	$x, -1+y, z$	2.911
(2)	N(1)	$\frac{1}{2}-x, -\frac{1}{2}+y, z$	2.932
(3)	O(2)	x, y, z	2.767
(4)	O(2)	$\frac{1}{2}-x, -\frac{1}{2}+y, z$	2.796
(5)	O(1W)	$x, -1+y, z$	2.979
(6)	O(1W)	$1-x, -1+y, \frac{1}{2}-z$	2.738
(7)	O(1W)	$\frac{1}{2}+x, -\frac{1}{2}+y, \frac{1}{2}-z$	2.900
(1)—K—(2)	83.25	(3)—K—(6)	136.24
(1)—K—(5)	74.43	(4)—K—(6)	79.23
(2)—K—(3)	81.38	(5)—K—(7)	111.82
(2)—K—(6)	125.76	(1)—K—(4)	81.28
(3)—K—(5)	106.19	(1)—K—(7)	156.20
(4)—K—(5)	132.65	(2)—K—(5)	156.86
(5)—K—(6)	63.44	(3)—K—(4)	121.04
(1)—K—(3)	124.52	(3)—K—(7)	76.99
(1)—K—(6)	94.90	(4)—K—(7)	78.07
(2)—K—(4)	46.77	(6)—K—(7)	69.76
(2)—K—(7)	91.08		

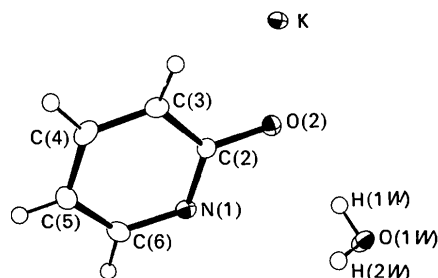


Fig. 1. A general view showing atom-numbering scheme: thermal ellipsoids are drawn at the 30% probability level.

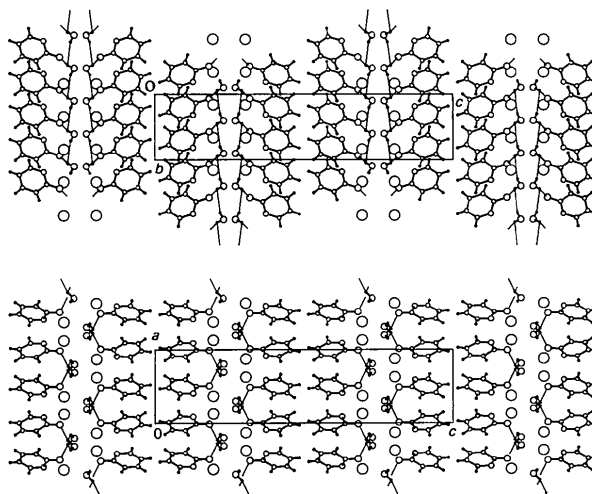


Fig. 2. Two views illustrating the hydrogen-bonded network and the alternation of hydrophilic and hydrophobic zones in the crystal.

as a monodentate oxygen donor (Blake, Gould & Winpenny, 1991).

We thank the SERC for a research studentship (PEYM) and for funding towards the purchase of a diffractometer.

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Acta Cryst. (1991). **C47**, 2463–2465

Structure of *trans*-Perhydro-4,1-benzoxazepin-2-one

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(Received 9 December 1990; accepted 9 May 1991)

Abstract. $C_9H_{15}NO_2$, $M_r = 169.225$, monoclinic, $P2_1/n$, $a = 12.148$ (5), $b = 5.2735$ (9), $c = 14.559$ (4) Å, $\beta = 106.48$ (3)°, $V = 894.4$ (5) Å³, $Z = 4$, $D_x = 1.256$ Mg m⁻³, m.p. 430–431 K, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 0.52$ cm⁻¹, $F(000) = 368$, $T = 293$ K, final $R = 0.082$ for 842 reflections with $F > 2\sigma(F)$. The bond lengths and angles are normal. There is an intermolecular hydrogen bond N(1)—H···O(10): N(1)···O(10) = 2.943 (6), H···O(10) = 1.999 Å, N(1)—H···O(10) = 144.4 (3)°. Conformational analysis of the cyclohexane ring indicates a nearly ideal chair conformation ¹C₄. The 4,1-benzoxazepine hetero-ring also adopts a chair conformation.

Experimental. Diffraction measurements were carried out using a Nicolet R3m/E automatic diffractometer, with graphite-monochromated Mo $K\alpha$ radiation. A crystal of dimensions 0.12 × 0.17 × 0.44 mm was used for the X-ray data collection at 293 K. The lattice parameters and orientation matrix were obtained from 25 reflections in the range $2 < 2\theta < 32^\circ$. Intensity data were measured by the ω - 2θ scan technique, scan width 2°, scan speed to 29.3° min⁻¹(max.). Check reflections 404 and 101 were monitored every 100 reflections. 1538 unique reflections were measured [842 with $F > 2\sigma(F)$]. h, k, l

range: 0 to 13, 0 to 16 and -16 to 16, $\theta_{\text{max}} = 25^\circ$. Neither absorption nor extinction corrections were applied. Structures were solved by using *SHELXTL* (Sheldrick, 1984) and refined by *SHELX76* (Sheldrick, 1976). Full-matrix refinement, $\sum w(\Delta F)^2$ minimized, anisotropic non-H atoms. Positions of H atoms were generated from the assumed geometries, checked in Fourier maps and refined in the riding mode with an overall isotropic temperature factor [final value 0.079 (4) Å²] (110 parameters). Final $R = 0.082$, $wR = 0.069$, $w = [\sigma^2(F) + 9 \times 10^{-4}(F)^2]^{-1}$. The value of the R factor reflects the poor quality of the crystal sample. Difference Fourier maps showed no significant peaks (maximum 0.34, minimum -0.28 e Å⁻³). Maximum Δ/σ is 0.189. Scattering factors were taken as in *SHELX76*. Calculations were performed on the Nicolet R3m/E diffractometer system with structure solution package Nicolet *SHELXTL* and on an IBM 43/41 computer.

The final atomic coordinates for non-H atoms are listed in Table 1.* Fig. 1 shows a perspective view of

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54249 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.