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3-Hydroxypyridinium-1-acetate dihydrate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.113
Data-to-parameter ratio = 14.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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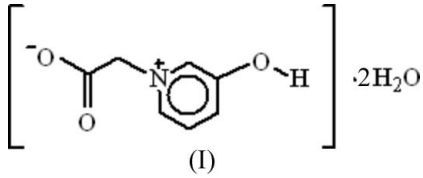
The title compound, $C_7H_7NO_3\cdot 2H_2O$, exists as a betaine. The O atoms of the hydroxy and carboxylate groups of 3-hydroxypyridinium-1-acetate interact with water molecules to form a three-dimensional hydrogen-bonded supramolecular network.

Comment

Pyridine betaine and its derivatives, as zwitterions containing anionic carboxylate groups and positively charged pyridinium groups, are good proton acceptors and easily form acceptor hydrogen bonds with water molecules and hydrohalic acids (Wu & Mak, 1995). The crystal structures of some pyridine betaine compounds have been determined to elucidate the interaction of hydrogen bonds (Chen & Mak, 1990, 1991; Buczak *et al.*, 1997; Gao *et al.*, 2004). Recent studies have shown that a hydroxy-substituted pyridine betaine, which crystallizes as 1-carboxymethyl-3-hydroxypyridinium chloride–3-hydroxypyridinium-1-acetate (1/1) (Zhao *et al.*, 2004), exists in either a zwitterionic or an uncharged configuration and involves a hydrogen-bonded chain. Our interest has been directed toward the synthesis of a metal complex based on 3-hydroxypyridinium-1-acetate; however, the reaction yielded the title organic compound, (I), whose crystal structure is reported here.



(I)

As shown in Fig. 1, the title compound, (I), also exists as a zwitterion. The O1—C1 and O2—C1 bond lengths are 1.2475 (18) and 1.230 (2) Å, respectively, suggesting electron delocalization (Table 1). The pyridine ring, in which bond lengths are nearly equal, is aromatic. The carboxylate group (O1—C7—O2) is twisted out of the attached pyridine ring plane, with a dihedral angle of 78.7 (3)°. The carboxylate and hydroxy groups and the water molecules (O1*W* and O2*W*) form a three-dimensional supramolecular framework *via* intermolecular hydrogen bonds (Table 2 and Fig. 2).

Experimental

Zinc acetate dihydrate (3.00 g, 15 mmol) was added to an aqueous solution of 1-carboxymethyl-3-hydroxypyridinium chloride–3-hydroxypyridinium-1-acetate (1/1) (6.86 g, 20 mmol). The mixture was stirred for 0.5 h and then filtered. Colorless crystals of (I) sepa-

rated from the solution over a period of several days. Analysis calculated for $C_7H_{11}NO_5$: C 44.45, H 5.86, N 7.40%; found: C 44.49, H 5.84, N 7.41%.

Crystal data

$C_7H_7NO_3\cdot 2H_2O$	$Z = 2$
$M_r = 189.17$	$D_x = 1.446$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 6.9055$ (14) Å	Cell parameters from 3575 reflections
$b = 7.0086$ (14) Å	$\theta = 3.2$–$27.5°$
$c = 10.074$ (2) Å	$\mu = 0.12$ mm^{-1}
$\alpha = 71.62$ (3)°	$T = 295$ (2) K
$\beta = 76.09$ (3)°	Prism, colorless
$\gamma = 71.93$ (3)°	0.32 × 0.25 × 0.18 mm
$V = 434.34$ (18) Å^3	

Data collection

Rigaku R-AXIS RAPID diffractometer	1964 independent reflections
ω scans	1540 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	$R_{int} = 0.013$
$T_{min} = 0.961$, $T_{max} = 0.978$	$\theta_{max} = 27.5°$
4289 measured reflections	$h = -8 \rightarrow 7$
	$k = -9 \rightarrow 9$
	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 0.076P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.040$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.113$	$\Delta\rho_{max} = 0.28$ e Å^{-3}
$S = 1.09$	$\Delta\rho_{min} = -0.22$ e Å^{-3}
1964 reflections	
133 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.2475 (18)	C1—C2	1.525 (2)
O2—C1	1.230 (2)	C3—C4	1.3834 (19)
O3—C4	1.3301 (17)	C4—C5	1.385 (2)
N1—C2	1.4746 (17)	C5—C6	1.376 (2)
N1—C3	1.3369 (17)	C6—C7	1.370 (2)
N1—C7	1.3422 (19)		
O1—C1—C2	114.54 (13)	N1—C7—C6	119.14 (13)
O2—C1—O1	126.86 (14)	C3—N1—C7	121.76 (12)
O2—C1—C2	118.59 (13)	C3—N1—C2	118.20 (12)
O3—C4—C3	116.58 (13)	C3—C4—C5	118.65 (12)
O3—C4—C5	124.77 (13)	C6—C5—C4	118.90 (13)
N1—C2—C1	112.77 (11)	C7—N1—C2	120.00 (12)
N1—C3—C4	120.64 (13)	C7—C6—C5	120.90 (14)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1*W*—H1*W*1···O2^{i}	0.86 (2)	1.87 (2)	2.704 (2)	164 (2)
O1*W*—H1*W*2···O1^{ii}	0.87 (2)	2.18 (2)	2.9147 (18)	143 (2)
O1*W*—H1*W*2···O2^{ii}	0.87 (2)	2.41 (2)	3.239 (2)	159 (2)
O2*W*—H2*W*1···O1*W*^{iii}	0.86 (2)	1.87 (2)	2.725 (2)	178 (2)
O2*W*—H2*W*2···O1^{iv}	0.86 (2)	1.94 (3)	2.785 (2)	168 (2)
O3—H8···O2*W*	0.86 (1)	1.72 (2)	2.5764 (19)	177 (2)

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

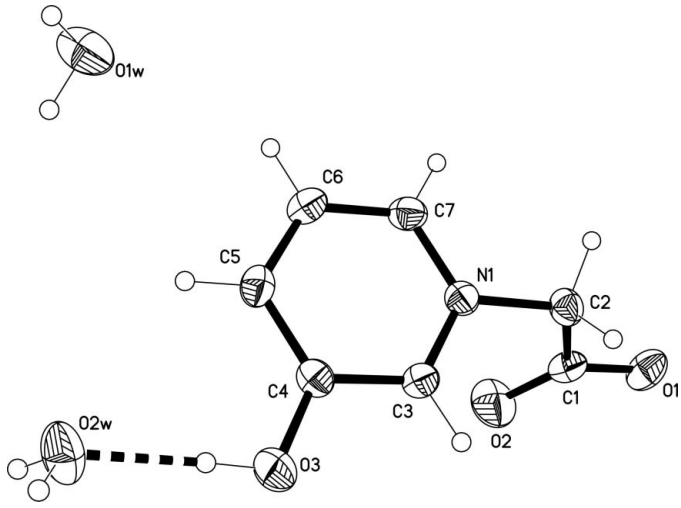


Figure 1
The atom-numbering scheme for (I), showing displacement ellipsoids at the 30% probability level. One hydrogen bond is shown as a dashed line.

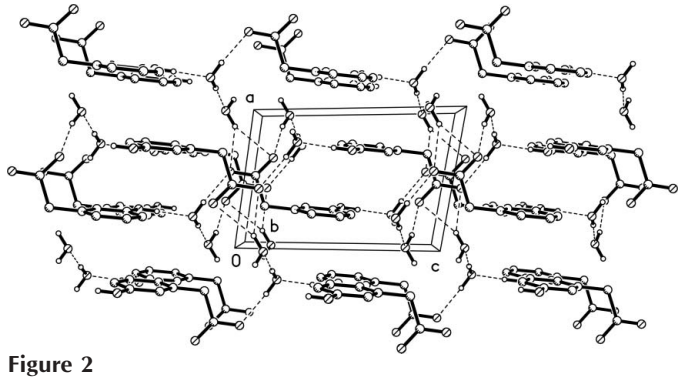


Figure 2
A plot of the hydrogen-bonding network of (I). Hydrogen bonds are shown as dashed lines.

The H atoms of the water molecules and hydroxy group were located in a difference map and refined with O—H and H···H distances restrained to 0.85 (1) and 1.39 (1) Å, respectively, and with $U_{iso}(H) = 1.5U_{eq}(O)$. All other H atoms were placed in calculated positions, with aromatic C—H = 0.93 Å and aliphatic C—H = 0.97 Å, and were included in the refinement using the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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