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Hexaaquazinc(II) bis[(4-oxo-4*H*-pyridin-1-yl)-acetate] dihydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.027
wR factor = 0.075
Data-to-parameter ratio = 15.5

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

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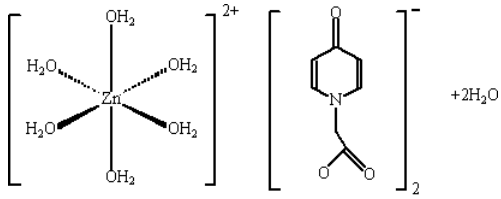
The title complex, $[Zn(H_2O)_6](C_7H_6NO_3)_2\cdot 2H_2O$, was synthesized and characterized by X-ray crystallography. The zinc(II) ion, which lies on a center of symmetry, is coordinated by six water molecules, forming an octahedron. A three-dimensional supramolecular framework is formed *via* hydrogen bonds between the anions and cations.

Comment

There is interest in metal complexes containing nitrogen-heterocycle carboxylic acid ligands (Kondo *et al.*, 2002; Premkumar & Govindarajan, 2003). (4-Oxo-4*H*-pyridin-1-yl)acetic acid, an important medical intermediate (Edwards *et al.*, 1977), is a multidentate ligand with a versatile binding mode, but there is little information available about the structure of its metal complexes. The reaction of (4-oxo-4*H*-pyridin-1-yl)acetic acid and zinc(II) acetate yielded a new zinc^{II} complex, $[Zn(H_2O)_6](C_7H_6NO_3)_2\cdot 2H_2O$, (I), the crystal structure of which is reported here, and in which the organic anion does not act as a ligand.



(I)

As shown in Fig. 1, the asymmetric unit of (I) consists of one-half of a $[Zn(H_2O)_6]^{2+}$ cation, a (4-oxo-4*H*-pyridin-1-yl)-acetate anion and an uncoordinated water molecule. In the cation, the zinc^{II} atom occupies an inversion site and is coordinated by six water molecules in an octahedral geometry. The Zn—O bond lengths range from 2.076 (1) to 2.096 (1) Å (Table 1). The cation interacts with the uncoordinated water

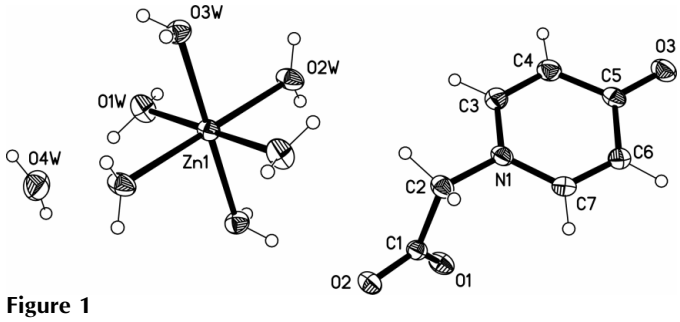


Figure 1
The components of the title compound, with 30% probability displacement ellipsoids.

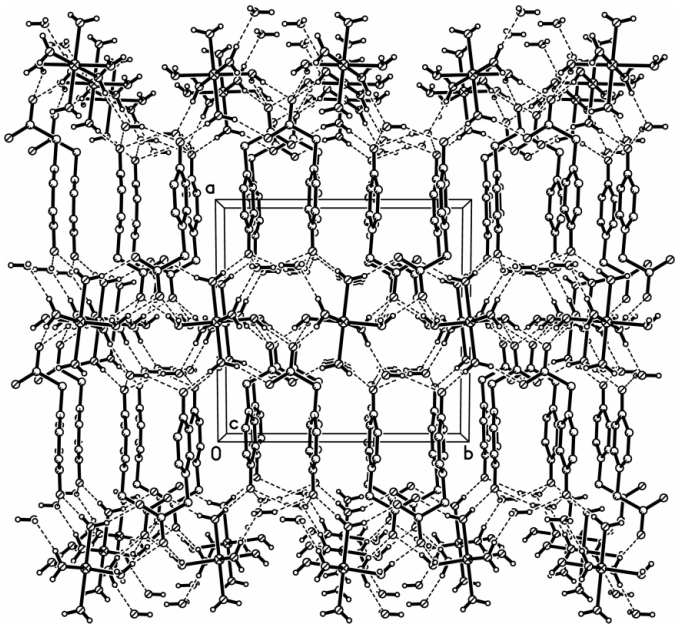


Figure 2
Packing diagram of the complex, viewed along the *c* axis. Hydrogen bonds are shown as dashed lines.

molecules *via* intermolecular hydrogen bonds (Table 2). The carboxy group and pyridine ring in the (4-oxo-4*H*-pyridin-1-yl)acetate anion are not coplanar, and the N1—C2—C1 bond angle is 114.0 (1)°. The C3—C4, C6—C7 and C5—O3 bond lengths are 1.359 (2), 1.356 (2) and 1.276 (2) Å, respectively. The structure can be envisaged as one in which layers of anions alternate with layers of cations (Fig. 2), and the layers are linked by hydrogen bonds, giving rise to a three-dimensional supramolecular network.

Experimental

The title complex was prepared by the addition of $Zn(CH_3COO)_2{\cdot}2H_2O$ (4.40 g, 20 mmol) to an aqueous solution of (4-oxo-4*H*-pyridin-1-yl)acetic acid (58.40 g, 40 mmol). The pH was adjusted to 7 with 0.2 M NaOH solution. Colorless single crystals were obtained from the filtered solution over a period of several days. Analysis calculated for $C_{14}H_{28}N_2O_{14}Zn$: C 32.73, H 5.49, N 5.45%; found: C 32.95, H 5.60, N 5.29%.

Crystal data

$[Zn(H_2O)_6](C_7H_6NO_3)_2{\cdot}2H_2O$
M_r = 513.75
Monoclinic, $P2_1/c$
a = 12.427 (3) Å
b = 12.833 (3) Å
c = 6.7816 (14) Å
β = 98.62 (3)°
V = 1069.3 (4) Å^3
Z = 2
D_x = 1.596 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8214 reflections
θ = 3.2–27.5°
μ = 1.22 mm^{-1}
T = 293 (2) K
Prism, colorless
0.31 × 0.25 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.703, T_{max} = 0.810
9929 measured reflections
2447 independent reflections
2251 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 27.5°
$h = -16 \rightarrow 16$
$k = -16 \rightarrow 16$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.075
S = 1.05
2447 reflections
158 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.3156P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.040 (2)

Table 1
Selected geometric parameters (Å, °).

Zn1—O3*W*	2.076 (1)	O2—C1	1.262 (2)
Zn1—O2*W*	2.077 (1)	O3—C5	1.276 (2)
Zn1—O1*W*	2.096 (1)	C3—C4	1.359 (2)
O1—C1	1.242 (2)	C6—C7	1.356 (2)
O3*W*—Zn1—O2*W*^{v}	91.52 (5)	O3*W*—Zn1—O1*W*	89.07 (5)
O3*W*—Zn1—O2*W*	88.48 (5)	O2*W*—Zn1—O1*W*	92.22 (6)
O2*W*—Zn1—O1*W*^{v}	87.78 (6)	N1—C2—C1	114.0 (1)
O3*W*^{v}—Zn1—O1*W*	90.93 (5)		

Symmetry code: (v) $1-x, -y, -z$.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H8*A*···O4*W*	0.82	1.93	2.720 (2)	161
O1*W*—H8*B*···O2^{i}	0.82 (2)	2.04 (1)	2.836 (2)	166 (2)
O2*W*—H9*A*···O1^{ii}	0.82	1.89	2.694 (2)	164
O2*W*—H9*B*···O3^{iii}	0.80 (2)	1.94 (1)	2.730 (2)	177 (2)
O3*W*—H10*A*···O2^{iv}	0.82	1.88	2.690 (2)	170
O3*W*—H10*B*···O2^{v}	0.84 (2)	2.09 (1)	2.837 (2)	147 (1)
O4*W*—H11*B*···O3^{vi}	0.84 (2)	2.00 (1)	2.794 (2)	159 (2)
O4*W*—H11*A*···O3^{vii}	0.83 (2)	2.20 (1)	2.980 (2)	157 (2)

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

Carbon-bound H atoms were placed in calculated positions, with C—H = 0.93 and 0.97 Å, and U_{iso}(H) = 1.2U_{eq}(C), and were included in the refinement in the riding-model approximation. Water atoms H8*A*, H9*A* and H10*A* were located in difference Fourier synthesis maps and refined with a riding model, with the O—H distances restrained to 0.82 (1) Å and U_{iso}(H) = 1.5U_{eq}(O).

Data collection: *RAPID-AUTO* (Rigaku Corporation, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXL*97; software used to prepare material for publication: *SHELXL*97.

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