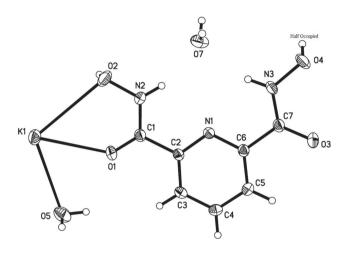
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Jian Zuo*

Crystal structure of *catena*-poly[diaqua-bis(di(N^2 , N^6 -dihydroxy-pyridine-2,6-dicarboxamide))potassium(I)]tetrahydrate, $C_{14}H_{25}N_6O_{14}K$



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Abstract

C₁₄H₂₅N₆O₁₄K, triclinic, $P\bar{1}$ (no. 2), a = 6.9630(15) Å, b = 9.2770(17) Å, c = 9.6100(18) Å, $\alpha = 69.822(2)^\circ$, $\beta = 87.225(2)^\circ$, $\gamma = 85.559(2)^\circ$, V = 580.8(2) Å³, Z = 1, $R_{\rm gt}(F) = 0.0366$, $wR_{\rm ref}(F^2) = 0.1052$, T = 293(2) K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless block	
Size:	$0.52\times0.48\times0.11~\text{mm}$	
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)	
μ :	$0.31 \ \text{mm}^{-1}$	
Diffractometer, scan mode:	Bruker APEX-II, $oldsymbol{arphi}$ and $oldsymbol{\omega}$ -scans	
$ heta_{ ext{max}}$, completeness:	25°, >99%	
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	2977, 2006, 0.013	
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{\rm obs} > 2 \ \sigma(I_{\rm obs})$, 1598	
N(param) _{refined} :	167	
Programs:	Bruker programs [1], SHELX [2, 3]	

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\mathring{A}^2).

Atom	х	у	Z	U _{iso} */U _{eq}
K1	0.0000	0.0000	1.0000	0.0457(2)
N1	0.2659(2)	0.50644(17)	0.44865(16)	0.0279(4)
N2	0.2144(3)	0.21064(18)	0.60315(18)	0.0383(4)
H2	0.2534	0.2431	0.5120	0.046*
N3	0.3508(3)	0.57664(19)	0.16023(17)	0.0382(4)
Н3	0.3462	0.4845	0.2226	0.046*
01	0.1590(2)	0.26057(16)	0.81500(15)	0.0471(4)
02	0.1690(3)	0.05813(16)	0.67222(17)	0.0521(4)
H2A	0.2643	0.0126	0.7186	0.078*
03	0.3412(2)	0.83185(15)	0.12336(15)	0.0422(4)
04	0.3809(3)	0.6006(2)	0.01137(16)	0.0504(5)
H4	0.5000	0.5000	0.0000	0.112(18)
05	-0.0111(2)	0.2782(2)	1.07787(16)	0.0573(5)
H5A	-0.1223	0.3229	1.0552	0.086*
H5B	0.0600	0.3384	1.0124	0.086*
06	0.4822(2)	0.92820(16)	0.83050(15)	0.0474(4)
H6A	0.4434	0.8701	0.9150	0.071*
H6B	0.5376	1.0003	0.8446	0.071*
07	0.2657(2)	0.25409(18)	0.28948(16)	0.0524(4)
H7A	0.1676	0.2581	0.2387	0.079*
H7B	0.3536	0.1989	0.2637	0.079*
C1	0.1965(3)	0.3040(2)	0.6806(2)	0.0319(4)
C2	0.2274(3)	0.4698(2)	0.59373(19)	0.0278(4)
C3	0.2111(3)	0.5763(2)	0.6655(2)	0.0401(5)
H3A	0.1868	0.5457	0.7673	0.048*
C4	0.2316(4)	0.7281(2)	0.5831(2)	0.0471(6)
H4A	0.2200	0.8025	0.6281	0.056*
C5	0.2699(3)	0.7691(2)	0.4324(2)	0.0399(5)
H5	0.2833	0.8715	0.3743	0.048*
C6	0.2877(3)	0.6550(2)	0.3699(2)	0.0293(4)
C7	0.3293(3)	0.6940(2)	0.2069(2)	0.0319(4)

Source of materials

Hydroxylamine hydrochloride (0.695 g, 10 mmol) was added to potassium hydroxide (0.841 g, 15 mmol) in deionized water (10 mL). The solution was then added dropwise to dimethyl 2,6-pyridinedicarboxylate (0.976 g, 5 mmol) in methanol (10 mL). The reaction was stirred for 24 h. The resulting colourless solution was allowed to evaporate slowly at room temperature for a week, yielding colourless block crystals.

Experimental details

All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were assigned with common isotropic displacement factors $U_{\rm iso}({\rm H}) = 1.2$ times $U_{\rm eq}$ (C, pyridyl),

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and $U_{\rm iso}({\rm H}) = 1.5$ times $U_{\rm eq}$ (O, water), (N, imino group) and refined as riding on their parent atom.

Comment

Hydroxamic acids have a high binding affinity to a range of metal ions, and are ligands ubiquitous in coordination chemistry and chemical biology [4]. The dominant coordination mode in metal-hydroxamic acid complexes is the 0.0'bidentate chelate in which the ligand is either singly deprotonated (hydroxamato) or doubly deprotonated (hydroximato) [5]. Studies of hydroxamic acids continue to reveal potential uses in heavy metal extraction procedures and nuclear fuel reprocessing, in materials chemistry and in biomedical applications [6]. Many hydroxamic acid transition metal complexes have been reported. The title compound is one of the rare examples in the literature in which hydroxamic acids have been reacted with alkali metal [7].

The asymmetric unit of the title structure contains one half of potassium ion, one 2,6-picolinedihydroxamic acid ligand, one coordinated water molecule and two more water molecules. In the asymmetric unit, potassium is three-coordinated by two O atoms from one 2,6picolinedihydroxamic acid ligand and one coordinated water O atom. Among them, the K-O bond lengths range from 2.7367(14) Å to 3.1880(17) Å. Bond lengths are in the expected ranges [8–11]. K atoms are linked by 2,6-picolinedihydroxamic acid into one-dimensional chains.

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