Li-ping Xue*

Crystal structure of *catena*-poly[tetraaqua(μ_2 -4-(1*H*-imidazol-1-yl)pyridine- κ^2 N:N')]zinc(II)] thiophene-2,5-dicarboxylate, $C_{14}H_{17}N_3O_8ZnS$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.38 × 0.31 × 0.29 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.64 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω -scans
$\theta_{\max}$, completeness:	25°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8683, 2935, 0.024
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2757
$N(\text{param})_{\text{refined}}$:	251
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], OLEX2 [4]

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Abstract

$C_{14}H_{17}N_3O_8ZnS$, triclinic, $P\bar{1}$ (no. 2), $a = 6.3962(5)$ Å, $b = 6.9439(7)$ Å, $c = 20.4208(19)$ Å, $\alpha = 93.040(8)^\circ$, $\beta = 96.337(7)^\circ$, $\gamma = 110.970(8)^\circ$, $V = 837.54(13)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0390$, $wR_{\text{ref}}(F^2) = 0.0769$, $T = 288(10)$ 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.

Source of material

A mixture of $Zn(OAc)_2 \cdot 2H_2O$ (22.0 mg, 0.1 mmol), 2,5-thiophenedicarboxylic acid (17.2 mg, 0.1 mmol), 4-imidazol-1-yl-pyridine (14.5 mg, 0.05 mmol) and 5 mL distilled water in a 25 mL Teflon-lined autoclave was kept under autogenous pressure at 353 K for 3 days. After cooling to room temperature at a rate of 5 K h⁻¹, light yellow block crystals were collected

by filtration in 45% yield. Elemental analysis calculated for $C_{14}H_{17}N_3O_8ZnS$: C 37.11%, H 3.76%, N 9.28%; found C 37.15%, H 3.79%, N 9.22%.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Discussion

Coordination polymers built from metal ions with multifunctional organic ligands are an important class of functional materials with potential functionalities [5–8]. It is a long-term challenge to construct new structures with desired properties due to many complicated factors. Among, the reasonable selection of metal ions and the design of organic ligands are crucial to the construction and structural tuning of the complexes. Recently, many multicarboxylate or heterocyclic carboxylic acids are used for this purpose. The 2,5-thiophenedicarboxylic acid ligand (H_2L) with C_2 symmetry is a ligand bearing two carboxylate groups and may show versatile coordination modes, which makes it a useful bridge to construct coordination polymers [9–11].

The title compound, $C_{14}H_{17}N_3O_8ZnS$, was prepared under mild hydrothermal condition. The asymmetric unit of the title crystal structure consists of two half of Zn(II) ion, one L^{2-} anion, one 4-imidazol-1-yl-pyridine and four coordinated water molecules. Both Zn(II) ions, located on an inversion centres are octahedrally coordinated by two nitrogen atoms

*Corresponding author: Li-ping Xue, College of Food and Drug, Luoyang Normal University, Luoyang, Henan 471934, P.R. China, e-mail: lpxue@163.com

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Zn1	0.5000	0.5000	0.5000	0.02520(15)
Zn2	0.5000	0.5000	1.0000	0.02506(15)
O5	0.7031(4)	0.3207(4)	0.51899(11)	0.0292(5)
H1W	0.7130	0.2572	0.4834	0.044*
H2W	0.6467	0.2309	0.5452	0.044*
O6	0.7742(4)	0.7902(4)	0.53645(12)	0.0351(6)
H3W	0.7283	0.8633	0.5618	0.053*
H4W	0.8829	0.7683	0.5584	0.053*
O7	0.6560(4)	0.2833(4)	0.97156(11)	0.0302(6)
H5W	0.5557	0.1706	0.9531	0.045*
H6W	0.7114	0.2366	1.0040	0.045*
O8	0.2142(4)	0.2447(4)	1.01949(12)	0.0376(6)
H7W	0.2162	0.1319	1.0018	0.056*
H8W	0.2155	0.2370	1.0609	0.056*
N1	0.4211(5)	0.4627(4)	0.59765(13)	0.0261(6)
N2	0.2824(5)	0.4355(4)	0.79368(13)	0.0250(6)
N3	0.3416(5)	0.4556(4)	0.90277(12)	0.0271(6)
C7	0.2108(6)	0.3878(6)	0.61115(17)	0.0347(9)
H7	0.0936	0.3431	0.5760	0.042*
C8	0.1563(6)	0.3724(6)	0.67465(16)	0.0314(8)
H8	0.0061	0.3166	0.6818	0.038*
C9	0.3295(6)	0.4418(5)	0.72784(16)	0.0246(7)
C10	0.5500(6)	0.5177(5)	0.71469(16)	0.0283(8)
H10	0.6703	0.5630	0.7490	0.034*
C11	0.5877(6)	0.5247(6)	0.64961(16)	0.0308(8)
H11	0.7365	0.5751	0.6410	0.037*
C12	0.0748(6)	0.3913(6)	0.81494(17)	0.0332(8)
H12	−0.0637	0.3595	0.7886	0.040*
C13	0.1124(6)	0.4034(6)	0.88154(17)	0.0333(8)
H13	0.0014	0.3803	0.9092	0.040*
C14	0.4376(6)	0.4733(6)	0.84949(16)	0.0315(8)
H14	0.5919	0.5072	0.8491	0.038*
S1	0.86112(14)	0.10859(13)	0.26641(4)	0.0243(2)
O1	0.7375(4)	0.0484(4)	0.07198(11)	0.0313(6)
O2	1.0640(4)	0.1782(4)	0.13995(11)	0.0357(6)
O3	0.4102(4)	−0.0276(4)	0.38905(11)	0.0349(6)
O4	0.7843(4)	0.1399(4)	0.40980(11)	0.0391(6)
C1	0.8540(6)	0.1023(5)	0.12876(16)	0.0242(7)
C2	0.7241(5)	0.0717(5)	0.18644(15)	0.0208(7)
C3	0.4961(6)	0.0197(5)	0.18553(16)	0.0285(8)
H3	0.3940	−0.0042	0.1471	0.034*
C4	0.4349(6)	0.0066(5)	0.24949(16)	0.0258(7)
H4	0.2863	−0.0271	0.2576	0.031*
C5	0.6097(5)	0.0468(5)	0.29834(15)	0.0230(7)
C6	0.6025(6)	0.0521(5)	0.37171(16)	0.0277(8)

from two 4-imidazol-1-yl-pyridine and four oxygen atoms of coordinated water molecules, furnishing octahedral geometries. The title compound is a salt. The cationic substructure the [Zn(4-imidazol-1-yl-pyridine)(H₂O)₄]²⁺, is a 1-D chain in which the 4-imidazol-1-yl-pyridine is a polymeric bridging ligand. The separation distance of Zn⋯Zn is 10.2104(10) Å. The pyridyl ring and imidazol ring of the 4-imidazol-1-yl-pyridine ligands are not coplanar showing a dihedral angle of

3.6%. The thiophene-2,5-dicarboxylate anion is fully deprotonated but uncoordinated and only serves as a counterion. The bond lengths of Zn—O range from 2.118(2) Å to 2.171(2) Å which are in the normal range of Zn(II)-aqua complexes. The bond distances of Zn—N are 2.081(3), 2.117(3) Å, respectively. In crystal structure of the title compound, there are intermolecular or intramolecular H-bond interactions O(5)—H(1W)⋯O(4), O(5)—H(2W)⋯O(3^v), O(6)—H(3W)⋯O(3ⁱ), O(6)—H(4W)⋯O(4^{vi}), O(7)—H(5W)⋯O(1^v), O(7)—H(5W)⋯O(1ⁱⁱⁱ), O(8)—H(5W)⋯O(1^v), O(8)—H(5W)⋯O(2^{iv}) (symmetry code: (i) $-x+1, -y+1, -z+1$; (iii) $x, y, z+1$; (iv) $x-1, y, z+1$; (v) $-x+1, -y, -z+1$; (vi) $-x+2, -y+1, -z+1$) with O⋯O distances of 2.692, 2.801, 2.818, 2.774, 2.752, 2.805, 2.811 and 2.741 Å, respectively. Then the one dimensional chains are expanded into a three-dimensional structure by intermolecular H-bond interactions.

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