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Crystal structure of *catena*-poly[(4-hydroxyl-5-(methoxycarbonyl)thiophene-2-carboxylato- $\kappa^1 O$)-(μ_2 -piperazine-1,4-diylbis(pyridin-4-ylmethanone)- $\kappa^2 N:N'$)silver(I)] monohydrate, $C_{23}H_{23}AgN_4O_8S$

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Abstract

$C_{23}H_{23}AgN_4O_8S$, monoclinic, $P2_1/n$ (no. 14), $a = 8.5356(4)$ Å, $b = 27.9494(15)$ Å, $c = 10.3796(6)$ Å, $V = 2474.1(2)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0347$, $wR_{ref}(F^2) = 0.0744$, $T = 289$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.35 \times 0.29 \times 0.25$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.96 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
$\theta_{\max}$, completeness:	25.5° , 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	14,043, 4526, 0.026
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3860
$N(\text{param})_{\text{refined}}$:	339
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], Olex2 [4]

Source of material

All chemicals were used without further purification. The title compound was prepared under the hydrothermal conditions. A mixture of AgOAc (16.7 mg, 0.1 mmol),

methyl-3-hydroxy-5-carboxy-2-thiophenecarboxylate (H_2L) (20.2 mg, 0.1 mmol), piperazine-1,4-diylbis(pyridin-4-ylmethanone) (29.6 mg, 0.1 mmol) and 8 mL distilled water in a 25 mL L Teflon-lined autoclave was kept under autogenous pressure at 373 K for 3 days. After cooling to room temperature at a rate of 5 K h^{-1} , colourless block crystals were collected by filtration and washed with distilled water in 43% yield.

Experimental details

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

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
Ag1	0.23668 (3)	0.70745 (2)	0.69656 (2)	0.04102 (9)
S1	−0.26145 (8)	0.79146 (3)	0.51422 (7)	0.03459 (18)
O1	0.0101 (2)	0.74194 (9)	0.6123 (2)	0.0568 (6)
O2	−0.0853 (3)	0.66793 (9)	0.6153 (2)	0.0570 (6)
O3	−0.6978 (3)	0.81908 (11)	0.3964 (2)	0.0690 (8)
O4	−0.4886 (3)	0.86761 (9)	0.4300 (2)	0.0530 (6)
O5	−0.6502 (3)	0.72473 (11)	0.4329 (3)	0.0738 (8)
H5	−0.7012	0.7475	0.4046	0.111*
O6	−0.2878 (4)	0.59049 (10)	0.5550 (4)	0.0915 (11)
H2W	−0.2292	0.6113	0.5921	0.137*
H1W	−0.2376	0.5765	0.4968	0.137*
O7	0.3420 (2)	0.46787 (8)	0.9045 (2)	0.0487 (6)
O8	1.0627 (2)	0.44695 (7)	0.6141 (2)	0.0503 (6)
N1	0.3106 (3)	0.63213 (8)	0.7196 (2)	0.0366 (6)
N2	1.1541 (3)	0.28096 (8)	0.7644 (2)	0.0324 (5)
N3	0.5289 (3)	0.46344 (9)	0.7565 (2)	0.0412 (6)
N4	0.8419 (3)	0.43732 (9)	0.7205 (3)	0.0437 (7)
C1	−0.0957 (4)	0.71145 (12)	0.5960 (3)	0.0395 (7)
C2	−0.2499 (3)	0.73127 (11)	0.5446 (3)	0.0331 (7)
C3	−0.3865 (4)	0.70796 (12)	0.5143 (3)	0.0438 (8)
H3	−0.4009	0.6752	0.5243	0.053*
C4	−0.5025 (3)	0.73927 (13)	0.4664 (3)	0.0450 (8)
C5	−0.4551 (3)	0.78594 (12)	0.4597 (3)	0.0387 (7)
C6	−0.5586 (4)	0.82501 (14)	0.4248 (3)	0.0470 (8)
C7	−0.5859 (5)	0.90856 (15)	0.3963 (4)	0.0785 (13)
H7A	−0.6371	0.9034	0.3133	0.118*
H7B	−0.5218	0.9367	0.3934	0.118*
H7C	−0.6634	0.9127	0.4598	0.118*
C8	0.4377 (3)	0.62033 (11)	0.7927 (3)	0.0438 (8)
H8	0.5034	0.6447	0.8234	0.053*
C9	0.4756 (3)	0.57399 (11)	0.8245 (3)	0.0423 (8)
H9	0.5646	0.5675	0.8763	0.051*
C10	0.3813 (3)	0.53698 (10)	0.7796 (3)	0.0321 (6)
C11	0.2501 (3)	0.54887 (11)	0.7033 (3)	0.0398 (7)
H11	0.1832	0.5251	0.6707	0.048*
C12	0.2197 (3)	0.59638 (11)	0.6761 (3)	0.0400 (7)
H12	0.1312	0.6038	0.6247	0.048*
C13	0.4155 (3)	0.48628 (10)	0.8186 (3)	0.0364 (7)
C14	0.6131 (4)	0.48281 (12)	0.6483 (3)	0.0451 (8)
H14A	0.5747	0.5147	0.6281	0.054*
H14B	0.5936	0.4628	0.5729	0.054*
C15	0.7844 (4)	0.48481 (11)	0.6800 (3)	0.0488 (8)
H15A	0.8392	0.4953	0.6049	0.059*
H15B	0.8053	0.5077	0.7487	0.059*
C16	0.7564 (3)	0.41722 (12)	0.8285 (3)	0.0464 (8)
H16A	0.7759	0.4367	0.9049	0.056*
H16B	0.7940	0.3851	0.8471	0.056*
C17	0.5838 (4)	0.41579 (11)	0.7955 (3)	0.0481 (8)
H17A	0.5627	0.3933	0.7258	0.058*
H17B	0.5280	0.4051	0.8698	0.058*
C18	0.9808 (3)	0.42187 (11)	0.6808 (3)	0.0361 (7)
C19	1.0343 (3)	0.37234 (10)	0.7151 (3)	0.0315 (6)
C20	1.1831 (3)	0.36585 (10)	0.7711 (3)	0.0363 (7)
H20	1.2456	0.3921	0.7931	0.044*
C21	1.2371 (3)	0.32026 (10)	0.7939 (3)	0.0352 (7)

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
H21	1.3370	0.3165	0.8319	0.042*
C22	1.0117 (3)	0.28756 (10)	0.7101 (3)	0.0361 (7)
H22	0.9521	0.2607	0.6883	0.043*
C23	0.9476 (3)	0.33196 (11)	0.6845 (3)	0.0389 (7)
H23	0.8471	0.3347	0.6470	0.047*

Comment

Owing to structural diversity, novel topology, distinctive property and potential application in different fields, the syntheses of coordination polymers have been extensively studied [5–12]. Many synthetic factors, especially the design and judicious selection of organic ligands, could contribute to structural diversities of coordination polymers. Recently, the methyl-3-hydroxy-5-carboxy-2-thiophenecarboxylate (H_2L) ligand with multiple potential coordination sites was synthesized and many related coordination polymers structurally characterized [13–16]. On the other hand, piperazine-1,4-diylbis(pyridin-4-ylmethanone), as a long-spanning, conformationally flexible dipyrindyl ligand, has attracted significant attention during the construction of coordination polymers [17–20].

The title compound was prepared under mild hydrothermal conditions. Single crystal X-ray structural analysis shows that the compound is a one-dimensional chain structure and crystallizes in the monoclinic space group $P2_1/n$. The asymmetric unit of the title structure consists of a silver ion, one partially deprotonated HL^{-1} anion, one piperazine-1,4-diylbis(pyridin-4-ylmethanone) molecule and one water molecule. Each Ag(I) atom is coordinated by two nitrogen atoms from two different bpfp ligands, and an oxygen atom from a HL^{-1} anion with Ag–N, Ag–O bond distances of 2.208 (2), 2.285 (2) and 2.301 (2) Å, respectively. The bond angles about the silver ion range from 91.21 (8) to 132.25 (9)°, which are all within a reasonable range [21, 22]. Thus, the three-coordinated Ag(I) center has a distorted trigonal-planar coordination geometry. The central silver ions are interconnected by two pyridyl groups of μ_2 -bridged piperazine-1,4-diylbis(pyridin-4-ylmethanone) ligands with *anti* conformation, giving a linear $[Ag(bpfp)]_n$ chain with a Ag...Ag distance of 16.5077(7) Å. Then the one-dimensional chain structure was further decorated by the partially deprotonated HL^{-1} anions. The one-dimensional chain is further assembled into a two-dimensional supramolecular layer structure through intramolecular hydrogen-bonding interactions involving the carboxylate oxygen atom from HL^{-1} anion,

carboxylate oxygen atom of piperazine-1,4-diylbis(pyridin-4-ylmethanone) molecule and water molecule. Meanwhile, intermolecular hydrogen-bonding interaction between the hydroxyl oxygen atom and carboxylate oxygen atom is also observed.

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