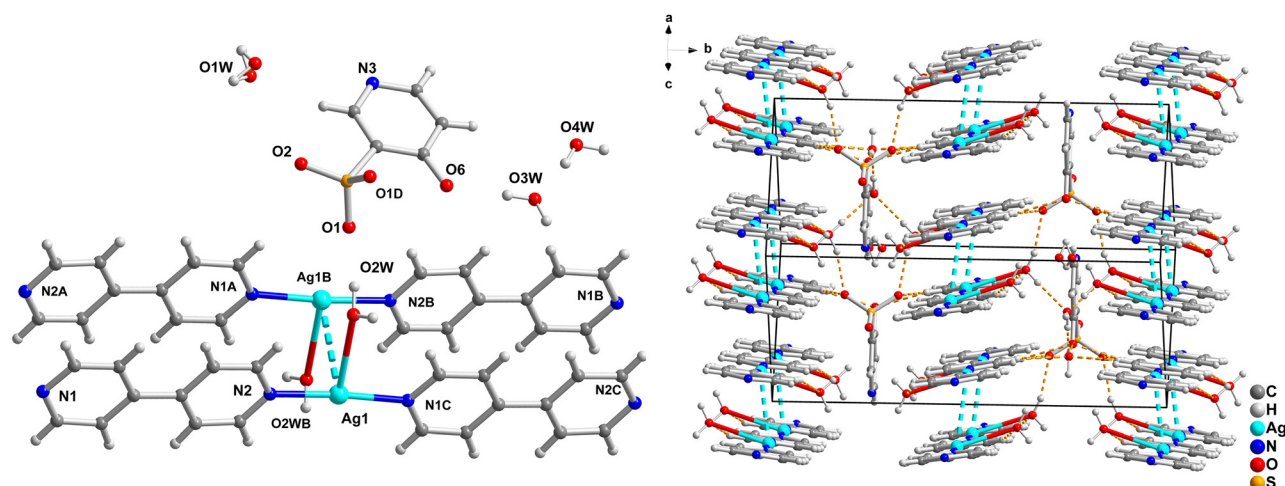


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Crystal structure of *catena*-poly[*diaqua*-bis(μ_2 -4,4'-bipyridyl- $\kappa^2 N:N'$) disilver(I)] 4-oxidopyridine-3-sulfonate trihydrate, $C_{25}H_{29}Ag_2N_5O_9S$



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Abstract

$\text{C}_{25}\text{H}_{29}\text{Ag}_2\text{N}_5\text{O}_9\text{S}$, monoclinic, $P2_1/m$ (no. 11), $a = 8.5437(16)$ Å, $b = 17.852(4)$ Å, $c = 9.3335(13)$ Å, $\beta = 101.380(16)^\circ$, $V = 1395.6(5)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0271$, $wR_{\text{ref}}(F^2) = 0.0653$, $T = 293(2)$ K.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.14 × 0.11 × 0.07 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.54 mm ⁻¹
Diffractometer, scan mode:	XtaLAB Pro, ω
$\theta_{\max}$, completeness:	25.0°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8980, 2524, 0.022
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2260
$N(\text{param})_{\text{refined}}$:	235
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], WinGX/ORTEP [4]

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of Ag_2CO_3 (0.138 g, 0.5 mmol) and 4-hydroxypyridine-3-sulfonic acid (0.088 g, 0.5 mmol) in 15 mL EtOH/ H_2O (2:1, v/v) was reacted for 15 min. An aqueous solution of ammonia was added to make the solution clear. A methyl alcohol solution of 4,4'-bipyridine (0.078 g, 0.5 mmol) was added to the system at the same time. The solution was kept in a dark room and crystals were obtained.

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

Atom	x	y	z	U _{iso} [*] /U _{eq}
C1	−0.1153 (4)	0.44867 (18)	−0.3552 (3)	0.0504 (8)
H1	−0.165162	0.403996	−0.389298	0.061*
C2	0.0077 (4)	0.44610 (16)	−0.2370 (3)	0.0488 (8)
H2	0.039168	0.400314	−0.193059	0.059*
C3	0.0859 (3)	0.51092 (14)	−0.1819 (3)	0.0292 (5)
C4	0.0347 (4)	0.57633 (16)	−0.2565 (3)	0.0441 (7)
H4	0.084441	0.621541	−0.226066	0.053*
C5	−0.0892 (4)	0.57467 (17)	−0.3751 (3)	0.0461 (8)
H5	−0.120368	0.619329	−0.423771	0.055*
C6	0.3762 (4)	0.44860 (17)	0.1533 (3)	0.0501 (8)
H6	0.405918	0.404021	0.202779	0.060*
C7	0.2608 (4)	0.44609 (16)	0.0284 (3)	0.0494 (8)
H7	0.214718	0.400504	−0.004748	0.059*
C8	0.2127 (3)	0.51087 (14)	−0.0483 (3)	0.0280 (5)
C9	0.2878 (4)	0.57545 (17)	0.0082 (4)	0.0560 (10)
H9	0.260878	0.620843	−0.039117	0.067*
C10	0.4025 (4)	0.57356 (18)	0.1342 (4)	0.0541 (9)
H10	0.450735	0.618338	0.169692	0.065*
C11	0.1113 (5)	0.250000	0.8923 (5)	0.0345 (9)
C12	0.2578 (4)	0.250000	0.8503 (4)	0.0270 (7)
C13	0.4015 (4)	0.250000	0.9585 (4)	0.0271 (8)
C14	0.3766 (5)	0.250000	1.1047 (4)	0.0344 (8)
H14	0.464115	0.250000	1.181843	0.041*
C15	0.2273 (5)	0.250000	1.1331 (5)	0.0417 (10)
H15	0.217585	0.250000	1.230628	0.050*
Ag1	0.63265 (3)	0.51108 (2)	0.39791 (2)	0.04697 (11)
N1	−0.1674 (3)	0.51182 (13)	−0.4241 (2)	0.0351 (5)
N2	0.4476 (3)	0.51144 (13)	0.2073 (2)	0.0335 (5)
N3	0.0925 (4)	0.250000	1.0315 (4)	0.0417 (8)
O1	0.3464 (3)	0.31775 (12)	0.6346 (2)	0.0482 (5)
O2	0.0984 (4)	0.250000	0.5851 (3)	0.0571 (9)
O1W ^a	−0.2622 (6)	0.2663 (7)	0.7345 (6)	0.069 (4)
O2W	0.5527 (3)	0.35965 (13)	0.4459 (3)	0.0574 (6)
H2WA	0.496332	0.338942	0.500089	0.086*
H2WB	0.626564	0.328587	0.441817	0.086*
O3W	0.8106 (4)	0.250000	1.1360 (3)	0.0371 (6)
O4W	0.7897 (4)	0.250000	1.4180 (4)	0.0561 (9)
O6	0.5412 (3)	0.250000	0.9290 (3)	0.0368 (6)
S1	0.26302 (11)	0.250000	0.66201 (10)	0.0319 (2)
H1WA	−0.338 (9)	0.250000	0.762 (8)	0.09 (3)*
H1WB	−0.286 (13)	0.250000	0.648 (3)	0.18 (5)*
H3WA	0.727 (3)	0.250000	1.071 (3)	0.030 (11)*
H3WB	0.894 (5)	0.250000	1.103 (6)	0.09 (2)*
H4WA	0.875 (5)	0.250000	1.480 (6)	0.10 (2)*
H4WB	0.805 (6)	0.250000	1.331 (3)	0.062 (16)*
H11	0.017 (5)	0.250000	0.824 (5)	0.036 (11)*

^aOccupancy: 0.5.

Experimental details

The detailed information of the single crystal were collected by CrysAlis^{PRO} 1.171.39.7e [1], were solved by SHELXT [2] program with WinGX [4], refined by SHELXL [3].

Comment

As is depicted in the Figure, the Ag cation is three-coordinated by two nitrogen atoms coming from two 4,4'-bipyridine ligands and one oxygen atom of a water molecule. The bond lengths and angles are similar to those observed in other coordination polymers [5–8]. The 4-hydroxypyridine-3-sulfonic acid is the counter ion in this structure. The distance of Ag...Ag is 3.260 Å. Lastly, the title compound forms a three-dimensional structure due to the existence of free water molecules. It should be mentioned that the structure of the ylidic educt has been reported [9].

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