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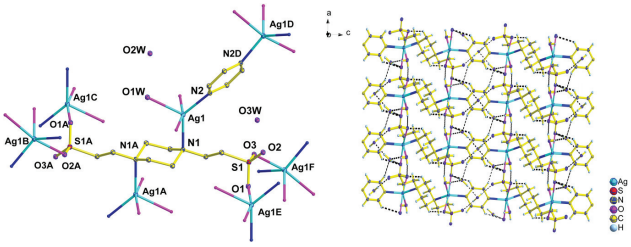
Crystal structure of *catena*-poly[aqua(μ_6-piperazine-1,4-bisethanesulfonato-κ^6*N*:*N*′:*O*:*O*′:*O*″:*O*‴)(μ_2-pyrazinyl-κ^2*N*:*N*′)disilver(I) sesquihydrate], $C_{12}H_{30}Ag_2N_4O_{11}S_2$



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Abstract

$C_{12}H_{30}Ag_2N_4O_{11}S_2$, monoclinic, *I*2/*a* (no. 15), $a = 12.2643(8)$ Å, $b = 7.1481(4)$ Å, $c = 26.2858(16)$ Å, $\beta = 93.119(6)°$, $V = 2301.0(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0186$, $wR_{ref}(F^2) = 0.0462$, $T = 298(2)$ 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.

Source of materials

Ag_2O (0.1159 g, 0.5 mmol) and piperazine-1,4-bisethanesulfonic acid (0.1512 g, 0.5 mmol) were added to 10 mL H_2O/DMF (1:1, *v*/*v*) and reacted for 25 min. Pyrazine (0.0810 g, 1 mmol) was then added to the solution. Single crystals suitable for diffraction were obtained after one month.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.13 × 0.15 × 0.15 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	1.94 mm^{-1}
Diffractometer, scan mode:	XtaLAB Pro, ω-scans
θ_{max}, completeness:	50°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	22891, 3056, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2866
$N(param)_{refined}$:	201
Programs:	CrysAlis PRO [1], WinGX [2], SIR 2004 [3], SHELXL [4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Ag1	0.74424(2)	0.14837(2)	0.13957(2)	0.03044(5)
O1	0.91787(10)	0.91285(18)	0.15938(6)	0.0407(3)
O2	1.03937(9)	0.67953(17)	0.12808(5)	0.0323(2)
O3	0.89452(12)	0.8042(2)	0.07213(5)	0.0467(3)
O1W	0.61930(12)	−0.1128(2)	0.15559(8)	0.0456(3)
H1A	0.551(3)	−0.058(5)	0.1607(13)	0.083(9)*
H1B	0.615(2)	−0.160(4)	0.1305(11)	0.045(8)*
O2W	0.58423(15)	−0.3538(2)	0.07500(8)	0.0539(4)
H2A	0.571(3)	−0.455(6)	0.0867(14)	0.092(12)*
H2B	0.632(4)	−0.371(6)	0.050(2)	0.134(18)*
O3W	0.750000	0.6456(4)	0.000000	0.0622(7)
H3A	0.789(2)	0.711(4)	0.0191(11)	0.063(8)*
S1	0.92925(3)	0.75737(5)	0.12405(2)	0.02419(8)
N1	0.80529(9)	0.33573(15)	0.20804(4)	0.0184(2)
N2	0.74687(11)	0.1798(2)	0.05292(5)	0.0291(3)
C1	0.84017(12)	0.5771(2)	0.14293(6)	0.0282(3)
H1	0.8368(16)	0.489(3)	0.1163(8)	0.036(5)*
H2	0.772(2)	0.642(3)	0.1450(10)	0.051(7)*
C2	0.88267(11)	0.4795(2)	0.19126(6)	0.0252(3)
H3	0.8949(17)	0.569(3)	0.2178(8)	0.036(5)*
H4	0.9498(16)	0.423(3)	0.1862(8)	0.030(5)*
C3	0.86360(10)	0.2192(2)	0.24752(5)	0.0209(2)
H5	0.8924(15)	0.302(3)	0.2751(7)	0.022(4)*
H6	0.9247(16)	0.160(2)	0.2318(8)	0.023(4)*
C4	0.71163(11)	0.42612(19)	0.23144(5)	0.0210(2)
H7	0.7383(15)	0.508(3)	0.2590(7)	0.026(4)*
H8	0.6714(15)	0.499(3)	0.2059(8)	0.029(5)*
C5	0.84104(13)	0.1822(2)	0.02983(6)	0.0311(3)
H9	0.9054(19)	0.182(3)	0.0510(9)	0.043(6)*
C6	0.65587(13)	0.1817(3)	0.02273(6)	0.0314(3)
H10	0.5876(19)	0.186(3)	0.0384(9)	0.042(6)*

Experimental details

Data were collected by CrysAlis^{PRO}, version 1.171.39.7e [1]; the structure was solved by SIR2004 [3], as incorporated within the WinGX suite [2], and refined by SHELXL [4]. The position and isotropic displacement parameters of H atoms were freely refined.

Comment

As is shown in the figure, the silver(I) atom is coordinated by two nitrogen atoms from a pyrazine ligand and a piperazine-1,4-bisethanesulfonic acid (ppiS) ligand and an oxygen atom of a water molecule, giving rise to a distorted trigonal planar geometry around the metal cation. The 1D chains, the figure shows the main repeating unit, are interlinked by longer 2.742(1) and 2.799(1) Ag$\cdots$O(ppiS) contacts. More specifically, the asymmetric unit contains half a ppiS ligand (which sits on an inversion centre), half a piperazine ligand (which sits on a two-fold rotation axis), one metal cation, two fully occupied water molecules, O1W and O2W and half a water molecule, with O3W sitting on a 2-fold rotation axis. All bond lengths and angles are in the normal range [5–7]. The existence of intramolecular interactions such as OH$\cdots$O and CH$\cdots$O hydrogen bonds give rise to a 3D supramolecular structure [8, 9].

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