Crystal structure of *catena*-(bis(μ_2-4,4′-bipyridine)dissilver(I) dihydroxide dimethyl terephthalate dihydrate, $Ag_2(C_{10}H_8N_2)_2(OH)_2 \cdot C_{10}H_{10}O_4 \cdot 2H_2O$

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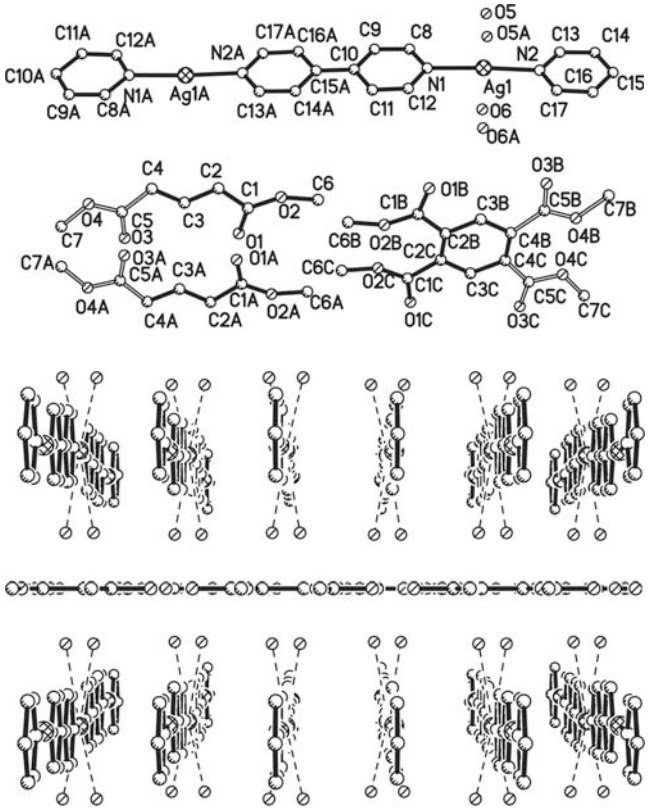

Abstract

$C_{15}H_{16}AgN_2O_4$, orthorhombic, *Cmcm* (no. 63), a = 18.309(2) Å, b = 22.831(2) Å, c = 7.180(1) Å, V = 3001.2 Å^3, Z = 8, $R_{gt}(F)$ = 0.052, $wR_{ref}(F^2)$ = 0.177, T = 293 K.

Source of material

An ammonia solution (25 mL) containing 0.0169 g $AgNO_3$ (0.10 mmol) and 0.0166 g benzene-1,4-dicarboxylate acid (0.10 mmol) was added dropwise to an methanol solution (25 mL) of 0.039 g 4,4′-bipyridine (bpy, 0.25 mmol). The clear mixture was stirred for a few minutes and then allowed to evaporate at room temperature slowly. Needlelike colorless crystals of the title compound appeared after several weeks. The crystals must be protected by vaseline oil as soon as possible after taken from mother liquid because they were unstable when exposed under air.

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Experimental details

All H atoms were fixed geometrically and allowed to ride on their parent carbon atoms, with d(C—H) of 0.93 Å and U_{iso}(H) = 1.2 U_{eq}(C). The COO^- groups attached on the terephthatate ligand are disordered over two positions, the O1, O2, O3, O4, C1, C5 and corresponding H positions were split during refinement resulting in a site occupancy ratio of 0.50/0.50(1). The C6 and corresponding H atoms of the methanol are also disordered over two positions, and their site occupancy factor ratio was set to 0.50/0.50.

Discussion

4,4′-Bipyridine is a flexible and versatile spacer to built novel coordination polymers. Few complexes of Ag(I) and bpy-like ligands with different counterions have been reported [1-6].

The crystal structure of the title compound contains 1D cationic wave-like chains $Ag(bpy)]^+_n$, dimethyl terephthatate, OH^- anions and lattice water molecules. In the $Ag(bpy)]^+_n$ chains (figure, top), Ag(I) in a bent linear coordination geometry are coordinated by two nitrogen atoms from different bpy ligands with Ag—N bond distances being 2.167 - 2.172 Å and N–Ag–N bond angles being 176.2°, as expected for Ag(I) complex with pyridine-type ligands [1,2]. The bpy act as bidentate ligand to bridge silver ions. The cationic charge of $Ag(bpy)]^+_n$ is balanced by OH^- anions. The dimethyl terephthatate is formed from the methanol and terephthatate during the preparation and is located layer-wise between the ion layers (figure, bottom). The lattice water molecules are situated within the framework and stablized by multiple hydrogen bonds.

Table 1. Data collection and handling.

Crystal:	colorless needle, size 0.30 × 0.32 × 0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	13.63 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	49.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2627, 1491
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1161
$N(param)_{refined}$:	164
Programs:	SHELXS-97 [7], SHELXL-97 [8], SHELXTL [9]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(5C)	8*g*		0.9230	0.4051	¾	0.089
H(5D)	16*h*	0.50	0.9251	0.4288	0.5675	0.089

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(6F)	16*h*	0.50	0.5752	0.4159	0.5994	0.107
H(2)	8*f*	0.50	½	0.8511	0.4086	0.046
H(3)	8*f*		½	0.7638	0.5716	0.062
H(4)	8*f*	0.50	½	0.6758	0.4128	0.079
H(6A)	16*h*	0.25	0.4815	0.9842	0.5432	0.050
H(6B)	16*h*	0.25	0.4695	0.9911	0.3282	0.050
H(6C)	16*h*	0.25	0.5489	0.9885	0.4093	0.050
H(7A)	16*h*	0.25	0.5067	0.5305	0.5571	0.076
H(7B)	16*h*	0.25	0.4542	0.5710	0.6718	0.076

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(7C)	16*h*	0.25	0.5391	0.5780	0.6914	0.076
H(8)	8*g*		0.8667	0.3164	¾	0.066
H(9)	8*g*		0.8655	0.2173	¾	0.059
H(11)	8*g*		0.6476	0.2229	¾	0.060
H(12)	8*g*		0.6543	0.3223	¾	0.065
H(13)	8*g*		0.8571	0.5349	¾	0.097
H(14)	8*g*		0.8546	0.6336	¾	0.098
H(16)	8*g*		0.6400	0.6259	¾	0.091
H(17)	8*g*		0.6488	0.5268	¾	0.091

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ag(1)	8*g*		0.76160(5)	0.42501(3)	¾	0.0664(6)	0.0174(4)	0.1028(8)	–0.0002(3)	0	0
N(1)	8*g*		0.7612(4)	0.3299(3)	¾	0.049(4)	0.019(3)	0.074(5)	–0.002(3)	0	0
N(2)	8*g*		0.7541(4)	0.5197(3)	¾	0.048(4)	0.023(4)	0.090(6)	0.001(3)	0	0
O(1)	8*f*	0.50	½	0.8535(5)	0.656(2)	0.080(8)	0.040(6)	0.039(7)	0	0	–0.004(5)
O(2)	8*f*	0.50	½	0.9154(5)	0.404(2)	0.084(9)	0.036(6)	0.058(9)	0	0	–0.004(6)
O(3)	8*f*	0.50	½	0.6710(5)	0.688(2)	0.078(8)	0.044(7)	0.07(1)	0	0	0.010(6)
O(4)	8*f*	0.50	½	0.6123(6)	0.451(2)	0.11(1)	0.052(9)	0.08(1)	0	0	0.004(8)
O(5)	16*h*	0.50	0.9002(5)	0.4270(4)	0.667(2)	0.050(5)	0.059(6)	0.11(1)	0.008(4)	0.002(5)	0.024(5)
O(6)	16*h*	0.50	0.6050(6)	0.4273(4)	0.683(2)	0.076(6)	0.067(7)	0.13(2)	–0.024(5)	–0.014(7)	0.003(6)
C(1)	8*f*	0.50	½	0.8647(7)	0.480(3)	0.051(9)	0.028(8)	0.05(1)	0	0	–0.001(7)
C(2)	8*f*		½	0.8157(3)	0.344(1)	0.040(4)	0.035(4)	0.040(4)	0	0	–0.004(4)
C(3)	8*f*		½	0.7634(4)	0.442(1)	0.047(4)	0.058(5)	0.051(5)	0	0	0.019(5)
C(4)	8*f*		½	0.7110(4)	0.348(2)	0.045(5)	0.039(4)	0.113(9)	0	0	0.022(5)
C(5)	8*f*	0.50	½	0.6652(8)	0.505(3)	0.06(1)	0.032(8)	0.06(1)	0	0	0.003(9)
C(6)	8*f*	0.50	½	0.9740(5)	0.422(2)	0.057(9)	0.013(6)	0.030(8)	0	0	–0.003(6)
C(7)	8*f*	0.50	½	0.5691(7)	0.607(3)	0.06(1)	0.033(8)	0.06(1)	0	0	–0.008(8)
C(8)	8*g*		0.8219(5)	0.2973(4)	¾	0.042(5)	0.029(4)	0.093(8)	–0.003(3)	0	0
C(9)	8*g*		0.8215(4)	0.2376(3)	¾	0.035(4)	0.025(4)	0.087(7)	–0.002(3)	0	0
C(10)	8*g*		0.7567(4)	0.2070(4)	¾	0.043(4)	0.022(4)	0.059(5)	0.000(3)	0	0
C(11)	8*g*		0.6930(4)	0.2410(3)	¾	0.033(4)	0.022(4)	0.094(7)	0.000(3)	0	0
C(12)	8*g*		0.6974(5)	0.3008(4)	¾	0.042(5)	0.028(4)	0.093(8)	0.001(3)	0	0
C(13)	8*g*		0.8117(5)	0.5532(4)	¾	0.040(5)	0.032(5)	0.17(1)	0.005(4)	0	0
C(14)	8*g*		0.8107(5)	0.6131(4)	¾	0.036(5)	0.031(5)	0.18(1)	0.000(4)	0	0
C(15)	8*g*		0.7471(4)	0.6430(4)	¾	0.037(4)	0.020(4)	0.090(7)	0.000(3)	0	0
C(16)	8*g*		0.6860(5)	0.6085(4)	¾	0.039(5)	0.029(5)	0.16(1)	–0.001(4)	0	0
C(17)	8*g*		0.6918(5)	0.5485(4)	¾	0.041(5)	0.033(5)	0.15(1)	–0.003(4)	0	0

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