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Synthesis and Crystal Structure of Three Mixed-Ligand Silver(I) Complexes Constructed from 1,2-Di(4-Pyridyl)ethylene and Different Organic Carboxylate Anions[1]

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Abstract—The reaction of $AgNO_3$ with 1,2-di(4-pyridyl)ethylene (Dpe) and 2,3-pyrazinedicarboxylic acid (H_2Pzdc) or 2,2'-bipyridyl-4,4'-dicarboxylic acid (H_2Bpdc) or salicylic acid (HSa) in alcohol aqueous solution produces block-like crystals of $[Ag_2(Dpe)_2]Ppzdc) \cdot 6H_2O$ (**I**), $[Ag_3(Dpe)_3](Bpdc)(NO_3) \cdot 4H_2O$ (**II**), and $[Ag_2(Dpe)_2](Sa)(NO_3) \cdot 3H_2O$ (**III**) at room temperature. Crystal **I** is monoclinic: space group $P2_1/m$, $a = 7.5370(7)$, $b = 17.2309(16)$, $c = 12.5131(11)$ Å, $\beta = 98.3780(10)°$, $V = 1607.7(3)$ Å^3, $Z = 2$. Crystal **II** is triclinic: space group $P\bar{1}$, $a = 8.8260(8)$, $b = 11.1149(11)$, $c = 13.3751(14)$ Å, $\alpha = 102.0140(10)°$, $\beta = 105.696(2)°$, $\gamma = 99.5940(10)°$, $V = 1200.4(2)$ Å^3, $Z = 1$. Crystal **III** is triclinic: space group $P\bar{1}$, $a = 7.3600(6)$, $b = 2.7549(11)$, $c = 18.2241(16)$ Å, $\alpha = 106.225(2)°$, $\beta = 98.0150(10)°$, $\gamma = 99.5400(10)°$, $V = 1588.5(2)$ Å^3, $Z = 2$. All the three complexes contain sandwich-like crystal structures, in which anionic sheets built up from different anions ($Pzdc^{2-}$, $Bpdc^{2-}$, Sa^-) and lattice water molecules *via* rich hydrogen-bonding interactions are inserted between the cationic silver complexes layers. The lattice water molecules are situated among the framework of the crystal structure and stabilized by rich hydrogen-bonding interactions, and lattice water molecules may play a role in the orientation of the organic anions in the crystal packing.

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INTRODUCTION

The supramolecular chemistry of silver(I) coordination polymers have been received much attention, not only due to its ability to form complexes with coordination numbers between two to six, resulting in many different architectures [1–5], but also due to their potential applications, such as gas storage [6], antimicrobial [7, 8], conductive material [9, 10], luminescent [11–15] and magnetic materials [16]. The geometrical flexibility of silver(I) results in intricate coordination architectures, and also afforded an opportunity to explore how the self-assembly process can be influenced by some factors, like the structural characteristic of the polydentate organic ligands, the metal-ligand ratio, solvents and the possible counter-ions [2–4, 11, 17–19]. The combination of center metal ions with neutral N-donor ligands and anionic O-donor ligands can generate more interesting structures which can not be obtained only *via* one type of ligands [12–16]. Furthermore, the prospect of introducing the second or more organic ligands into a reaction system provides further impetus for research on metal-organic supramolecular frameworks. And with the aid of supramolecular interactions, such as hydrogen-bonding interactions, π–π staking interactions, Ag···Ag and Ag···N contacts [2–4, 17–24], various high dimensional silver(I) coordination polymers can be built up from the low-dimensional Ag(I) assemblies.

In this paper, we report the synthesis and crystal structures of three new coordination complexes assembled by $AgNO_3$, 1,2-di(4-pyridyl)ethylene (Dpe) and 2,3-pyrazinedicarboxylic acid (H_2Pzdc) or 2,2'-bipyridyl-4,4'-dicarboxylic acid (H_2Bpdc) or salicylic acid (HSa), namely $[Ag_2(Dpe)_2](Pzdc) \cdot 6H_2O$ (I), $[Ag_3(Dpe)_3](Bpdc)(NO_3)_2 \cdot 4H_2O$ (II), and $[Ag_2(Dpe)_2](Sa)(NO_3) \cdot 3H_2O$ (III). The single-crystal diffraction results revealed that all three complexes contain fascinating sandwich-like framework, in which the anionic sheets built up from different anions ($Pzdc^{2-}$, $BPdc^{2-}$, Sa^-) and lattice water molecules *via* rich hydrogen-bonding interactions are inserted between the cationic silver complexes layers.

EXPERIMENTAL

Materials and general methods. All commercially available chemicals are reagent grade and used as received without further purification. Elemental analysis

[1] The article is published in the original.

for the title complexes was performed by Elementar Vario EL-III. IR spectra were recorded on PerkinElmer Spectrum 100 Fourier Transform infrared spectrophotometer in the region 400–4000 cm^{-1}.

Synthesis of I. An ammonia solution (25 mL) containing $AgNO_3$ (0.0085 g, 0.05 mmol) and H_2Pzdc (0.0084 g, 0.05 mmol) was added drop-wise to an alcohol solution (25 mL) of Dpe (0.0091 g, 0.05 mmol). The clear mixture was stirred for a few minutes and then allowed to evaporate at room temperature slowly. Block-like colorless crystals of **I** appeared after several weeks.

IR spectrum (KBr pellet; ν, cm^{-1}): 3435.64, 1601.29, 1498.18, 1425.05, 1384.82, 1355.36, 1204.64, 1158.99, 1110.77, 1073.63, 1010.17, 997.80, 973.05, 884.25, 827.22, 548.06.

For $C_{30}H_{34}N_6O_{10}Ag_2$

anal. calcd., %:	C, 42.17;	H, 4.01;	N, 9.84.
Found, %:	C, 42.72;	H, 4.12;	N, 9.77.

Synthesis of II. Synthesis of block-like colorless crystals of **II** followed the same procedure as for **I** except that H_2Pzdc was replaced with H_2Bpdc (0.05 mmol).

IR spectrum (KBr pellet; ν, cm^{-1}): 3435.64, 1601.29, 1498.18, 1425.05, 1384.82, 1355.36, 1204.64, 1158.99, 1110.77, 1073.63, 1010.17, 997.80, 973.05, 884.25, 827.22, 548.06.

For $C_{48}H_{44}N_{10}O_{14}Ag_3$

anal. calcd., %:	C, 44.06;	H, 3.39;	N, 10.70.
Found, %:	C, 44.56;	H, 3.51;	N, 10.82.

Synthesis of III. Synthesis of block-like colorless crystals of **III** followed the same procedure as for **I** except that H_2Pzdc was replaced with H_2Sa (0.05 mmol).

IR spectrum (KBr pellet; ν, cm^{-1}): 3435.62, 3039.01, 1627.56, 1600.38, 1557.10, 1488.58, 1462.89, 1384.61, 1302.14, 1254.07, 1205.10, 1144.74, 1073.93, 1036.41, 998.10, 989.65, 972.56, 955.11, 861.82, 838.12, 826.85, 760.30, 745.66, 703.43, 669.10, 548.11, 492.49.

For $C_{31}H_{31}N_5O_9Ag_2$

anal. calcd., %:	C, 44.68;	H, 3.75;	N, 8.40.
Found, %:	C, 44.93;	H, 3.81;	N, 8.48.

X-ray crystallography. Diffraction intensities for complexes **I**–**III** were recorded with a Bruker CCD area detector diffractometer with a graphite-monochromatized MoK_α radiation (λ = 0.71073 Å) using φ–ω mode at 298(2) K. Semi-empirical absorption correction were applied using the SADABS program [25]. The structures were solved by direct methods [26] and refined by full-matrix least-squares on F^2 using SHELXS-97 and SHELXL-97 programs respectively [26, 27]. All non-hydrogen atoms were refined anisotropically and hydrogen atoms were placed in geometrically calculated positions. Crystallographic data and structural refinements for compound **I**–**III** are summarized in Table 1. Selected bond lengths and angles are listed in Table 2.

Supplementary material for structures **I**–**III** has been deposited with the Cambridge Crystallographic Data Centre (nos. 837004–837006; deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

In the IR spectrum of complex **I**, a strong and broad absorption at 3435.64 cm^{-1} is assigned to stretching vibration of hydroxyl, showing the presence of lattice water molecules. The asymmetric and symmetric vibrations of carboxylate group in the complex appear at about 1601.29 and 1425.05 cm^{-1}. Observed from the IR spectrum of the complex **II**, a strong band at 3433.14 cm^{-1} results from stretching vibration of hydroxyl, indicating the existence of lattice water molecules. The sharp bands at 1597.81 and 1406.67 cm^{-1} are attributed to the asymmetric and symmetric vibrations of carboxylate group, respectively. While in the IR spectrum of complex **III**, a strong and broad absorption at 3435.62 cm^{-1} is assigned to stretching vibration of hydroxyl, showing the presence of lattice water molecules. The asymmetric and symmetric vibrations of carboxylate group in the complex appear at about 1600.38 and 1384.61 cm^{-1}.

The crystal structure of **I** is made up of infinite cationic chains of $\left[Ag_2(Dpe)_2\right]_\infty^{2n+}$, $Pzdc^{2-}$ anions, and H_2O molecules, as illustrated in Fig. 1a. In the cationic chains of $\left[Ag_2(Dpe)_2\right]_\infty^{2n+}$, the Ag(I) atoms are coordinated in a linear coordination geometry with two nitrogen atoms from two different Dpe ligands (Ag–N ranging from 2.133(4) to 2.139(4) Å; NAgN 174.94(14)°), forming the simple topology of a single-strand chains. Dpe acts as typical bidentate ligand, linking two Ag atoms *via* nitrogen atoms from two pyridyl rings. Deprotonated H_2Pzdc act as counter-ions, balancing the cationic charge of $\left[Ag_2(Dpe)_2\right]_\infty^{2n+}$ cationic chains.

In complex **I**, the adjacent $\left[Ag_2(Dpe)_2\right]_\infty^{2n+}$ chains are interconnected by ligand-unsupported Ag···Ag (3.4074(6) Å) and Ag···N interactions (3.6879(39) and 3.9877(38) Å) to build up the 2D cationic sheet (A) (Fig. 1c). While the $Pzdc^{2-}$ anions and water molecules are linked into 2D anionic sheet (B) *via* rich hydrogen bonding interactions, as shown in Fig. 1b. The cationic A sheets and anionic B sheets were stacked by A···B···A mode *via* electrostatic interactions to construct 3D sandwich-like framework, as illustrated in Fig. 1d.

Table 1. Crystallographic data and refinement for compounds **I–III**

Parameter	Value		
	I	**II**	**III**
M	854.37	1308.54	833.35
Crystal system	Monoclinic	Triclinic	Triclinic
Space group	$P2_1/m$	$P\bar{1}$	$P\bar{1}$
a, Å	7.5370(7)	8.8260(8)	7.3600(6)
b, Å	17.2309(16)	11.1149(11)	12.7549(11)
c, Å	12.5131(11)	13.3751(14)	18.2241(16)
α, deg	90	102.0140(10)	106.225(2)
β, deg	98.3780(10)	105.696(2)	98.0150(10)
γ, deg	90	99.5940(10)	99.5400(10)
V, Å^3	1607.7(3)	1200.4(2)	1588.5(2)
Z	2	1	2
ρ_{calcd}, g cm^{-3}	1.765	1.810	1.742
$F(000)$	860	655	836
$\mu(MoK_\alpha)$, mm^{-1}	1.285	1.292	1.295
Crystal size, mm	0.49 × 0.41 × 0.40	0.42 × 0.19 × 0.16	0.40 × 0.32 × 0.13
θ Range, deg	2.73–25.02	2.75–25.02	2.37–25.02
Index ranges h, k, l	$-8 \le h \le 8$	$-10 \le h \le 10$	$-8 \le h \le 8$
	$-20 \le k \le 18$	$-13 \le k \le 13$	$-15 \le k \le 15$
	$-14 \le l \le 11$	$-15 \le l \le 14$	$-21 \le l \le 21$
Total reflections	7907	6072	7958
Unique (R_{int})	2904 (0.0459)	4182 (0.0160)	5502 (0.0265)
Reflections with $I > 2\sigma(I)$	2129	3165	2987
Parameters	238	359	424
GOOF	1.103	1.026	1.044
Final R indices ($I > 2\sigma(I)$)	$R_1 = 0.0445$	$R_1 = 0.0420$	$R_1 = 0.0589$
	$wR_2 = 0.0965$	$wR_2 = 0.1117$	$wR_2 = 0.1400$
R indices (all data)	$R_1 = 0.0668$	$R_1 = 0.0637$	$R_1 = 0.1080$
	$wR_2 = 0.1117$	$wR_2 = 0.1332$	$wR_2 = 0.1690$
Largest diff. peak and hole, e/Å^3	0.873 and –0.573	0.688 and –1.030	0.791 and –0.557

The crystal structure **II** is made up of cationic chains of $\left[Ag_3(Dpe)_3\right]_\infty^{2n+}$, $Bpdc^{2-}$ ions, NO_3^- anions, and water molecules. In the cationic chains of $\left[Ag_3(Dpe)_3\right]_\infty^{2n+}$, each Ag(I) atom, in linear coordination geometry, is ligated with two nitrogen atoms from two different Dpe ligands (Ag–N 2.111(4) and 2.134(4) Å; NAgN 174.60(18)° and 180.000(1)°), as illustrated in Fig 2a and Table 2, comparable to complex **I** and the reported Ag(I) complexes [3–5]. The cationic charge of $\left[Ag_3(Dpe)_3\right]_\infty^{2n+}$ chains is balanced by the counterions of deprotonated $Bpdc^{2-}$ anions and NO_3^- anions.

In complex **II**, the adjacent $\left[Ag_3(Dpe)_3\right]_\infty^{2n+}$ chains are packed into cationic sheets (A) *via* ligand-unsupported Ag–Ag (3.5827(7) Å) and Ag–N (3.7076(47) Å) (Fig. 2c). The anionic sheets (B) are constructed from $Bpdc^{2-}$, NO_3^- anions, and water molecules *via* rich intermolecular hydrogen bonding interactions (Table 3 and Fig. 2b). The cationic A sheets and B anionic sheets are further interconnected by electrostatic interactions to build up the 3D sandwich-like crystal

Table 2. Selected bond lengths (Å) and angles (deg) for compounds **I**–**III***

Bond	*d*, Å	Bond	*d*, Å
	I		
Ag(1)–N(2)$^{\#1}$	2.133(4)	Ag(1)–N(1)	2.139(4)
	II		
Ag(1)–N(1)	2.111(4)	Ag(1)–N(3)	2.121(4)
Ag(2)–N(2)$^{\#1}$	2.134(4)	Ag(2)–N(2)	2.134(4)
	III		
Ag(1)–N(1)$^{\#1}$	2.138(6)	Ag(1)–N(2)	2.144(6)
Angle	ω, deg	Angle	ω, deg
	I		
N(2)$^{\#1}$Ag(1)N(1)	174.94(14)		
	II		
N(1)Ag(1)N(3)	174.60(18)	N(2)$^{\#1}$Ag(2)N(2)	180.000(1)
	III		
N(3)$^{\#1}$Ag(2)N(4)	173.4(2)	N(1)$^{\#1}$Ag(1)N(2)	175.7(2)

* Symmetry codes: $^{\#1}$ $x + 1, y, z + 1$ for I; $-x + 2, -y + 2, -z + 2$ for II; $x - 1, y - 1, z$ for III.

Table 3. Geometric parameters for hydrogen bonds for compounds **I**–**III***

Contact D–H···O	Distance, Å		Angle DHA, deg	Symmetry operations for A
	H···A	D···A		
	I			
O(4)–H(4*C*)··· O(3)	1.876	2.722	173	$x - 1, y, z + 1$
O(4)–H(4*D*)···O(8)	2.037	2.881	172	$x - 1, y, z$
O(5)–H(5*C*)···O(4)	1.986	2.830	172	x, y, z
O(5)–H(5*D*)···N(4)	2.026	2.871	174	$x, y, z + 1$
O(6)–H(6*C*)···O(1)	2.260	2.980	143	x, y, z
O(6)–H(6*C*)···O(8)	2.380	2.808	112	x, y, z
O(6)–H(6*D*)···N(3)	2.304	3.024	143	x, y, z
O(7)–H(7*C*)···O(6)	2.125	2.930	158	x, y, z
O(7)–H(7*D*)···O(1)	1.908	2.714	158	$x - 1, y, z$
O(8)–H(8*C*)···O(6)	1.963	2.808	172	x, y, z
O(8)–H(8*D*)···O(7)	2.458	3.303	173	$x + 1, y, z$
	II			
O(1)–H(1*C*)···O(4)	1.978	2.819	170	x, y, z
O(1)–H(1*D*)···O(2)	2.033	2.875	170	$-x + 1, -y + 1, -z + 2$
O(2)–H(2*C*)···O(4)	1.907	2.757	179	x, y, z
O(2)–H(2*D*)···O(6)	1.961	2.811	179	$-x + 1, -y, -z + 1$
	III			
O(1)–H(1*C*)···O(3)	2.049	2.896	174	x, y, z
O(1)–H(1*D*)···O(4)	1.639	2.487	174	$-x + 1, -y + 1, -z + 1$
O(2)–H(2*B*)···O(9)	2.340	3.022	138	x, y, z
O(2)–H(2*C*)···O(1)	2.282	3.067	154	$-x + 1, -y, -z + 1$
O(3)–H(3*C*)···O(5)	2.254	2.983	144	$-x, -y + 1, -z + 1$
O(3)–H(3*D*)···O(9)	2.483	3.210	144	$-x + 1, -y, -z + 1$
O(6)–H(6)···O(5)	1.657	2.401	150	x, y, z

* *d*(D–H) 0.85 Å.

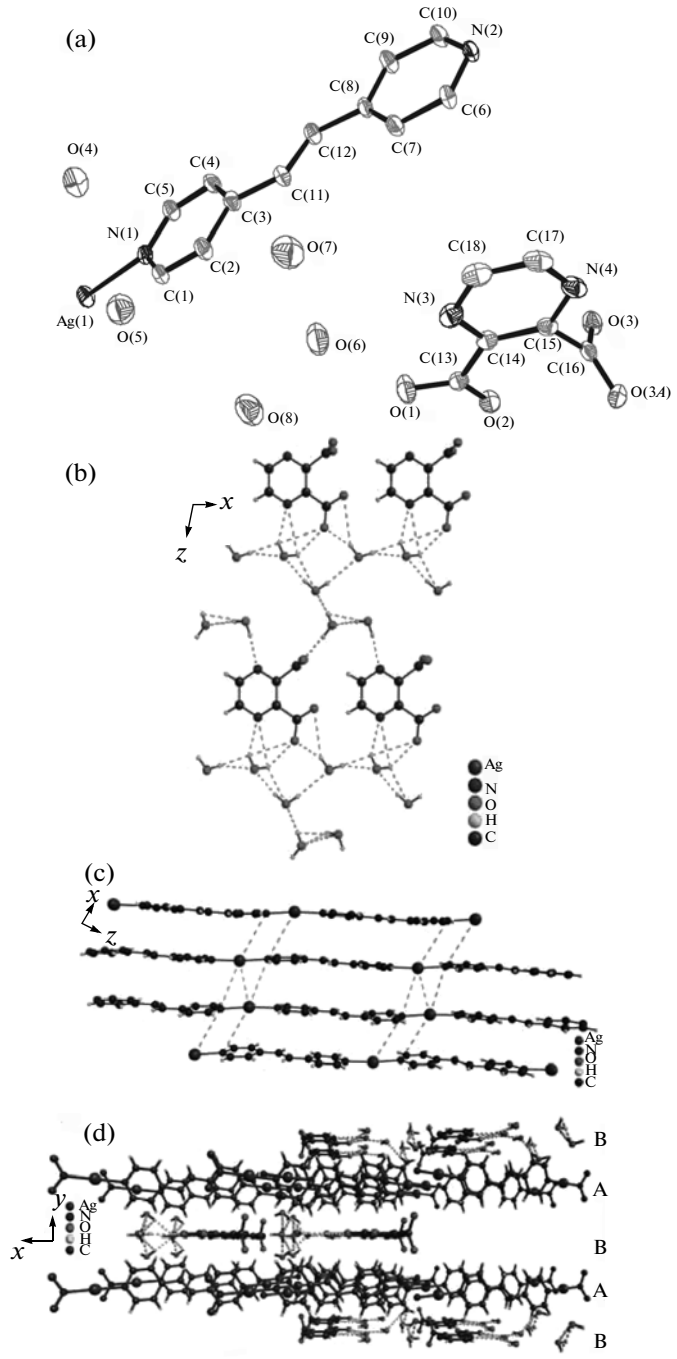

Fig. 1. Ortep view of the $[Ag_2(Dpe)_2](Pzdc) \cdot 6H_2O$ (**I**) structure with atomic labeling of one asymmetric unit, the H atoms are omitted for clarity (a); the anionic sheets built up from $Pzdc^{2-}$ and lattice water molecules (b); the cationic sheet formed by the $\left[Ag_2(Dpe)_2\right]_\infty^{2n+}$ chains *via* Ag···Ag and Ag···N interactions (c); and packing view along *z* axis direction of 3D sandwich-like framework (d).

framework (Fig. 2d). From other point of view, the lattice water molecules may play a role in the orientation of the $Bpdc^{2-}$ in the crystal structure.

As shown in Fig. 3a, in complex **III**, the coordination environment of Ag(I), just similar to the one in complex **I**, is coordinated in a linear coordination geometry by two nitrogen atoms from two different Dpe ligands, with bond length of Ag–N being 2.138(6) and 2.144(6) Å and bond angles of NAgN being 173.4(2)° to 175.7(2)°. The presence of deprotonated Sa^- and NO_3^-

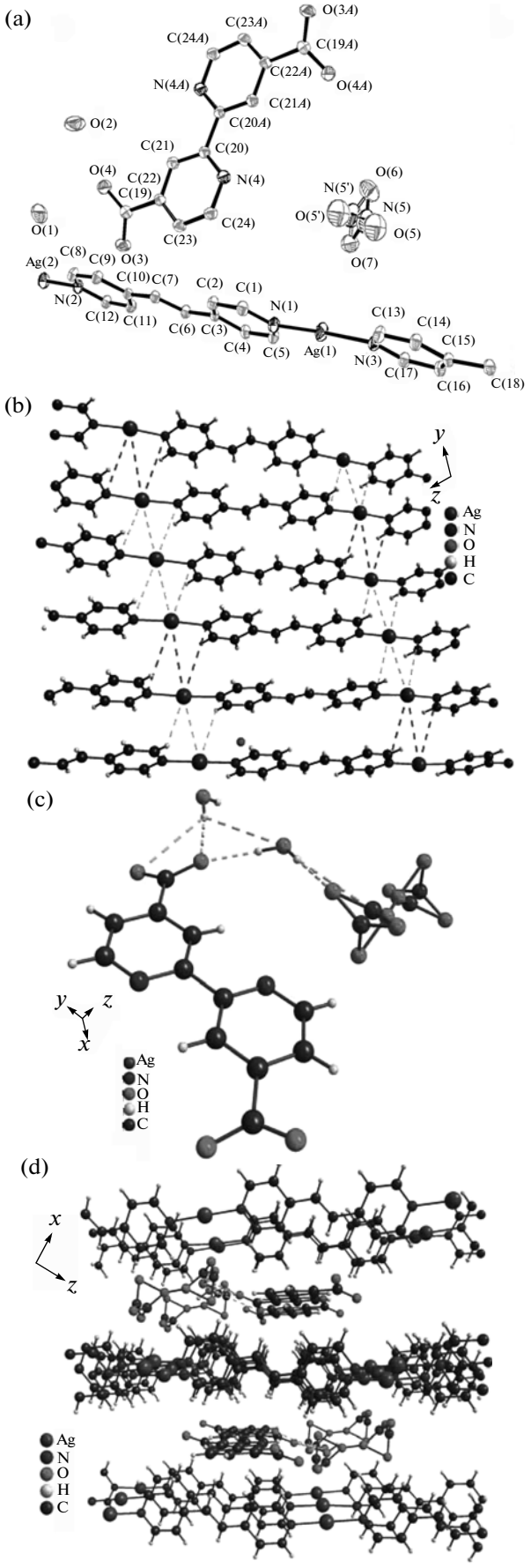

Fig. 2. Ortep view of the $[Ag_3(Dpe)_3](Bpdc)(NO_3) \cdot 4H_2O$ (**II**) structure with atomic labelling of one asymmetric unit, the H atoms are omitted for clarity. Both the position occupancy factor ratio of N5/N5' and O5/O5' are 0.82(2)/0.18(2) (a); the anionic sheets built up from $Bpdc^{2-}$, NO_3^- and lattice water molecules (b); the cationic sheet formed by the $\left[Ag_3(Dpe)_3\right]_\infty^{2n+}$ chains *via* Ag···Ag and Ag···N interactions (c); and packing view along *y* axis direction of 3D sandwich-like framework (d).

is to balance the charge of cationic $\left[Ag_2(Dpe)_2\right]_n^{2n+}$ chains.

In complex **III**, the adjacent cationic $\left[Ag_2(Dpe)_2\right]_n^{2n+}$ chains are weakly connected by the ligand-unsupported Ag···Ag interactions (ranging from 3.3775(10) and 3.9528(61) Å) into 2D cationic sheets (A) (Fig. 3c). While the deprotonated Sa^- and NO_3^- anions are linked into anionic sheets (B) with the aid of lattice water molecules *via* intermolecular rich hydrogen-bonding interactions (Fig. 3b and Table 3). The adjacent cationic A sheets and anionic B sheets are further joined into 3D sandwich-like framework by electrostatic interactions, viewed from *x* axis (Fig. 3d).

A few complexes of silver(I) with sandwich-like structure have been reported, like $[Ag_2(Bipy)_2](HA)_2 \cdot 6H_2O$, $[Ag_2(Bipy)_2(H_2O)](Pdc) \cdot 3H_2O$, $[Ag_4(Bipy)_4](Bptc) \cdot 14H_2O$, and $[Ag_2(Dpe)_2(H_2O)_2](HA) \cdot 6H_2O$ [3]; $[Ag_2(Bipy)_2(HDa)_2](HAc)_2 \cdot 2H_2O$ and $[Ag_2(Dpe)_2(Da)] \cdot 4H_2O$ [4]; $(NH_4)[Ag(Dpe)(H_2Bptc)]$ and $Ag_2(Bipy)(Ox) \cdot 7H_2O$ [5] (Bipy = 4,4'-bipyridine, Dpe = 1,2-di(4-pyridyl)ethylene, HA = hexanedioic acid, Pdc = pyridine-3,5-dicarboxylic acid, Bptc = 3,3'4,4'-biphenyltetracarboxylic acid, Da = diphenic acid, Ox = oxalic acid). All the above-stated complexes consist of 2D cationic sheets constructed from parallel 1D infinite Bipy/Dpe-silver cationic chains *via* ligand-unsupported Ag···Ag and Ag···N, interspersed with anionic sheets constructed from organic anions and water molecules *via* rich hydrogen bonding interactions, which play the role of charge compensation in the crystal structure.

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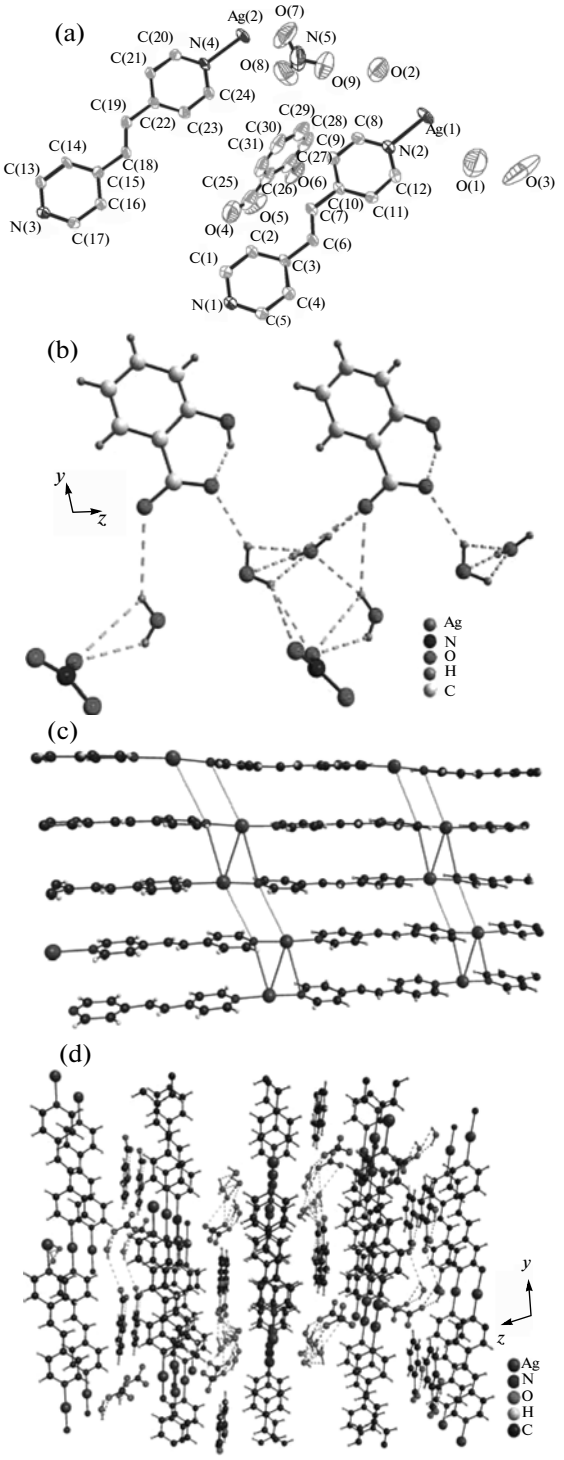

Fig. 3. Ortep view of the $[Ag_2(Dpe)_2](Sa)(NO_3) \cdot 3H_2O$ (**III**) structure with atomic labeling of one asymmetric unit, the H atoms are omitted for clarity (a); the anionic sheets built up from Sa^-, NO_3^- and lattice water molecules (b); the cationic sheet formed by the $\left[Ag_2(Dpe)_2\right]_\infty^{2n+}$ chains *via* Ag···Ag and Ag···N interactions (c); packing view along *x* axis direction of 3D sandwich-like framework (d).

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