

# 2D and 3D Silver(I)-Ethylenediamine Coordination Polymers with Ag–Ag Argentophilic Interactions

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Two complexes of silver(I) salts with ethylenediamine (etda) as a ligand were prepared and characterized. The study of the crystal structures (of the 2-hydroxy-4-nitro-benzoate trihydrate (**1**) and nitrate (**2**)) has shown that the formation of 2D and 3D coordination polymer networks results from etda ligands bridging the silver atoms which are connected *via* Ag–Ag argentophilic interactions.

**Key words:** Silver Compound, Ethylenediamine, Coordination Polymer, Crystal Structure, Ag–Ag Interaction

## Introduction

The programmed self-assembly of coordination polymers has attracted intense interest not the least because of the intricate structural topologies of the networks that may be manipulated to dramatically influence the overall physical properties and functions of the material [1–4]. This has stimulated a great deal of attention towards the design and construction of coordination frameworks to perform highly specific functions [5, 6]. For example, the utilization of rigid multidentate poly-pyridyl-based ligands, of which 4,4'-bipy is a particularly often used example, in combination with metal centers of varying geometries can give rise to a large number of network architectures and interpenetrating structures [7–10]. As it is well known from the recent literature, silver(I) in particular has been used to advantage to construct a great number of geometrically and stereochemically interesting 1D, 2D, and 3D coordination polymeric frameworks [11–13]. In contrast to many poly-pyridyl-based ligands and a number of highly rigid aliphatic amine ligands [7–14], a few alkyldiamines as soft ligands have been found to bind to silver(I) to form coordination polymers [15, 16]. Moreover, it is interesting to note that silver(I) was shown to accept non-chelated coordination with alkylidenediamines [17, 18], affording new coordination networks with different topologies. Recently we have isolated and structurally character-

ized a series of silver(I)-alkylidenediamine coordination polymers [19–22]. In the course of our current research on silver(I) coordination polymeric complexes, we herein report the syntheses of 2D and 3D silver-ethylenediamine coordination polymers with silver–silver interactions, and their characterization by single crystal X-ray diffraction.

## Experimental Section

### Materials and measurements

Silver 2-hydroxy-4-nitro-benzoate, Ag(hba), was synthesized from the reaction of AgNO<sub>3</sub> and 5-nitro-2-hydroxy-benzoic acid (hbaH) in methanol and water. All other reagents and solvents were used as received without further purification. Infrared spectra were recorded on a Nicolet 170sx FT-IR spectrophotometer with use of pressed KBr pellets. Elemental analyses were performed on a Perkin-Elmer 240C Elemental Analyzer.

### Preparation of [Ag<sub>2</sub>(μ-etda)<sub>2</sub>](hba)<sub>2</sub> · 3 H<sub>2</sub>O (**1**)

To a suspension of Ag(hba) (58 mg, 0.20 mmol) in aqueous methanol (5 mL) was added dropwise a methanolic solution (5 mL) of ethylenediamine (50 mg, 0.83 mmol). The mixture was stirred for *ca.* 2 h to give a clear light-yellow solution. Single crystals suitable for X-ray diffraction were grown by diffusion of diethyl ether. Yield: 67 mg, 55 %. – Selected IR bands (KBr, cm<sup>−1</sup>): ν = 3461 br (H<sub>2</sub>O), 3315m br (OH), 1601s 1453s (CO<sub>2</sub><sup>−</sup>), 1367s (NO<sub>2</sub>), 526m (etda). –

Table 1. Crystal data, data collection parameters and details of the structure refinement.

Complex	1	2
Empirical formula	C <sub>18</sub> H <sub>30</sub> N <sub>6</sub> O <sub>13</sub> Ag <sub>2</sub>	C <sub>2</sub> H <sub>8</sub> N <sub>3</sub> O <sub>3</sub> Ag
Formula weight	754.22	229.98
Color, habit	yellow, block	yellow, prism
Crystal size, mm <sup>3</sup>	0.25 × 0.15 × 0.10	0.17 × 0.14 × 0.10
Crystal system	monoclinic	monoclinic
Space group	<i>P</i> 2 <sub>1</sub> / <i>n</i>	<i>C</i> 2/ <i>c</i>
<i>a</i> , Å	7.094(1)	19.202(16)
<i>b</i> , Å	28.999(6)	21.012(18)
<i>c</i> , Å	12.828(3)	6.277(5)
β, deg	92.61(3)	101.011(11)
Volume, Å <sup>3</sup>	2636.2(9)	2486(4)
<i>Z</i>	4	16
Density (calc.), g cm <sup>-3</sup>	1.90	2.46
Absorption coefficient, mm <sup>-1</sup>	1.6	3.2
Temperature, K	293(2)	293(2)
<i>F</i> (000), e	1512	1792
Radiation, wavelength	— MoK <sub>α</sub> , λ = 0.71073 Å —	
Reflections collected	12124	6269
Independent reflections, <i>R</i> <sub>int</sub>	5367, <i>R</i> <sub>int</sub> = 0.053	2138, <i>R</i> <sub>int</sub> = 0.037
Reflections with <i>I</i> ≥ 2σ( <i>I</i> )	3709	560
Weighting scheme	$w = [\sigma^2(F_o^2) + 0.0439P^2 + 0.0000P]^a$	$w = [\sigma^2(F_o^2) + 0.0594P^2 + 0.0000P]^a$
Parameters refined	352	160
Final <i>R</i> indices (all data) <sup>b</sup>	<i>R</i> 1 = 0.094, <i>wR</i> 2 = 0.142	<i>R</i> 1 = 0.075, <i>wR</i> 2 = 0.134
Goodness of fit (GoF) <sup>c</sup>	1.01	0.98
Final difference peaks, e Å <sup>-3</sup>	+0.89/−0.74	+0.90/−0.95

<sup>a</sup>  $P = (F_o^2 + 2F_c^2)/3$ ; <sup>b</sup>  $R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ ,  $wR2 = [\Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma wF_o^2]^1/2$ ; <sup>c</sup> GoF =  $[2\Sigma w(|F_o| - |F_c|)^2/(N_{\text{obs}} - N_{\text{param}})]^{1/2}$ .

Anal. for C<sub>18</sub>H<sub>24</sub>Ag<sub>2</sub>N<sub>6</sub>O<sub>10</sub> · 3 H<sub>2</sub>O: calcd. C 28.6, H 3.98, N 11.1; found C 27.9, H 3.92, N 10.7.

#### Preparation of [Ag(μ-*etda*)](NO<sub>3</sub>) (2)

AgNO<sub>3</sub> (50 mg, 0.29 mmol), dissolved in degassed MeCN (8 mL), was added to ethylenediamine (30 mg, 0.50 mmol) in MeCN (5 mL) and the mixture stirred for *ca.* 4 h. The light-yellow solid that precipitated was filtered and dried in vacuum. Yield: 42 mg, 62 %. Yellow X-ray quality crystals were grown by slow diffusion of a MeCN solution of **en** layered with a MeCN solution of AgNO<sub>3</sub>. Selected IR bands (KBr, cm<sup>-1</sup>): ν = 1352s (NO<sub>3</sub><sup>-</sup>), 521m (*etda*). — Anal. for C<sub>2</sub>H<sub>8</sub>Ag N<sub>3</sub>O<sub>3</sub>: calcd. C 10.4, H 3.48, N 18.3; found C 10.2, H 3.44, N 17.8.

#### X-Ray crystallographic studies

Single crystals of **1** (0.25 × 0.15 × 0.10 mm<sup>3</sup>) and **2** (0.17 × 0.14 × 0.10 mm<sup>3</sup>) were mounted in random orientation on a glass fiber. Diffraction data were collected on a Bruker SMART Apex CCD diffractometer with MoK<sub>α</sub> radiation (λ = 0.71073 Å) at 293 K using an ω scan mode. The collected frames were processed with the software SAINT [23]. The data were corrected for absorption using the program SADABS [24]. The structures were solved by Direct Methods and refined by full-matrix least-squares on *F*<sup>2</sup> using the SHELXTL software package [25]. All non-

hydrogen atoms were refined anisotropically. The positions of all hydrogen atoms were generated geometrically (*C*<sub>sp<sup>3</sup></sub>-H = 0.96, *C*<sub>sp<sup>2</sup></sub>-H = 0.93, N-H = 0.90, and O-H = 0.85 Å) and included in the structure factor calculations with fixed isotropic displacement parameters but were not refined. The water molecules were also refined anisotropically, their oxygen atoms being treated with hydrogen atoms found in difference Fourier density maps. Crystal data, data collection parameters and details of the structure refinement are given in Table 1.

Crystal data (excluding structure factors) for the structures in this paper have been deposited with The Cambridge Crystallographic Data Centre as supplementary publications CCDC 249111 and CCDC 249112. Copies of the data can be obtained free of charge *via* [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).

## Results and Discussion

Treatment of a suspension of Ag(*hba*) with an excess of ethylenediamine (*etda*) in aqueous methanol afforded a light-yellow solution from which yellow blocks of the trihydrate [Ag<sub>2</sub>(μ-*etda*)<sub>2</sub>](*hba*)<sub>2</sub> · 3 H<sub>2</sub>O (**1**) were isolated in a yield of 55 %. Microanalytical data were consistent with the solid having a 1 : 1 : 1 of Ag : *etda* : *hba* ratio. The peaks at 1601 and 1453 cm<sup>-1</sup> for ν(CO<sub>2</sub><sup>-</sup>) and at 1367 cm<sup>-1</sup> for ν(NO<sub>2</sub>) in the

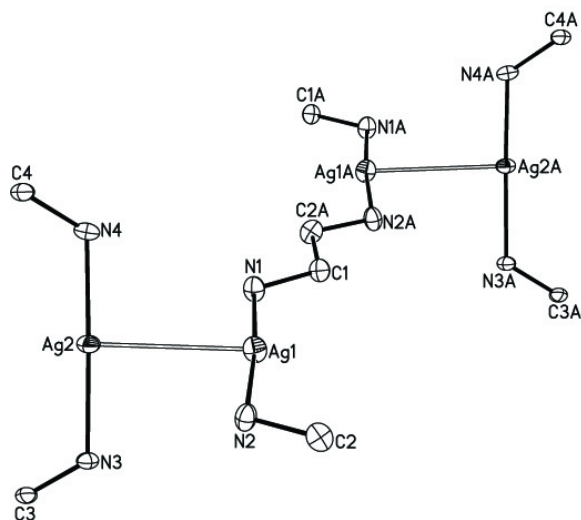


Fig. 1. A view of the edta ligand and the  $\text{Ag}^{\text{I}}$  coordination environment in the cation  $[\text{Ag}_2(\mu\text{-etda})_2]^{2+}$  of **1**. Displacement ellipsoids are drawn at the 40 % probability level. Hydrogen atoms are omitted for clarity. Selected bond lengths (Å) and angles ( $^\circ$ ):  $\text{Ag}(1)\text{--N}(1)$  2.140(6),  $\text{Ag}(1)\text{--N}(2)$  2.138(6),  $\text{Ag}(2)\text{--N}(3)$  2.124(6),  $\text{Ag}(2)\text{--N}(4)$  2.115(6),  $\text{Ag}(1)\cdots\text{Ag}(2)$  3.0523(11);  $\text{N}(1)\text{--Ag}(1)\text{--N}(2)$  175.3(3),  $\text{N}(3)\text{--Ag}(2)\text{--N}(4)$  171.0(2),  $\text{N}(1)\text{--Ag}(1)\cdots\text{Ag}(2)$  88.85(18),  $\text{N}(2)\text{--Ag}(1)\cdots\text{Ag}(2)$  86.49(17),  $\text{N}(3)\text{--Ag}(2)\cdots\text{Ag}(1)$  88.18(17),  $\text{N}(4)\text{--Ag}(2)\cdots\text{Ag}(1)$  93.81(19).

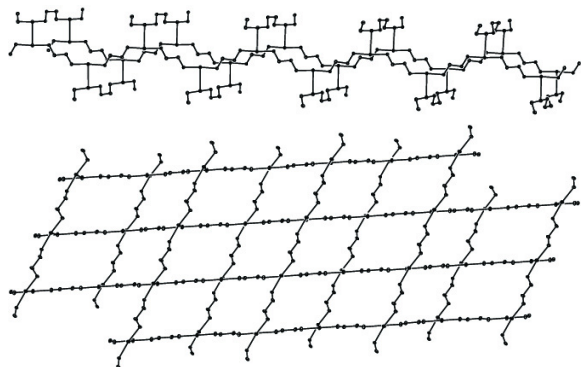


Fig. 2. View of a 2D sheet of cations  $[\text{Ag}_2(\mu\text{-etda})_2]^{2+}$  in **1**. Top view (top) and side view (bottom).

IR spectrum indicate the presence of hba in complex **1**, complemented by a peak at  $526\text{ cm}^{-1}$  indicative of the ligand edta. X-Ray structural analysis revealed that complex **1** forms a two-dimensional (2D) ladder coordination polymer and crystallizes in the monoclinic space group  $P2_1/n$ . The asymmetric unit consists of two silver(I) cations, two complete en ligands, two hba anions, and three water molecules. Each silver(I) atom adopts a T-shape coordination of

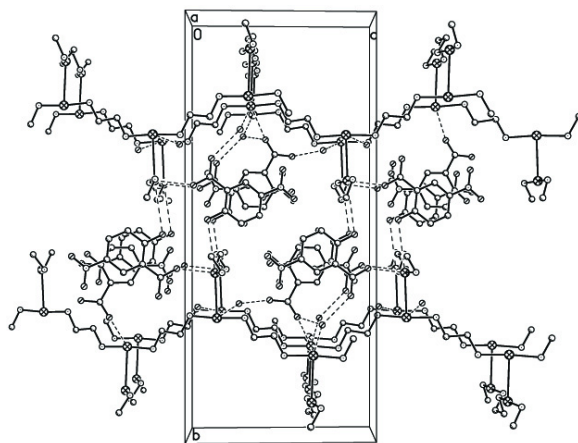


Fig. 3. Packing diagram of complex **1** in projection onto the  $bc$  plane.

two nitrogen atoms of different edta ligands and one neighboring Ag atom (see Fig. 1). The average Ag–N bond length and N–Ag–N bond angle are 2.129(6) Å and  $173.2(2)^\circ$ , respectively. The Ag $\cdots$ Ag distance is 3.0523(11) Å in **1**, indicative of the silver(I) ions being interacting, as the separation between them is obviously shorter than the sum (3.40 Å) of the van der Waals radii [26]. The sawtooth-shaped edta ligand bridges two silver atoms to form two zig-zag chains that are connected through Ag $\cdots$ Ag interactions, resulting in the formation of a non-coplanar ladder network, as shown in Fig. 2. Furthermore, the structure of complex **1** contains hba counterions and water molecules, both of which are involved in hydrogen-bonded interactions with the coordinating edta groups. Fig. 3 displays the packing of complex **1** viewed in the  $bc$  plane. The N–H $\cdots$ O( $\text{H}_2\text{O}$ ) distances are in the range of 2.14–2.59 Å, with corresponding N $\cdots$ O separations ranging from 3.039(9) to 3.367(10) Å. The N–H $\cdots$ O(hba) distances are in the range of 2.23–2.61 Å, with corresponding N $\cdots$ O separations ranging from 3.082(10) to 3.207(10) Å. There are also hydrogen-bonded interactions between hba and water (see Table 2). The ( $\text{H}_2\text{O}$ )O–H $\cdots$ O(hba) distances are 1.73 and 2.50 Å, with corresponding O $\cdots$ O separations between 2.741(9) and 3.166(10) Å. The ( $\text{H}_2\text{O}$ )O–H $\cdots$ O( $\text{H}_2\text{O}$ ) distances are in the range of 1.76–1.92 Å, with corresponding O $\cdots$ O separations ranging from 2.740(9) to 2.754(9) Å. Strong intramolecular hydrogen bonds are observed in the hba anions, as indicated by O(8) $\cdots$ O(7) and O(3) $\cdots$ O(2) distances of 2.490(9) and 2.519(10) Å, respectively

Table 2. Hydrogen bond lengths (Å) and angles (°).

D–H...A	<i>d</i> (D–H)	<i>d</i> (H...A)	<i>d</i> (D...A)	∠(DHA)
<b>Complex 1</b>				
N(1)–H(1A)...O(2) <sup>#5</sup>	0.90	2.23	3.082(10)	157.9
N(1)–H(1D)...O(1W)	0.90	2.31	3.145(10)	155.1
N(4)–H(4A)...O(1W)	0.90	2.42	3.127(9)	135.0
N(4)–H(4A)...O(2W)	0.90	2.59	3.367(10)	145.1
N(4)–H(4D)...O(9) <sup>#5</sup>	0.90	2.34	3.198(9)	159.9
N(2)–H(2A)...O(6)	0.90	2.32	3.207(10)	166.9
N(2)–H(2D)...O(2W) <sup>#1</sup>	0.90	2.56	3.384(10)	152.0
N(2)–H(2D)...O(5)	0.90	2.61	3.176(10)	121.5
N(3)–H(3A)...O(1W) <sup>#1</sup>	0.90	2.14	3.039(9)	172.8
N(3)–H(3D)...O(5)	0.90	2.28	3.179(9)	174.1
O(2W)–H(2WA)...O(5) <sup>#2</sup>	0.85	2.50	3.166(10)	136.2
O(3)–H(3)...O(2)	0.85	1.75	2.519(10)	149.1
O(8)–H(8B)...O(7)	0.85	1.72	2.490(9)	148.6
O(3W)–H(3WA)...O(6)	1.06	1.73	2.741(9)	157.1
O(3W)–H(3WB)...O(1W) <sup>#4</sup>	1.08	1.86	2.740(9)	136.4
O(1W)–H(1WA)...O(3W) <sup>#3</sup>	1.01	1.76	2.740(9)	160.9
O(1W)–H(1WB)...O(2) <sup>#6</sup>	0.98	1.92	2.754(9)	141.2
<b>Complex 2</b>				
N(1)–H(1A)...O(4) <sup>#12</sup>	0.90	2.39	3.099(10)	135.5
N(1)–H(1B)...O(3) <sup>#13</sup>	0.90	2.33	3.171(9)	156.2
N(2)–H(2A)...O(7) <sup>#14</sup>	0.90	2.35	3.192(7)	154.8
N(2)–H(2B)...O(4) <sup>#15</sup>	0.90	2.33	3.075(9)	139.6
N(3)–H(3A)...O(5) <sup>#16</sup>	0.90	2.35	3.199(7)	157.2
N(3)–H(3B)...O(6) <sup>#17</sup>	0.90	2.37	3.080(9)	135.3
N(4)–H(4C)...O(1)	0.90	2.40	3.121(10)	137.8
N(4)–H(4D)...O(3) <sup>#13</sup>	0.90	2.37	3.207(9)	154.2

Symmetry transformations used to generate equivalent atoms: for complex 1: <sup>#1</sup>  $x+1, y, z$ ; <sup>#2</sup>  $x-1, y, z$ ; <sup>#3</sup>  $x-1/2, 1/2-y, z-1/2$ ; <sup>#4</sup>  $x+1/2, 1/2-y, z+1/2$ ; <sup>#5</sup>  $x, y, z-1$ ; <sup>#6</sup>  $x-1, y, z-1$ ; for complex 2: <sup>#12</sup>  $x+1/2, 3/2-y, z-1/2$ ; <sup>#13</sup>  $x, 2-y, z-1/2$ ; <sup>#14</sup>  $x+1/2, y+1/2, z$ ; <sup>#15</sup>  $x+1/2, 3/2-y, z+1/2$ ; <sup>#16</sup>  $x+1, y, z$ ; <sup>#17</sup>  $1-x, 1-y, -z$ .

(Table 2). The separations of Ag...O(H<sub>2</sub>O) (3.211 Å) and Ag...O(O<sub>2</sub>C) (3.207 Å) are considered non-bonding.

The reaction of AgNO<sub>3</sub> and etda produced a light yellow solid in a yield of 62 % which gave microanalytical data consistent with a 1 : 1 metal-to-ligand ratio. The IR spectrum shows a strong peak at 1352 cm<sup>-1</sup>, which suggests that the NO<sub>3</sub><sup>-</sup> anion is not coordinated. A peak at 521 cm<sup>-1</sup> is indicative of the ligand etda. Yellow prism-shaped X-ray quality crystals formed at the interface between organic ligand and inorganic salt in MeCN solution by slow diffusion. X-Ray structural analysis revealed that this complex forms a three-dimensional (3D) coordination network, which crystallizes in the monoclinic space group *C2/c*. The asymmetric unit consists of one silver(I) cation, one complete etda ligand, and one nitrate anion. Each silver atom exhibits a square planar coordination geometry of two nitrogen atoms of two different etda ligands and two neighboring Ag atoms (see Fig. 4). The average Ag–N bond length of 2.110(5) Å

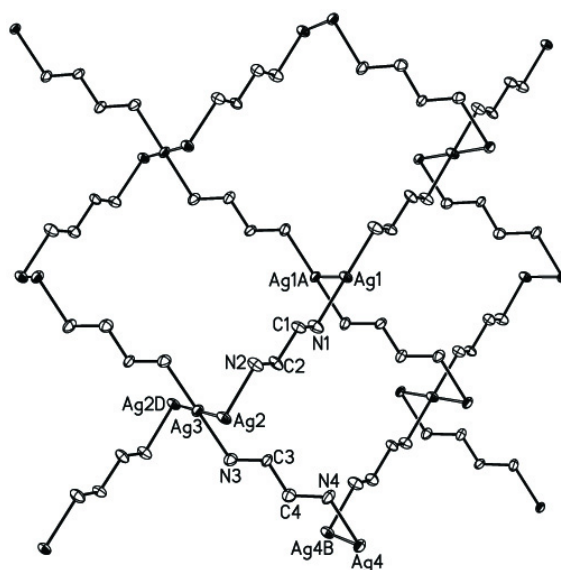


Fig. 4. A view of the etda ligand and the Ag<sup>I</sup> coordination environment in [Ag(μ-etda)]<sup>+</sup> of 2. Displacement ellipsoids at the 40 % probability level, hydrogen atoms omitted for clarity. Selected bond lengths (Å) and angles (°): Ag(1)–N(1) 2.124(5), Ag(2)–N(2) 2.096(5), Ag(3)–N(3) 2.127(5), Ag(4)–N(4) 2.091(5), Ag(2)···Ag(3) 3.139(3); N(1)<sup>#1</sup>–Ag(1)–N(1) 179.999(1), N(3)–Ag(3)–N(3)<sup>#6</sup> 180.0(3), N(3)–Ag(3)···Ag(2) 87.50(16), N(1)–Ag(1)···Ag(1)<sup>#2</sup> 92.11(16), N(1)–Ag(1)···Ag(1)<sup>#3</sup> 87.89(16), N(2)–Ag(2)···Ag(3) 87.50(16), N(3)<sup>#6</sup>–Ag(3)···Ag(2) 92.50(16), N(4)–Ag(4)···Ag(4)<sup>#9</sup> 88.06(16), N(4)–Ag(4)···Ag(4)<sup>#10</sup> 91.94(16), Ag(3)···Ag(2)···Ag(3)<sup>#5</sup> 180.0, Ag(1)<sup>#2</sup>···Ag(1)···Ag(1)<sup>#3</sup> 180.0. (Symmetry codes: <sup>#1</sup>  $1-x, 2-y, -z$ ; <sup>#2</sup>  $1-x, y, -1/2-z$ ; <sup>#3</sup>  $1-x, y, 1/2-z$ ; <sup>#6</sup>  $3/2-x, 3/2-y, -z$ ; <sup>#9</sup>  $2-x, y, 1/2-z$ ; <sup>#10</sup>  $2-x, y, 3/2-z$ ).

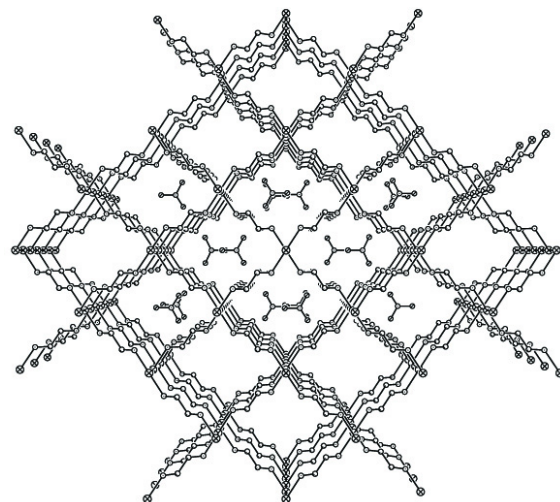


Fig. 5. View of the crystal structure of complex 2, showing a 3D topological network and nitrate anions encapsulated in the channels.

in **2** is slightly shorter than that of 2.129(6) Å in **1**. The etda ligands are bridging the silver atoms featuring Ag...Ag connections, with both N–Ag–N and Ag...Ag...Ag angles of 180°, resulting in the formation of a 3D topological network, as shown in Fig. 5. The Ag...Ag distance of 3.139(3) Å in **2** is slightly longer than that of 3.0523(11) Å in **1**, but it is still shorter than twice the van der Waals radius (1.7 Å) of the silver atoms, indicative of weak Ag...Ag interactions [26, 27]. The nitrate anions occupy the channels and interact with the walls of the channels *via* N–H...O hydrogen bonds. The N–H...O(NO<sub>3</sub><sup>−</sup>) distances are in the range of 2.33–2.40 Å, with corresponding N...O separations ranging from 3.080(9) to 3.207(9) Å. It is interesting to note that the nitrate oxygen atoms show an average O...H–N angles of 156° (see Table 2), which combined with the O...H...O angles of 54°, leads to a description of these

interactions as three-centered, bifurcated hydrogen bonds [28].

In summary, two different 1 : 1–etda silver(I) complexes have been prepared and characterized with hba<sup>−</sup> and NO<sub>3</sub><sup>−</sup> counterions, respectively. The crystal structures of the two complexes indicate that etda acts as a bridging ligand for silver atoms rather than as a chelating ligand. The formation of the 2D and 3D coordination polymer networks results from the Ag–Ag argentophilic interactions. Such supramolecular metal-metal interactions in the topology of the poly-dimensional networks are in active study in this laboratory.

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