



## Note

## A novel 3D silver(I) supramolecular framework assembled from hybrid ligands incorporating Ag···C interactions

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## ABSTRACT

A novel silver(I) complex with mixed 2-amino-4-methylpyrimidine (mapym) and naphthalene-2,6-dicarboxylic acid (H<sub>2</sub>npd),  $\{[Ag(mapym)(npd)_{0.5} \cdot H_2O]\}_n$  (**1**), has been synthesized and characterized by elemental analysis, IR spectroscopy and single crystal X-ray diffraction. In **1**, Ag(I) ions exhibit a T-shaped coordination geometry and are doubly clamped by two npd anions to form  $[Ag_2C_2O_4]$  units incorporating a short Ag···Ag contact of 2.8192(15) Å. The coordination bonds link Ag(I) ions and npd ligands to form one-dimensional (1D) fish-bone chains which are extended into a two-dimensional (2D) layer by inter-chain hydrogen bonds. Interestingly, Ag···C<sub>npd</sub>( $\eta^2$ ) interactions of av. 2.88 Å combine inter-layer hydrogen bonds and  $\pi\cdots\pi$  stacking interactions to give **1** a three-dimensional (3D) supramolecular framework. The photoluminescence properties of **1** were investigated in the solid state at room temperature.

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## 1. Introduction

The design and generation in the crystalline phase of infinite periodic self-assembled architectures called molecular networks is the subject of active research. In particular, coordination networks based on mutual bridging between organic and metallic tectons or building blocks, have attracted considerable interest because of their potential applications in many fields, such as storage, conductivity, catalysis, photophysics, photochemistry, fluorescence, magnetism, biological properties [1–9]. Although the self-assembly of these periodic molecular networks is mainly controlled by the nature of the metal ions and the structural characterization of organic ligands, it is also vastly influenced by other factors such as the solvent systems, the temperatures, the counter ions with different bulk or coordination ability, the templates and metal/ligand ratio [10–17]. Parallel to the above aspects, the non-covalent forces such as hydrogen bond,  $\pi\cdots\pi$  stacking, anion··· $\pi$ , metal···metal interactions based on  $d^{10}$  closed-shell metal cations (Cu<sup>1</sup>, Ag<sup>1</sup>, Au<sup>1</sup>), and metal···aromatic interactions can also intensively impact the supramolecular topology and dimensionality [18–22]. Despite some recent improvement, prediction and control the supramolecular assembly remain long-standing challenges, and much more work is required to do to understand the intra- or intermolecular interactions that direct the patterns of supramolecular structures and crystal packing in the solid state.

As we known, both *N*-donor heterocyclic ligands such as pyridyl, pyrimidyl, and pyrazinyl ligands and *O*-donor ligands such as di-, tri-, tetra-carboxylates are excellent synthons for the rational design of coordination polymers [23–34], so as a continuation of our research on silver/2-aminopyrimidine/dicarboxylates system, we focus on the silver/H<sub>2</sub>npd/2-aminopyrimidyl derivative system, and successfully obtained a novel 3D supramolecular framework constructed from 1D Ag-npd chains *via* Ag···C<sub>npd</sub>( $\eta^2$ ), hydrogen bonds and  $\pi\cdots\pi$  interactions.

## 2. Experimental procedure

## 2.1. Materials and methods

All the reagents and solvents employed were commercially available and used as received without further purification. Infrared spectra were recorded on a Nicolet AVATAT FT-IR360 spectrometer as KBr pellets in the frequency range 4000–400 cm<sup>−1</sup>. The elemental analyses (C, H, N contents) were determined on a CE instruments EA 1110 analyzer. Photoluminescence measurements were performed on a Hitachi F-4500 fluorescence spectrophotometer with solid powder on a 1 cm quartz round plate.

2.2. Synthesis of complex  $[Ag(mapym)(npd)_{0.5} \cdot H_2O]_n$  (**1**)

A mixture of AgNO<sub>3</sub> (167 mg, 1 mmol), H<sub>2</sub>npd (216 mg, 1 mmol) and mapym (109 mg, 1 mmol) was stirred in CH<sub>3</sub>CN–H<sub>2</sub>O mixed solvent (10 mL, v/v: 1/1). Then aqueous NH<sub>3</sub> solution (25%, 0.5 mL) was dropped into the mixture to give a clear solution under

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**Table 1**  
Crystallographic data for complex **1**.

Complexes	<b>1</b>
Formula	AgC <sub>11</sub> H <sub>12</sub> N <sub>3</sub> O <sub>3</sub>
<i>M</i> <sub>r</sub>	342.11
Crystal system	triclinic
Space group	<i>P</i> 1
<i>a</i> (Å)	6.959(4)
<i>b</i> (Å)	8.524(5)
<i>c</i> (Å)	11.012(7)
$\alpha$ (°)	68.269(10)
$\beta$ (°)	76.299(10)
$\gamma$ (°)	69.283(10)
<i>Z</i>	2
<i>V</i> (Å <sup>3</sup> )	563.3(6)
<i>D</i> <sub>calc</sub> (g cm <sup>-3</sup> )	2.017
$\mu$ (mm <sup>-1</sup> )	1.793
<i>F</i> (0 0 0)	340
Number of unique reflections	1868
Number of observed reflections [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )]	1678
Parameters	164
GOF	1.030
Final <i>R</i> indices [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] <sup>a,b</sup>	<i>R</i> <sub>1</sub> = 0.0391, <i>wR</i> <sub>2</sub> = 0.0997
<i>R</i> indices (all data)	<i>R</i> <sub>1</sub> = 0.0428, <i>wR</i> <sub>2</sub> = 0.1017
Largest difference peak and hole (e Å <sup>-3</sup> )	1.323 and -0.57

<sup>a</sup>  $R_1 = \sum ||F_o| - |F_c|| / \sum |F_o|$ .

<sup>b</sup>  $wR_2 = [\sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)]^{0.5}$ .

ultrasonic treatment. The resultant solution was allowed to evaporate slowly in darkness at room temperature for several days to give colorless crystals of **1** (yield, 46%). They were washed with a small volume of cold CH<sub>3</sub>OH and diethyl ether. *Anal.* Calc. for AgC<sub>11</sub>H<sub>12</sub>N<sub>3</sub>O<sub>3</sub>: C, 38.62; H, 3.53; N, 12.28. Found: C, 38.58; H, 3.41; N, 12.35%. IR (KBr):  $\nu$  (cm<sup>-1</sup>) = 3405 (m), 3317 (m), 3149 (m), 1592 (s), 1561 (s), 1479 (s), 1360 (m), 1315 (m), 1217 (m), 750 (m), 561 (w), 540 (w), 476 (m).

### 2.3. X-ray crystallography

Single crystal of the complex **1** with appropriate dimensions was mounted on a glass fiber and used for data collection. Data were collected on a Bruker-AXS CCD diffractometer equipped with a graphite-monochromated Mo K $\alpha$  radiation source ( $\lambda = 0.71073$  Å). Absorption correction was performed with the SADABS program [35]. The structure was solved by direct methods using SHELXS-97 [36] and refined by full-matrix least-squares techniques using SHELXL-97 [37]. All H atoms were refined using a riding model, with C<sub>aromatic</sub>–H = 0.95 and C<sub>methyl</sub>–H = 0.98 Å. The positions of the water H atoms were refined with the O–H bond length restrained to 0.85 Å. The crystallographic details of **1** are summarized in Table 1. Selected bond lengths and angles are collected in Table 2.

## 3. Results and discussion

### 3.1. Syntheses and IR

The synthesis of complex **1** was carried out in the darkness to avoid photodecomposition. The formation of the products is not significantly affected by changes of the reaction mole ratio of organic ligands to metal ions, and the resultant crystals are insoluble in water and common organic solvents. The IR spectrum and elemental analyses of **1** are fully consistent with its formation. Its IR spectrum (Fig. S1) exhibits the absorptions in the range of ~3411 cm<sup>-1</sup> to ~3317 cm<sup>-1</sup>, corresponding to the N–H stretching vibrations of the amino group. Strong characteristic bands of deprotonated carboxylic groups are observed in the range of ~1658 cm<sup>-1</sup> to ~1560 cm<sup>-1</sup> for the asymmetric vibrations and ~1475 cm<sup>-1</sup> to ~1356 cm<sup>-1</sup> for symmetric vibrations, respectively.

**Table 2**  
Selected bond distances (Å) and angles (°) for **1**.

Bond lengths (Å)			
Ag1–O2 <sup>i</sup>	2.243(4)	Ag1…Ag1 <sup>i</sup>	2.8192(15)
Ag1–O1	2.250(3)	Ag1–N1	2.316(4)
Ag1…C8 <sup>v</sup>	2.936(6)	Ag1…C9 <sup>v</sup>	2.814(6)
Angles (°)			
N1–Ag1…Ag1 <sup>i</sup>	172.44(10)	O2 <sup>i</sup> –Ag1–O1	153.83(14)
O2 <sup>i</sup> –Ag1…Ag1 <sup>i</sup>	82.15(9)	O1–Ag1–N1	93.97(14)
O1–Ag1…Ag1 <sup>i</sup>	79.71(10)	O2 <sup>i</sup> –Ag1–N1	102.41(14)
D–H…A		D…A (Å)	
N2–H2B…O2 <sup>i</sup>	3.072(6)	N2–H2C…O1W <sup>iii</sup>	2.888(6)
O1W–H1WB…O1 <sup>iv</sup>	2.880(6)	O1W–H1WA…N3	2.930(6)

Symmetry codes: (i) 3 – *x*, –*y*, 1 – *z*; (iii) 4 – *x*, –1 – *y*, –*z*; (iv) 3 – *x*, –*y*, –*z*; (v) 2 – *x*, –*y*, 1 – *z*.

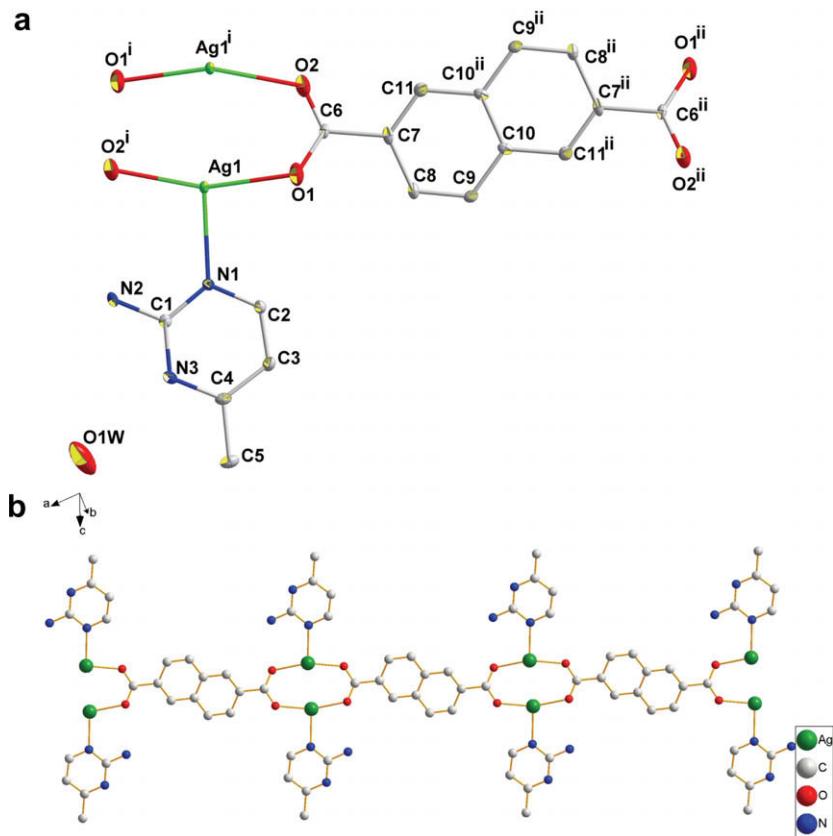
The absence of the characteristic bands at around ~1700 cm<sup>-1</sup> attributed to the carboxylic groups, indicating that the complete deprotonation of all carboxylate groups in **1** upon reaction with Ag ions [38].

### 3.2. Crystal structure of $\{[\text{Ag}(\text{mapym})(\text{npd})_{0.5}\cdot\text{H}_2\text{O}]\}_n$ (1)

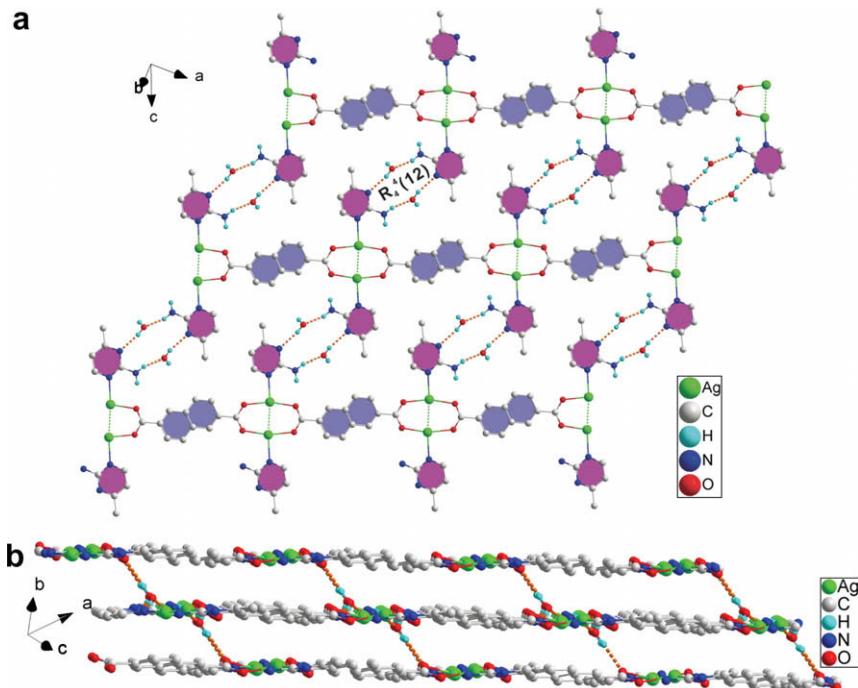
The X-ray crystallographic analysis shows that **1** crystallizes in the triclinic space group *P*1 and has an infinite 1D fish-bone-like chain structure. As shown in Fig. 1a, a crystallographically independent Ag(I) ion is surrounded with a T-shaped geometry by two oxygen atoms belonged to two npd anions and a heterocyclic nitrogen atom [Ag1–O2<sup>i</sup> = 2.243(4), Ag1–O1 = 2.250(3), Ag1–N1 = 2.316(4) Å] without consideration of Ag…Ag interaction. It is noteworthy that the ndc anion locates on an inversion center and clamps a pair of Ag(I) ions with a *syn*-*syn* mode to form an [Ag<sub>2</sub>C<sub>2</sub>O<sub>4</sub>] eight-membered ring. On the other hand, as depicted in Fig. 1b, the binuclear Ag(I) units were linked to form 1D infinite chains by npd anions with a symmetrical  $\mu_4\text{-}\eta^1\text{:}\eta^1\text{:}\eta^1\text{:}\eta^1$  bridging mode and the mapym ligands monodentately coordinated to Ag(I) ions in a direction nearly perpendicular to the ribbon axis. Another heterocyclic nitrogen atom doesn't participant in coordinating to Ag(I) ion which may be due to the steric factor of methyl group on pyrimidine.

Hydrogen bonds also exist in the adjacent 1D chains. Lattice water molecules (both donor and acceptor), amino group of mapym (donor) and heterocyclic nitrogen atoms (acceptor) are hydrogen-bonded to form a  $R_4^4(12)$  hydrogen motif centered at crystallographic inversion centers according to the graph-set notation [39]. This kind of hydrogen bonding motif extends the 1D chains into a 2D layer (Fig. 2a and Table 2). The carboxylate groups of npd anions and lattice water molecules are hydrogen bonded between adjacent layers with O1W…O1<sup>iv</sup> of 2.880(6) Å (Fig. 2b).

After carefully analyzing the crystal packing of the molecules, we surprisingly found the existence of the closest contacts between Ag and naphthyl ring which is associated with the C8 and C9 atoms (Fig. 3). The Ag…C contacts are 2.936(6) and 2.814(6) Å for C8 and C9, respectively, both of which fall in the range of Ag…C interactions and are obviously shorter than the sum of the van der Waals radii of Ag and C (3.42 Å) [40,41]. The next closest contact (Ag1…C10<sup>v</sup> = 3.272(6) Å) between the silver and carbon atoms is above 3.13 Å, well beyond the values reported for the Ag(I)/aromatic compounds [42–44]. Furthermore, in the previously documented Ag(I)/aromatic compounds, Ag(I) ions have high affinity to ligate at the shortest carbon–carbon bond [45], and this tendency is basically maintained in **1** (C8–C9 = 1.359(7) Å, the second shortest C–C bond in npd), indicative of the high  $\pi$ -electron density accumulated on this bond. Herein the Ag…C interaction sites are



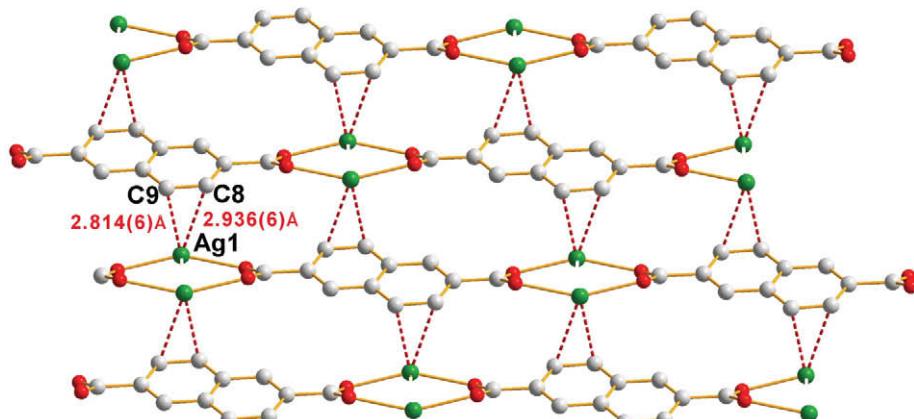
**Fig. 1.** (a) The coordination environment of the Ag(I) ion and the linkage modes of ligands in **1** with 50% thermal ellipsoid probability, hydrogen atoms are omitted for clarity. (b) 1D fish-bone chain in **1** (symmetry code: (i)  $3 - x, -y, 1 - z$ ; (ii)  $1 - x, 1 - y, 1 - z$ ).



**Fig. 2.** (a) The 2D layer incorporating  $R_4^4(12)$  hydrogen bond motif (golden dashed lines). (b) Inter-layer hydrogen bonds. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

not necessarily the shortest C–C bonded carbon atoms which may be dominated by various factors, such as the size and geometry of the aromatic, steric hindrance, molecular packing energy, and

other structural details. These interactions in **1** may be considered as a medium dihapto aromatic coordination of the benzene ring of npd to the Ag(I) ion, and are very significant, in the present case, for



**Fig. 3.** The Ag...C interactions (red dashed lines) and mapym ligands are omitted for clarity. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

the packing of **1** in the solid state. Although Ag...C interactions have been widely observed [46–47], such interactions involving npd unit in the solid state have rarely been documented in the literatures [48]. The Ag...C<sub>npd</sub>( $\eta^2$ ) interactions as well as inter-layer hydrogen bonds and  $\pi\cdots\pi$  stacking interactions play important roles in extending the 1D chains to 3D supramolecular framework (Fig. S2) and finally contribute to the stabilization of the crystal packing (symmetry code: (i) 3 – x, –y, 1–z; (iv) 3 – x, –y, –z; (v) 2 – x, –y, 1 – z).

### 3.3. Photoluminescence properties

The solid-state photoluminescence data for both free ligands and complex **1** at room temperature are shown in Fig. 4 and Table S2. Complex **1** exhibits photoluminescence in the solid state, with an emission maximum at 423 nm upon excitation at 330 nm at room temperature. To understand the nature of the emission band, we analyzed the photoluminescence properties of the corresponding free ligands and found that mapym and H<sub>2</sub>npd ligand emit photoluminescence in 350 and 458 nm, respectively. The emission bands of H<sub>2</sub>npd can be assigned to the  $\pi^* \rightarrow n$  transition as previously reported [49], which is very weak compared to that of the

$\pi^* \rightarrow \pi$  transition of the mapym, so the carboxylate ligands almost have no contribution to the fluorescent emission of **1** [50]. The shifted emissions and enhancement of luminescence of **1** were attributed to ligand coordination to the metal center, which effectively increases the rigidity of the ligand and reduces the loss of energy by radiationless decay. Therefore, the emission band of **1** can be assigned to the intraligand transitions photoluminescence emission [48].

## 4. Conclusions

A novel Ag(I) complex with mixed N- and O-donor ligands has been synthesized and structurally characterized. In **1**, a pair of Ag(I) ions are clamped by two npd anions leading to a strong argentophilic interaction. The molecules of **1** are packed into a 3D supramolecular framework via Ag...C interactions between the Ag(I) ion and the phenyl ring of npd,  $\pi\cdots\pi$  interactions and inter-layer hydrogen bonds. The Ag...C interaction is a key factor controlling packing of molecules as well as the structure dimensionality. The photoluminescence properties were also investigated in the solid state at room temperature.

## Acknowledgments

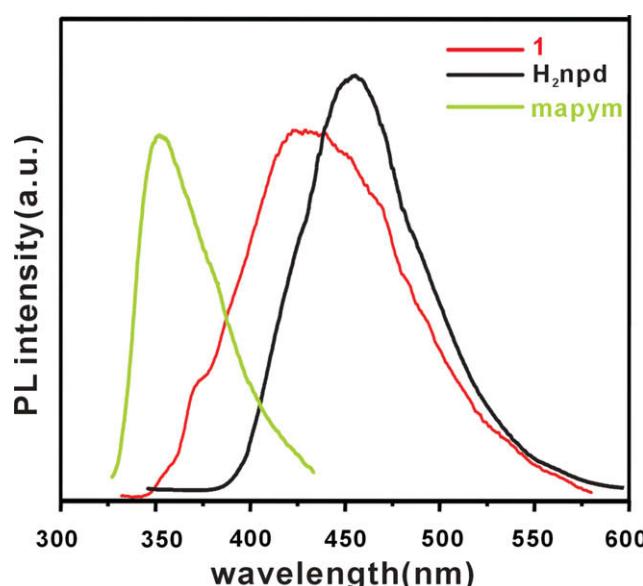
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## Appendix A. Supplementary material

CCDC 742809 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif). Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorgchem.2010.02.026.

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**Fig. 4.** Emission spectra of the complex **1** and free ligands.

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