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Guest inclusion by Borromean weave coordination networks

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ABSTRACT

Reaction of *N,N″* -ethylene-1,2-diylbis(3-pyridin-3-ylurea) (**L**) with AgNO$_3$ in a variety of solvents gives a total of five 2-D Borromean weave coordination polymer networks that adopt three structural types depending on the interactions to the solvent pocket. The Borromean network is of formula [Ag$_2$(**L**)$_3$](NO$_3$)$_2$·*solvent* where the solvent is either a cluster of water molecules, mixtures of water and acetonitrile, water and methanol or chloroform and methanol. The Borromean structure is a thermodynamic sink and under fast crystallization conditions an alternative 2 + 2 metallomacrocycle forms that can result in metallogel formation. The metallogel structure transforms into the crystalline Borromean network over time.
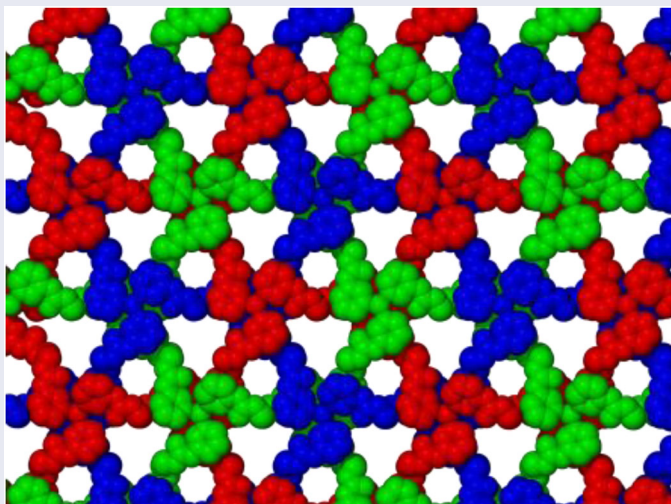
1. Introduction

The Borromean motif is an example of a prime link, formally 6_2^3 in Alexander–Briggs notation [1, 2]. Borromean topologies in chemistry are rare but have been

experimentally realized for DNA nanostructures [3], discrete metallosupramolecular assemblies [4, 5], 2-D coordination polymer sheets [6–12], and hydrogen- and halogen-bonded structures [5, 13–15]. Mutually entangled topologies in chemical systems are of considerable interest because of changes in properties such as mechanical robustness, framework cooperativity, and dynamical non-trivial behavior that can arise from mechanical bonding. Interest in this topic increased significantly with the award of the 2016 Nobel Prize in chemistry in the area of molecular machines [16–18]. While the Borromean topology and topological links in general such as the 2_1^2 (Hopf), 4_1^2 (Solomon), 6_1^2 (Star of David), 6_3^3 (cyclic [3]catenane), and a 12_1^4 [4]catenane have been engendered in chemical systems [19, 20], it is more unusual for molecular link systems to display host-guest chemistry. Previously, Barbour reported 2-D Borromean weave coordination polymers based on silver(I) complexes of extended difunctional ligands that are held together with argentophilic interactions [7], while we have reported a related example that includes a discrete water heptamer in a well-defined guest binding pocket [21]. This latter compound is of formula $[Ag_2(\mathbf{L})_3](NO_3)_2 \cdot 7H_2O$ (1) and arises from the reaction of $AgNO_3$ and N,N″ -ethylene-1,2-diylbis(3-pyridin-3-ylurea) (L) in aqueous DMF or aqueous THF. Both Ag^I ions in the asymmetric unit act as nodes in a network made up of large hexanuclear rings (Figure 1) formed from the doubly connected, dipyridyl ligands. The 2-D coordination polymer structure is supported by argentophilic interactions between pairs of stacked Ag^I cations (Ag···Ag 3.54 Å). The nitrate anions form infinite stacks, with each anion surrounded by a total of three urea groups from three different ligands, each belonging to a separate sheet (Figure 1). Each urea···nitrate interaction is an eight-membered hydrogen-bonded ring, and the hydrogen bonding is saturated [22] as each anion is surrounded by six NH hydrogen-bond donors. Along with the nitrate cavity, there is an additional cavity that is occupied by a seven-membered water cluster that forms long Ag···O interactions and strong OH···O interactions with the urea carbonyl groups. TGA analysis indicates that the water guest is very tightly bound in this system [21]. We now report a series of related host-guest complexes based on the same Borromean motif as in 1 in which the basic 2-D Borromean weave framework is able to accommodate a variety of guest species.

L
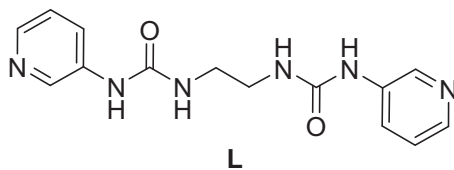
2. Results and discussion

In total, N,N″ -ethylene-1,2-diylbis(3-pyridin-3-ylurea) (L) forms five Borromean weave inclusion complexes upon slow crystallization in various solvent mixtures with silver(I) nitrate. Within each structure, the basic Borromean weave is similar but the argentophilic interactions and the way in which the 2-D Borromean sheets stack is governed by the included solvent guests. When the nitrate counter anion is replaced by either
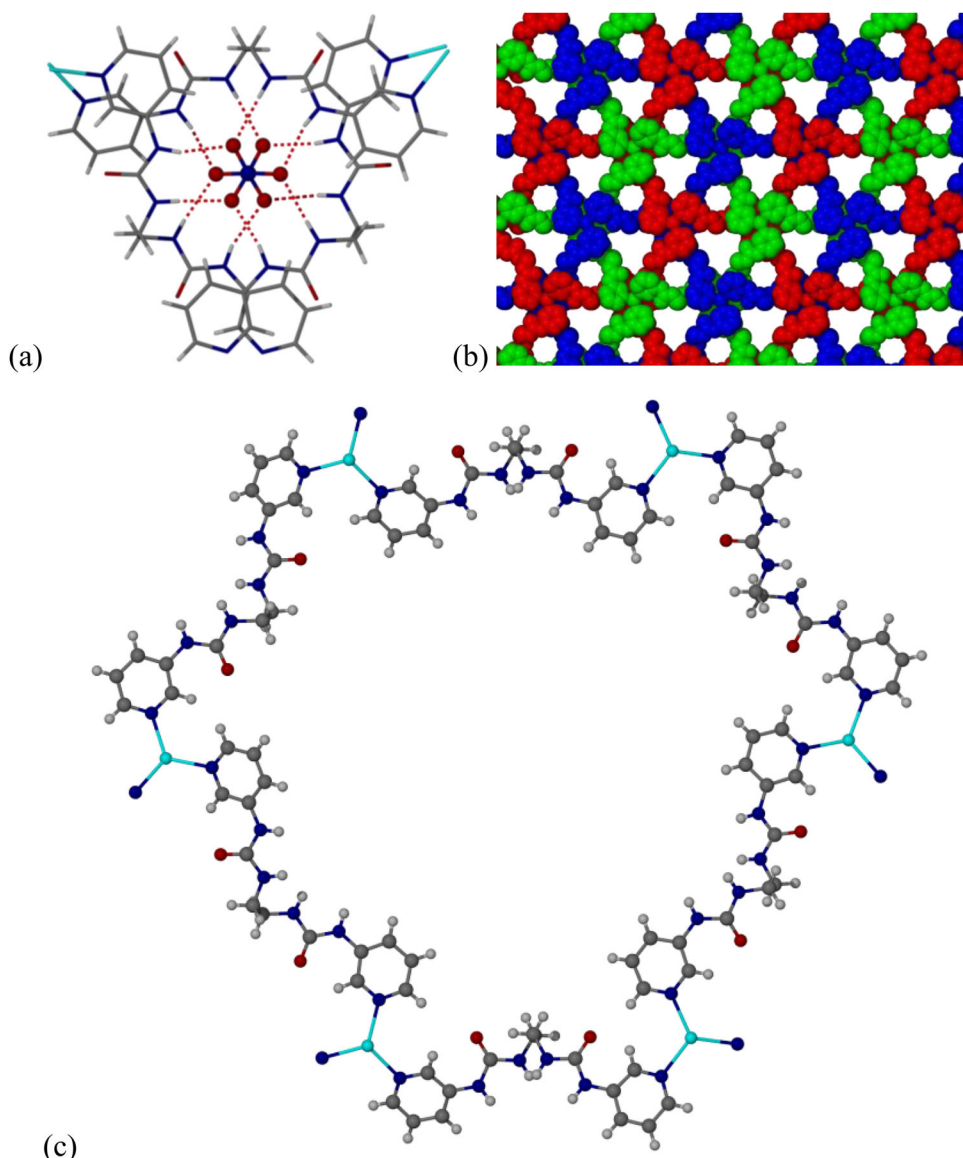
(a)

(b)

(c)

Figure 1. Structure of $[Ag_2(\mathbf{L})_3](NO_3)_2 \cdot 7H_2O$ (**1**) (a) saturated hydrogen bonding of the nitrate anions, (b) the three independent metallocyclic mesh networks linked in a Borromean topology. Circular voids contain stacks of nitrate anions, while the triangular voids are occupied by the ordered water heptamer. (c) The macrocyclic ring in **1** (approximately 31.5 Å diameter) comprising six Ag(I) ions and six ligands. Three ligands have their NH groups pointing into the macrocycle, and three further ligands point outside to give an approximately threefold symmetry [21]. (Color code: red = oxygen, blue = nitrogen, grey = carbon, white = hydrogen, cyan = silver).

acetate, tetrafluoroborate, or hexafluorophosphate, several coordination polymers can be prepared, but the Borromean structure is not reproduced and hence, the Borromean topology is likely to arise from the very specific saturated hydrogen bonding interaction to the trigonal nitrate anion. A similar dependency on counter anion

Table 1. Formulas of five Borromean inclusion complexes prepared from *N,N″*-ethylene-1,2-diyl-bis(3-pyridin-3-ylurea) and silver(I) nitrate in a variety of solvent mixtures.

Borromean [Ag$_2$(L)$_3$] (NO$_3$)$_2$·solvent	Solvent content	Solvent system
1	7H$_2$O	DMF and water (1:1, 6 mL)
2	5H$_2$O·MeOH	MeOH and water (1:1, 6 mL)
3	Analysis: 2MeCN·1.25CHCl$_3$·0.675MeOH X-ray: 0.16CHCl$_3$·0.08MeOH 384 Å^3, 86 e$^-$ per unit cell	MeCN, CHCl$_3$ and MeOH (3:3:4, 15 mL)
4	5H$_2$O·MeCN	MeCN and water (1:1, 6 mL)
5	Analysis: 7H$_2$O·2MeCN X-ray: 4.2H$_2$O·1.2MeCN 1816 Å^3 547 e$^-$ per unit cell	MeCN and CHCl$_3$ (1:1, 9 mL)

was observed with *N,N″*-pentylene-1,5-diylbis(3-pyridin-3-ylurea), which forms a range of coordination polymer systems including a quintuple helix but without any interpenetration or link formation [23].

When the crystallization of **L** in the presence of silver(I) nitrate was performed in a selection of other solvents in addition to the conditions used for **1**, four additional Borromean solvate materials were obtained. Their formulas were determined by elemental analysis and/or X-ray crystallography and are shown in Table 1. For **2** and **4**, the solvent content is more or less ordered and unambiguous. Solvent proved to be highly disordered for **3** and **5**, and in a preliminary report of the structure of **3**, solvent was not modeled [21]. The solvent was handled using the solvent masking implementation of the SQUEEZE [24] procedure in Olex2 [25]. This gave an estimate of the number of electrons in the structure arising from solvent. For both complexes, this proved to be lower than the solvent content implied by elemental analysis. It is likely that the complexes can include variable amounts of solvent within the crystal void and that the amount from elemental analysis (which used samples with limited drying in order to preserve solvent content) is an overestimate as a result of surface solvent, while the solvent content from the solvent masking procedure is correct for each crystal studied but likely an underestimate of the total capacity as a result of partial desolvation during crystal handling.

The four Borromean inclusion complexes **2**–**5** are closely related to **1** (although **3**–**5** are not isomorphous with **1**) with pairs of silver(I) ions linked by argentophilic interactions within each 2-D Borromean sheet. Complex **2** is isomorphous with **1** in *P*2$_1$/*c* with a similar crystal packing arrangement in which two of the ordered water guests have been replaced by one molecule of methanol. The methanol hydrogen bonds only to water molecules, while the water forms one long coordinate bond to one of the two independent silver ions in the same way as **1**; Ag–O 2.79 Å. Complex **3** has a similar-sized unit cell to **1** and **2** but is more symmetrical with a single independent silver(I) ion in space group *C*2/*c*. The ordered acetonitrile forms a long coordinate bond to the silver ion Ag–N 2.71 Å, and the remaining solvent occupies a disordered pocket. The lack of specific hydrogen bonding to the carbonyl ligands and guest--guest interactions is likely to be the cause of the higher symmetry. The Ag···Ag distance is reduced to 3.40 Å (from 3.54 Å in **1** and 3.59 Å in **2**). A single solvent cluster is able to coordinate to two Ag$_2$ argentophilically bonded units in adjacent weaves. Thus, channels of nitrate anions are produced and each Ag$_2$ unit stacks alternately with a solvent cluster.
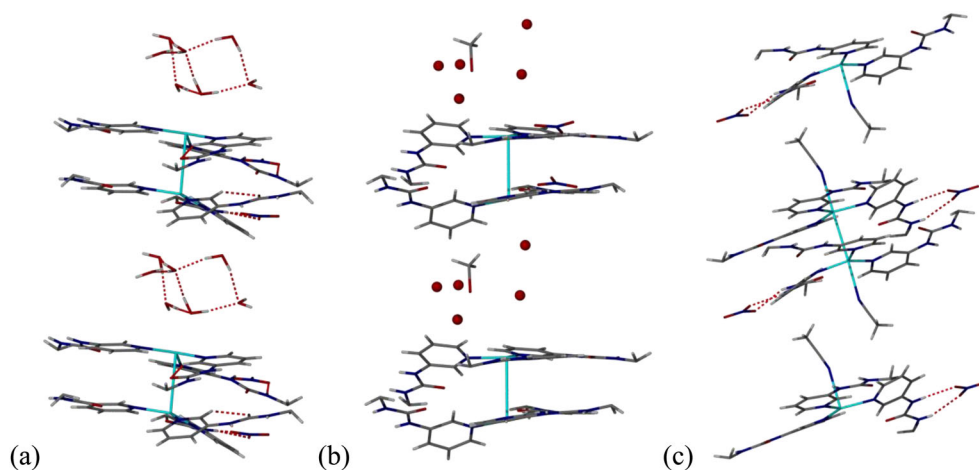
Figure 2. The stacked solvent cluster–Ag$_2$ units of (a) **1**, (b) **2**, and (c) **3**.

Complex **4** combines elements of **2** and **3**. As in **3**, the acetonitrile molecules are coordinated to the silver ion Ag–N 2.56 Å allowing a short Ag···Ag interaction of 3.40 Å. The space between the two back-to-back acetonitrile molecules in adjacent Borromean sheets is occupied by a discrete, ordered (H$_2$O)$_8$ water cluster, and hence, the whole (MeCN)$_2$(H$_2$O)$_8$ solvent pocket is similar in size to twice that of the (H$_2$O)$_7$ cluster in **1**. Creating this "double cavity" results in significantly different crystal packing in which the two Borromean sheets are shifted with respect to one another.

Structure **5** is particularly interesting since it involves the shortest argentophilic interaction of just 3.25 Å. This arises because the silver ions are not coordinated to any solvent molecule on the other side of the Ag···Ag pair and instead, a urea nitrogen atom of a ligand on an adjacent Borromean sheet forms a 3.48 Å long contact to the silver ions. This lack of solvation serves to increase the strength of the argentophilic interactions and give rise to a further different packing type in which the 2-D Borromean sheets are more closely interdigitated. However, a total of four disordered solvent-filled voids per unit cell remain, each of volume 454 Å^3 filled with disordered water and acetonitrile. While the solvent was refined using the solvent mask procedure, the position of the individual Fourier peaks suggests that there is a water molecule in each layer that is hydrogen bonded in between a pair of urea carbonyl groups and in turn interacts with another solvent water molecule, which lies between the layers, an occupant of both.

Within all the structures, the solvent clusters are either at the end of or beside the Ag···Ag argentophilically bonded units and depending on how the layers stack, each Borromean contains a minimum of a four-anion nitrate channel. As with **1**, the layers in **2** and **3** are stacked to form infinite nitrate channels and the solvent clusters always have an Ag$_2$ unit adjacent to them on both sides, as shown in Figure 2.

For **4** and **5**, the alternate stacking of Ag$_2$ units and solvent clusters is not present and a translation between the layers removes the nitrate channels. In **4** where the solvent cluster comprises five molecules of water and one acetonitrile, the layers are arranged so that the Ag$_2$–solvent clusters are stacked alternately with a discrete four-
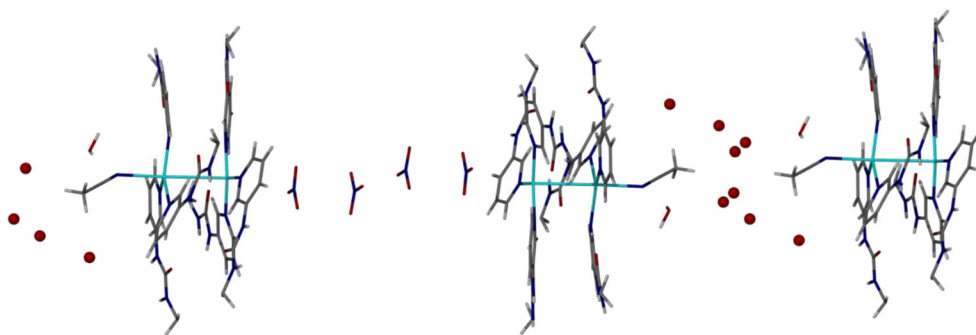
Figure 3. Stacking of the layers in **4**. The Ag$_2$–solvent clusters from one layer are alternately stacked with the four-membered nitrate channel of the adjacent layers.
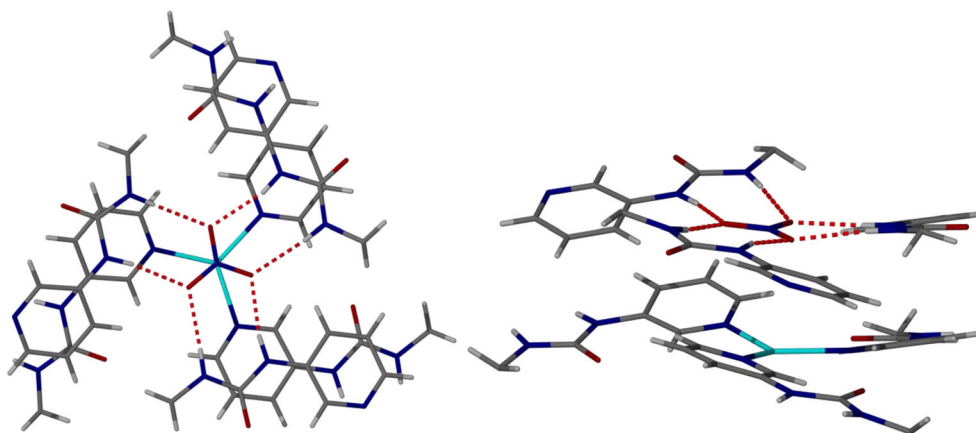

Figure 4. Two views of the π-π stacking interaction between adjacent layers of **4**.

anion nitrate channel, as shown in Figure 3, possibly driven by the lack of a second molecule of acetonitrile to allow adjacent layers to interact. This arrangement, as well as the out-of-plane twisting of the pyridyl groups at the Ag^I centers, allows adjacent layers to stack *via* π-π stacking (Figure 4).

Interestingly, the Borromean topology is not an inevitable outcome of the reaction of AgNO$_3$ with **L**. We have previously reported that the reaction of **L** with AgNO$_3$ gives an apparently kinetic product from a mixture of acetonitrile, chloroform, and methanol (3:3:4) in which crystals form within five minutes of standing [26]. This solvent mixture is essentially the same as that used to form Borromean **3** but using a more concentrated sample. This material is a [2 + 2] metallomacrocycle and seems to be the basis for metallogel formation (Figure 5). Interestingly, crystals of Borromean **1** form upon standing a metallogel sample in aqueous tetrahydrofuran. However, the XRPD pattern of the dried xerogel implies that the gel structure is based on the [2 + 2] metallomacrocycle, not the Borromean motif [26]. Hence, gels based on metallomacrocycles and Borromean weave coordination networks represent alternative outcomes of the reaction with the former likely to be the kinetic product and the latter the thermodynamic sink.
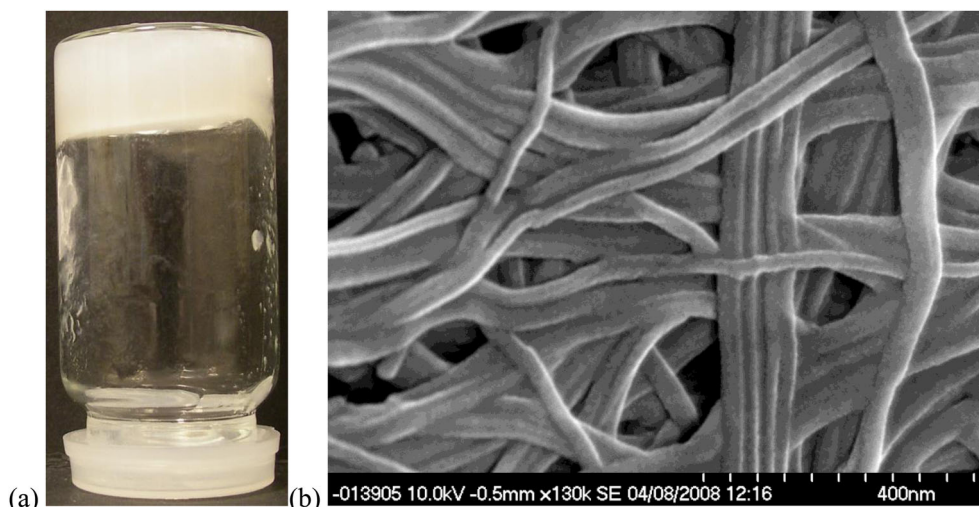
Figure 5. (a) Metallogel formed from a mixture of AgNO₃ and **L** in aqueous tetrahydrofuran from which crystals of Borromean structure **1** form over time. (b) SEM image of the dried gel (xerogel) in (a).

3. Conclusion

The isolation of the same core Borromean structure from five different solvent media under slow crystallization conditions suggests that the Borromean weave is a robust structural motif and represents the thermodynamic product of the AgNO₃/**L** system. Under fast crystallization conditions, previously reported metallomacrocyclic or metallogel products are isolated [26]. The Borromean weave 2-D network is based on pairs of silver(I) ions linked by argentophilic interactions and can adopt three different structure types. The most common involves a solvent pocket in between silver pairs capable of binding ordered clusters of water or mixtures of water and other solvents. An alternative structure allows two such pockets to merge giving a larger pocket capable of accommodating an $(MeCN)_2(H_2O)_8$ cluster. Ordering is driven by weak interactions to the silver(I) ions and hydrogen bonding to the urea carbonyl oxygen atoms. A third structure type involves significantly stronger argentophilic interactions with $d(Ag\cdots Ag) = 3.25\,\text{Å}$ with the exposed face of the silver occupied by a long contact to a urea nitrogen. The hexanuclear ring in **1** is approximately threefold symmetric with Ag⋯Ag separations of 15.75 Å, 16.20 Å, and 16.48 Å and a diameter of *ca.* 31.5 Å. These dimensions vary by up to 0.3 Å across the various solvates as the Borromean weave motif adjusts to the different included solvents and packing arrangement. The reproducibility of this topologically complex network and its tolerance to a variety of guests highlight the complexity of the crystallization process in these labile systems, and the time needed to engender topological complexity may allow alternative, simpler kinetic products including gels based on a metallomacrocyclic motif to form.

4. Experimental

4.1. Preparations

The preparation of ligand **L** and Borromean weave $[Ag_2(\mathbf{L})_3](NO_3)_2\cdot 7H_2O$ (**1**) have been reported previously [21]. Crystallographic data have been deposited

with the Cambridge Structural Database [27], deposition numbers: CCDC 2044637-2044640.

$[Ag_2(L)_3](NO_3)_2 \cdot MeOH \cdot 5H_2O$ (**2**). N,N''-ethylene-1,2-diylbis(N'-pyridin-3-ylurea) (15 mg, 0.05 mmol) was dissolved in a 1:1 mixture of MeOH and water (6 mL), which was then added to silver(I) nitrate (8.5 mg, 0.05 mmol). Upon slow evaporation of the solvent, crystals formed which were analyzed using X-ray crystallography. Yield: 19.7 mg, 0.01 mmol (87%). Anal. Calcd for $C_{43}H_{62}Ag_2N_{20}O_{18}$: C, 37.90; H, 4.59; N, 20.56%. Found: C, 37.31; H, 4.18; N, 20.58%. IR (v/cm^{-1}): 3338 (w, $(N–H)_{str}$), 1686 (strong, sharp, $(C = O)_{str}$), 1547 (strong, sharp, $(N–H)_{def}$). Crystal data $M = 1351.77$ g mol^{-1}, colorless block, $0.2 \times 0.12 \times 0.1$ mm^3, monoclinic, space group $P2_1/c$ (No. 14), $a = 16.0110(3)$, $b = 26.9940(5)$, $c = 12.6123(3)$ Å, $\beta = 91.3200(10)°$, $V = 5449.60(19)$ Å^3, $Z = 4$, $D_c = 1.648$ g cm^{-3}, $F_{000} = 2748$, SMART 6k, MoKα radiation, $\lambda = 0.71073$ Å, $T = 120(2)$ K, $2\theta_{max} = 52.0°$, 37442 reflections collected, 10717 unique ($R_{int} = 0.0907$). Final $GooF = 1.041$, $R1 = 0.0678$, $wR2 = 0.1606$, R indices based on 5853 reflections with $I > 2\sigma(I)$ (refinement on F^2), 744 parameters, 0 restraints. Lp and absorption corrections applied, $\mu = 0.809$ mm^{-1}.

$[Ag_2(L)_3](NO_3)_2 \cdot 2MeCN \cdot xCHCl_3 \cdot yMeOH$ (**3**). N,N''-ethylene-1,2-diylbis(3-pyridin-3-ylurea) (30 mg, 0.10 mmol) was dissolved in a mixture of acetonitrile, chloroform, and methanol (3:3:4, 15 mL) and added to silver(I) nitrate (17 mg, 0.10 mmol). Crystals formed upon slow evaporation of the solvent. Yield: 18.0 mg, 0.01 mmol (40%). Anal. Calcd for $C_{42}H_{48}N_{20}O_{12}Ag_2(MeCN)_2(CHCl_3)_{1.25}(CH_3OH)_{0.675}$: C, 38.54; H, 3.90; N, 20.65%. Found: C, 38.30; H, 3.94; N, 20.56%. The elemental analysis was carried out on a fresh sample, while some desolvation occurred on the crystal studied. Crystal data $M = 661.42$, colorless prism-shaped needles, $0.43 \times 0.24 \times 0.23$ mm^3, monoclinic, space group $C2/c$ (No. 15), $a = 15.727(2)$, $b = 27.878(4)$, $c = 12.9629(18)$ Å, $\beta = 103.922(6)°$, $V = 5516.6(13)$ Å^3, $Z = 8$, $D_c = 1.593$ g cm^{-3}, $F_{000} = 2696$, Smart-6K, MoKα radiation, $\lambda = 0.71073$ Å, $T = 120.15$ K, $2\theta_{max} = 70.1°$, 31608 reflections collected, 11136 unique ($R_{int} = 0.0419$). Final $GooF = 0.995$, $R1 = 0.0452$, $wR2 = 0.1041$, R indices based on 5993 reflections with $I > 2\sigma(I)$ (refinement on F^2), 371 parameters, 0 restraints. Lp and absorption corrections applied, $\mu = 0.791$ mm^{-1}. Disordered chloroform and methanol solvent was treated using the solvent mask procedure in Olex2 [25]. Note this structure determination was previously reported without solvent masking [21]. The present refinement results in significantly improved precision.

$[Ag_2(L)_3](NO_3)_2 \cdot 5H_2O \cdot MeCN$ (**4**). N,N''-ethylene-1,2-diylbis(3-pyridin-3-ylurea) (30 mg, 0.10 mmol) was dissolved in a mixture of MeCN and water (1:1, 6 mL) and added to silver(I) nitrate (17 mg, 0.10 mmol). Crystals formed upon evaporation of the solvent. Crystal data $M = 1371.87$ g mol^{-1}, colorless block, $0.200 \times 0.150 \times 0.100$ mm^3, triclinic, space group $P-1$ (No. 2), $a = 15.4333(12)$, $b = 15.5503(12)$, $c = 15.6241(12)$ Å, $\alpha = 113.907(2)$, $\beta = 118.481(2)$, $\gamma = 95.751(2)°$, $V = 2805.0(4)$ Å^3, $Z = 2$, $D_c = 1.624$ g cm^{-3}, $F_{000} = 1404$, SMART 6k, MoKα radiation, $\lambda = 0.71073$ Å, $T = 120(2)$ K, $2\theta_{max} = 60.0°$, 16342 reflections collected, 16342 unique ($R_{int} = 0.062$). Final $GooF = 0.983$, $R1 = 0.0565$, $wR2 = 0.1272$, R indices based on 8680 reflections with $I > 2\sigma(I)$ (refinement on F^2), 789 parameters, 15 restraints. Lp and absorption corrections applied, $\mu = 0.786$ mm^{-1}.

$[Ag_2(L)_3](NO_3)_2 \cdot xH_2O \cdot yMeCN$ (**5**). N,N''-ethylene-1,2-diylbis(3-pyridin-3-ylurea) (30 mg, 0.10 mmol) was dissolved in a mixture of MeCN and CHCl$_3$ (1:1, 9 mL) and added to a

solution of silver(I) nitrate (17 mg, 0.10 mmol) in MeOH (6 mL). Crystals formed upon evaporation of the solvent. Yield: 7.2 mg, 0.01 mmol (15%). Anal. Calcd for $Ag_2C_{42}H_{48}N_{20}O_{12}(H_2O)_7(CH_3CN)_2$: C, 38.13; H, 4.73; N, 21.27%. Found: C, 38.03; H, 4.20; N, 21.18%. The elemental analysis was carried out on a fresh sample, while some desolvation occurred on the crystal studied. Disordered chloroform and methanol solvent was treated using the solvent mask procedure in Olex2 [25]. Crystal data, $M = 1365.67$ g mol^{-1}, colorless block, $0.3 \times 0.2 \times 0.1$ mm^3, monoclinic, space group $C2/c$ (No. 15), $a = 27.1990(13)$, $b = 16.0780(8)$, $c = 26.2515(13)$ Å, $\beta = 108.261(2)°$, $V = 10901.8(9)$ Å^3, $Z = 8$, $D_c = 1.511$ g cm^{-3}, $F_{000} = 5032$, SMART 6 K, MoKα radiation, $\lambda = 0.71073$ Å, $T = 120$ K, $2\theta_{max} = 65.1°$, 58030 reflections collected, 19634 unique ($R_{int} = 0.0714$). Final $GooF = 0.987$, $R1 = 0.0584$, $wR2 = 0.1289$, R indices based on 10646 reflections with $I > 2\sigma(I)$ (refinement on F^2), 691 parameters, 0 restraints. Lp and absorption corrections applied, $\mu = 0.794$ mm^{-1}.

Disclosure statement

No potential conflict of interest was reported by the authors.

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