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Molecular tectonics: from a binuclear metallamacrocycle to a 1D isostructural coordination networks based on tetracyanomethyl[1.1.1.1]metacyclophane and silver cation

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Depending on the nature of the anion (NO₃ or BF₄) and stoichiometry, combinations, under self-assembly conditions, of the [1.1.1.1]metacyclophane **2 adopting a blocked *1,3*-alternate conformation and bearing four cyanomethylene groups, with silver cations, lead either to the formation of a 1D coordination network or a discrete binuclear metallamacrocycle.**

Coordination Polymers (CPs)^{1,2} or coordination networks³ are extended periodic architectures generated by self-assembly processes. Since the pioneering work of Robson,^{Erreur ! Signet non défini.} this class of crystalline materials has been extensively studied, in particular for their applications.⁴

Following principles established in *molecular tectonics*,^{2,5} coordination networks may be regarded as infinite assemblies displaying translational symmetry. Thus, they might be designed by combining coordinating tectons and metal centres or metal complexes. The reliability of the design of coordination networks depends on the flexibility of the coordination tecton, the coordination requirement of the metal centre and the nature of the anion when neutral coordinating tectons and metal cations are used.⁶

For the design of coordination networks, the macrocyclic [1.1.1.1]metacyclophane backbone is of particular interest. Indeed, this rigid macrocyclic platform, owing to the presence of methyl groups, adopts a stable *1,3*-alternate conformation both in the solid state and in solution in the -60 to 150°C range.⁷ The *1,3*-alternate conformation allows to position in a divergent manner up to four coordinating sites leading thus to a variety of coordinating tectons. Many tetrasubstituted [1.1.1.1]metacyclophane bearing hydroxyl,^{Erreur ! Signet non défini.} cyano,⁸ mercapto,⁹ pyridyl,¹⁰ bipyridyl and quinoleinyl,¹¹ and imidazolyl¹² have been reported. Based on some of the above mentioned tectons, several coordination networks have been obtained upon their combinations with Hg(II) or Ag(I) cations.^{8,12,13}

Here, we report on the synthesis of the tecton **2** (Fig. 1) and the formation of silver coordination networks generated upon its combinations with two different silver salt AgNO₃ and AgBF₄, differing by the nature of their anions.

Figure 1. [1.1.1.1]metacyclophane *1,3*-Alternate conformation **1** and **2**.

Compound **2** is based on the [1.1.1.1]metacyclophane adopting a *1,3*-alternate conformation and bearing four nitrile units. The spacer connecting the coordinating sites with the backbone is a methylene group. Owing to the conformation adopted by **2**, the four nitrile units are located below and above the main plane of the macrocycle in an alternate fashion and thus occupy the apexes of a pseudo-tetrahedron. The analogue of the compound **2**, for which the nitrile units are directly

connected to the backbone, has been already reported.

The synthesis of **2** was achieved in 81% yield upon substitution of all four chlorine atoms in **1**^{7c} by CN groups by refluxing in a DMF/acetone mixture in presence of 10 fold excess of KCN[†].

As connecting metallic node, silver cation was used since on one hand it forms kinetically labile complexes and on the other it requires a rather loose coordination demand both in terms of coordination number, geometry and bond distance.

Under self-assembly conditions, combinations of tecton **2** with two different silver salts (AgNO₃ and AgBF₄) using different metal:tecton stoichiometry, crystalline materials were obtained and studied by X-ray diffraction techniques on single crystals.

Using a 2:1 metal:tecton stoichiometry during de synthesis, combinations of the tecton **2** with AgBF₄ or AgNO₃ lead to the formation of isostructural 1D coordination polymers **2**-AgX (X= NO₃ or BF₄), as shown in Fig. 2a.

Figure 2. Schematic representations of an extended 1D silver (in black) coordination network (a) and a discrete metallamacrocycle (b) formed upon combining the tecton **2** with silver cation.

[†] *Synthesis of 4,11,18,25-tetrakis(chloromethyl)-3,5,7,10,12,14,17,19,21,24,26,28-dodecamethyl[1.1.1]metacyclophane 1* was achieved by modifying the previously reported procedure,¹⁴ using CHCl₃ instead of CS₂.

Synthesis of 4,11,18,25-Tetracyanomethyl-3,5,7,10,12,14,17,19,21,24,26,28-dodecamethyl[1.1.1]metacyclophane 2. 1g (1,38 mmol) of **1** was suspended in a DMF/acetone 3:1 mixture (40 mL). 0.72 g of KCN (720 mg, 11,06 mmol) was added. The reaction mixture was stirred 30 hours at reflux. After cooling to room temperature, the solvent was evaporated under reduced temperature and the residue was treated with 50 ml of CH₂Cl₂. After filtration, the organic solvent was evaporated to dryness. After addition of 50 ml of MeOH to the residue and filtration, the pure compound **2** was obtained as a white powder (0.77g, yield 81%). ¹H-NMR (CDCl₃, 400 MHz, 25 °C): δ(ppm) = 1.07 (s, 12H, *para*-CH₃), 2.43 (s, 24H, *ortho*-CH₃), 3.73 (s, 8H, -CH₂-CN), 4.02 (s, 8H, Ar-CH₂-Ar); ¹³C-NMR (CDCl₃, 125 MHz, 25 °C) δ: 17.8, 18.9, 20.1, 33.3, 118.3, 126.1, 132.4, 135.8, 138.2. IR, ν (C≡N) = 2246 cm⁻¹. Mp: 246 °C, MALDI TOF: m/z = 684.24 [M⁺] (calculated 684.96). Elemental Analysis for C₄₈H₅₂N₄, % found: C, 84.02; H, 7.62; N, 8.15; calc.: C, 84.17; H, 7.65; N, 8.18; %.

Synthesis of [Ag₃(C₄₈H₅₂N₄)₃(BF₄)₃] 2-AgBF₄: In a crystallization tube (diameter 4 mm), a solution of **2** (5 mg, 7.3 × 10⁻³ mmol) in CHCl₃ (1 mL) was layered with a CHCl₃/MeOH (1/1) mixture (0,5 mL). A solution of AgBF₄ (3.39 mg, 14.6 × 10⁻³ mmol) in MeOH (1 mL) was then carefully added. At room temperature, slow diffusion in the dark afforded colourless crystals suitable for X-ray diffraction after several days. Elemental Analysis for C₁₄₄H₁₅₆Ag₃B₃F₁₂N₁₂, % found: C, 65.76 %; H, 5.81 %; N, 6.25 %; Calc.: C, 65.54 %; H, 5.96 %; N, 6.37 %.

Synthesis of [Ag₃(C₄₈H₅₂N₄)₃(NO₃)₃] 2-AgNO₃: In a crystallization tube (diameter 4 mm), a solution of **2** (5 mg, 7.3 × 10⁻³ mmol) in CHCl₃ (1 mL) was layered with a CHCl₃/MeOH (1/1) mixture (0,5 mL). A solution of AgNO₃ (2.48 mg, 14.6 × 10⁻³ mmol) in MeOH (1 mL) was then carefully added. Slow diffusion in the dark and at room temperature afforded colourless crystals suitable for X-ray diffraction after several days. Elemental Analysis for C₁₄₄H₁₅₆Ag₃N₁₅O₉: % found: C, 67.68 %; H, 6.05 %; N, 8.12 %; Calc.: C, 67.44 %; H, 6.13 %; N, 8.19 %.

Synthesis of [Ag₂(C₄₈H₅₂N₄)₂(NO₃)₂·6(CHCl₃)] (2)₂-(AgNO₃)₂: In a crystallization tube (diameter 4 mm), a solution of **2** (5 mg, 7.3 × 10⁻³ mmol) in CHCl₃ (1 mL) was layered with a CHCl₃/MeOH (1/1) mixture (0,5 mL). A solution of AgNO₃ (4,96 mg, 29.2 × 10⁻³ mmol) in MeOH (1 mL) was then carefully added. At room temperature, slow diffusion in the dark produced colourless crystals suitable for X-ray diffraction after several days. Elemental Analysis for C₁₀₂H₁₁₀Ag₂Cl₁₈N₁₀O₆, % found: C, 50.86 %; H, 4.42 %; N, 5.56 %; Calc.: C, 50.50 %; H, 4.57 %; N, 5.77 %.

[‡] *Crystallographic data for 2-AgBF₄ :* 3(C₄₈H₅₂AgN₄) 3(BF₄), *M_r* = 2638.84, cubic, space group *Ia*3*d*, *a* = *b* = *c* = 29.4672(11) Å, α = β = γ = 90°, *Z* = 8, *d*_{calc} = 1.370 Mg/m³, μ = 0.529 mm⁻¹, *F*(000) = 10944, the final *R* = 0.1043, *wR* = 0.3306. 24812 collected reflections and 3125 independent reflections (*R*_{int} = 0.0567) were used in further refinement.

Crystallographic data for 2-(AgNO₃)₂ : 2(C₄₈H₅₂AgN₄)₂(NO₃)₂ 6(CHCl₃), *M_r* = 2564.44, cubic, space group *Ia*3*d*, *a* = *b* = *c* = 29.2699(9) Å, α = β = γ = 90°, *Z* = 8, *d*_{calc} = 1.359 Mg/m³, μ = 0.530 mm⁻¹, *F*(000) = 10704, the final *R* = 0.0885, *wR* = 0.2272. 3064 collected and independent reflections (*R*_{int} = 0.0616) were used in further refinement.

Crystallographic data for (2)₂-(AgNO₃)₂ : 2(C₄₈H₅₂AgN₄)₂(NO₃)₂ 6(CHCl₃), *M_r* = 2425.83, triclinic, space group *P*1, *a* = 12.7017(9) Å, *b* = 14.8810(9) Å and *c* = 15.5594(10) Å, α = 73.798(3)°, β = 69.159(3)°, γ = 86.320(4)°, *Z* = 1, *d*_{calc} = 1.527 Mg/m³, μ = 0.886 mm⁻¹, *F*(000) = 1240, the final *R* = 0.0885, *wR* = 0.2272. 3064 collected and independent reflections (*R*_{int} = 0.0616) were used in further refinement.

All crystallographic data for X-Ray analysis were collected at 173(2) K on a Bruker APEX8 CCD Diffractometer equipped with an Oxford Cryosystem liquid N₂ device, using graphite-monochromated Mo-Kα (λ = 0.71073 Å) radiation. For all structures, diffraction data were corrected for absorption. Structures were solved using SHELXS-97 and refined by full matrix least-squares on *F*² using SHELXL-97.¹⁵ The hydrogen atoms were introduced at calculated positions and not refined (riding model). They can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/datarequest/cif. CCDC: 1504654-1504656.

It is worth noting that the same architecture is obtained independently of the nature of the anion (trigonal NO_3^- or tetrahedral BF_4^-). In both cases, the crystal (**2**-AgNO₃ and **2**-AgBF₄) is composed of the neutral tecton **2**, Ag⁺ cation and anions (BF₄⁻ or NO₃⁻). Within the 1D network, a 1:1 metal:tecton ratio is observed. The crystal (space group *1a-3d*) is composed of 1D cationic silver coordination networks separated by BF₄⁻ or NO₃⁻ anions (Fig. 3a). No specific and strong interactions between the anions and the cationic architectures are spotted. The cationic part of the crystal is generated by mutual interconnection between organic tectons **2** and silver cations through C≡N–Ag⁺ interactions with Ag–N distances of 2.257(5) and 2.265(5) Å for **2**-AgBF₄ and **2**-AgNO₃ respectively (see table 1). The silver cation, in a deformed tetrahedral coordination geometry, is surrounded by four N atoms belonging to two consecutive tectons **2** with the N–Ag–N angle in the 96.9(3)° – 132.7(3)° range.

Figure 3 A portion of isostructural 1D networks formed upon combinations of the tecton **2** with either AgBF₄ or AgNO₃ (a), their packing in a cubic arrangement (c) and schematic representation of the latter packing mode (b). Hydrogen atoms are not presented for clarity. For bonds distances and angles see text and table 1.

The cubic packing of the 1D networks is rather unusual.¹⁶ Three identical 1D networks are aligned along the *a*, *b* and *c* axes (Fig. 3 b and c). The packing is compact preventing thus the presence of solvent molecules.

Table 1 Bond distance and angles for **2**-AgBF₄, **2**-AgNO₃ and [(**2**)₂-(AgNO₃)₂].

	2 -AgBF ₄	2 -AgNO ₃	[(2) ₂ -(AgNO ₃) ₂]
Ag–N Distance (Å)	2.257(5)	2.265(5)	2.238(5) 2.272(5)
Ag–O Distance (Å)	-	-	2.371(4) 2.382(4)
O–Ag–N Angle (°)	-	-	95.80(17) 111.07(16) 123.88(18)
N–Ag–N Angle (°)	96.9(3) 102.9(3) 131.1(3)	98.2(2) 100.3(3) 132.7(3)	87.45(17) 110.24(16) 122.3(2)
d _{eq} (Å)	1.190(8)	1.169(7)	1.134(7)

The formation of 1D coordination networks based on combinations of cyano[1.1.1.1]metacyclophane¹⁶ or cyano appended calixarene derivatives¹⁷ with silver cations, that present the same connectivity, has been previously documented.

Surprisingly, in contrast with the above mentioned case, upon using a 4:1 metal:tecton stoichiometry, the combination of **2** with AgNO₃ afforded a discrete binuclear metallamacrocycle¹⁸ [(**2**)₂-(AgNO₃)₂] (Fig. 4a). As depicted in figure 2b, the

metallamacrocycle is of the [22] type and composed of two ligands **2** and two Ag⁺ cations. The metallamacrocycle crystallises in the *P*-1 space group and the crystal, in addition to Ag⁺ cations and ligands **2**, contains NO₃⁻ anions and chloroform molecules. The compound **2** behaves as a tridentate ligand with only three of the four coordinating C≡N groups participating in the binding of Ag⁺ cation. The two silver cations, equivalent by a centre of symmetry, are pentacoordinated and their coordination sphere is composed of 3 N atoms belonging to three C≡N moieties of two different ligands (Ag-N distances of 2.238(5) and 2.272(5) Å) and 2 O atoms belonging to a chelating NO₃⁻ anion with Ag-O distances of 2.371(4) and 2.382(4) Å (see table 1). NAgN, NAgO and OAgO angles are given in table 1. Within the rectangular shape metallamacrocycle, the Ag-Ag distances is 13.843(19) Å.

In the crystals, the packing of the metallamacrocycles leads to ribbons along the *b* axis and consecutive ribbons are packed in a parallel fashion along the *a* and *c* axes (Fig. 4b). No specific interaction for the CHCl₃ solvent molecules present in the crystal is observed.

Using a variety of other ligands, we have previously reported several metallamacrocycles based on silver and other cations.^{12,18}

Figure 4 Solid state structure of the metallamacrocycle [(**2**)-(AgNO₃)] (a) and the packing of the molecular units in the crystal (b). CHCl₃ solvent molecules and Hydrogen atoms are omitted for clarity. For bond angles and distances and description of packing see text and table 1.

In conclusion, we have investigated the synthesis and the propensity of the neutral [1,1,1,1]metacyclophane **2** adopting a 1,3-alternate conformation and bearing four nitrile units connected to the macrocyclic backbone by methylene units to form coordination networks using silver cation as the metallic connecting node. We have shown that independent of the nature of the anion (trigonal NO₃⁻ or tetrahedral BF₄⁻), combinations of the tetra dentate tecton **2** with Ag⁺ lead to the formation of isostructural 1D silver coordination networks. However, by varying the metal:tecton ratio, we have observed that the above mentioned extended architectures are obtained for 2:1 stoichiometry. In marked contrast, by increasing the Ag:**2** ratio to 4:1 during the synthetic procedure, instead of the extended assembly, a discrete [22] type metallamacrocycle based on two Ag⁺ cations and two ligands **2** is formed. It appears that for the design of coordination networks, the control of the rigidity of the coordinating organic tecton, the nature of the anion when required and the M:tecton ratio play important roles. Thus, even for simple cases, the reliability of the design requires systematic studies. Combinations of the tecton **2** with other metallic nodes are currently under investigation.

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