



# Appealing Copper(I) and Silver(I) Coordination Polymers with an Unprecedented Betainoid Assembling Ligand

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## Abstract

Two coordination polymers of coinage metals with a rare pyridinium-betainoid **L** assembling ligand are reported. These polymers are obtained by self-assembly of the linker **L** and copper(I) or silver(I) ions in acetonitrile. The compounds were characterized by spectroscopic methods and by elemental analysis. The solid-state structures were unambiguously confirmed by single crystal diffraction studies. These assemblies exhibit original helicoidal arrangements. The UV-Vis. absorption and photoluminescence properties are reported as well.

**Keywords:** Ligand, Polymers, Coordination polymer, Crystallization, Spectroscopy

**Abbreviations:** MOFs-Metal-Organic Frameworks; CPs-Coordination Polymers; L-ligand

## Introduction

Metal-Organic Frameworks (MOFs) or Coordination Polymers (CPs) have attracted a tremendous interest in the past decade owing to their potential applications as functional materials in various fields [1-4]. In particular luminescent CPs are attractive candidates for chemical sensing, light-emitting devices or biomedical imaging [5-9]. CPs are also fascinating from a structural point of view since depending on the nature of the ligand and the metallic bricks, a huge number of 1-3D frameworks can be obtained such as helicates [10], zeotype structures [11] for instance. Numerous types of assembling ligands have been designed and reported in the literature, however we are not aware of any example using pyridinium-betaine like assembling ligands. We were therefore intrigued to explore the assembling ability of this family of ligands. It is noteworthy to mention that betaines possess a very rich optical behaviour; indeed, these molecules were used as non-linear optical materials [12-15], as sensors [16-18] and dyes [19-20] for instance. We believe that CPs with betaine based assembling linkers may provide interesting materials with unusual properties.

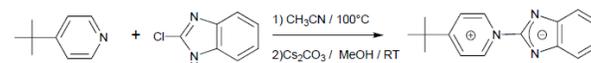
In this work we describe an original approach to the design of CPs using an unprecedented type of assembling ligand noted (**1**) that relies on a betaine scaffold. This ligand consists of a benzimidazolite moiety linked through the C<sub>2</sub> carbon atom to the nitrogen of a 4-tert-butylpyridinium core. It has a zwitterionic electronic structure, a positively charged pyridinium moiety and an anionic benzimidazolite core that we believed to be capable of coordination to transition metals through the benzimidazolite nitrogen atoms. We describe in this work for the first time the preparation and structures of two CPs obtained by self-assembly of copper(I) and silver(I) ions with this unique assembling ligand. The copper(I) polymer can be described as a

helicate type network in which the assembling ligand **L** acts as the bridge between tetrahedrally coordinated Cu<sup>+</sup> cations; while the silver(I) assembly consists of a honey-comb like 2D network with the ligand linking two environmentally different Ag<sup>+</sup> cations; one silver cation is linearly coordinated while the neighboring one has a trigonal environment with three ligands forming a propeller structure around the metal center.

## Results and Discussion

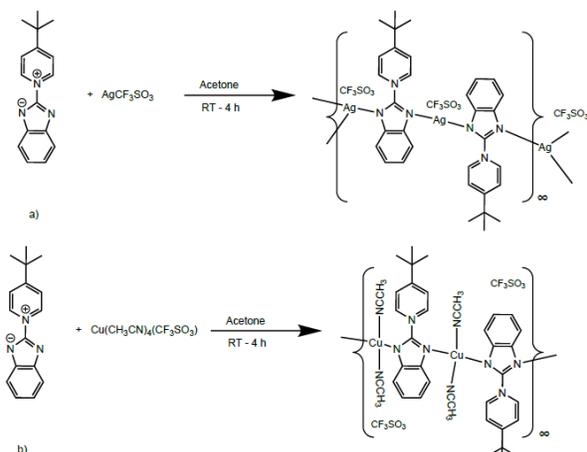
### Synthesis and Characterization

The ligand has been prepared by reaction of 2-chlorobenzimidazole with 4-tert-butylpyridine in refluxing acetonitrile followed by deprotonation of the NH group in methanol with cesium carbonate. Compound **1** was isolated as a yellow solid in good yields and characterized spectroscopically and by elemental analysis. The structure was clearly identified by <sup>1</sup>H-NMR spectroscopy for instance an AA'BB' set of signals attributable to the benzimidazole protons is observable at δ = 6.95 ppm and δ = 7.48 ppm, finally two doublets due to the pyridinium are visible at δ = 8.16 ppm and δ = 9.86 ppm. The composition of the target molecule was determined by mass spectroscopy and by elemental analysis.



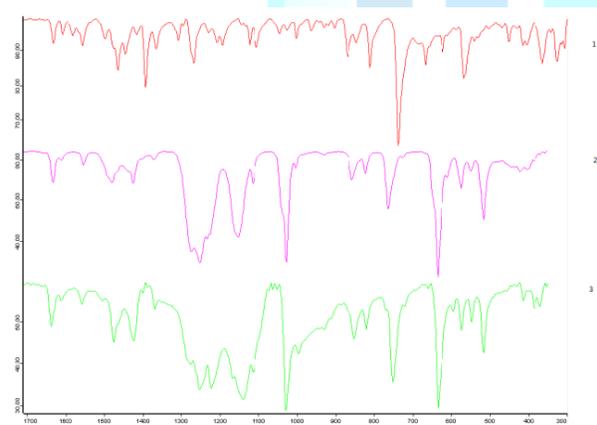
Scheme 1: Preparation of the assembling ligand **L**.

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**Scheme 2:** Self-assembly of the coordination polymers  $[\text{CuL}(\text{CH}_3\text{CN})_2]\text{CF}_3\text{SO}_3$  (**2**) and  $[\text{AgL}]\text{CF}_3\text{SO}_3$  (**3**).

Mixing a yellow acetone solution of the ligand **L** to a slight excess of  $[\text{Cu}(\text{CH}_3\text{CN})_4]\text{CF}_3\text{SO}_3$  in acetone (colourless) at room temperature leads to the immediate formation of a light-yellow precipitate during Four hours. The precipitate was recovered on a sintered glass funnel and washed with small amounts of ice-cold acetone. The infrared spectrum of this solid is clearly different than that of the free ligand; it shows in particular presence of triflate anions. **Figure 1** shows comparison of infrared spectra of compounds **1**, **2** and **3**.

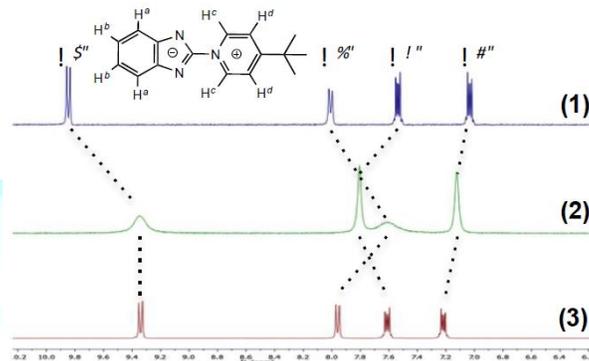


**Figure 1:** Impact of coordination of **L** to copper(I) and silver(I) on its infrared spectrum.

The solid obtained upon reaction with copper(I) was then dissolved in acetonitrile and analysed by  $^1\text{H-NMR}$  and  $^{13}\text{C-NMR}$  spectroscopy. The  $^1\text{H-NMR}$  spectrum displays important changes with regard to the free ligand (**Figure 2**) but the symmetry pattern of the free ligand is maintained. First one can note the important broadening of the aromatic signal due to coordination to copper (I); it is well known that copper(I) causes broadening of  $^1\text{H-NMR}$  signals due to its quadrupolar nucleus [21]. This broadening is certainly also amplified by a dynamic exchange with the coordinating acetonitrile solvent. The chemical shifts of the pyridinium protons are the most affected upon coordination. This observation confirms that coordination of the ligand to copper(I) occurs in solution even in acetonitrile which is a coordinating solvent, however a dynamic equilibrium cannot be

excluded as it will be shown with the absorption and emission data in this solvent.

Solution  $^1\text{H-NMR}$  study of the silver(I) solid shows a similar behaviour. Although without significant broadening of the signals, both the benzimidazole moiety and the pyridinium protons undergo important shifts presumably due to coordination to the cations. Compound  $[\text{AgL}]\text{CF}_3\text{SO}_3$  (**3**) exhibited lower solubility in acetonitrile compared to the copper compound  $[\text{CuL}(\text{CH}_3\text{CN})_2]\text{CF}_3\text{SO}_3$  (**2**).



**Figure 2:** Impact of coordination of **L** to copper(I) and silver(I) ions on the  $^1\text{H-NMR}$  spectrum in  $\text{CD}_3\text{CN}$ .

These solids were then dissolved in acetonitrile and crystallized by slow diffusion of diethyl ether into these solutions. Single-crystals suitable for an X-Ray diffraction structure determination were obtained to unravel the structural features of the novel compounds.

In both cases the analysis shows formation of coordination polymers with the assembling ligand bridging copper(I) or silver(I) ions providing compounds of general formulae  $[\text{CuL}(\text{CH}_3\text{CN})_2]\text{CF}_3\text{SO}_3$  (**2**) and  $[\text{AgL}]\text{CF}_3\text{SO}_3$  (**3**) respectively. The structural features are discussed in the next section.

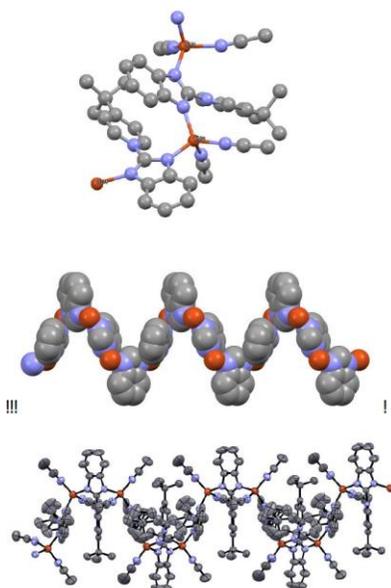
### Structural determination and analysis

The copper(I) compound crystallizes in the orthorhombic space group  $\text{Iba}2$ , with unit-cell dimensions  $a = 26.8106(5)$ ,  $b = 30.8824(6)$ ,  $c = 13.7036(3)$  Å. The structure clearly shows that the assembling ligand is bridging two tetrahedral coordinated copper(I) ions through each nitrogen of the anionic benzimidazolite ring providing a 1D helicoidal structure. Every copper(I) center is therefore linked to two benzimidazolite nitrogen of the benzimidazolite cores with two coordinated acetonitrile molecules completing the coordination sphere. The copper atoms are in a slightly distorted tetrahedral environment. The two aromatic parts of the coordinated ligand are not coplanar but twisted by approximately  $45^\circ$ . Cu-N bond distances lie in the range of reported values for related compounds in the literature. Thus the coordination polymer obtained with copper(I) ions can be formulated as  $[\text{CuL}(\text{CH}_3\text{CN})_2]\text{CF}_3\text{SO}_3$  (**2**). The asymmetric unit consists of subunits containing two copper(I) atoms, two ligands and four coordinated acetonitrile molecules. These views clearly show the helix feature of this structure that is grown along the  $c$  axis of the unit cell. This asymmetric unit is repeated to build the polymeric chains; the helical repeat distance is twice the  $c$  axis translation. This compound is obtained as a racemic mixture no spontaneous resolution was observed during crystallization.

Reaction using silver(I) triflate instead of copper(I) ions provided upon crystallization from acetonitrile suitable single-crystals for an X-Ray diffraction analysis. The obtained silver(I) coordination polymer can be

formulated as  $\{[AgL]CF_3SO_3\}_n$  (3) and crystallizes in the R-3 space group. The structure shows that this silver(I) assembly consists of a honey-comb like 2D network with ligand bridging two environmentally different silver (I) cations; one silver cation is linearly coordinated while the neighbouring one has a trigonal environment with three ligands forming a propeller structure around the metal centre.

These two alternating coordination environments lead to formation of six subunits membered cyclic subunits that are repeated to describe a honeycomb type 2D network. The Tricoordinated silver cations exhibit a chiral P or M propeller structure; the arrangement is homochiral in one plane leading to a racemic crystal i.e. no spontaneous resolution occurs in the crystallization process.

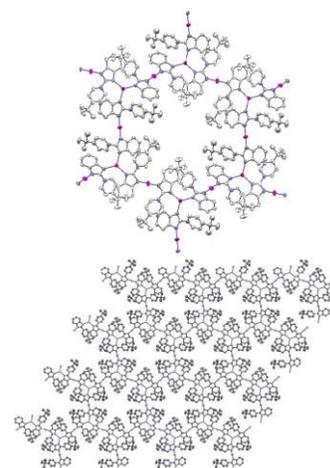


**Figure 3:** Views of the cationic part of the coordination polymer  $\{[CuL(CH_3CN)_2]CF_3SO_3\}_n$  (2). Hydrogen atoms and triflate anions are omitted for clarity.

These results show that the benzimidazolato moiety of the novel ligand is nucleophilic enough to coordinate transition metal cations despite presence of the neighbouring cationic pyridinium ring. Consequently we can reasonably believe to obtain complexes in which this (pro)-ligand could behave as a chelating C<sup>+</sup>N upon C-H bond activation (deprotonation) in position as to the pyridinium nitrogen.

The resulting carbon centre will be a pyridylidene that might impact the electronic properties of the target complexes. In particular, the desired metal complexes could exhibit interesting luminescent properties giving the luminescence ability of ligand. The absorption spectra of the ligand and the metal assemblies were recorded in acetonitrile at  $C = 10^{-5} \text{ m}^{-1}$  and are assembled in **Figure 4**.

The spectrum of the free ligand shows a high-energy absorption band centered at 273 nm with high molar absorptivity ( $> 80000 \text{ m}^{-1} \cdot \text{cm}^{-1}$ ) indicating a  $\pi-\pi^*$  process; the spectrum also exhibits a lower energy transition centered at 395 nm ascribed to an intense charge transfer absorption in line with observed values in related betainoid compounds reported in the literature [22].



**Figure 4:** View of the cationic part of the coordination polymer  $\{[AgL]CF_3SO_3\}_n$  (3). Hydrogen atoms and triflate anions are omitted for clarity.

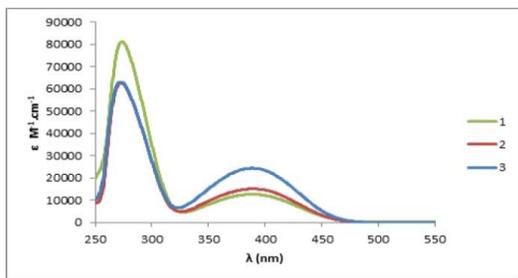
Compound	2	3
<b>Empirical formula</b>	$C_{44}H_{51}Cu_2F_6N_{10}O_{6.5}S_2$	$C_{101}H_{105}Ag_5F_{15}N_{18}O_{17}S_5$
<b>Formula weight</b>	1129.15	2827.79
<b>Temperature/K</b>	200	200
<b>Crystal system</b>	orthorhombic	trigonal
<b>Space group</b>	Iba2	R-3
<b>a/Å</b>	26.8106(5)	20.9167(6)
<b>b/Å</b>	30.8824(6)	20.9167(6)
<b>c/Å</b>	13.7036(3)	23.5245(7)
<b><math>\alpha/^\circ</math></b>	90	90
<b><math>\beta/^\circ</math></b>	90	90
<b><math>\gamma/^\circ</math></b>	90	120
<b>Volume/Å<sup>3</sup></b>	11346.2(4)	8913.3(6)
<b>Z</b>	8	3
<b><math>\rho_{\text{calc}} \text{ g/cm}^3</math></b>	1.322	1.580
<b><math>\mu/\text{mm}^{-1}</math></b>	2.225	0.988
<b>F(000)</b>	4648.0	4269.0
<b>Crystal size/mm<sup>3</sup></b>	$0.21 \times 0.09 \times 0.09$	$0.2 \times 0.18 \times 0.13$
<b>Radiation</b>	$\text{CuK}\alpha$ ( $\lambda = 1.54178$ )	$\text{MoK}\alpha$ ( $\lambda = 0.71073$ )
<b>2<math>\theta</math> range for data collection/<math>^\circ</math></b>	6.594 to 135.564	3.894 to 60.39
<b>Index ranges</b>	$-31 \leq h \leq 31, -36 \leq k \leq 36, -15 \leq l \leq 16$	$-24 \leq h \leq 24, -29 \leq k \leq 29, -33 \leq l \leq 33$
<b>Reflections collected</b>	52164	33853
<b>Independent reflections</b>	9606 [ $R_{\text{int}} = 0.0216$ , $R_{\text{sigma}} = 0.0166$ ]	5882 [ $R_{\text{int}} = 0.0235$ , $R_{\text{sigma}} = 0.0184$ ]
<b>Data/restraints/parameters</b>	9606/10/784	5882/1/259
<b>Goodness-of-fit on <math>F^2</math></b>	1.069	1.039
<b>Final R indexes [<math>I \geq 2\sigma(I)</math>]</b>	$R_1 = 0.0362, wR_2 = 0.1015$	$R_1 = 0.0486, wR_2 = 0.1277$
<b>Final R indexes [all data]</b>	$R_1 = 0.0381, wR_2 = 0.1036$	$R_1 = 0.0680, wR_2 = 0.1483$
<b>Largest diff. peak/hole / <math>e \text{ \AA}^{-3}</math></b>	0.37/-0.21	1.51/-0.90
<b>Flack parameter</b>	0.009(4)	

**Table 1:** Crystallographic parameters for 2 and 3.

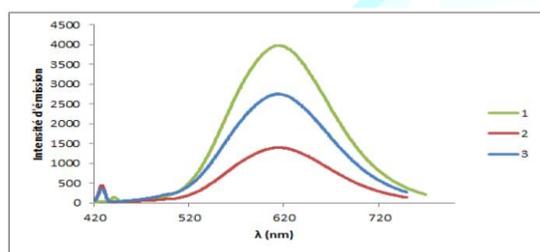


## Absorption and photoluminescence properties

The metal assemblies show spectra with a similar pattern to the free ligand showing weakly metal perturbed transitions. This behavior indicates a fast dynamic equilibrium involving free and coordinated ligand **L** competing with the acetonitrile solvent molecules (Figures 5,6).



**Figure 5:** UV-Vis. Absorption spectra of the free ligand **L** and coordination polymers  $[\text{CuL}(\text{CH}_3\text{CN})_2]\text{CF}_3\text{SO}_3$  (**2**) and  $[\text{AgL}]\text{CF}_3\text{SO}_3$  (**3**).



**Figure 6:** Photoluminescence spectra of the free ligand **L** and coordination polymers  $[\text{CuL}(\text{CH}_3\text{CN})_2]\text{CF}_3\text{SO}_3$  (**2**) and  $[\text{AgL}]\text{CF}_3\text{SO}_3$  (**3**). ( $\lambda_{\text{exc}} = 390 \text{ nm}$ )

Acetonitrile solution ( $10^{-5} \text{ M}$ ) of the free ligand shows a strong emission at  $\lambda_{\text{em}}$  around 620 nm upon excitation in the charge transfer absorption region. This emission is ascribed to a CT process according to studies on related molecules [22]. The metal assemblies both still exhibit this emission almost not affected by the coordination maybe due to fast dynamic equilibrium in the coordinating acetonitrile solvent as suggested by the absorption behavior as well.

These preliminary photoluminescence experiments show that luminescence can be maintained in metal assemblies with this type of ligands, which is promising for the development of luminescent metal complexes with various potential applications.

## Experimental

All experimental manipulations were carried out under an argon atmosphere by using Schlenk tube techniques. Solvents were dried and distilled under argon by standard procedures. All reagents obtained from commercial sources were used as received. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded in  $\text{CD}_3\text{CN}$  using a Bruker Avance 300 NMR spectrometer at 300.13 MHz, and 75.47 MHz respectively. IR spectra were recorded on a Bruker Tensor 27 equipped with an ATR Harricks apparatus. UV-Vis. spectra were recorded on a JASCO V-670 Spectrometer. Photoluminescence spectra were recorded using a JASCO J-815 CD Spectrometer.

A single crystal of each compound was selected, mounted onto a cryoloop, and transferred in a cold nitrogen gas stream. Intensity data were collected with a BRUKER Kappa-APEXII diffractometer with

either graphite-monochromated Mo-K $\alpha$  or copper micro-focused radiation. Data collection was performed with APEX2 suite (BRUKER). Unit-cell parameters refinement, integration and data reduction were carried out with SAINT program (BRUKER). SADABS (BRUKER) was used for scaling and multi-scan absorption corrections. In the Olex2 [23], the structure were solved with SHELXT-14 [24] program and refined by full-matrix least-squares methods using SHELXL-14 [25].

### Compound 1

2-chlorobenzimidazole (540 mg, 3.54 mmol) and anhydrous 4-tert-butylpyridyl (1 ml) were placed in a Schlenk tube and acetonitrile (20 ml) was added. The mixture was refluxed overnight under an argon atmosphere at 100°C. Then cooling the solution resulted in the precipitation of unreacted 2-chlorobenzimidazole as white crystalline needles that can be recovered on a glass frit. The filtrate is then evaporated to dryness and to the oily residue is added anhydrous diethyl ether (50 ml), this precipitates and anhydrous  $\text{Cs}_2\text{CO}_3$  (219 mg) were placed in a Schlenk tube and methanol (20 ml) was added. The mixture was stirred under an atmosphere of argon at room temperature for two hours. The solvent is then evaporated under reduced pressure. The residue is extracted with dichloromethane (50 ml), compound 1 is obtained as a yellow crystalline solid (553 mg, 1.92 mmol). Yield: 54 %.

$^1\text{H}$  NMR (300.13 MHz, DMSO):  $\delta = 9.84$  (d, 2H,  $^2J = 6.9 \text{ Hz}$ ,  $\text{H}_\alpha$  pyridinium), 8.01 (d, 2H,  $^2J = 6.9 \text{ Hz}$ ,  $\text{H}_\beta$  pyridinium), 7.53 (dd, 2H,  $^2J = 9.0 \text{ Hz}$ ,  $^3J_{\text{H-H}} = 2.7 \text{ Hz}$ ,  $\text{H}_\beta$  benzimidazol), 7.03 (dd, 2H,  $^2J = 9.0 \text{ Hz}$ ,  $^3J_{\text{H-H}} = 2.7 \text{ Hz}$ ,  $\text{H}_\alpha$  benzimidazol), 1.46 (s, 9H,  $\text{CH}_3$  pyridinium).  $^{13}\text{C}$  ( $^{-1}\text{H}$ ) NMR (75.47 MHz, DMSO)  $\delta = 29.1, 36.2, 100.3, 119.8, 124.3, 138.3, 146.7, 154.3$ . HRMS  $[\text{L} - \text{K}]^+$  calc. 252.1495; Found. 252.1505

### Compound 2

A colorless solution of  $[\text{Cu}(\text{CH}_3\text{CN})_4]\text{CF}_3\text{SO}_3$  (100 mg, 0.40 mmol) was added to a yellow acetone solution of the ligand (150 mg, 0.39 mmol) at room temperature during four hours. Immediate formation of a light-yellow precipitate was observed. The precipitate was separated on a sintered glass funnel and washed with small amounts of ice-cold acetone. The precipitate was dissolved in the minimum of acetonitrile (ca 4-5ml) and slow diffusion of diethylether into this solution provided light yellow crystals of compound 2, separated by filtration on a sintered glass funnel and dried under vacuum overnight (184 mg, 0.34 mmol). Yield: 86%. anal. Calcd (%) for  $\text{C}_{17}\text{H}_{17}\text{CuF}_3\text{N}_3\text{O}_3\text{S}$  (463, 9  $\text{g}\cdot\text{mol}^{-1}$ ): C 44.01, H 3.69; N 9.06; found: C 43.42, H 3.73; N 8.81. (Precipitate before crystallization in acetonitrile)

$^1\text{H}$  NMR (300.13 MHz,  $\text{CD}_3\text{CN}$ ):  $\delta = 9.35$  (br, 2H,  $\text{H}_\alpha$  pyridinium), 7.81 (m br, 2H,  $\text{H}_\beta$  benzimidazol), 7.61 (m br, 2H,  $\text{H}_\beta$  benzimidazole  $\text{H}_\beta$  pyridinium), 7.12 (m br, 2H,  $\text{H}_\alpha$  benzimidazol), 1.36 (s, 9H,  $\text{CH}_3$  pyridinium).  $^{13}\text{C}$  ( $^{-1}\text{H}$ ) NMR (75.47 MHz,  $\text{CD}_3\text{CN}$ )  $\delta = 29.0, 36.4, 121.1, 122.8, 124.0, 148.4, 171.8, 191.8$ .

### Compound 3

This compound was prepared in a similar way to compound 2 mixing  $\text{AgCF}_3\text{SO}_3$  (145 mg, 0.56 mmol) instead of  $[\text{Cu}(\text{CH}_3\text{CN})_4]\text{CF}_3\text{SO}_3$  with ligand (145 mg, 0.58 mmol). The obtained precipitate was recrystallized from acetonitrile/ diethylether (271 mg, 0.54 mmol). Yield: 95 %.

Anal. calcd. (%) for  $\text{C}_{17}\text{H}_{17}\text{AgF}_3\text{N}_3\text{O}_3\text{S}$  (508,3  $\text{g}\cdot\text{mol}^{-1}$ ): C 40.17, H 3.37; N 8.27; found: C 42.89, H 3.86; N 8.48. (Precipitate before crystallization in acetonitrile).

$^1\text{H}$  NMR (300.13 MHz,  $\text{CD}_3\text{CN}$ ):  $\delta = 9.38$  (d, 2H,  $^2J = 6.9 \text{ Hz}$ ,  $\text{H}_\alpha$  pyridinium), 7.88 (d, 2H,  $^2J = 6.9 \text{ Hz}$ ,  $\text{H}_\beta$  pyridinium), 7.58 (dd, 2H,  $^2J = 9.0 \text{ Hz}$ ,  $^3J_{\text{H-H}} = 2.7 \text{ Hz}$ ,  $\text{H}_\beta$  benzimidazol), 7.16 (dd, 2H,  $^2J = 9.0 \text{ Hz}$ ,

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$^3J_{\text{H-H}} = 2.7$  Hz,  $H_{\alpha}$  benzimidazol), 1.40 (s, 9H,  $\text{CH}_3$  pyridinium).  $^{13}\text{C}\{^1\text{H}\}$  NMR (75.47 MHz,  $\text{CD}_3\text{CN}$ )  $\delta = 29.0, 36.6, 122.0, 124.7, 140.9, 144.03$  (complete data could not be obtained due to precipitation occurring during the spectrum record).

## Conclusion

In summary we have reported two coordination polymers with a rare pyridinium-betaine based assembling ligand and coinage metals. The self-assembly of this rare ligand with copper(I) or silver ions provides appealing coordination networks with helicoidal structures. Our future objectives will focus on the C-H bond activation in the  $\alpha$  position to the pyridinium nitrogen to generate bidentate pyridylidene chelates which is a still a challenging task.

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