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# Two acetylenedicarboxylato-bridged 4-styrylpyridine appended 1D coordination polymers: synthesis, structural characterization and variable temperature magnetism

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**Abstract.** Two hitherto unknown one-dimensional coordination polymers (1D CPs)  $[M(\text{adc})(4\text{-spy})_2(\text{H}_2\text{O})_2]_n$ , ( $M = \text{Mn(II)}$  (**1**) and  $\text{Co(II)}$  (**2**);  $\text{H}_2\text{adc} = \text{acetylenedicarboxylic acid}$  and  $4\text{-spy} = 4\text{-styrylpyridine}$ ) have been characterized by infrared spectra, elemental analysis, thermogravimetric analyses (TGA), powder X-ray diffraction (PXRD) patterns and single-crystal X-ray diffraction techniques. Both these compounds **1** and **2**, are isostructural and construct 3D supramolecular networks by the combination of hydrogen bonding and  $\text{C-H}\cdots\pi$  interactions. Interestingly, these two materials exhibit variable temperature magnetic moment.

**Keywords.** Coordination polymers; crystal structure; H-bonding; transition metals; variable temperature magnetic moment.

## 1. Introduction

The coordination polymer (CP), introduced by J. C. Bailar in 1967, has been immensely popular over the last few decades and is becoming interdisciplinary in nature because of the potential applications in the fields of science and technology.<sup>1–15</sup> The CP is designed by coordination of organic linkers to metal nodes where linkers may bind more than one metal ion to form a polymer. Upon extending the polymerization in one direction 1D polymer is formed; the propagation simultaneously in two directions has generated 2D polymer and spreading in three directions would synthesize 3D polymers.<sup>16–25</sup> In most cases, mixed organic ligand system has been used for the synthesis of structurally diverse CPs. For mixed ligand system, aromatic polycarboxylic acids are generally used

along with N or S donor auxiliary ligands. The aromatic polycarboxylates have been chosen not only due to strong coordination but also their thermal stability and ability to form various interactions to generate higher-dimensional supramolecular architecture. However, fabrication of CPs using aliphatic polycarboxylic acids remains largely unexplored. In this regard, our group has been interested in using aliphatic polycarboxylic acid. Recently, we have reported a series of CPs by using various aliphatic (ene and yne) carboxylates which are proven to be electrically conducting materials.<sup>26–30</sup> These materials have the potential for the fabrication of electronic devices. However, the judicious selection of organic building blocks, as well as metal ions, play significant role for the prospective application in various fields.<sup>31–33</sup> Variable temperature magnetism is also a striking area

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regarding coordination polymer.<sup>34–40</sup> In this work, we have used acetylenedicarboxylic acid ( $H_2adc$ ) as an organic linker to generate coordination polymer and end-capping ligand 4-styrylpyridine (4-spy). Vittal and MacGillivray used 4-spy as a coordinating ligand which undergoes [2+2] cycloaddition reaction upon light irradiation.<sup>41–43</sup> Here, we have directed our attention to synthesize 4-spy based transition metal complexes such as Mn(II) and Co(II) complexes,  $\{[M(adc)(4-spy)_2(H_2O)_2]\}_n$ , (M = Mn(II), **1**; Co(II), **2**) and characterized by single-crystal X-ray diffraction measurement. Although these complexes are photo-inactive but show temperature-dependent magnetic properties.

## 2. Experimental

### 2.1 General

All chemicals purchased were reagent grade and were used without further purification. Elemental analysis (carbon, hydrogen and nitrogen) of the samples were performed in Perkin–Elmer 240C elemental analyzer. Infrared spectrum in KBr ( $4500–500\text{ cm}^{-1}$ ) was recorded from a Perkin–Elmer FT-IR spectrum RX1 spectrometer. Thermogravimetric analyses (TGA) were obtained on a Perkin–Elmer Pyris Diamond TG/DTA in a broad temperature range (between  $30\text{ }^\circ\text{C}$  and  $600\text{ }^\circ\text{C}$ ) under nitrogen atmosphere at a heating rate of  $12\text{ }^\circ\text{C min}^{-1}$ . The PXRD data of the powdered sample was collected on a Bruker D8 Advance X-ray diffractometer using  $\text{CuK}\alpha$  radiation ( $\lambda = 1.548\text{ \AA}$ ) generated at 40 kV and 40 mA in a  $2\theta$  range of  $5–50$ . Magnetic properties were investigated using a Quantum Design MPMS-XL superconducting quantum interference device magnetometer (SQUID) at an applied field 0.5 T in a temperature range  $5–300\text{ K}$ . The diamagnetic correction was carried out by using Pascal constants.

### 2.2 Synthesis of complexes

**2.2a**  $[Mn(adc)(4-spy)_2(H_2O)_2]_n$  (**1**): A 5 mL aqueous solution of  $Mn(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$  (0.049 g, 0.2 mmol) was added dropwise with the 5 mL methanolic solution of 4-spy (0.031 g, 0.2 mmol) and it was stirred for 30 min. After that  $H_2adc$  (0.023 g, 0.2 mmol) neutralized with  $\text{Et}_3\text{N}$  (0.021 g, 0.2 mmol) in 5 mL EtOH was added and it was stirred for another 5 h. The unclear solution was filtered and the filtrate was kept in desk. The yellow coloured prism-shaped crystals of  $[Mn(adc)(4-spy)_2(H_2O)_2]$ , (**1**) were obtained after seven days (0.067 g, Yield 60%). Elemental analysis (%) calcd. for  $\text{C}_{30}\text{H}_{26}\text{MnN}_2\text{O}_6$ : C 63.72, H 4.63, N 4.95; found: C 63.86, H 4.58, N 5.12. IR (KBr pellet,  $\text{cm}^{-1}$ ): 1595  $\nu_{\text{as}}(\text{COO}^-)$ , 1322  $\nu_{\text{sys}}(\text{COO}^-)$ .

**2.2b**  $[Co(adc)(4-spy)_2(H_2O)_2]_n$  (**2**): It was synthesized by a similar procedure adopted for **1** using  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (0.058 g, 0.2 mmol) instead of  $\text{Mn}(\text{CH}_3\text{COO})_2$ . The pink coloured prism-shaped crystals of  $[Co(adc)(4-spy)_2(H_2O)_2]$ , (**2**) were obtained after three days (0.078 g, Yield 70%). Elemental analysis (%) calcd. for  $\text{C}_{30}\text{H}_{22}\text{CoN}_2\text{O}_6$ : C 63.72, H 3.92, N 4.95; found: C 62.98, H 4.21, N 4.86. IR (KBr pellet,  $\text{cm}^{-1}$ ): 1588  $\nu_{\text{as}}(\text{COO}^-)$ , 1309  $\nu_{\text{sys}}(\text{COO}^-)$ .

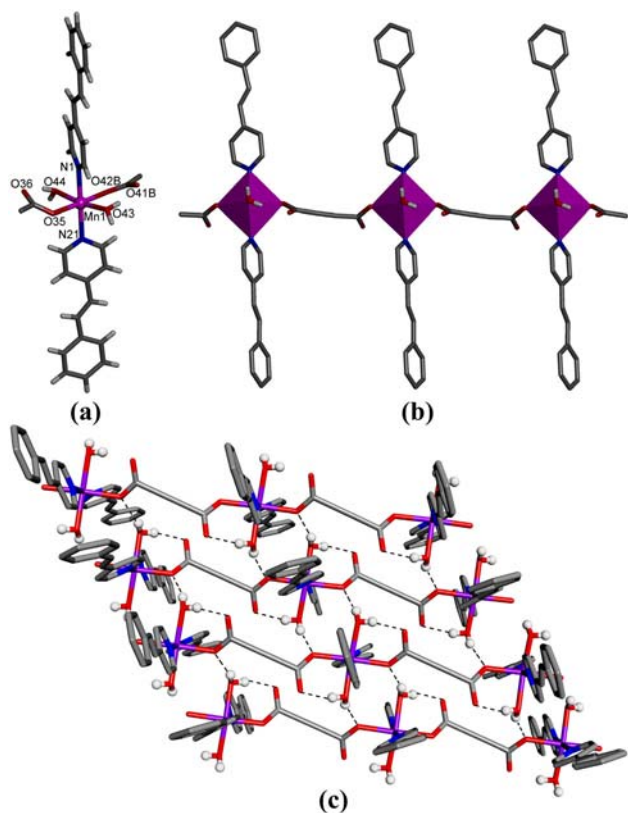
### 2.3 Crystal structure determination

Suitable single crystals of compound **1** and **2** with proper dimensions, were used for data collection using a Bruker SMART APEX II diffractometer furnished with graphite-monochromated  $\text{MoK}\alpha$  radiation ( $\lambda = 0.71073\text{ \AA}$ ). The molecular structure of the single crystal was solved using the SHELX-97 package.<sup>44</sup> Non-hydrogen atoms of the compound were refined with anisotropic thermal parameters. All the Hydrogen atoms were located in their geometrically perfect positions and constrained to ride on their parent atoms. The crystallographic data for compound **1** and **2** are summarized in Table S1 (Supplementary Information) and selected bond lengths and bond angles are also given in Tables S2–S3 (Supplementary Information).

## 3. Results and Discussion

### 3.1 Description of structures

**3.1a**  $[Mn(adc)(4-spy)_2(H_2O)_2]_n$  (**1**): Single crystal X-ray crystallographic structure determination has revealed that the compound **1** crystallizes in the triclinic space group  $P\bar{1}$  with  $Z = 2$ . Each Mn(II) centre in **1** adopts a distorted octahedral geometry and is coordinated by two O atoms from two adc anions in monodentate  $\eta^1$ -fashion (Mn–O, 2.186(5) – 2.194(4)  $\text{\AA}$ ) and O atoms from two aqua ligands (Mn–O, 2.186(5) – 2.187(5)  $\text{\AA}$ ) in the equatorial plane, and by two N atoms from two 4-spy ligands (Mn–N, 2.295(5) – 2.301(5)  $\text{\AA}$ ) at the axial positions (Figure 1a). The connectivity of the adc ligands with Mn(II) centers results in a 1D linear coordination polymeric chain structure (Figure 1b). The aqua ligands undergo intermolecular hydrogen-bonding interactions with coordinated O atom of bridging adc ligands with the O...O separation of 2.695  $\text{\AA}$  leading to the formation of 2D sheet in *ab* plane (Figure 1c). Here, the 1D chains are also self aggregated through weak C–H... $\pi$  interactions along the *ac* plane among the 4-spy ligands with an edge-to-face pattern. These hydrogen bonding and C–H... $\pi$  interactions designed a 3D supramolecular arrangement by stacking the 1D



**Figure 1.** (a) A perspective view of **1** showing coordination environment around Mn(II). (b) A portion of 1D coordination polymeric chain of **1**. (c) Hydrogen bonding in 1D chains forming 2D sheet structure.

chains together (Figure S1, Supplementary Information).

**3.1b**  $[Co(adc)(4-spy)_2(H_2O)_2]_n$  (**2**): Compound **2** is isostructural with **1** and crystallizes also in triclinic space group  $P\bar{1}$  with  $Z = 2$ . Each Co(II) center in **2** is in distorted octahedral geometry and ligated to two O atoms from two *adc* anions in monodentate fashion (Co–O, 2.114(4) Å) and O atoms from two aqua ligands (Co–O, 2.093(3) – 2.101(3) Å) in the equatorial plane, and by two N atoms from two 4-*spy* ligands (Co–N, 2.149(4) Å) in axial positions. Compound **2** also fabricates 3D supramolecular assembly by the combination of hydrogen bonding and edge-to-face C–H $\cdots$  $\pi$  interactions. However, intermolecular H-bonded O $\cdots$ O separation is 2.702 Å, which is a little higher than that of **1**.

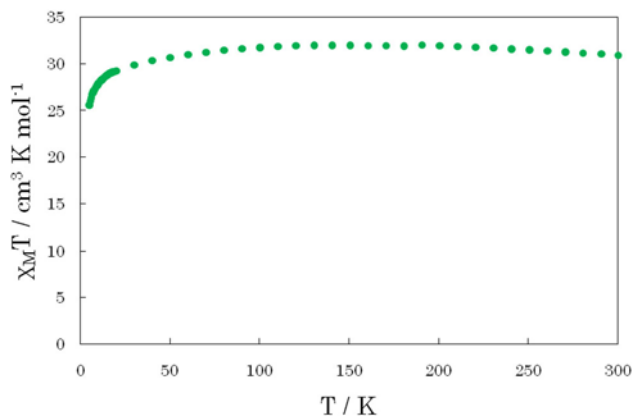
### 3.2 TGA and PXRD Analyses

Thermogravimetric analyses (TGA) of both the compounds were performed within the temperature range 30 °C to 600 °C under  $N_2$  atmosphere. The analyses

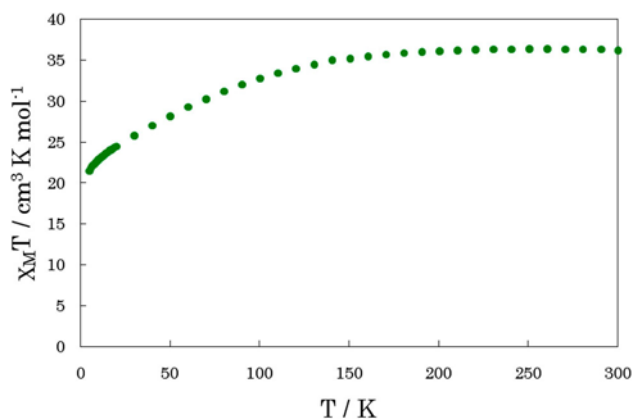
indicate that both the compounds are thermally stable and start decomposing  $\sim 110$  °C (Figures S2 and S3, Supplementary Information). Bulk samples of as-synthesized **1** and **2** were measured by powder X-ray diffraction (PXRD) at room temperature which matches well with those simulated from single-crystal data (Figures S4 and S5, Supplementary Information).

### 3.3 Magnetic properties

These two compounds have paramagnetic central metal ions, Mn(II) ( $d^5$ ) and Co(II) ( $d^7$ ), and may expect, from the crystal structures, the antiferromagnetic coupling between metal centres due to the bridging delocalized linear dicarboxylato bridging group, *adc*. To evaluate the nature of the magnetic coupling among the metal centres in these complexes, the magnetic susceptibility measurements as a function of the temperature (2–300 K) were carried out. The  $\chi_M T$  vs  $T$  curves for **1** and **2** are shown in Figures 2 and 3 respectively. The molar susceptibilities



**Figure 2.** The  $\chi_M$  vs  $T$  plot of compound **1** under 0.5 T.



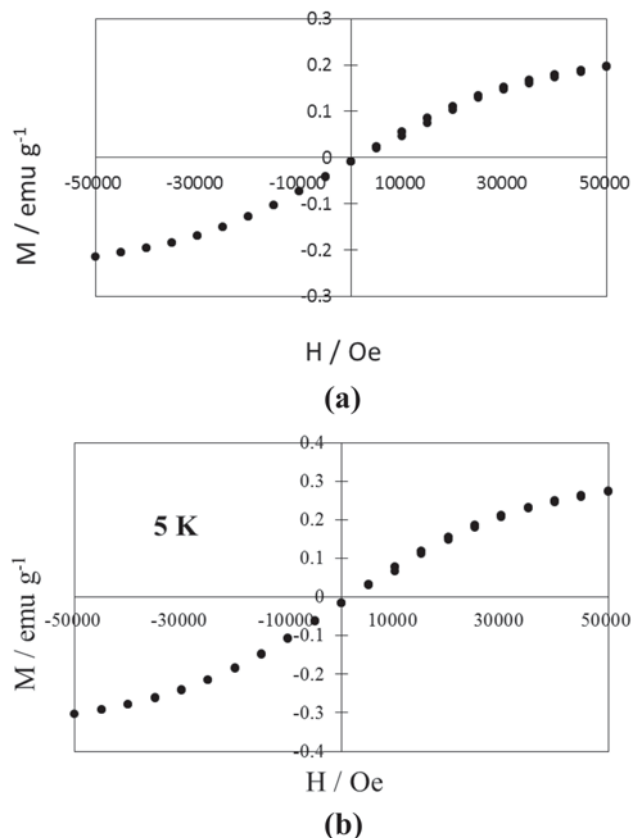
**Figure 3.** The  $\chi_M T$  vs  $T$  plot of compound **2** under 0.5 T.

are calculated as per Mn or Co atoms. At 300 K, the effective magnetic moment for **1** and **2** are 4.44 and 4.80 B.M. respectively, which is less than that of spin only values; for Mn(II),  $d^5$ , theoretical  $\mu_s$  is 5.92 BM and for Co(II),  $d^7$  (high spin), 4.7–5.2 BM. The antiferromagnetic effect is more prominent in Mn(II) than that of Co(II) CP. As shown in Figure 2, compound **1** exhibits  $\chi_M T$  value  $25.6 \text{ cm}^3 \text{ K mol}^{-1}$  at 5 K and it becomes almost constant value until around 30 K. With increasing temperature, the  $\chi_M T$  value reaches to  $30.9 \text{ cm}^3 \text{ K mol}^{-1}$  at 300 K. However, the compound **2** exhibits  $\chi_M T$  value  $21.5 \text{ cm}^3 \text{ K mol}^{-1}$  at 5 K and increases to  $36.2 \text{ cm}^3 \text{ K mol}^{-1}$  at 300 K (Figure 3). Lowering the temperature, there is a rapid fall of  $\chi_M T$  in case of **2** which has started from 150 K, while in case of **1**, optimum  $\chi_M T$  is noted at 110 K and significant decrease is observed below 15 K. This feature indicates the noteworthy stability of magnetic coupling of spins of Mn(II)-based CP than that of Co(II)-based CP. In octahedral symmetry, Co(II) is high-spin  $d^7$  configuration with  $(t_{2g})^5(eg)^2$  of  $S=3/2$  (unpaired electrons are in  $(t_{2g})^1(eg)^2$ ), while Mn(II) is high-spin  $d^5$  configuration with  $(t_{2g})^3(eg)^2$  of  $S=5/2$  (unpaired electrons are in  $(t_{2g})^3(eg)^2$ ). When magnetic orbitals (that contain unpaired electrons) are orthogonal, theoretically ferromagnetic interaction may occur. In this case, the geometrical condition does not allow ferromagnetic interaction, and Mn(II) has many unpaired electrons in  $t_{2g}$  orbitals than Co(II). However, the strength of superexchange interaction depends not only on the number of unpaired electrons but also geometrical conditions as well as supramolecular interactions. Therefore, the experimental results were truly observed.

The antiferromagnetic behavior of the CPs are further proved by the absence of hysteresis widths of ferromagnetism in  $H$  vs  $M$  plots (Figure 4). It seems that spin flop transition has not occurred. However, little magnetic interaction was expected for Mn. Based on the Curie-Weiss equation, Weiss constants of Co(II) and Mn(II)-based CPs are  $-0.00081$  and  $-8.8 \text{ K}$ , respectively (Figures S6–S7, Supplementary Information). As the small negative value indicates strong antiferromagnetic interaction, Co(II)-based CP exhibits stronger magnetic interaction than Mn(II)-based CP.

#### 4. Conclusions

Two linear acetylenedicarboxylato based CPs with 4-styrylpyridine axial ligand have been synthesized using Mn(II) and Co(II) ions and characterized by



**Figure 4.**  $H$  vs  $M$  plots for (a) **1** and (b) **2**.

X-ray crystallography. Both the compounds form 1D polymeric chain structure which generates 3D supramolecular assembly by hydrogen bonding and C–H $\cdots$  $\pi$  interactions. Interestingly, both the compounds exhibit variable temperature magnetic properties. However, compound **1** has higher  $\chi_M T$  value as compared to **2** ( $< 50 \text{ K}$ ). Therefore, compound **1** has better magnetic susceptibility.

#### Supplementary Information (SI)

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos. 1870379 & 1870380. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via [http://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 <http://dx.doi.org/>. Supplementary information for this article is available at [www.ias.ac.in/chemsci](http://www.ias.ac.in/chemsci).

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