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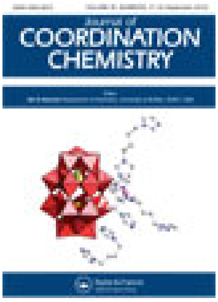

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Syntheses, crystal structures, and photoluminescence of two new coordination polymers derived from dicarboxylate and N-donor ligands

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Syntheses, crystal structures, and photoluminescence of two new coordination polymers derived from dicarboxylate and N-donor ligands

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Two metal-organic coordination polymers, $[Co(tda)(ip)(H_2O)_2]_n$ (**1**) and $[Mn(tda)(ip)(H_2O)]_n$ (**2**) [H_2tda = thiophene-2,5-dicarboxylic acid, ip = 1H-imidazo[4,5-f][1,10]-phenanthroline], have been synthesized and characterized by elemental analyses, IR, PXRD, and X-ray diffraction. Single-crystal X-ray analyses reveal that 2,5-tda is a bridging ligand, exhibiting two coordination modes to link metal ions: μ_1-$\eta^1:\eta^0/\mu_1$-$\eta^1:\eta^0$ and μ_2-$\eta^1:\eta^1/\mu_1$-$\eta^1:\eta^0$. Compound **1** demonstrates a 1-D structure in which Co^{2+} centers are connected *via* tda anions into 1-D chains; the chains are further connected *via* hydrogen-bonding and $\pi\cdots\pi$ interactions. Compound **2** displays a 2-D structure in which tda connects two Mn ions forming a dinuclear molecule. In **2** the 3-D supramolecular structure arrays through hydrogen-bonding and $\pi\cdots\pi$ interactions. In addition, photoluminescence for **1** and **2** is also investigated in the solid state at room temperature.

Keywords: Coordination Polymers; Crystal structures; Photoluminescence

1. Introduction

Design and synthesis of coordination polymers constructed with transition metals and ligands have attracted attention for diverse topologies and potential applications in magnetism, porosity, and luminescence [1–9]. Many metal-organic complexes with good properties were successfully prepared by the selection of carboxylates and N-containing ligands due to their various coordination modes and structural features [10–14]. However, it is still difficult to design and construct coordination polymers from mixed ligands and metal ions from variable composition or structure of the product [15]. Therefore, much work is required for the development of strategies leading to desirable architectures and useful properties.

In this article, thiophene-2,5-dicarboxylic acid (H_2tda) and 1H-imidazo[4,5-f][1,10]-phenanthroline (ip) were selected as ligands based on the following considerations: (a) The ip contains an extended π-system and potential hydrogen bonding groups

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involving heteroaromatic rings; (b) H_2tda is a useful bridging ligand. Herein, we report syntheses, crystal structures, and photoluminescence properties of two new compounds, $[Co(tda)(ip)(H_2O)_2]_n$ (**1**) and $[Mn(tda)(ip)(H_2O)]_n$ (**2**).

2. Experimental

2.1. *General materials and methods*

All reagents and solvents were commercially available and used without purification. Elemental analyses for C, H, and N were performed on an Elementar Vario Micro Cube analyzer. IR spectra were recorded on a Thermo Nicolet IR200 FT-IR spectrometer as KBr pellets ($4000–400\,cm^{-1}$). Powder X-ray diffraction (PXRD) analysis was performed on a MiniFlex2 X-ray Diffractometer with Cu-Kα radiation ($\lambda = 0.1542$ nm) in the 2θ range of 5–50° with a scanning rate of $5°\,min^{-1}$. Fluorescence spectra were obtained on a Hitachi F-4600 spectrofluorometer.

2.2. *Synthesis*

2.2.1. Synthesis of 1. The hydrothermal reaction of a mixture of $CoCl_2 \cdot 6H_2O$ (0.0238 g, 0.1 mmol), H_2tda (0.0172 g, 0.1 mmol), NaOH (0.004 g, 0.1 mmol), ip (0.0220 g, 0.1 mmol), and distilled water (8 mL) was heated to 160°C for 72 h in a 25 mL stainless steel reactor with a Teflon liner, followed by slow cooling to room temperature in 24 h. The resulting reaction products were red block crystals that were washed with distilled water to give pure samples (yield: 52% based on Co). Experimental PXRD data match well with the simulated powder pattern (figure S1). Elemental Anal. Calcd for $C_{19}H_{14}CoN_4O_6S$ (%): C, 47.02; H, 2.91; N, 11.54. Found (%): C, 46.82; H, 2.78; N, 11.08. IR: 3341s, 1576s, 1525s, 1394s, 1336s, 1307s, 1251m, 1078m, 780s, 732s, 638m, 536w.

2.2.2. Synthesis of 2. Compound **2** was synthesized in a similar manner to that described for **1** using $MnCl_2 \cdot 4H_2O$ instead of $CoCl_2 \cdot 6H_2O$. The resulting reaction products were orange block crystals that were washed with distilled water to give pure samples (yield: 62% based on Mn). Experimental PXRD data match well with the simulated powder pattern (figure S2). Elemental Anal. Calcd for $C_{19}H_{12}MnN_4O_5S$ (%): C, 49.25; H, 2.61; N, 12.09. Found (%): C, 48.86; H, 2.49; N, 11.75. IR: 3414s, 1600s, 1576m, 1545m, 1518 w, 1391m, 1343s, 1248 w, 1114 w, 1074m, 779m, 731m, 635w.

2.3. *X-ray crystallography*

Diffraction intensity data of the single crystals of the two compounds were collected on a Bruker Smart Apex II CCD area-detector diffractometer with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å) at 23°C using ω- and φ-scan techniques. The diffraction data were integrated using SAINT [16], which was also used for the intensity corrections for the Lorentz and polarization effects. Semiempirical absorption

Table 1. Summary of crystallographic data for **1** and **2**.

	1	**2**
Empirical formula	$C_{19}H_{14}CoN_4O_6$ S	$C_{19}H_{12}MnN_4O_5$ S
Formula weight	485.33	463.33
Crystal system	Monoclinic	Monoclinic
Space group	*P2(1)/c*	*C2/c*
Unit cell dimensions (Å, °)		
a	8.522(5)	25.531(5)
b	28.661(5)	7.059(5)
c	7.451(6)	19.749(6)
α	90.000	90.000
β	95.875(5)	101.158(5)
γ	90.000	90.000
Volume (Å^3), Z	1810.3(16), 4	3492(3), 8
Temperature (K)	296(2)	296(2)
Calculated density (g cm^{-3})	1.781	1.763
F(000)	988	1880
Reflections collected	11,103	10,335
Independent reflections	4056 [R(int) = 0.0167]	3974 [R(int) = 0.0151]
No. of parameters	320	311
Final R indices [$I > 2\sigma(I)$]^{a,b}	$R_1 = 0.0274$, $wR_2 = 0.0686$	$R_1 = 0.0262$, $wR_2 = 0.0711$
R indices (all data)	$R_1 = 0.0297$, $wR_2 = 0.0699$	$R_1 = 0.0282$, $wR_2 = 0.0728$
Goodness of fit on F^2	1.026	1.028
Largest difference peak and hole (e Å^{-3})	0.355 and −0.284	0.785 and −0.424

$^{a}R = \Sigma(||F_o| - F_c||)/\Sigma|F_o|$. $^{b}wR_2 = [\Sigma w(|F_o| - F_c|^2)^2]/\Sigma w(|F_o|^2)]^{1/2}$.

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

Compound **1**			
Co(1)–O(3)	2.0703(15)	Co(1)–O(2W)	2.1256(13)
Co(1)–N(2)	2.1155(14)	Co(1)–O(1W)	2.1411(17)
Co(1)–N(1)	2.1194(16)	Co(1)–O(1A)$^{\#1}$	2.1694(17)
O(3)–Co(1)–N(2)	91.56(5)	N(1)–Co(1)–O(1W)	89.70(6)
O(3)–Co(1)–N(1)	169.85(5)	O(2W)–Co(1)–O(1W)	88.13(5)
N(2)–Co(1)–N(1)	78.34(6)	O(3)–Co(1)—O(1A)$^{\#1}$	92.33(7)
O(3)–Co(1)–O(2W)	90.26(5)	N(2)–Co(1)–O(1A)$^{\#1}$	91.21(5)
N(2)–Co(1)–O(2W)	176.51(5)	N(1)–Co(1)–O(1A)$^{\#1}$	86.92(7)
N(1)–Co(1)–O(2W)	99.77(6)	O(2W)–Co(1)–O(1A)$^{\#1}$	85.74(5)
O(3)–Co(1)–O(1W)	92.20(7)	O(1W)–Co(1)–O(1A)$^{\#1}$	172.39(5)
N(2)–Co(1)–O(1W)	94.78(5)		
Compound **2**			
Mn(1)–O(3)	2.1189(11)	Mn(1)–O(2B)$^{\#2}$	2.2386(16)
Mn(1)–O(4A)$^{\#1}$	2.1575(11)	Mn(1)–N(2)	2.2798(12)
Mn(1)–O(1W)	2.2166(17)	Mn(1)–N(1)	2.3374(13)
O(3)–Mn(1)–O(4A)$^{\#1}$	117.36(5)	O(1W)–Mn(1)–N(2)	92.29(5)
O(3)–Mn(1)–O(1W)	91.84(5)	O(2B)$^{\#2}$–Mn(1)–N(2)	87.31(5)
O(4A)$^{\#1}$–Mn(1)–O(1W)	86.02(4)	O(3)–Mn(1)–N(1)	83.67(4)
O(3)–Mn(1)–O(2B)$^{\#2}$	94.03(5)	O(4A)$^{\#1}$–Mn(1)–N(1)	156.43(4)
O(4A)$^{\#1}$–Mn(1)–O(2B)$^{\#2}$	81.09(4)	O(1W)–Mn(1)–N(1)	104.86(4)
O(1W)–Mn(1)–O(2B)$^{\#2}$	167.11(4)	O(2B)$^{\#2}$–Mn(1)–N(1)	87.21(4)
O(3)–Mn(1)–N(2)	154.63(4)	N(2)–Mn(1)–N(1)	71.09(4)
O(4A)$^{\#1}$–Mn(1)–N(2)	87.90(4)		

Symmetry codes: (**1**) $^{\#1}$: $x+1$, y, z. (**2**) $^{\#1}$: $-x+1/2$, $-y+1/2$, $-z+2$; $^{\#2}$: x, $-y$, $z+1/2$.

correction was applied using SADABS [17]. The crystal structures of **1** and **2** were solved by direct methods using SIR97 program [18] and refined anisotropically for all non-hydrogen atoms by full-matrix least squares on all F^2 data using SHELXL97 [19]. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were located by

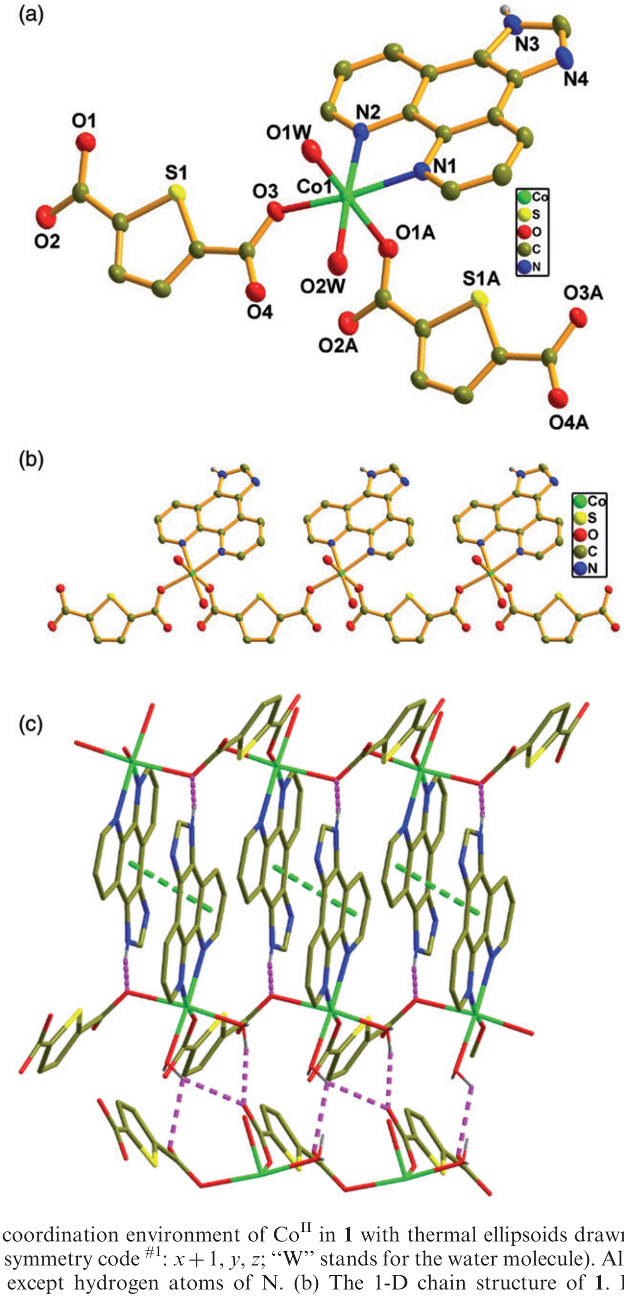


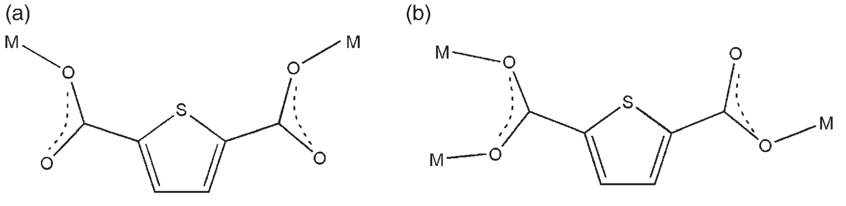
Figure 1. (a) The coordination environment of Co^{II} in **1** with thermal ellipsoids drawn at 50% probability ("A" stands for the symmetry code $^{\#1}$: $x+1$, y, z; "W" stands for the water molecule). All hydrogen atoms are omitted for clarity except hydrogen atoms of N. (b) The 1-D chain structure of **1**. Hydrogen atoms are omitted for clarity except hydrogen atoms of N. (c) The 2-D supramolecular structure of **1** showing hydrogen-bonding interactions (pink dashed line involving tda, ip and water molecules) and $\pi\cdots\pi$ interactions (green dashed line between parallel benzene rings).

geometric calculations, and their positions and thermal parameters were fixed during the structure refinement. The crystallographic data and experimental details of structural analyses are summarized in table 1. Selected bond and angle parameters are listed in table 2.

3. Results and discussion

3.1. *Description of crystal structures*

3.1.1. Compound 1. Single crystal X-ray diffraction reveals that **1** crystallizes in the monoclinic system, with *P2(1)/c* space group. In the crystal structure, there is only one crystallographically unique Co. Each Co(II) is coordinated by two nitrogen atoms from one ip [Co(1)–N(2) = 2.1155(14) Å and Co(1)–N(1) = 2.1194(16) Å] and four oxygen atoms of which two are from two tda anions [Co(1)–O(3) = 2.0703(15) Å and Co(1)–O(1 A) = 2.1694(17) Å] and two from water molecules [Co(1)–O(1W) = 2.1411(17) Å and Co(1)–O(2W) = 2.1256(13) Å] to furnish a distorted octahedral geometry (figure 1a). O1W and O1A are axial and the equatorial plane was occupied by O3, O2W, N1, and N2. The out-of-plane angles in **1** range from 78.34° to 176.51°, which is identical to the ranges of some reported complexes [20]. The tda anions connect Co ions in a μ_1-$\eta^1:\eta^0/\mu_1$-$\eta^1:\eta^0$ coordination mode (scheme 1a) to form a 1-D chain (figure 1b). The ip ligands are attached on the same side of the chain, and the centroid distance of parallel benzene rings of ip between neighboring chains is about 3.704 Å, which means there are weak $\pi\cdots\pi$ interactions between them [21]. As a result, the 2-D



μ_1 - η^1: η^0/ μ_1- η^1: η^0

μ_2 - η^1: η^1/ μ_1 - η^1: η^0

Scheme 1. Coordination modes of tda anion in **1** and **2**.

Table 3. Hydrogen bond lengths (Å) and angles (°) for **1** and **2**.

Compound	D–H···A	*d*(D–H)	*d*(H···A)	*d*(D···A)	∠(DHA)
1	O(2W)–H(14W)···O(4)$^{\#1}$	0.85	2.33	3.047(2)	141.6
	O(2W)–H(14W)···O(2)$^{\#2}$	0.85	2.48	3.016(2)	122.0
	O(1W)–H(13W)···O(4)$^{\#1}$	0.85	1.95	2.7695(19)	162.8
	N(3)–H(4)···O(1)$^{\#3}$	0.89	1.91	2.791(2)	166
2	N(3)–H(11)···O(2)$^{\#1}$	0.87	1.96	2.8189(19)	169

Symmetry codes: (**1**) $^{\#1}$: x, $-y+1/2$, $z-1/2$; $^{\#2}$: $x+1$, $-y+1/2$, $z-1/2$; $^{\#3}$: $-x$, $-y$, $-z+1$. (**2**) $^{\#1}$: $-x$, y, $-z+3/2$.

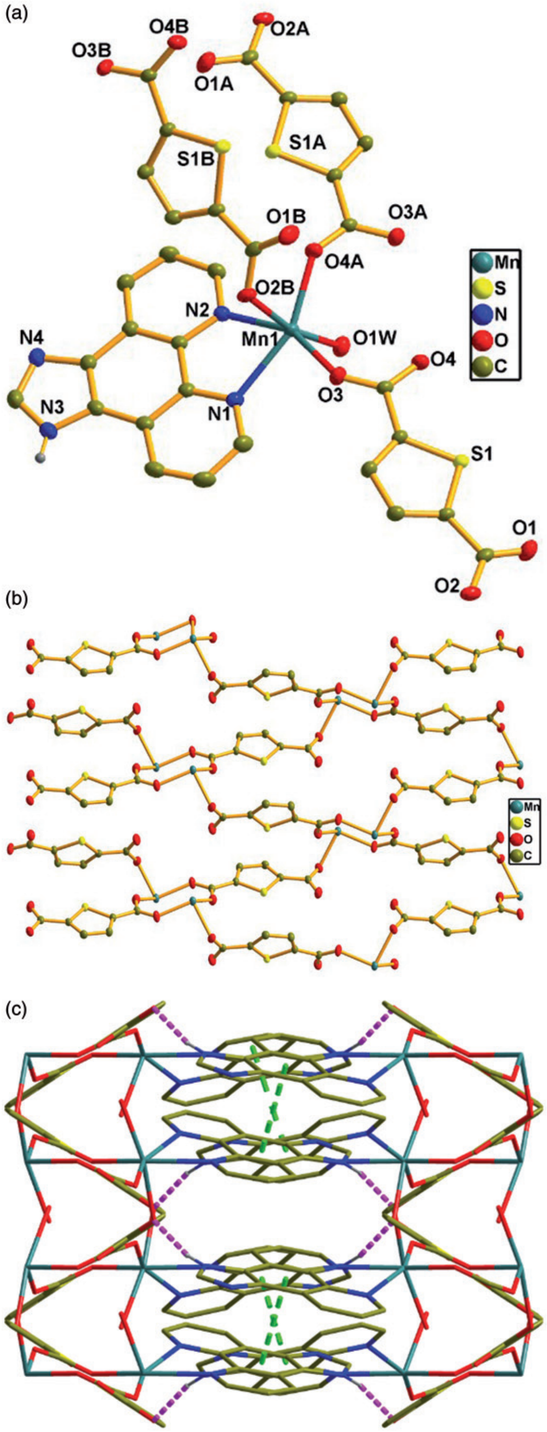


Figure 2. (a) The coordination environment of Mn^{II} in **2** with thermal ellipsoids drawn at 50% probability ("A" stands for the symmetry code $^{\#1}$: $-x+1/2, -y+1/2, -z+2$; "B" stands for the symmetry code $^{\#2}$: $x, -y, z+1/2$; "W" stands for the water molecule). All hydrogen atoms are omitted for clarity except hydrogen atoms of N. (b) The 2-D supramolecular structure of **2** assembled through tda anions. All ip molecules are omitted for clarity. (c) The 3-D supramolecular structure of **2** showing hydrogen bonding interactions (pink dashed line involving ip and tda molecules) and $\pi \cdots \pi$ interactions (green dashed line between parallel benzene rings).

structure was constructed *via* weak $\pi\cdots\pi$ interactions and hydrogen bonds involving tda anions and water and ip molecules (figure 1c, table 3).

3.1.2. Compound 2. Compound **2** crystallizes in the monoclinic system, with space group *C2/c*; the symmetric unit contains one Mn, one tda, one ip, and one water molecule. The Mn(II) is coordinated by two nitrogen atoms from one ip [Mn(1)–N(2) = 2.2798(12) Å and Mn(1)–N(1) = 2.3374(13) Å] and four oxygen atoms of which three are from three tda anions [Mn–O, ranging from 2.1189(11) to 2.2386(16) Å] and one from water molecule [Mn(1)–O(1W) = 2.2166(17) Å] to form a distorted octahedral geometry (figure 2a). O1W and O2B are axial and the equatorial plane is occupied by N1, N2, O3, and O4A. The tda anion is a μ_3-ligand (μ_2-$\eta^1:\eta^1/\mu_1$-$\eta^1:\eta^0$, shown in scheme 1b) with one carboxyl bridging two Mn(II) in a monodentate, leading to a dinuclear molecule, similar to other Mn complexes [22]. The 2-D supramolecular structure was assembled and stabilized *via* the carboxylate of tda anions (figure 2b). The centroid distance of parallel benzene rings between neighboring ip ligands is 3.60 Å. Therefore, the 2-D layer was built into 3-D supramolecular structure *via* weak $\pi\cdots\pi$ interactions and interchain N–H···O hydrogen bonds (figure 2c, table 3).

3.2. *Photoluminescent properties*

Organic–inorganic coordination polymers have been investigated for their fluorescent properties and potential applications as fluorescent-emitting materials [23]. Photoluminescence for **1** and **2** is investigated in the solid state at room temperature. The emission spectra of **1** and **2** are depicted in figure 3. Excited at 367 nm, the peak maxima occur at 433 nm for **1** and 497 nm for **2**. To further understand the origin of these emission bands, the fluorescence spectrum of ip has also been measured, showing an emission band at 473 with excitation at 340 nm, which can be attributed to π^*–π transition of aromatic rings. Fluorescent emission of carboxylates deriving from the π^*–n transition is much weaker than that of the π^*–π transition of aromatic ligands,

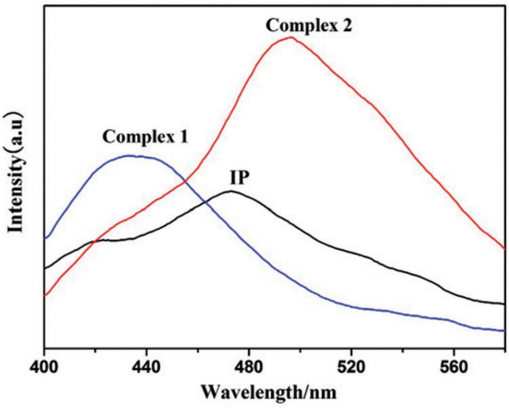


Figure 3. The solid-state photoluminescent spectra of free ip ligand, **1** and **2** at room temperature.

thus the carboxylate ligands have almost no contribution to fluorescent emission of the complexes [23]. Compound **1** shows a 40 nm blue-shift compared to free ip. Taking the crystal structure of **1** into consideration, the blue-shift of 433 nm is probably ascribed to the intraligand charge transfer of the coordinated ip [24]. Compound **2** has a 24 nm red-shift emission peak at 497 nm compared to free ip, which may be assigned to ligand-to-metal charge transfer, whereas the significant red shift of the band in comparison to that of ip would be attributed to metal–ligand coordination and its structure [25]. The differences of the peak positions are a result of the unequal coordination environments of the metal centers with the incorporation of co-ligands, which may also be involved with energy transfer involved in the luminescence. The enhancement of luminescence may result from coordination, heightening the rigidity of the ligands, and therefore reducing the non-radiative decay of the intraligand (π^*–π) excited state [26].

4. Conclusions

We obtained two new metal coordination polymers using H_2tda and ip as ligands. X-ray analyses reveal that **1** shows a 1-D chain with tda anions connecting Co^{2+} centers and **2** shows a 2-D structure propagating *via* the carboxyl groups of tda anions. Preparation of **1** and **2** provides an approach for the construction of other MOFs from dicarboxylate and N-donor ligands. Solid-state luminescence spectrum demonstrates that **1** and **2** are candidates for fluorescent materials.

Supplementary material

Crystallographic information of the two compounds has been deposited with the Cambridge Crystallographic Data Centre as a supplementary material with the publication numbers CCDC $[Co(tda)(ip)(H_2O)_2]_n$ (859968) and CCDC $[Mn(tda)(ip)(H_2O)]_n$ (859971). These data can be obtained free of charge at http://www.ccdc.cam.ac.uk (or Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44-1223-336-033; Email: deposit@ccdc.cam.ac.uk).

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