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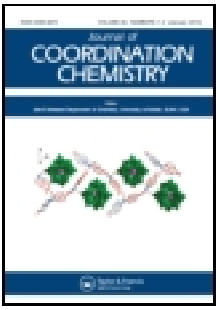

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Synthesis, crystal structures, and fluorescence properties of two 1-D zinc(II) coordination polymers constructed from a bis(benzimidazole) ligand

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Synthesis, crystal structures, and fluorescence properties of two 1-D zinc(II) coordination polymers constructed from a bis(benzimidazole) ligand

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$[Zn(L)(HCOO)Cl]_n$ (**1**) and $\{[Zn_2(L)_2(OH)(HCOO)Cl]\cdot I\}_n$ (**2**) have been obtained by hydrothermal methods of zinc chloride with 1,3-bis(5,6-dimethylbenzimidazole-1-ylmethyl)benzene (L) and potassium bromide (or potassium iodide), and are characterized by elemental analysis, IR spectra, X-ray powder diffraction, and single-crystal X-ray diffraction. The zinc(II) centers display tetrahedral coordination in **1** and **2**. Solid **1** forms a one-dimensional (1-D) helical chain bridged by L, which is further arranged into a two-dimensional supramolecular structure through intermolecular π–π stacking interactions. A 1-D ladder-like chain of **2** connected by hydroxyl and L is presented. Solid-state fluorescent properties of **1** and **2** have been investigated.

Keywords: Bis(benzimidazole); Crystal structure; Fluorescence properties; Zinc(II) coordination polymers

1. Introduction

Metal coordination polymers have attracted interest for a variety of structures, interesting properties, and potential applications in catalysis, luminescence, gas adsorption, and magnetic materials [1–6]. However, control of structures in hydrothermal reactions is still a challenge since the assembly of such polymers can be easily influenced by factors such as selection of metal ions with different coordination geometry, ligands, counteranions with different coordination abilities, solvent, and supramolecular interactions such as hydrogen bonding and π–π stacking [7–12]. A key step for the construction of such complexes is selection of ligands which possess strong coordination ability and can provide hydrogen-bond-acceptors/donors and π-conjugated systems for extending networks. A variety of studies indicate that anions with different sizes, coordination abilities and electronic properties play important roles in assembly of metal–organic coordination polymers, because the anions not only function as counterions and structure-directing agents but also function as ligands and secondary building units [13–18]. Using anions in assembly of new functional materials is a rapidly emerging field [19]. For example, Wang *et al*. reported three coordination polymers based on a flexible bis(benzimidazole) ligand, $[Cd(bbbi)(ita)(H_2O)]\cdot H_2O$, $[Cd(bbbi)(fma)]$, $[Cd(bbbi)(fma)(H_2O)]\cdot 2H_2O$ (bbbi = 1,1-(1,4-butanediyl)bis-1H-benzimidazole, H_2ita = itaconic acid, and H_2fma = fumaric acid); the significant differences of these

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metal-organic frameworks indicate that the carboxylates with different substituents play an important role in formation of the final frameworks and coordination of Cd(II) [20]. Compared with common rigid N-donors [21–28], flexible bis(5,6-dimethylbenzimidazole) derivatives with arylaliphatic core have advantages: (i) the bis(benzimidazole) nitrogens have strong coordination; (ii) benzimidazole ligands contain both the imidazole ring and a larger conjugated π-system, capable of acting as hydrogen bond donors and for π–π stacking interactions; (iii) among the series of benzimidazole derivatives, the most prominent compound is 5,6-dimethylbenzimidazole, which serves as an axial ligand for cobalt in vitamin B_{12}; and (iv) the flexible nature of –(Ph-CH_2)– spacers allows the ligand to bend and rotate upon coordination to metal centers to conform to the coordination geometries of metals. However, coordination polymers based on flexible bis(5,6-dimethylbenzimidazole) ligands with various metal salts have scarcely been reported [29–32]. Zn(II) with d^{10} configuration exhibits a wide variety of coordination geometries and modes [33].

In this work, we selected two inorganic anions in the presence of L to react with Zn(II) chloride and obtained two new Zn(II) coordination polymers, $[Zn(L)(HCOO)Cl]_n$ (**1**) and $\{[Zn_2(L)_2(OH)(HCOO)Cl]\cdot I\}_n$ (**2**). Solid **1** has a one-dimensional (1-D) helical chain while **2** shows a 1-D ladder-like chain bridged by L and oxygens from hydroxyl group.

2. Experimental

2.1. *Materials and physical measurements*

Solvents and materials for synthesis were commercially available and used without purification. L was prepared according to the literature (chart 1) [34]. Elemental analyses (C, H and N) were taken on a Perkin-Elmer 240C analyzer. The IR spectra were measured on a Tensor 27 (Bruker) FT-IR spectrometer with KBr pellets from 4000 to 400 cm^{-1}. X-ray powder diffraction (XRPD) was recorded on a Rigaku D/Max-2500 diffractometer at 40 kV, 100 mA for a Cu-target tube and a graphite monochromator. Thermogravimetric (TG) measurements were carried out on a Netzsch TG 209 thermal analyzer from room temperature to 800 °C under N_2 with a heating rate of 10 °C min^{-1}. Fluorescence spectra were obtained with a Hitachi F-7000 fluorescence spectrophotometer at room temperature.

2.2. *General synthesis procedure for coordination polymers 1 and 2*

2.2.1. $[Zn(L)(HCOO)Cl]_n$ (1). A mixture of $ZnCl_2$ (4.09 mg, 0.03 mmol), L (11.82 mg, 0.03 mmol), and KBr (5.95 mg, 0.05 mmol) in 5 mL H_2O and 2 mL N,N-dimethylformamide (DMF) was sealed in a Teflon-lined stainless vessel, heated to 140 °C, held for 72 h, and then, cooled to room temperature at a descent rate of 10 °C h^{-1}. Colorless block-shaped

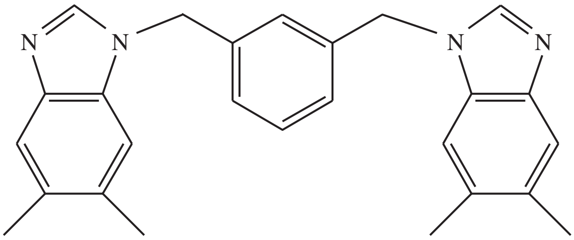

Chart 1. Structure of L.

crystals were obtained by filtration and washed with distilled water and dried in air. Yield: 0.0107 g, 66.4% (based on $ZnCl_2$). Anal. Calc. for $C_{27}H_{27}O_2N_4ClZn$ ($M_r = 540.37$): C, 60.01; H, 5.04; N, 10.37. Found: C, 59.71; H, 4.89; N, 10.24. The FT-IR (KBr pellets, cm^{-1}): 3430s, 3139w, 2929w, 1642m, 1497s, 1360m, 1084m, 850s, 714w, 615w, 540w.

2.2.2. $\{[Zn_2(L)_2(OH)(HCOO)Cl]\cdot I\}_n$ (2).

By employing the above-described procedure with KI (8.3 mg, 0.05 mmol) instead of KBr (5.95 mg, 0.05 mmol), colorless block-shaped crystals were obtained with a yield of 0.0257 g, 75.1% (based on $ZnCl_2$). Anal. Calc. for $C_{53}H_{54}N_8O_3ClIZn_2$ ($M_r = 1144.17$): C, 55.59; H, 4.72; N, 9.79. Found: C, 55.43; H, 4.62; N, 9.82. The FT-IR (KBr, cm^{-1}): 3450s, 3080w, 2999w, 1629s, 1510s, 1250w, 1080m, 860m, 716w, 622m, 549w.

2.3. *Single-crystal X-ray diffraction determination*

Single-crystal X-ray data for **1** and **2** were collected on a Bruker SMART 1000 CCD diffractometer with graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) at 298 K. The structures were solved by direct methods and refined by full-matrix least-squares on F^2 using SHELXL-97 [35]. All nonhydrogen atoms were refined anisotropically. Hydrogens of the ligand were generated theoretically and refined isotropically with fixed thermal factors. Crystallographic data and other pertinent information for the coordination polymers are summarized in table 1. Selected bond lengths and angles are listed in table 2.

3. Results and discussion

3.1. *Structure description of 1 and 2*

3.1.1. $[Zn(L)(HCOO)Cl]_n$ (1).

Single-crystal X-ray diffraction analysis shows that **1** crystallizes in the monoclinic space group $P2_1/c$. The asymmetric unit of **1** contains one

Table 1. Crystallographic data and structure refinement for **1** and **2**.

Coordination polymers	1	2
Formula	$C_{27}H_{27}ClN_4O_2Zn$	$C_{53}H_{54}ClIN_8O_3Zn_2$
Formula weight	540.37	1144.17
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/c$	$P{-}1$
a (Å)	150.0176(10)	12.5742(8)
b (Å)	9.7204(6)	13.5236(8)
c (Å)	21.4398(10)	17.3990(11)
α (°)	90	98.2070(9)
β (°)	124.856(3)	108.9340(8)
γ (°)	90	105.755(1)
V (Å^3)	2568.2(3)	2605.4(3)
Z	4	2
ρ_{calcd} (g cm^{-3})	1.397	1.459
μ (mm^{-1})	1.092	1.614
F(0 0 0)	1120	1164
Goodness-of-fit on F^2	1.152	1.073
R_1, wR_2 ($I > 2\sigma(I)$)	0.0737, 0.2081	0.0575, 0.1552
Largest diff. peak and hole /e Å^{-3}	2.250, −1.269	2.108, −1.450

$^a R_1 = \Sigma||F_o|-|F_c||/\Sigma|F_o|$; $^b R_1 = \{\Sigma[wF_o{}^2 - F_c{}^{2\ 2}]/\Sigma[wF_o{}^{2.2}]\}^{1/2}$.

zinc, one formyl, one chloride, and one L as shown in figure 1(a). Each Zn(II) is four-coordinate by two nitrogens from different L [Zn1–N1 = 2.035(4) Å, Zn1–N4 = 2.015(5) Å], one oxygen from formate (decomposition of DMF) [Zn1–O1 = 1.969(6) Å], and one chloride [Zn1–Cl1 = 2.311(3) Å] to furnish a distorted tetrahedral geometry. In **1**, adjacent Zn (II) ions are connected by L via Zn–N bonds to form a 1-D polymeric chain, which is not linear but wrapped in a helical form with Zn···Zn distance of 12.606(8) Å and Zn–Zn–Zn angle of 165.67° (figure 1(b)). Each L adopts *cis* form with dihedral angle between the planes of the two benzimidazole planes of 105.81(18)°. The crystal structure is further stabilized by strong intermolecular π–π stacking interactions between benzimidazole rings from adjacent chains with a center-to-center distance of 3.519(4) Å, an interplanar angle $\alpha = 1.7(4)°$ and slipping angles β or γ of 15.55° or 15.26°, and an averaged $d_{\pi-\pi}$ distance of 3.395(3) Å, as shown in figure 1(c). Thus, the helical chains are further assembled into a 2-D supramolecular network by intermolecular stacking interactions.

3.1.2. $\{[Zn_2(L)_2(OH)(HCOO)Cl]\cdot I\}_n$ (2). Single-crystal X-ray diffraction analysis indicates that **2** crystallizes in the triclinic $P-1$ space group and displays a 1-D infinite ladder-like structure. All bond lengths around Zn(II) are in the range of reported values [16,36]. The coordination environment of Zn(II) is depicted in figure 2(a). The structure motif is two Zn(II) cations, two L, and four anions including one hydroxide, one chloride, one formate from decomposition of DMF, and one uncoordinated iodide. The coordination environment of two independent Zn(II) cations is different. Zn1 is coordinated by two nitrogens of distinct L [Zn1–N5 = 2.021(4) Å, Zn1–N8A = 2.003(4) Å (symmetry mode: $A = -x, -y+1, -z$)] and one oxygen of formate [Zn1–O2 = 2.082(6) Å], while Zn2 is connected by two nitrogens of other distinct ligands [Zn2–N1 = 2.027(4) Å, Zn2–N3 = 2.019 (4) Å] and one chloride[Zn2–Cl1 = 2.3147(15) Å]. As two Zn(II) are bridged by one oxygen of hydroxide[Zn1–O3 = 1.927(4) Å, Zn2–O3 = 1.915(4) Å], dinuclear units are complete with Zn1···Zn2 distance of 3.5089 Å and Zn2–O3–Zn1 angle of 131.8(3)°. Therefore, both Zn(II) ions are four-coordinate and indicate slightly distorted tetrahedral geometries.

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

Complex **1**			
Zn1–O1	1.969(6)	Zn1–N1	2.035(4)
Zn1–N4	2.015(5)	Zn1–Cl1	2.311(3)
N4–Zn1–N1	105.9(2)	N1–Zn1–Cl1	102.07(16)
N4–Zn1–Cl1	108.00(17)	O1–Zn1–Cl1	112.0(2)
N4–Zn1–O1	113.5(2)	O1–Zn1–N1	114.5(2)
Complex **2**			
Zn1–O3	1.927(4)	Zn2–O3	1.915(4)
Zn1–O2	2.082(6)	Zn2–N3	2.019(4)
Zn1–N8A	2.003(4)	Zn2–N1	2.027(4)
Zn1–N5	2.021(4)	Zn2–Cl1	2.3147(15)
O3–Zn1–O2	104.6(3)	O3–Zn2–N3	109.30(17)
O3–Zn1–N8A	111.83(18)	O3–Zn2–N1	105.14(17)
O2–Zn1–N8A	108.88(17)	N3–Zn2–N1	109.92(17)
O3–Zn1–N5	103.92(16)	O3–Zn2–Cl1	116.60(15)
O(2)–Zn1–N5	113.4(2)	N3–Zn2–Cl1	105.28(12)
N8A–Zn1–N5	113.76(15)	N1–Zn2–Cl1	110.58(12)

Symmetry code: A: $-x$, $-y+1/2$, $-z$.

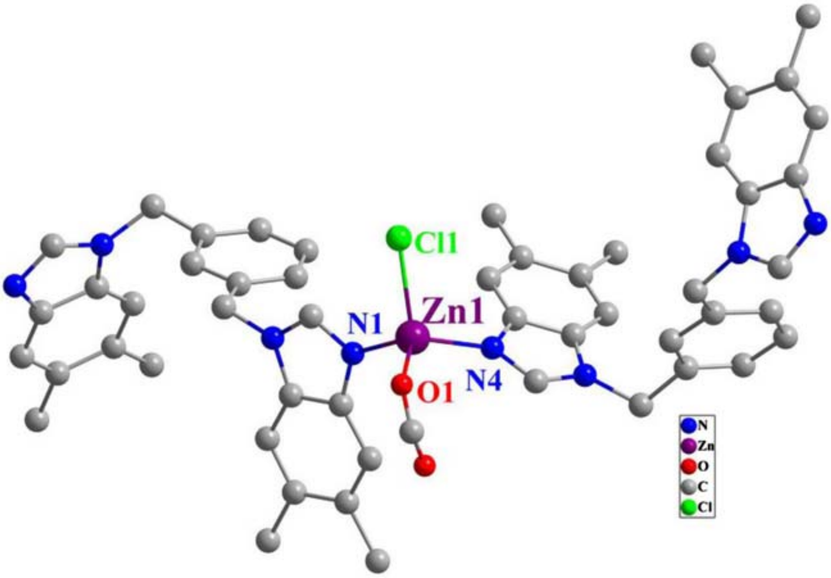

Figure 1(a). Representation of the coordination environment of Zn(II) in **1**; all hydrogens were omitted for clarity.

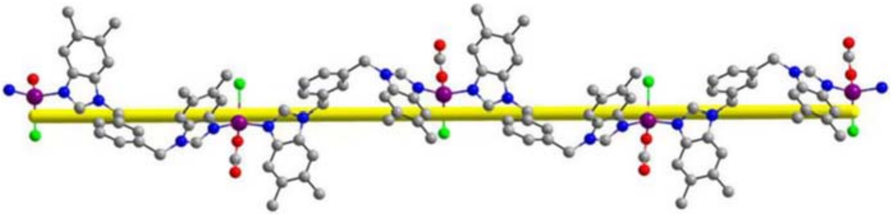

Figure 1(b). The 1-D helical chain of **1**.

Meanwhile, the dinuclear unit is further connected by L via Zn–N bonds and the Zn–O–Zn bridge to form a 1-D infinite ladder-like motif coordination polymer, in which L is a bidentate bridging ligand (two N donors) to bind Zn1 and Zn1A and Zn2 and Zn2B (symmetry mode: $B = x, y+1, z$) with Zn···Zn separation of 9.511 and 13.524 Å, respectively. I^- from KI contributes to form the ladder-like structure and results in no π–π stacking to form higher dimension structure compared with **1**. In one L, dihedral angles between the plane of benzene and benzimidazole rings are 110.6(4)° and 112.4(4)°, respectively (figure 2(b)).

3.2. *IR spectra*

In IR spectrum of **2**, a hydroxyl vibration is visible as a very broad band at 3450 $\mathrm{cm^{-1}}$ [37]. Strong ligand absorptions at 1497 $\mathrm{cm^{-1}}$ for **1** (1510 $\mathrm{cm^{-1}}$ for **2**) can be assigned to $\nu_{C=N}$ of benzimidazole rings. The band at 3139 $\mathrm{cm^{-1}}$ for **1** and 3080 $\mathrm{cm^{-1}}$ for **2** are $\nu_{C\text{–}H}$

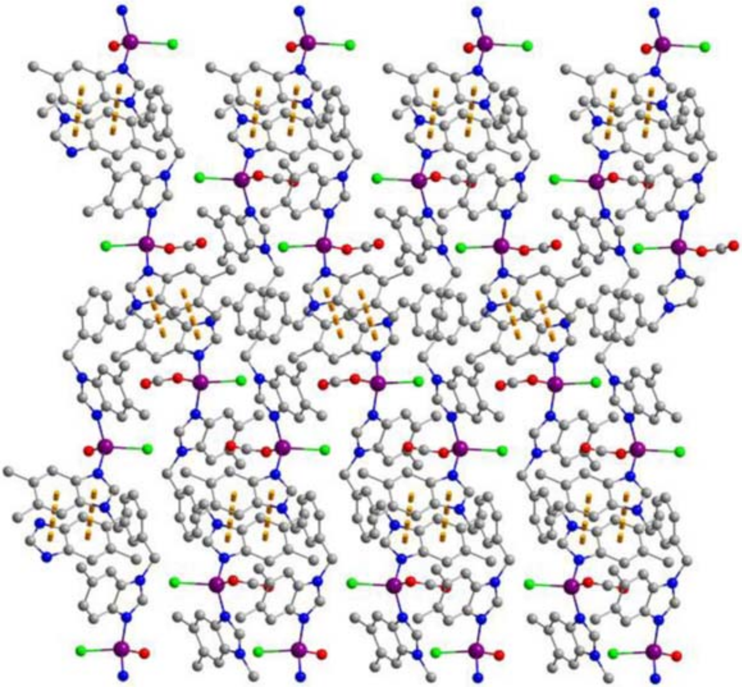

Figure 1(c). The 2-D supramolecular networks formed by intermolecular π–π stacking interactions.

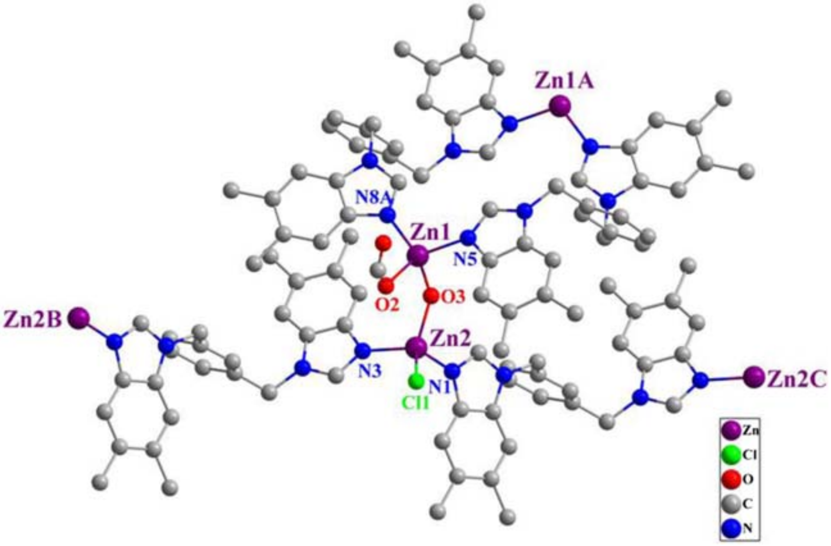

Figure 2(a). View of the coordination environment of Zn(II) in **2**; all hydrogens were omitted for clarity, Symmetry transformations used to generate equivalent atoms: *A* = −*x*, −*y* + 1, −*z*; *B* = *x*, *y* + 1, *z*; *C* = *x*, *y* − 1, *z*.

of aromatic structure. Weak bands below 500 cm^{-1} are due to Zn–O and Zn–N stretches

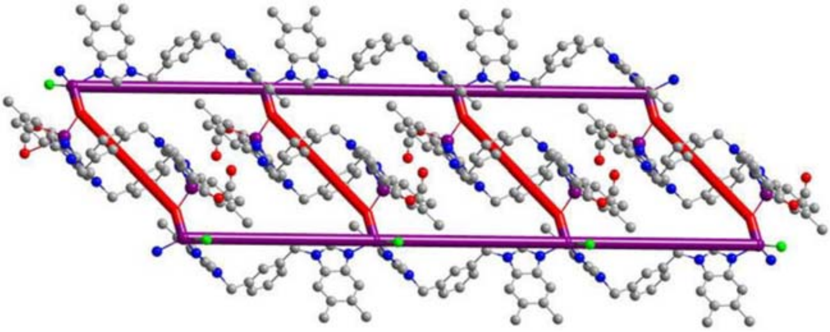

Figure 2(b). The 1-D infinite ladder-like structure of **2**.

[38].

3.3. *XRPD results*

The powder XRD pattern and simulated XRD pattern of **1** and **2** are depicted in Supplementary material. It is obvious that the measured powder XRD pattern is in good agreement with the simulated pattern by X-ray single-crystal data, indicating phase purities of these samples. The difference in reflection intensities between simulated and experimental patterns is due to the different orientation of the crystals in the powder samples.

3.4. *Thermal analysis*

Thermogravimetric analyses were performed to assess the thermal stabilities of **1** and **2**. The TG curves indicate that the two complexes have excellent thermal stability: **1** was stable up to 313 °C and **2** did not exhibit appreciable loss up to 356 °C.

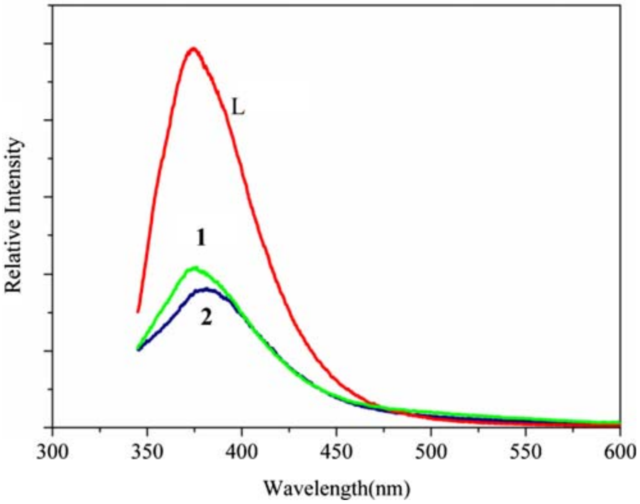

Figure 3. Solid-state photoluminescent spectra of free ligand, **1** and **2**.

3.5. *Luminescent properties*

Photoluminescence of **1** and **2**, together with L, were studied in the solid state at room temperature. Emission spectra of the polymers and L are depicted in figure 3. Excitation of solid samples at 345 nm produces luminescence peaks with a maximum at 376 nm for **1** and 380 nm for **2**, respectively. These emissions are neither metal-to-ligand charge transfer nor ligand-to-metal charge transfer, and can probably be assigned to intra-ligand (π–π^*) emissions since emission maximum is at 374.6 nm for free L when excited at 345 nm. The results are in reasonable agreement with literature examples [39].

4. Conclusion

Two new polymers have been obtained by hydrothermal reactions of zinc chloride, 1,3-bis(5,6-dimethylbenzimidazol-1-ylmethyl)benzene and different anions. Single-crystal structure analysis shows that **1** and **2** have 1-D coordination architectures but **1** is a helical structure, expanded into a 2-D supramolecular architecture through intermolecular π–π stacking interactions, while **2** presents a ladder-like structure with no intermolecular π–π interactions. The different anions and flexibilities of L may exert an important influence on formation of supramolecular architectures.

Supplementary material

X-ray crystallographic files in CIF format for **1** and **2** have been deposited at the Cambridge Crystallographic Data Center with CCDC No: 881023 and 881024. The data can be obtained free of charge via https://www.ccdc.cam.ac.uk/services/structure_deposit/ or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336 033; or E-mail: deposit@ccdc.cam.ac.uk.

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