



Anions behaviors for the dimensionalities of coordination polymers based on poly(imidazole) ligands

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ABSTRACT

To explore the influence for the dimensionalities of coordination polymers, in presence of different anions with different behaviors, three new coordination polymers with different dimensionalities have been prepared by reacting M^{2+} ($M = \text{Zn}$ or Co) with two structurally related rod-like poly(imidazole) ligands, 1,4-bis(benzimidazol-1-yl)-phenyl (L^1) and 1,4-bis(imidazol-1-yl)-phenyl (L^2). Such three new coordination polymers with different architectures, $[\text{Zn}(L^1)(\text{NO}_3)_2]_n$ (**1**), $[\text{Zn}(L^2)_2 \cdot 2\text{BF}_4 \cdot 2\text{HCCl}_3 \cdot 2\text{H}_2\text{O}]_n$ (**2**) and $[\text{Co}(L^2)(\text{SO}_4)]_n$ (**3**), have been determined by single-crystal X-ray diffraction analysis and characterized by elemental analysis, and IR analysis. Complex **1** is alternative linked by octahedral Zn centers and the ligand L^1 to exhibit a one-dimensional (1D) chain structure. However, complexes **2** and **3** both present layered structures contain a similar $-(\text{ligand})-M(\text{metal})-L-M-$ chain motifs that found in complex **1**. In complex **2**, each Zn(II) ion is coordinated by four different L^2 to form a undulated sheet. Complex **3** also present a undulated sheet structure, however, they were connected by the SO_4^{2-} group and the L^2 ligands, different from only of the L^2 ligands in complex **2**. The result indicates that the different anions behaviors could be the great influence on the degree of dimensionality of coordination polymers.

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1. Introduction

In the past decades, coordination polymers rapidly expanded and their structures vary from one-dimensional (1D) to 3D architectures [1–17]. Such research have shown the occurrence of a variety of high dimensional architectures, especially 3D architectures, containing more potential application, such as catalysis, drug delivery, gas sorption and storage, and separation [1–8]. Recently, increasing attention has been paid to investigate the progressive increase in dimensionality of the coordination polymers [18–22], and some factors have been developed. One of remarkable factors is the reaction temperature and time, which demonstrated by Rao et al. that the dimensionality of the structure increases by increasing the reaction temperature and time [18]. In our recent work, we have shown that larger metal center could lead to obtain higher dimensional frameworks [19–21]. However, it is still a great chal-

lenge for the exploration of transformation from low dimensional to high dimensional frameworks.

As one kind of interesting ligands, imidazole ligands could build up to some zeolite-like porous frameworks with divalent and tetrahedral metal ions, which are one contemporary aspect of metal–organic frameworks called zeolite imidazolate frameworks (ZIFs) [23–27]. Then, more attention has been paid to extensively study a large number of poly(imidazole) ligands, and many fascinating coordination polymers have been synthesized [28–36]. In our recent work, we have prepared a series of coordination polymers based on three related rod-like imidazole ligands with different backbones, and those results reveal that ligand backbones have great influence on the topology of products and used to control interpenetrations [28].

Accordingly, to further explore the influence facts of coordination polymers constructions, our research interest beginning to focus on the anions behaviors for the dimensionality of coordination polymers. Herein, we are employing the poly(imidazole) ligands, 1,4-bis(benzimidazol-1-yl)-phenyl (HL^1) and 1,4-bis(imidazol-1-yl)-phenyl (HL^2), as the ligands, three coordination polymers, $[\text{Zn}(L^1)(\text{NO}_3)_2]_n$ (**1**, 1D), $[\text{Zn}(L^2)_2 \cdot 2\text{BF}_4 \cdot 2\text{HCCl}_3 \cdot 2\text{H}_2\text{O}]_n$ (**2**, 2D) and $[\text{Co}(L^2)(\text{SO}_4)]_n$ (**3**, 2D), have been synthesized under solvothermally reactions with different anions. It is worth noting that a similar chain building units have been found in the three compounds, where complex **1** are present 1D chain structure for

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blocked by the coordinated NO_3^- group. However, complexes **2** and **3** are 2D layered structures by linking the chains building units by ligands or SO_4^{2-} . These results reveal that the different anions behaviors have great influences on the degree of dimensionality of coordination architectures.

2. Experimental section

2.1. Materials and general methods

All the solvents and reagents for syntheses were commercially available and used as received. The ligands 4,4'-di(1H-benzo[d]imidazol-1-yl)biphenyl (**L**¹) and 4,4'-di(1H-imidazol-1-yl)biphenyl (**L**²) were synthesized according to the literature procedure [36,37], as shown in Scheme 1. Elemental analyses of C, H and N were performed on a Perkin–Elmer 240C analyzer. IR spectra were measured on a TENSOR 27 (Bruker) FT-IR spectrometer with KBr pellets.

2.2. Synthesis of complexes **1** and **2**

2.2.1. $[\text{Zn}(\text{L}^1)(\text{NO}_3)_2]_n$ (**1**)

A mixture of CH_3OH and CHCl_3 (1:1, 8 mL), as a buffer layer, was carefully layered over a solution of **L**¹ (0.04 mmol, 15.46 mg) in CHCl_3 (6 mL). A solution of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.04 mmol, 11.88 mg) in CH_3OH (6 mL) was then layered over the buffer layer, and the resultant reaction was left to stand at room temperature. After 3 weeks, colorless block single crystals appeared at the boundary. Yield: ~25% (based on **L**¹). Anal. Calcd for $\text{C}_{26}\text{H}_{18}\text{N}_6\text{O}_6\text{Zn}$: C, 54.23; H, 3.15; N, 14.60%. Found: C, 54.86; H, 3.25; N, 14.28%. IR (KBr, cm^{-1}): 3449s, 2360m, 1610m, 1522s, 1314w, 1267w, 1082m, 964s, 821w, 744w, 652s.

2.2.2. $[\text{Zn}(\text{L}^2)_2] \cdot 2\text{BF}_4 \cdot 2\text{HCl}_3 \cdot 2\text{H}_2\text{O}$ (**2**)

Synthesis of **2** was similar to that of **1**. A mixture of CH_3OH and CHCl_3 (1:1, 8 mL), as a buffer layer, was carefully layered over a solution of **L**² (0.06 mmol, 17.16 mg) in CHCl_3 (6 mL). A solution of $\text{Zn}(\text{BF}_4)_2 \cdot 6\text{H}_2\text{O}$ (0.06 mmol, 20.7 mg) in CH_3OH (6 mL) was then layered over the buffer layer, and the resultant reaction was left to stand at room temperature. After 3 weeks, colorless block single crystals appeared at the boundary. The solvent molecules HCl_3 in this compound will lose after exposure in air. Yield: ~30% (based on **L**²). Anal. Calcd. for $\text{C}_{36}\text{H}_{32}\text{B}_2\text{F}_8\text{N}_8\text{O}_2\text{Zn}$ (the solvent molecules HCl_3 lost): C, 51.00; H, 3.81; N, 13.22%. Found: C, 50.58; H, 3.91; N, 13.12%. IR (KBr, cm^{-1}): 3445s, 2360m, 1603w, 1479s, 1287s, 1244m, 1115w, 1007s, 817s, 743s, 580w.

2.2.3. $[\text{Co}(\text{L}^2)(\text{SO}_4)]_n$ (**3**)

Synthesis of **3** was similar to that of **1** and **2**. A mixture of CH_3OH and CHCl_3 (1:1, 8 mL), as a buffer layer, was carefully layered over a solution of **L**² (0.04 mmol, 11.4 mg) in CHCl_3 (5 mL). A solution of $\text{CoSO}_4 \cdot 6\text{H}_2\text{O}$ (0.02 mmol, 5.36 mg) in CH_3OH (5 mL) was then layered over the buffer layer, and the resultant reaction

Table 1

Crystal data and structure refinement parameters for complexes **1–3**.

	1	2	3
Chemical formula	$\text{C}_{26}\text{H}_{18}\text{N}_6\text{O}_6\text{Zn}$	$\text{C}_{38}\text{H}_{34}\text{B}_2\text{Cl}_6\text{F}_8\text{N}_8\text{O}_2\text{Zn}$	$\text{C}_{18}\text{H}_{14}\text{CoN}_4\text{O}_4\text{S}$
Formula weight	575.83	1086.42	441.32
Space group	<i>C2/c</i>	<i>C2/c</i>	<i>Pbca</i>
<i>a</i> (Å)	13.173(3)	17.342(3)	14.440(3)
<i>b</i> (Å)	15.204(3)	19.559(3)	8.9377(18)
<i>c</i> (Å)	11.556(2)	14.258(2)	26.930(5)
β (°)	91.30(3)	98.359(3)	90
<i>V</i> (Å ³)	2313.9(8)	4784.8(13)	3475.7(12)
<i>Z</i>	4	4	8
<i>D</i> (g cm ^{−3})	1.653	1.508	1.687
μ (mm ^{−1})	1.121	0.922	1.143
GOF	1.072	1.059	1.222
<i>R</i> ^a / <i>wR</i> ^b	0.0542/0.1262	0.0940/0.2549	0.0679/0.1231

^a $R = \sum ||F_o| - |F_c|| / \sum |F_o|$.

^b $R_w = [\sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2]^{1/2}$.

Table 2

Selected bond distances (Å) and angles (°) for **1–3**.

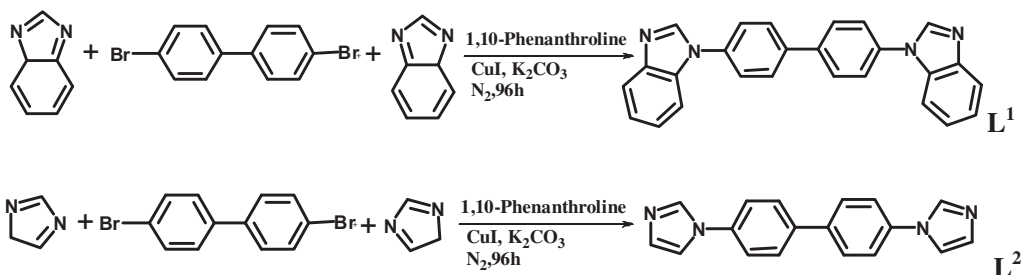
1			
Zn(1)–N(1)	2.009(3)	Zn(1)–O(1)	2.129(7)
Zn(1)–O(2)	2.341(6)		
N(1)#1–Zn(1)–N(1)	115.19(18)	N(1)#1–Zn(1)–O(1)	132.1(2)
N(1)–Zn(1)–O(1)	98.7(2)	N(1)–Zn(1)–O(1)#1	132.1(2)
O(1)–Zn(1)–O(1)#1	80.0(3)	N(1)#1–Zn(1)–O(2)	90.90(17)
N(1)–Zn(1)–O(2)	95.1(2)	O(1)–Zn(1)–O(2)	51.5(2)
O(1)#1–Zn(1)–O(2)	118.2(3)	O(2)–Zn(1)–O(2)#1	168.9(4)
2			
Zn(1)–N(3)	1.992(5)	Zn(1)–N(1)	1.997(6)
N(3)–Zn(1)–N(3)#2	111.2(3)	N(3)#2–Zn(1)–N(1)#2	111.5(2)
N(3)–Zn(1)–N(1)	111.5(2)	N(3)#2–Zn(1)–N(1)	108.2(2)
N(1)#2–Zn(1)–N(1)	106.2(3)		
3			
Co(1)–O(1)	1.960(4)	Co(1)–N(4)#3	1.989(4)
Co(1)–O(4)#4	1.991(3)	Co(1)–N(1)	1.995(4)
O(1)–Co(1)–N(4)#3	113.02(16)	O(1)–Co(1)–O(4)#4	93.48(13)
N(4)#1–Co(1)–O(4)#4	106.98(15)	O(1)–Co(1)–N(1)	107.39(15)
N(4)#1–Co(1)–N(1)	120.73(16)	O(4)#2–Co(1)–N(1)	111.91(14)

Symmetry codes: for **1** #1 $-x+1, y, -z+1/2$; for **2** #2 $-x, y, -z+3/2$; for **3** #3 $x, -y+3/2, z-1/2$; #4 $-x+1/2, y-1/2, z$.

was left to stand at room temperature. After 3 weeks, purple block single crystals appeared at the boundary. Yield: ~35% (based on **L**²). Anal. Calcd. for $\text{C}_{18}\text{H}_{14}\text{CoN}_4\text{O}_4\text{S}$: C, 48.98; H, 3.20; N, 12.70%. Found: C, 48.52; H, 3.50; N, 12.45%. IR (KBr, cm^{-1}): 2801w, 2711w, 2364s, 1579s, 1506s, 1359m, 1303m, 1255w, 1052s, 826s, 648m.

2.3. X-ray single crystal data collection and structure determinations

X-ray single-crystal diffraction data for complexes **1–3** were collected on a Rigaku SCX-mini diffractometer with graphite-monochromated Mo $\text{K}\alpha$ radiation ($\lambda = 0.71073$ Å). The program



Scheme 1.

SAINT [38] was used for integration of the diffraction profiles. All the structures were solved by direct methods using the SHELXS program of the SHELXTL package and refined by full-matrix least-squares methods with SHELXL [39]. The metal atoms (Zn or Co) in each complex were located from the *E*-maps and other non-hydrogen atoms were located in successive difference Fourier maps and refined with anisotropic thermal parameters on *F*². According to the Anal. Calcd. for **1**, the O atoms have been confirmed from the bidentate NO₃[−] groups although they have high *U*(eq) values compared to neighbors. The hydrogen atoms of the ligand were generated by geometrically, and refined in a riding model. Crystallographic data and experimental details for structural analyses are summarized in Table 1. Selected bond lengths and angles are listed in Table 2.

3. Results and discussion

3.1. Descriptions and comments on the crystal structures

3.1.1. [Zn(L¹)(NO₃)₂]_n (**1**)

Single-crystal X-ray diffraction analysis indicates that complex **1** is monoclinic and crystallizes in *C2/c* space group with *a* = 13.173(3) Å, *b* = 15.204(3) Å, *c* = 11.556(2) Å, β = 91.30(3)°, *Z* = 4. The structure of **1** is a 1D chain structure consisting of charge neutrality coordination [Zn(L¹)(NO₃)₂]_n chains involving ZnO₄N₂ octahedra and L¹ units. As shown in Fig. 1, the structure contains one type of Zn atom, which located on a crystallographic 2-fold axis of rotation. Each Zn atom is six coordinated and surrounded by two N atoms of benzoimidazol groups from two different L¹ ligands and four O atoms from two different NO₃[−] groups to adopt a distorted octahedral coordination geometry, with the Zn–N distance of 2.009(3) Å, the Zn–O distances of 2.128(7) and 2.341(6) Å, respectively. Each L¹ unit is two connected and linked two adjacent Zn atoms by two N atoms of the two benzoimidazol groups, as the bridging ligand. By this way, the octahedral Zn centers and L¹ units alternately linked, and each Zn center is further blocked by the coordinated NO₃[−] group give rise to a 1D chain [Zn(L¹)(NO₃)₂]_n, as shown in Fig. 2. Such chains are parallel arranged along (−101) direct.

3.1.2. {[Zn(L²)₂]•2BF₄•2HCCl₃•2H₂O}_n (**2**)

To better understand the effect of the ligand backbones and obtain higher dimensional structures, by the reaction of HL² with

lower terminal steric hindrance and Zn(BF₄)₂, complex **2**, {[Zn(L²)₂]•2BF₄•2HCCl₃•2H₂O}_n, was obtained under similar synthetic conditions as that for complex **1**. The structure of **2** crystallizes in monoclinic, space group *C2/c*, with *a* = 17.342(3) Å, *b* = 19.559(3) Å, *c* = 14.258(2) Å, β = 98.359(3)°. Different of the chain-like structure linked by L¹ with the terminal group of benzoimidazol in complex **1**, the structure of **2** exhibits a 2D layered structure by using L² with lower terminal steric hindrance as ligand, as shown in Fig. 3. It has the macrocationic [Zn(L²)₂]_n²⁺ undulated sheets linked by ZnN₄ tetrahedra and L² units. The counterions of BF₄[−], and the solvent HCCl₃ and free water molecules were located in the interlayer regions to balance the charge and fill the space.

As shown in Fig. 4, the ligand L² in compound **2** also displays 2-connected and links two adjacent Zn atoms by the N atoms of the imidazole groups, similar the linkage of L¹ found in compound **1**, which could be regarded as 2-connected node. However, the geometry of the Zn atom in this compound is tetrahedral, being bound to four N atoms of four imidazole groups from four different L² molecules, which is different of the 6-coordinated Zn atom in compound **1**, can be regarded as a distorted 4-connected node. Such two connected L² units and 4-connected Zn atoms are linked each other to form an undulated sheet (Fig. 3). Interesting, as shown in Fig. 3a, the 4-connected Zn atoms could be linked by L² ligands along *a* axis to form a strictly linear chains-like building unit, which is similar to the chains found in the compound **1**. The Zn atoms also could be linked by L² ligands along *c* axis to build an undulated chains-like building unit (Fig. 3b). In such a way, the 4-connected Zn atoms and the 2-connected L² ligands alternately linked to result in an undulated sheet along *ac* plane, which presents a 4,4-network. Those sheets are stacked along the *b* axis, with the counterions of BF₄[−] and the HCCl₃ and free water molecules residing in the interlayer region.

3.1.3. [Co(L²)(SO₄)]_n (**3**)

To further explore the role of anions during the reaction, a new complex **3**, [Co(L²)(SO₄)]_n, have been prepared by CoSO₄•6H₂O and L² with the similar synthetic conditions of complexes **1** and **2**. Single-crystal X-ray diffraction analysis indicates that **3** crystallizes in orthorhombic, space group *Pbca*, with *a* = 17.342(3) Å, *b* = 19.559(3) Å, *c* = 14.258(2) Å. Interestingly, its structure is charge neutrality, and it also presents 2D layered structure with 4,4-network by tetrahedral Co centers, L² ligands, and SO₄^{2−} groups.

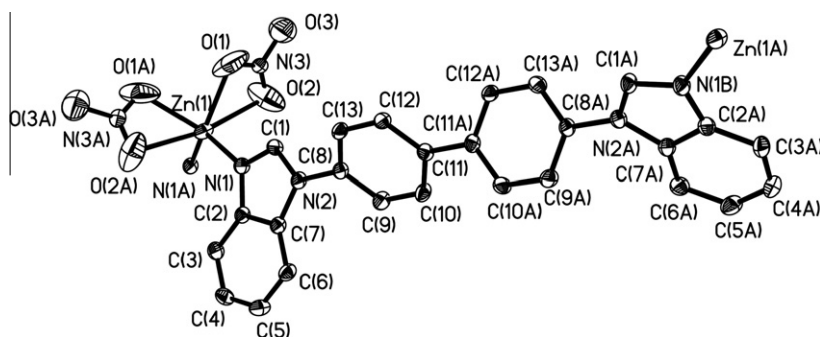


Fig. 1. Thermal ellipsoid plot (30%) showing atom in complex **1**.

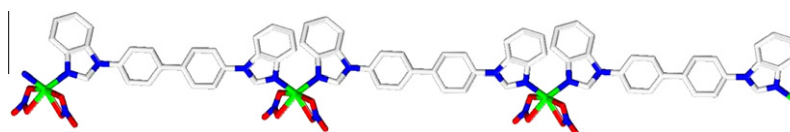


Fig. 2. The chain-like structure of complex **1** (all the H atoms are omitted). Color code: Zn, green; O, red; N, blue; C, light gray. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

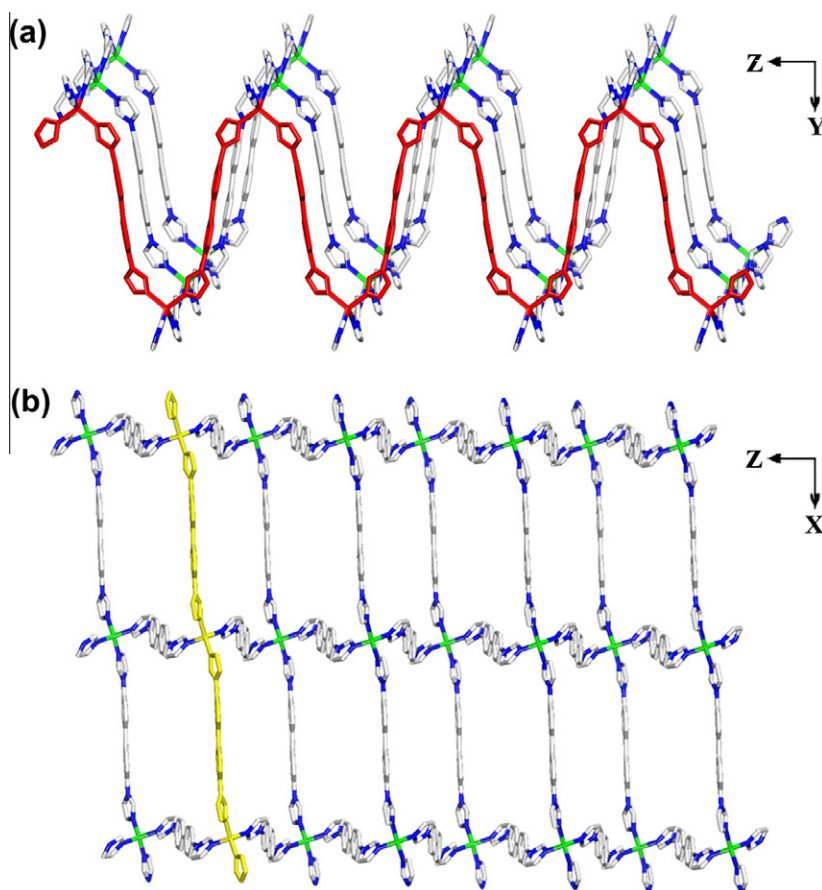


Fig. 3. One of the undulated sheet of complex **2** (all the H atoms are omitted) (a) contains a strictly linear chains-like building unit (red) along *a* axis, (b) contains an undulated chains-like building unit (yellow) along *b* axis. Color code: Zn, green; N, blue; C, light gray. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

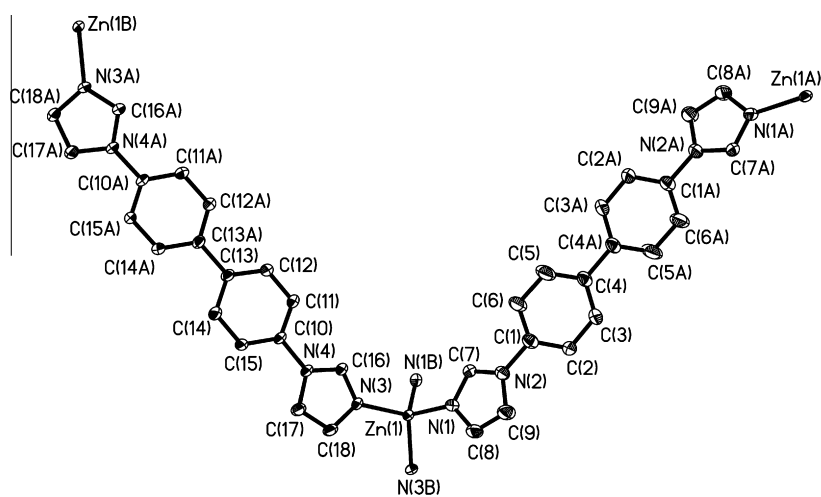


Fig. 4. Thermal ellipsoid plot (30%) showing atom in complex **2** (the solvent molecules and BF_4^- ions are ignored).

As shown in Fig. 5, each asymmetric unit contains one crystallographically independent Co center. Which connected with two N atoms from two different L^2 ligands and two O atoms from two different SO_4^{2-} groups to adopt a distorted tetrahedral coordination geometry, with the Co–O distances of 1.960(4) Å and 1.991(3) Å, and the Co–N distances of 1.989(4) Å and 1.995(4) Å. The structure contains one type L^2 unit and one type SO_4^{2-} group, the L^2 units display 2-connected and bind to two Co atoms as the

bridging ligands with the two N atoms of the imidazole groups, just like found in **2**. The SO_4^{2-} groups link two adjacent Co atoms by two O atoms, as a 2-connected bridging ligand. Interestingly, the Co atoms firstly alternately linked by the 2-connected L^2 units to form an undulated chain CoL^2 along *c* axis (see Fig. 6), which are similar to that found in **2**. Then, such chains are further linked by O1 and O4 atoms of the 2-connected SO_4^{2-} groups to result a undulated sheet along *bc* plane. Notable, which also presents a 4,4-network

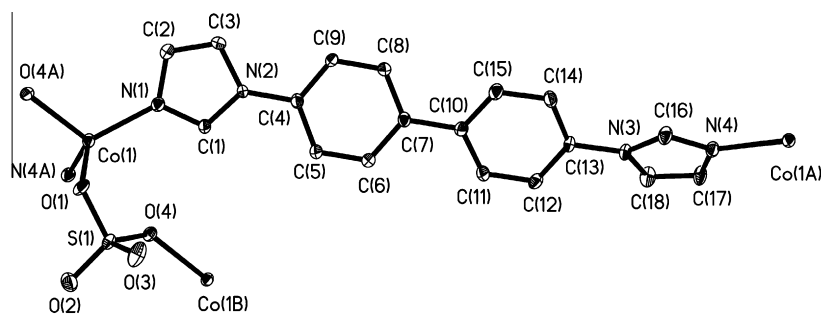


Fig. 5. Thermal ellipsoid plot (30%) showing atom in complex **3**.

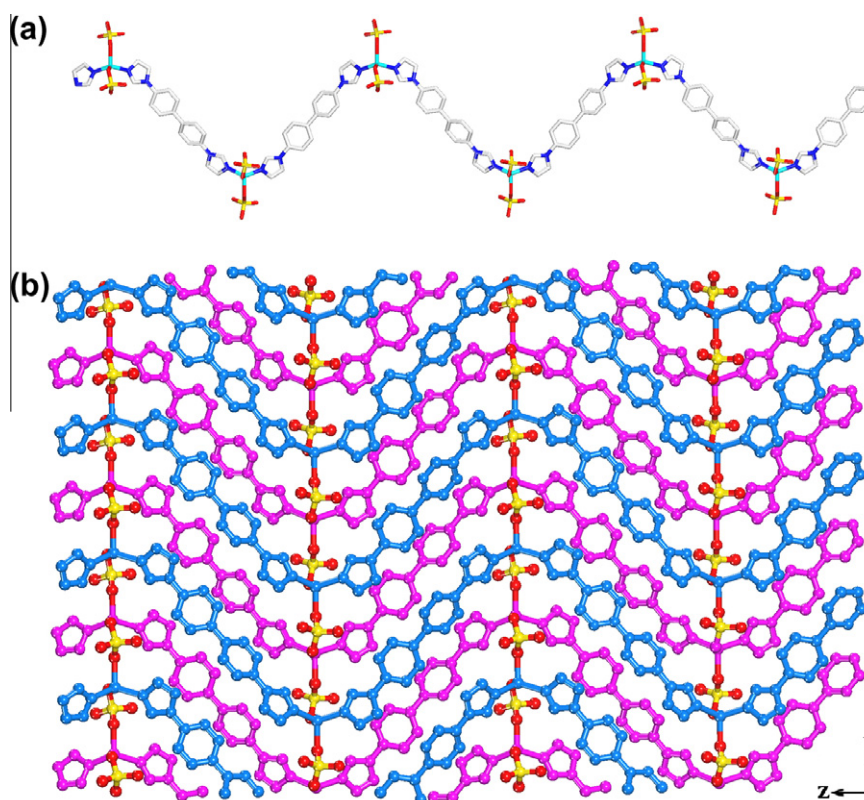


Fig. 6. (a) Undulated chain-like building unit of complex **3** (all the H atoms are omitted), Color code: O, red; N, blue; C, light gray; Co, cyan; S, yellow and (b) the undulated sheet of complex **3** along *bc* plane (all the H atoms are omitted). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

as found in **2**. Such sheets are stacked in an ABAB sequence along *a* axis.

3.2. Effects of anions behaviors

Complexes **1–3** with different dimensionality built up by structurally related rod-like ligands **L**¹ and **L**². In which two kinds of chain-like motifs, with similar linkage of -M(metal)-L(ligand)-M-L-, have been found, with the strictly linear chain-like motifs in complexes **1** and **2**, and the undulated chain-like motifs in complexes **2** and **3**. Interestingly, the metal centers in the three compounds are all linked by four ligands, even though they could be four connected or six connected. In **1**, the Zn atoms are surrounded by two **L**¹ ligands and two NO₃⁻ groups, and the Zn atoms are linked by **L**¹ ligands to form a strictly linear chain structure, due to the block of NO₃⁻ groups. Replaced **L**¹ by **L**², similar strictly linear chain-like motif has been found in **2**. Different to chain structure in **1**, the chain motifs in **2** have been further linked to present a layered

structure by **L**² ligands, for the anions BF₄⁻ preference for counterion behavior. Complex **3** also presents layered structure with 4,4'-network by using **L**² and SO₄²⁻ as the mixed ligands. Notably, the metal centers in **1** and **3** were linked by two poly(imidazole) ligands and two small anion groups, due to the anion groups exhibit different mode of linkage. The bidentate NO₃⁻ groups in **1** could be regarded as a block which direct to form 1D chain structure, however, due to the bridging SO₄²⁻ groups, the undulated chain-like motifs in **3** could be further linked to form a 2D layered structure. In summary, the dimensionalities of the coordination polymers could be influenced by the nature of the anions in the metal salt, for their different behaviors such as propensity for coordination and preference for counterion.

4. Conclusion

In presence of different anions, three new coordination polymers **1–3** based on two structurally related poly(imidazole) ligands

have been synthesized. It is worth noting that complexes **1–3** present 1D chain, 2D layered, 2D layered structures, for the presence of bidentate NO_3^- groups in **1**, BF_4^- counterions in **2**, bridging SO_4^{2-} groups in **3**, respectively. These results indicate that the different behaviors of anions could be the great influence on the degree of dimensionality of coordination polymers, and it should be a one more effective strategy to increase the dimensionality of coordination polymers.

Acknowledgments

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Appendix A. Supplementary materials

The supplementary crystallographic data for the structure analyses have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos. 840893, 840894 and 840895 for **1**, **2** and **3**. Copies of the data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: C44 1223 336033).

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