

# 由羧酸配体和铁(II)构筑的两个过渡金属配位聚合物的结构、表征和性质

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**摘要:** 采用水热法合成了 2 个铁的过渡金属配位聚合物  $[\text{Fe}(\text{Medpq})(\text{BDC})\text{H}_2\text{O}]_n$  (**1**) 和  $[\text{Fe}(\text{Medpq})(\text{QUI})\text{H}_2\text{O}] \cdot 2\text{H}_2\text{O}$  (**2**) (Medpq=2-methyldipyrido[3,2-*f*:2',3'-*h*]quinoxaline; H<sub>2</sub>BDC=terephthalic acid; H<sub>2</sub>QUI=2,3-pyridinedicarboxylic acid), 并对其进行了元素分析、红外光谱和热重表征, 并用 X 射线单晶衍射测定结构。2 个配位聚合物中的中心铁(II)离子, 都呈现一个稍微扭曲的八面体几何构型。

**关键词:** 配位聚合物; 水热合成; 羧酸配体

中图分类号: O614.81\*1

文献标识码: A

文章编号: 1001-4861(2016)02-0313-07

DOI: 10.11862/CJIC.2016.049

## Two Coordination Polymers Based on Carboxylate Ligands and Fe(II): Preparation, Structural Characterization and Properties

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**Abstract:** Two iron(II) coordination polymers  $[\text{Fe}(\text{Medpq})(\text{BDC})\text{H}_2\text{O}]_n$  (**1**) and  $[\text{Fe}(\text{Medpq})(\text{QUI})\text{H}_2\text{O}] \cdot 2\text{H}_2\text{O}$  (**2**) (H<sub>2</sub>BDC=terephthalic acid, H<sub>2</sub>QUI=2,3-pyridinedicarboxylic acid and Medpq=2-methyldipyrido [3,2-*f*:2',3'-*h*]quinoxaline) were prepared by hydrothermal method. They were characterized by elemental analysis, IR and thermogravimetric analysis, and their structures were determined by single-crystal X-ray diffraction. The Fe(II) ions of the two coordination polymers all assumed a slightly distorted octahedral geometry. CCDC: 1436495, **1**; 1436494, **2**.

**Keywords:** coordination polymer; hydrothermal processing; carboxylate ligand

## 0 Introduction

At this stage, owing to the possibility of structural variations and the controllability of the assembly process, the combination of anionic O-donor and N-donor ligands mixed ligands with metal cations in designing coordination polymers should be considered as an attractive design strategy<sup>[1]</sup>.

In particular, aromatic multicarboxylate ligands, for example 1,4-Benzenedicarboxylic acid, 2,3-pyridinedicarboxylic acid (H<sub>2</sub>QUI) are well used in the construction of MOFs with interesting structures and special topologies due to their structural rigidity, chemical stability and appropriate connectivity<sup>[2-13]</sup>.

On the other hand, there are a number of so-called “anomalies” of reactivity of N-heterocyclic complexes

收稿日期: 2015-04-16。收修改稿日期: 2015-12-28。

吉林省教育厅“十二五”科学技术研究项目(No.2013396)和吉林省科技厅自然科学基金(No.20130102002JC)资助。

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in aqueous solutions, such as bipyridine(bpy) and 1,10-phenanthroline(phen) complexes. So far, phen has been widely used to build supramolecular architectures because of its excellent coordinating ability and large conjugated system that can easily form  $\pi$ - $\pi$  interactions<sup>[14-16]</sup>. However, far less attention has been given to their derivatives. 2-Methyldipyrido[3,2-*f*:2',3'-*h*]quinoxaline (Medpq) as an important phen derivative possesses fruitful aromatic systems and is a good candidate for the construction of metal-organic supramolecular architectures. In this work, we designed and prepared the polymers using Medpq, namely  $[\text{Fe}(\text{Medpq})(\text{BDC})\text{H}_2\text{O}]_n$  (**1**) and  $\{[\text{Fe}(\text{Medpq})(\text{QUI})\text{H}_2\text{O}] \cdot 2\text{H}_2\text{O}\}_n$  (**2**).

## 1 Experimental

### 1.1 Materials

The Medpq ligand was synthesized according to the literature method<sup>[17]</sup>. Other chemicals from commercial sources were of reagent grade and used without further purification.

### 1.2 Instruments and measurements

Elemental analysis was carried out with a Perkin-

Elmer 240C analyzer. TG measurements were performed on a NETZSCH STA 449C analyzer. The Infrared (IR) spectrum was recorded from KBr pellets in the range of 4 000~400  $\text{cm}^{-1}$  on a Nicolet FTIR 170SX spectrometer.

### 1.3 Syntheses and measurements

#### 1.3.1 Synthesis of $[\text{Fe}(\text{Medpq})(\text{BDC})\text{H}_2\text{O}]_n$ (**1**)

Coordination polymer **1** was prepared from a mixture of  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  (0.12 g), Medpq (0.10 g),  $\text{H}_2\text{BDC}$  (0.08 g) and  $\text{H}_2\text{O}$  (25 mL) while stirring at room temperature. When the pH value of the mixture was adjusted to about 6.5 with NaOH, the cloudy solution was put into a 30 mL Teflon-lined autoclave under autogenous pressure at 165  $^{\circ}\text{C}$  for six days (Fig.1). After cooling to room temperature, light yellow block crystals of **1** were collected by filtration and washed with distilled water in 45% yield (based on Fe). Anal. Calcd. for  $\text{C}_{23}\text{H}_{16}\text{FeN}_4\text{O}_5$  (%): C, 57.05; H, 3.33; N, 11.57. Found(%): C, 57.08; H, 3.35; N, 11.49. IR (KBr,  $\text{cm}^{-1}$ ): 3 392m (ascribed to the stretching vibrations of  $\text{H}_2\text{O}$ , which indicates the existence of water molecules in coordination polymer **1**), 1 634s, 1 624s, 1 580s, 1 485 m, 1 355s, 1 368m, 1 258w, 1 133 m, 827m, 713m.

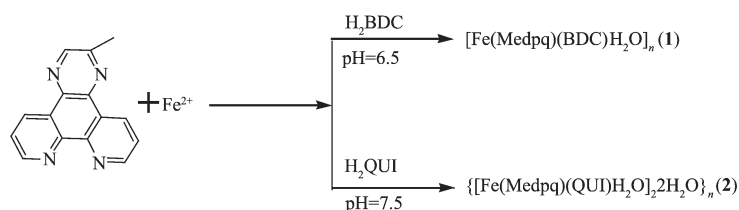


Fig.1 Synthetic routes of the coordination polymers

#### 1.3.2 Synthesis of $\{[\text{Fe}(\text{Medpq})(\text{QUI})\text{H}_2\text{O}] \cdot 2\text{H}_2\text{O}\}_n$ (**2**)

Coordination polymer **2** was synthesized by a method similar to that of **1**, except that the pH value of the reaction was adjusted to 7.5 with NaOH and using  $\text{H}_2\text{QUI}$  (0.1 g) instead of  $\text{H}_2\text{BDC}$ . Yellow block of **2** were collected by filtration and washed with distilled water in 40% yield (based on Fe). Anal. Calcd. (%) for  $\text{C}_{22}\text{H}_{19}\text{FeN}_5\text{O}_7$ : C, 50.69; H, 3.67; N, 13.44. Found (%): C, 50.76; H, 3.48; N, 13.45. IR (KBr,  $\text{cm}^{-1}$ ): 3 395m (ascribed to the stretching vibrations of  $\text{H}_2\text{O}$ , which indicates the existence of water

molecules), 1 634s, 1 610s, 1 580s, 1 523m, 1 355s, 1 368 m, 1 258w, 1 133m, 827m, 713m.

### 1.4 Crystal structure determination and physical measurements

Crystallographic data of the two coordination polymers were collected at 293 K on a Bruker SMART 1000 CCD X-ray diffractometer with a graphite-monochromatic  $\text{Mo } K\alpha$  radiation ( $\lambda=0.071\ 073\ \text{nm}$ ) by using  $\omega$  scan mode in the  $2^{\circ}$  range of  $6.0^{\circ}\sim 50.8^{\circ}$  (**1**) and  $6.3^{\circ}\sim 50.0^{\circ}$  (**2**). The structures of coordination polymers **1** and **2** were solved by direct methods with

SHELXS-97 program<sup>[18]</sup> and refined by SHELXL-97<sup>[19]</sup> using full-matrix least-squares techniques on  $F^2$ . All the hydrogen atoms were placed in the calculated sites and included in the final refinement in the riding model approximation with displacement parameters derived from the parent atoms to which they were bonded. All non-hydrogen atoms were refined anisotropic ally. All H atoms were positioned geometrically (C-H 0.093 nm for CH or 0.096 nm for

CH<sub>3</sub>) and refined as a riding mode, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . The H atoms of one uncoordination water molecule in coordination polymer **2** could not be positioned reliably. Other H atoms of water molecules were located from difference Fourier maps. Further crystallographic data and experimental details for structural analyses of both the two complexes are summarized in Table 1.

CCDC: 1436495, **1**; 1436494, **2**.

**Table 1** Crystallographic data for the coordination polymers **1** and **2**

Coordination polymer	<b>1</b>	<b>2</b>
Empirical formula	C <sub>23</sub> H <sub>16</sub> FeN <sub>4</sub> O <sub>5</sub>	C <sub>22</sub> H <sub>19</sub> FeN <sub>5</sub> O <sub>7</sub>
Formula weight	484.25	521.27
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/n$	$C2/c$
$a$ / nm	1.037 8(2)	2.485 8(5)
$b$ / nm	1.412 5(3)	1.257 0(3)
$c$ / nm	1.365 5(3)	1.396 6(3)
$\beta$ / (°)	96.00(3)	98.00(3)
$V$ / nm <sup>3</sup>	1.990 7(7)	4.321 4(17)
$Z$	4	8
$D_c$ / (g·cm <sup>-3</sup> )	1.616	1.599
Absorption coefficient / mm <sup>-1</sup>	0.805	0.755
$F(000)$	992	2 136
Crystal size / mm	0.20×0.20×0.22	0.18×0.21×0.22
Limiting indices ( $h, k, l$ )	-12~12, -16~16, -11~16	-29~23, -14~14, -16~16
Reflections collected	9 071	9 653
Independent reflections	3 604	3 787
$R_1, wR_2$ [ $I > 2\sigma(I)$ ]	0.059 4, 0.124 5	0.132 4, 0.233 5
Goodness-of-fit (GOF) on $F^2$	1.08	1.21

## 2 Results and discussion

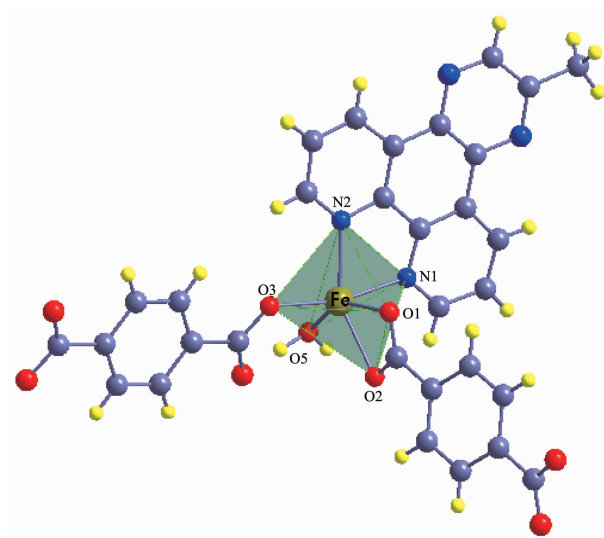
### 2.1 Description of crystal structures

#### 2.1.1 Crystal structure of [Fe(Medpq)(BDC)(H<sub>2</sub>O)]<sub>n</sub> (**1**)

Single-crystal X-ray diffraction analysis reveals that coordination polymer **1** crystallizes in  $P2_1/n$  space group and consists of a one-dimensional structure. There are one Fe(II) ion, one Medpq ligand, two half BDC ligands and one coordinate water molecule in the symmetric unit (Fig.2). The Fe (II) ion is hexa-coordinated with six atoms (N1, N2, O1, O2, O3, O5) from one Medpq ligand, two different BDC ligands and one coordinate water molecule, assuming a

slightly distorted octahedral geometry. The bond lengths are 0.217 0(3) and 0.216 8(3) nm for Fe-N, 0.205 9(2)~0.224 8(3) nm for Fe-O<sub>carboxylate</sub>, and 0.212 7(3) nm for Fe-O<sub>waters</sub>, respectively. The N(O)-Fe-O(N) angles range from 59.46(10)° to 166.00(11)°. The selected bond parameters are given in Table 2.

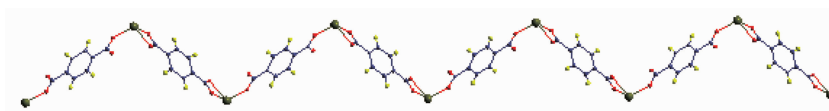
The coordination polymer adopt zigzag chain structures bridged by the BDC ligands in the bisbidentate and bis-monodentate modes with Fe-Fe distance of about 1.142 3 nm (Fig.3). Hydrogen bonding interactions are usually important in the synthesis of supramolecular architectures. There are persistent strong O-H...O hydrogen bonding

Fig.2 ORTEP drawing of **1** showing the local coordination environment of Fe(II)**Table 2** Selected bond lengths (nm) and angles ( $^{\circ}$ ) for the coordination polymers **1** and **2**

<b>1</b>					
Fe-O1	0.220 4(3)	Fe-O2	0.224 8(3)	Fe-O3	0.205 9(2)
Fe-O5	0.212 7(3)	Fe-N1	0.217 0(3)	Fe-N2	0.216 8(3)
O1-Fe-O3	103.07(10)	O1-Fe-O2	59.46(10)	O1-Fe-O5	147.34(11)
O1-Fe-N1	83.87(11)	O1-Fe-N2	90.26(11)	O2-Fe-O3	104.95(10)
O2-Fe-O5	88.10(10)	O2-Fe-N1	89.05(10)	O2-Fe-N2	147.86(11)
O3-Fe-O5	88.05(11)	O3-Fe-N1	166.00(11)	O3-Fe-N2	91.43(11)
O5-Fe-N1	92.49(12)	O5-Fe-N2	120.47(11)	N1-Fe-N2	76.24(11)
<b>2</b>					
Fe-O1	0.217 3(5)	Fe-O2	0.210 1(5)	Fe-O5A	0.209 3(5)
Fe-N1	0.221 2(6)	Fe-N2	0.219 5(6)	Fe-N5A	0.215 8(6)
O1-Fe-O2A	87.0(2)	O1-Fe-O5A	90.1(2)	O1-Fe-N1	169.6(2)
O1-Fe-N2	97.1(2)	O1-Fe-N5A	92.0(2)	O2-Fe-O5A	168.5(2)
O2-Fe-N1	87.6(2)	O2-Fe-N2	103.2(2)	O2-Fe-N5A	92.4(2)
O5A-Fe-N1	96.9(2)	O5A-Fe-N2	88.2(2)	O5A-Fe-N5A	76.5(2)
N1-Fe-N2	75.6(2)	N1-Fe-N5A	97.1(2)	N2-Fe-N5A	162.3(2)

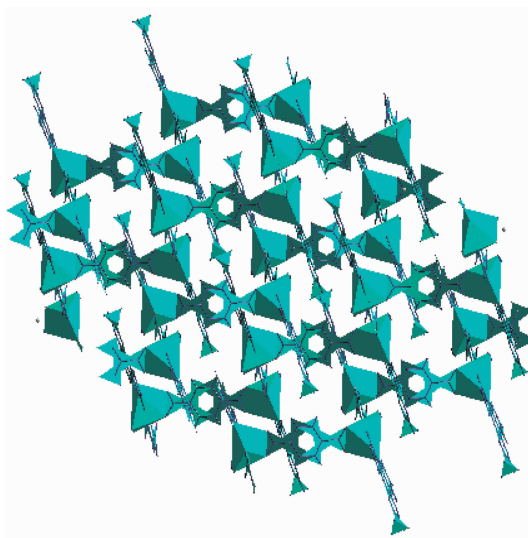
interactions between BDC ligands and coordinate water molecules and weak C-H $\cdots$ O hydrogen bonding interactions between BDC ligands and Medpq ligands (O5 $\cdots$ O2<sup>i</sup> 0.280 3(4) nm, O5 $\cdots$ O4 0.260 2(4) nm, C1 $\cdots$ O4<sup>i</sup> 0.314 9(5) nm, C2 $\cdots$ O4<sup>i</sup> 0.315 9(5) nm, C3 $\cdots$ O1<sup>ii</sup> 0.320 1(5) nm; O5-H5A $\cdots$ O2 165(4) $^{\circ}$ , O5-H5B $\cdots$ O4 158(5) $^{\circ}$ , C1-H1 $\cdots$ O4 124 $^{\circ}$ , C2-H2 $\cdots$ O4 121 $^{\circ}$ , C3-H3 $\cdots$ O1 133 $^{\circ}$ ; Symmetry codes: <sup>i</sup>  $-x, 1-y, -z$ ; <sup>ii</sup>  $1/2-x, 1/2+y, 1/2-z$ ), which play an important role in

stabilizing the network structure and controlling the orientation of ligands. At the same time, the aromatic ring of ligands (BDC ligand and Medpq ligand) and symmetry of the two adjacent equivalent of aromatic ring (Symmetry codes: <sup>i</sup>  $1/2-x, -1/2+y, 1/2-z$ ; <sup>ii</sup>  $-x, 1-y, 1-z$ ; <sup>iii</sup>  $1/2-x, 1/2+y, 1/2-z$ ; <sup>iv</sup>  $1/2+x, 1/2-y, 1/2+z$ ; <sup>v</sup>  $-1/2+x, 1/2-y, -1/2+z$ ) have  $\pi$ - $\pi$  interactions (Cg(4) $\rightarrow$ Cg(5)<sup>ii</sup> 0.361 6(2) nm, Cg(5) $\rightarrow$ Cg(6)<sup>ii</sup> 0.358 5(2) nm, Cg(5) $\rightarrow$ Cg(8)<sup>iii</sup> 0.363 6(2) nm, Cg(5) $\rightarrow$ Cg(8)<sup>iv</sup> 0.363 6(2)

Fig.3 View of 1D zigzag chain structure of the coordination polymer **1**

nm, Cg(8)→Cg(5)<sup>i</sup> 0.363 6(2) nm, Cg(8)→Cg(5)<sup>v</sup> 0.363 6(2) nm, Cg(6)→Cg(5)<sup>ii</sup> 0.358 5(2) nm, Cg(6)→Cg(6)<sup>ii</sup> 0.359 3(2) nm, Cg(6)→Cg(8)<sup>iv</sup> 0.372 4(2) nm, Cg(6)→Cg(8)<sup>iii</sup> 0.372 4(2) nm, Cg(8)→Cg(6)<sup>i</sup> 0.372 4(2) nm, Cg(8)→Cg(6)<sup>v</sup> 0.372 4(2) nm) for defined rings: Cg(4): N2→C12→C11→C10→C9→C14, Cg(5): N3→C5→C8→N4→C7→C6, Cg(6): C4→C5→C8→C9→

C14→C13 and Cg(8): C21→C22→C23→C21A→C22A→C23A. Through hydrogen bonding interactions and  $\pi$ - $\pi$  interactions between the adjacent aromatic ring of Medpq ligands and BDC ligands, coordination polymer **1** formed two-dimensional layer structure (Fig.4).

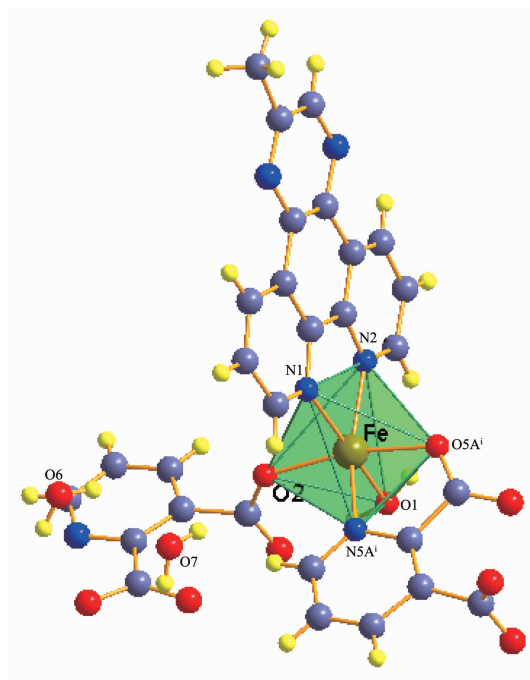
Fig.4 View of 2D layer structure of the coordination polymer **1**

### 2.1.2 Crystal structure of $[\text{Fe}(\text{Medpq})(\text{QUI})\text{H}_2\text{O}] \cdot 2\text{H}_2\text{O}]_n$ (**2**)

Single-crystal X-ray diffraction analysis reveals that coordination polymer **2** crystallizes in  $C2/c$  space group. The local coordination environment of Fe(II) consists of one Medpq ligand, one coordinate water molecule, two half QUI ligands and two free water (Fig.5). The Fe(II) ion is six-coordination with O1, N1, N2, N5A, O5A and O2 from coordinate water molecule, Medpq ligand, QUI ligand, and its symmetrical QUI ligand, respectively, assuming a slightly distorted octahedral geometry. The bond distances are Fe-N 0.215 8(6)~0.221 2(6) nm, Fe-O<sub>carboxylate</sub> 0.209 3(5) nm~0.210 1(5) nm and Fe-O<sub>water</sub> 0.217 3(5) nm, respectively. The N(O)-Fe-O(N) bond angles range from 75.6(2)° to 169.6(2)°. The selected

important bond parameters are given in Table 2. The interesting feature of coordination polymer **2** is that each QUI ligand links two symmetry-related iron atoms into an infinite 1D chain (Fig.6).

In coordination polymer **2**, strong O-H...O hydrogen bonds are observed between QUI ligands, water molecules and hydroxyl ions (O1...O3 0.263 8(9) nm, O1...O4<sup>i</sup> 0.278 7(8) nm, O6...O7 0.273 1(18) nm, O6...N4<sup>ii</sup> 0.302 4(15) nm; O1-H1B...O3 147.8°, O1-H1A...O4 138.2°, O6-H6D...O7 129.4°, O6-H6A...N4 124.2°). The weak C-H...O hydrogen bonds are observed between QUI ligands and Medpq ligands (C3...O2<sup>iii</sup> 0.335 2(11) nm, C11...O3<sup>i</sup> 0.302 0(13) nm, C20...O4<sup>iv</sup> 0.318 2(10) nm; C3-H3...O2 144.4°, C11-H11...O3 132.5°, C20-H20...O4 151.5°; Symmetry codes: <sup>i</sup> 1/2-x, -1/2+y, 1/2-z; <sup>ii</sup> -x, 1+y, 1/2-z; <sup>iii</sup> -x, y,



Symmetry codes: <sup>i</sup>  $x, 1-y, -1/2+z$

Fig.5 ORTEP drawing of **2** showing the local coordination environment of Fe(II)

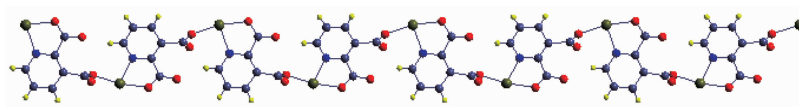


Fig.6 View of one-dimensional infinite chains of coordination polymer **2**

$1/2-z$ ; <sup>iv</sup>  $x, 1-y, 1/2+z$ ).

In coordination polymer **2**, intermolecular  $\pi$ - $\pi$  stacking interactions are found between the aryl ring of Medpq ligands and its equivalent symmetry (Symmetry codes: <sup>i</sup>  $-x, y, 1/2-z$ ; <sup>ii</sup>  $-x, -y, -z$ ) in an offset fashion. The distances for these intermolecular  $\pi$ - $\pi$  stacking interactions are 0.362 7(5) nm for Cg(4)  $\rightarrow$  Cg(4)<sup>i</sup>, 0.370 5(7) nm for Cg(5)  $\rightarrow$  Cg(6)<sup>i</sup>, 0.375 3(7) nm for Cg(5)  $\rightarrow$  Cg(6)<sup>ii</sup>, 0.370 5(7) nm for Cg(6)  $\rightarrow$  Cg(5)<sup>i</sup>, 0.375 2(7) nm for Cg(6)  $\rightarrow$  Cg(5)<sup>ii</sup>, 0.391 3(7) nm for Cg(6)  $\rightarrow$  Cg(8)<sup>i</sup>, 0.391 3(7) nm for Cg(8)  $\rightarrow$  Cg(6)<sup>i</sup> and 0.364 6(6) nm for Cg(8)  $\rightarrow$  Cg(8)<sup>i</sup> (Cg(4): N(1)  $\rightarrow$  C(2)  $\rightarrow$  C(3)  $\rightarrow$  C(4)  $\rightarrow$  C(13), Cg(5): N(2)  $\rightarrow$  C(12)  $\rightarrow$  C(11)  $\rightarrow$  C(10)  $\rightarrow$  C(9)  $\rightarrow$  C(14), Cg(6): N(3)  $\rightarrow$  C(5)  $\rightarrow$  C(8)  $\rightarrow$  N(4)  $\rightarrow$  C(7)  $\rightarrow$  C(6) and Cg(8): C(4)  $\rightarrow$  C(5)  $\rightarrow$  C(8)  $\rightarrow$  C(9)  $\rightarrow$  C(14)  $\rightarrow$  C(13)).

With the help of above two kinds of interactions, the title coordination polymer formed 2D layer

structure (Fig.7).

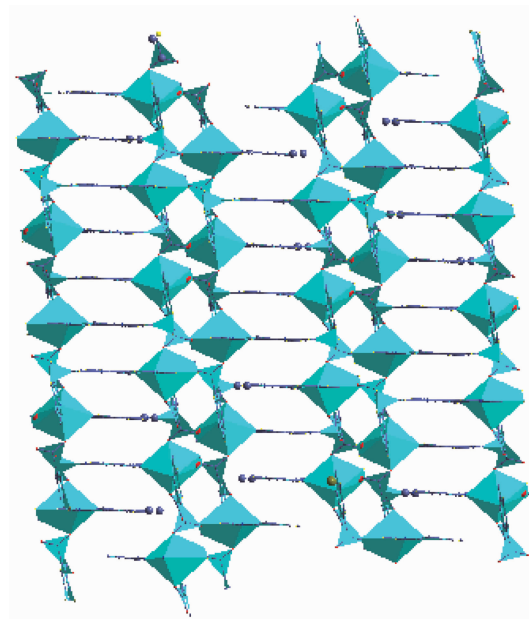


Fig.7 View of 2D layer structure of the coordination polymer **2**



## 2.2 Thermal analyses

The stability of **1** and **2** was investigated by thermal gravimetric analysis (Fig.8). The first weight loss of 3.9% (**1**) and 10.4% (**2**) are in the ranges of 139.8 ~347.6 °C and 111.8 ~226.3 °C, respectively, corresponding to the removal of water molecules (Calcd. 3.7% for **1** and 10.4% for **2**). The second weight loss of 31.8% (**1**) and 26.6% (**2**) are in the ranges of 347.6 ~408.6 °C and 226.3 ~424.9 °C, respectively, corresponding to the removal of carboxylic acid ligand (Calcd. 31.3 % for **1** and 28.1% for **2**). The last weight loss of 49.9% (**1**) and 48.1% (**2**) are in the ranges of 408.6 ~599.9 °C and 424.9 ~1197.9 °C, respectively, corresponding to the removal of Medpq ligand (Calcd. 50.2% for **1** and 47.6% for **2**). After 599.9 and 1197.9 °C, no weight loss is observed, indicating the complete decomposition of **1** and **2**. The residual weight of 14.4% (**1**) and 14.9% (**2**) (Calcd. 14.8% for **1** and 13.9% for **2**) correspond to the metal oxide FeO.

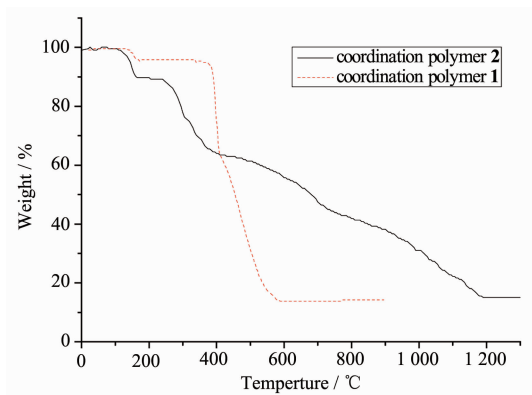


Fig.8 TG curves of the coordination polymers

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