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# 辅助配体导向合成两个锌金属配位聚合物:结构,稳定性和荧光性质

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**摘要**:以二苯-4,4′-二羧酸和六水硝酸锌为原料,在相同反应条件下,通过改变不同的辅助配体(2,2′-二甲基-4,4′-联吡啶和三乙烯二胺),合成出 2 个金属有机配位聚合物:{[Zn(DBA)(dmbpy)$_{0.5}$]·2H$_2$O}$_n$ (**1**)和[Zn$_2$(DBA)$_2$(dabco)]$_n$ (**2**)(H$_2$DBA=二苯-4,4′-二羧酸,dmbpy=2,2′-二甲基-4,4′-联吡啶,dabco=三乙烯二胺)。通过元素分析、红外光谱、差热分析、X 射线粉末衍射和 X 射线单晶衍射等手段对配合物进行了结构表征。结果显示,化合物 **1** 为二维(4,4)网络结构,2 个二维结构在平行方向上通过互锁进一步形成 2D→2D 的互锁网络结构。化合物 **2** 为一维带状结构。热稳定性表明化合物 **1** 能够稳定到 370 ℃;而化合物 **2** 稳定性差。荧光分析表明常温固态下配合物 **1** 和 **2** 均发射蓝色荧光,荧光寿命分别为 231.4 ns 和 2.33 ns。

**关键词**:配位聚合物;晶体结构;二苯-4,4′-二羧酸;荧光性质
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# Auxiliary Ligands Controlled Assembly of Two Zinc Coordination Polymers: Structures, Stability and Luminescence

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**Abstract:** By changing the auxilary ligands, two zinc-based coordination polymers (CPs), {[Zn(DBA)(dmbpy)$_{0.5}$]·2H$_2$O}$_n$ (**1**) and [Zn$_2$(DBA)$_2$(dabco)]$_n$ (**2**) (DBA=4,4′-methylenedibenzoate, dmbpy=2,2′-dimethyl-4,4′-bipyridine, dabco=triethylenediamine) have been prepared based on 4,4′-methylenedibenzoate and Zn(NO$_3$)$_2$·6H$_2$O under the same reaction conditions and were structurally characterized by elemental analysis, IR, TGA, powder X-ray diffraction and single-crystal X-ray diffraction. **1** is a 2D (4,4) network, and two nets interlock in parallel and give rise to a polycatenated layer (2D → 2D). **2** is a ribbon-like infinite chain with dabco ligands point alternately up and down with respect to the chain. The results of thermal analysis indicate that **1** is quite stable up to 370 ℃, whereas **2** shows low thermal stability. Finally, both of **1** and **2** emit the intensely blue characteristic luminescence at room temperature, with lifetimes of up to 231.4 ns and 2.33 ns, respectively. CCDC: 1450687, **1**; 1450688, **2**.

**Keywords:** coordination polymers; crystal structure; 4,4′-methylenedibenzoate; luminescent

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## 0 Introduction

Metal-organic coordination polymers (CPs), have received widespread attention over the past decade owing to their modular assembly, structural diversity and fascinating topology, chemical tailorability and tenability, as well as their excellent properties with promising applications such as gas storage and separation, nonlinear optics, catalysis, magnetism, luminescence, drug delivery, sensing, and detection[1-7]. During the attainment of CPs, many factors can influence the construction progress, e.g., metal ions, organic ligands, solvents, pH values, reaction temperatures, and so on[8-9]. Among many on-going efforts to develop high-performance CPs materials, selection and utilization of different organic ligands is considered to be the most significant factors that affect the structures and properties of final products[10-11]. Up to date, more attention has been paid to complicated organic ligands because of their various coordinated modes and ability to obtain novel structures [12]. However, it is fussy to obtain these ligands, which requires much more effort to achieve industrialization or practical application. As part of an on-going study related to functional CPs, 4,4′-methylenedibenzoic acid ($H_2$DBA), 2,2′-dimethyl-4,4′-bipyridine (dmbpy) and triethylenediamine (dabco) were chosen to afford two transition metal-based coordination polymers, {[Zn(DBA)(dmbpy)$_{0.5}$]·2$H_2O$}$_n$ (**1**) and [$Zn_2$(DBA)$_2$(dabco)]$_n$ (**2**), which are characterized by elemental analyses, IR spectrum, thermogravimetric analyses, powder X-ray diffraction and single-crystal X-ray crystallography. Furthermore, the stability and luminescence properties of **1** and **2** were also investigated.

## 1 Experimental

### 1.1 Materials and measurements

All chemicals purchased were of reagent grade and used without further purification. 2,2′-dimethyl-4,4′-bipyridine (dmbpy) was isolated based on the reported procedures[6]. All syntheses were carried out in 23 mL Teflon-lined autoclaves under autogenous pressure. Elemental analyses (C, H and N) were performed on a Perkin-Elmer 240 CHN elemental analyzer. Infrared spectra were taken on a Shimadzu IR-440 spectrometer with a KBr disk in the 4000~400 $cm^{-1}$ region. Thermogravimetric analyses (TGA) were carried out on an automatic simultaneous thermal analyzer (DTG-60, Shimadzu) under $N_2$ atmosphere at a heating rate of 10 ℃·$min^{-1}$ within a temperature range of 25~800 ℃. Powder XRD investigations were carried out on a Bruker AXS D8-Advanced diffractometer at 40 kV and 40 mA with Cu $K\alpha$ ($\lambda$=0.154 06 nm) radiation. Luminescence spectra and lifetime for crystal solid samples were recorded at room temperature on an Edinburgh FLS920 phosphorimeter.

### 1.2 Synthesis of 1

A mixture of $Zn(NO_3)_2\cdot 6H_2O$ (0.15 g, 0.5 mmol), $H_2$DBA (0.128 g, 0.5 mmol) and dmbpy (0.046, 0.25 mmol) in 12 mL DMF. The resulting solution was sealed in a 23 mL Teflon-lined stainless steel autoclave and heated at 130 ℃ for 3 days under autogenous pressure. Colorless single crystals were obtained (Yield: 68%, based on DBA) upon cooling the solution to room temperature at 5 ℃·$h^{-1}$. Anal. Calcd. for $C_{42}H_{31}N_2O_8Zn_2$(%): C, 61.28; H, 3.77; N, 3.40. Found (%): C, 62.51; H, 3.53; N, 3.31. IR (KBr, $cm^{-1}$): 3 442 (s), 3 063(w), 2 933(m), 1 674(m), 1 613(vs), 1 565(s), 1 535(m), 1 383(vs), 1 303(s), 1 172(s), 1 107(m), 1 016 (m), 967(w), 886(m), 854(m), 813(m), 762(vs), 743(m) m, 711(s), 699(m), 645(w), 637(w), 566(m), 512(m), 477(w), 428(s).

### 1.3 Synthesis of 2

The synthetic procedure of **2** was same as that of **1** except that dmbpy was replaced by dabco (Yield: 45%, based on DBA). Anal. Calcd. for $C_{36}H_{32}N_2O_8Zn_2$ (%): C, 57.49; H, 4.26; N, 3.73. Found(%): C, 57.61; H, 4.13; N, 3.80. IR (KBr, $cm^{-1}$): 3 423(s), 2 981(w), 2 935(w), 1 608(vs), 1 555(s), 1 504(m), 1 447(m), 1 367 (vs), 1 315(w), 1 268(w), 1 187(w), 1 150(w), 1 084 (m), 1 046(m), 857(m), 745(s), 659(m), 579(m), 517 (w), 442(w).

### 1.4 Crystal structure analysis

Single crystal X-ray diffraction analyses of compounds **1~2** were performed on a Bruker Apex Ⅱ CCD diffractometer operating at 50 kV and 30 mA

using Mo $K\alpha$ radiation ($\lambda$=0.071 073 nm). Data collection and reduction were performed using the APEX Ⅱ software[18]. Multi-scan absorption corrections were applied for all the data sets using SADABS, as included in the APEX Ⅱ program[18]. The structures were solved by direct methods and refined by least squares on $F^2$ using the SHELXTL program package[19]. The routine SQUEEZE (PLATON)[20] were applied to remove diffuse electron density caused by badly disordered water molecules of **1**. The formula unit of **1** was arrived at through combination of elemental analyses, IR spectra and thermogravimetric characterization. All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms attached to carbon and oxygen were placed in geometrically idealized positions and refined using a riding model. Crystallographic data and structural refinement detail of compounds **1**~**2** can be found in Table 1. Selected bond lengths and bond angles are given in Table 2.

CCDC: 1450687, **1**; 1450688, **2**.

**Table 1　Crystal data and structure refinement information for compounds 1~2**

| Complex | **1** | **2** |
|---|---|---|
| Empirical formula | $C_{42}H_{31}N_2O_8Zn_2$ | $C_{36}H_{33}N_3O_8Zn_2$ |
| Formula weight | 822.43 | 766.39 |
| Temperature / K | 296(2) | 296(2) |
| Crystal system | Orthorhombic | Tetragonal |
| Space group | *Cccm* | $P4_2/mnm$ |
| *a* / nm | 1.410 09(8) | 0.901 78(9) |
| *b* / nm | 2.113 49(13) | 0.901 78(9) |
| *c* / nm | 1.277 15(8) | 2.132 6(2) |
| *V* / nm$^3$ | 3.806 2(4) | 1.734 2(4) |
| *Z* | 4 | 2 |
| $D_c$ / (g·cm$^{-3}$) | 1.435 | 1.468 |
| $\mu$ / mm$^{-1}$ | 1.316 | 1.438 |
| *F*(000) | 1 684 | 788 |
| GOF | 1.152 | 1.090 |
| Reflections collected, unique | 11 919, 1 847 | 10 218, 710 |
| $R_{int}$ | 0.043 1 | 0.041 2 |
| $R_1$, $wR_2$ [$I>2\sigma(I)$] | 0.058 8, 0.182 0 | 0.062 0, 0.170 4 |
| $R_1$, $wR_2$ (all data) | 0.075 8, 0.211 2 | 0.072 6, 0.184 6 |

**Table 2　Selected bond distances (nm) and angles (°) of 1 and 2**

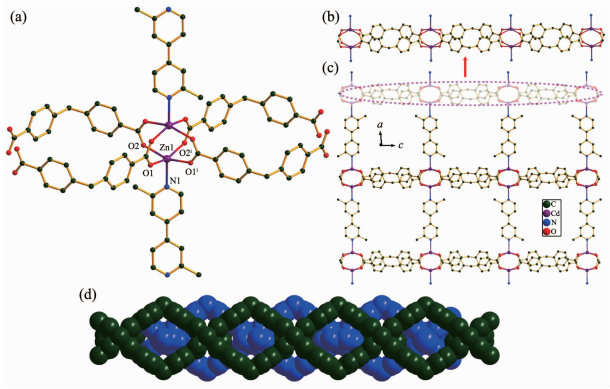
| | | | | | |
|---|---|---|---|---|---|
| | | | **1** | | |
| Zn1-N1 | 0.206 6(9) | Zn1-O1 | 0.204 2(5) | Zn1-O2 | 0.203 6(6) |
| O2i-Zn1-O2 | 87.4(5) | O2-Zn1-O1$^{i}$ | 157.5(3) | O2$^{i}$-Zn1-O1$^{i}$ | 88.2(3) |
| O2-Zn1-N1 | 97.8(2) | O1-Zn1-N1 | 104.7(2) | | |
| | | | **2** | | |
| Zn1-N1 | 0.203 5(9) | Zn1-O1 | 0.205 4(3) | | |
| O1$^{iii}$-Zn1-O1$^{i}$ | 88.0(2) | O1$^{iii}$-Zn1-O1$^{ii}$ | 159.3(2) | O1$^{ii}$-Zn1-O1$^{i}$ | 88.3(2) |
| N1-Zn1-O1 | 100.35(11) | | | | |

Symmetry codes: $^{i}$ $x$, $y$, $-z$ for **1**; $^{i}$ $x$, $y$, $1+z$; $^{ii}$ $1-y$, $1-x$, $z$; $^{iii}$ $1-y$, $1-x$, $1-z$ for **2**

## 2 Results and discussion

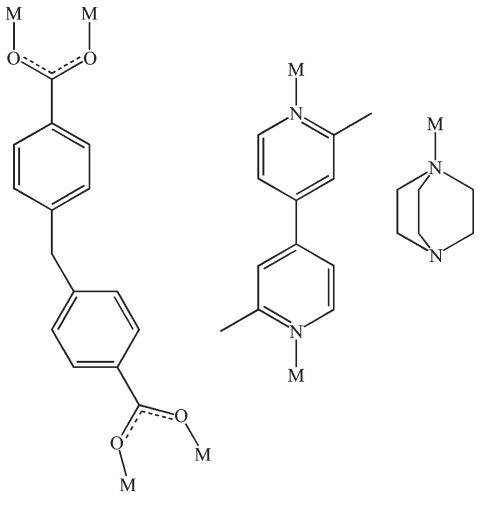
### 2.1 Structure description

Single-crystal X-ray diffraction analysis reveals that compound **1** has 2D (4,4) layered structure and crystallizes in orthorhombic *Cccm* space group. Coordination environment of Zn(Ⅱ) atom is shown in Fig.1a. In the asymmetric unit of **1**, there is one Zn(Ⅱ) cation, half a DBA anion and one quarter dmbpy ligand. The Zn(Ⅱ) center is five-coordinated by four oxygen atoms from four DBA ligands, one nitrogen atom from one dmbpy ligand, and displays a distorted trigonal bipyramidal geometry. The Zn-O, Zn-N bond lengths and O-Zn-O, O-Zn-N bond angles range from 0.202 9(4) to 0.205 6(4) nm and 87.7(3)° to 157.55(18)°, respectively, which is within the reasonable range of observed values for other five-coordinated Zn(Ⅱ) complexes with oxygen and nitrogen donating ligands[10,17]. In the crystal structure of **1**, the DBA ligands adopt one $\mu_4$ bridging mode to link four Zn(Ⅱ) ions, whereas dmbpy ligand acts as a *trans* bridging ligand to link two Zn(Ⅱ) ions (Scheme 1: modes Ⅰ and Ⅱ). In this manner, the dinuclear zinc units are linked into a linear chain through DBA anions with the separation of 1.277 2 nm between two dinuclear zinc cores (Fig. 1b). The dmbpy ligands also bridge the dinuclear zinc units to form a linear chain with the dinuclear zinc cores separation of 1.410 1 nm. Such two types of parallel linear chains are perpendicularly intersected by the dinuclear zinc nodes, resulting in a 2D (4,4) network (Fig.1c) when the dinuclear zinc unit and organic ligands are regarded as a node and linkers, respectively. Each 4-membered ring consists of four DBA ligands, two dmbpy ligands and eight Zn(Ⅱ) atoms. The large voids with dimensions of 0.75 nm×1.04 nm for each layer allow the formation of catenation between adjacent two layers in a parallel manner. As shown in Fig.1d, two (4,4) networks interlock in parallel and give rise to a polycatenated layer (2D→2D). Such parallel (4,4) layered interlocking modes are well know to belonging to the parallel-parallel (p-p) class[21].




All H atoms are omitted for clarity; Symmetry codes: $^{i}$ $x$, $y$, $-z$

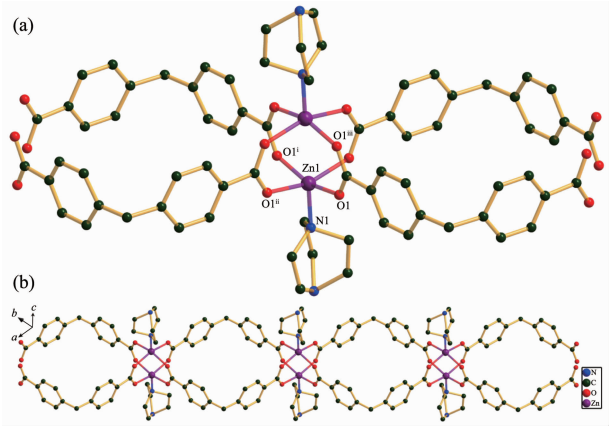
Fig.1 (a) Coordination environment of Zn(Ⅱ) in **1**; (b) View of a linear chain of 1; (c) View of the 2D layer structure of **1**; (d) View of the 2D polycatenation in **1** along the *a*-axis




Scheme 1 Coordination modes of DBA, dmbpy and dabco ligands in **1** and **2**

Compound **2** crystallizes in the tetragonal space group $P4_2/mnm$, with one Zn atom, one DBA anion and a dabco ligand in the asymmetric unit. The five-coordinated Zn(Ⅱ) center is surrounded by four oxygen atoms from four different DBA anions and one nitrogen atoms from a dabco ligand, which exhibits a distorted trigonal bipyramidal geometry (Fig.2a), with Zn-O, Zn-N distances and O-Zn-O, O-Zn-N bond angles range from 0.203 5(9) to 0.205 4(3) nm and from 88.0(2)° to 159.3(2)°, respectively. All of within the range of those found in other five-coordinated Zn(Ⅱ) complexes with oxygen and nitrogen donating ligands[10,17]. In the polymeric structure of **2**, the DBA and dabco ligands act in $\mu_4$ bridging and monodentate modes, respectively (Scheme 1: modes Ⅰ and Ⅲ). In

this manner, the $\mu_4$-DBA ligands link dinuclear zinc units to result in a ribbon-like infinite chain with dabco ligands point alternately up and down with respect to the chain (Fig.2b). The separation of two neighboring dinuclear zinc cores is 1.275 3 nm.




All H atoms are omitted for clarity; Symmetry codes: [i] $x$, $y$, $1+z$; [ii] $1-y$, $1-x$, $z$; [iii] $1-y$, $1-x$, $1-z$

Fig.2　(a) Coordination environment of Zn(Ⅱ) in **2**; (b) View of a ribbon-like infinite chain of **2**

## 2.2　IR spectra and TGA

The IR spectra shows broad bands in the 3 442 $cm^{-1}$ for **1**, 3 423 $cm^{-1}$ for **2**, which may be assigned to the $\nu$(O-H) stretching vibrations of the adsorbed water (impurity) and the free water molecules, respectively (Fig.S1). The absorption band observed at 2 933 $cm^{-1}$ for **1**, and 2 981, 2 935 $cm^{-1}$ for **2** are attributed to the $\nu(C_{methyl}\text{-H})$ vibration of dmbpy ligands.The features at 1 613 and 1 383 $cm^{-1}$ for **1**, 1 608 and 1 367 $cm^{-1}$ for **2**, are associated with the asymmetric $\nu$(COO) and symmetric $\nu$(COO) stretching vibrations.

Thermal gravimetric analyses (TGA) were carried out to examine the thermal stability of **1** and **2**. The samples were heated up in flowing $N_2$ with a heating rate of 10 ℃·$min^{-1}$. The TG curve for **1** shows a weight loss in the temperature range of 80~200 ℃ corresponding to the removal of two lattice water molecules (Calcd. 7.29%, Obsd. 7.52%). Upon further heating, the framework was stable up to 370 ℃ and then a sharp weight loss was observed above 370 ℃ due to the collapse of the framework (Fig.3). The TGA curve for **2** shows a weight loss between 80~200 ℃, which corresponds to the loss of one dabco ligand (Calcd. 14.93%, Obsd. 15.01%). Upon further heating, a sharp weight loss was observed above 200 ℃ due to the collapse of the framework.

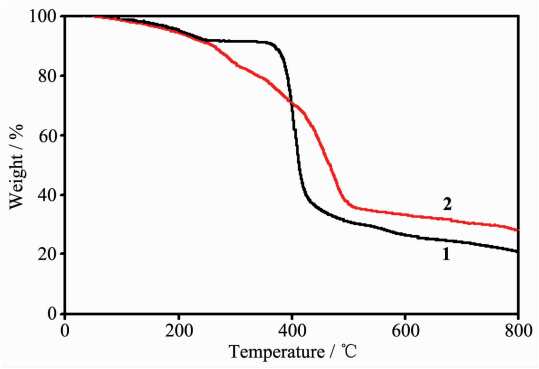



Fig.3　TGA curves of compounds **1** and **2**

## 2.3　Stability properties

It has demonstrated that the incorporation of hydrophobic functional groups (e.g. -$CH_3$) within frameworks might largely enhance the M-O bonds and thus improve the water resistance of the CPs[10]. The pH-dependent stability of **1** in aqueous solutions were investigated by PXRD (Fig.4). For these tests, 50 mg of as-synthesized compounds were soaked in aqueous

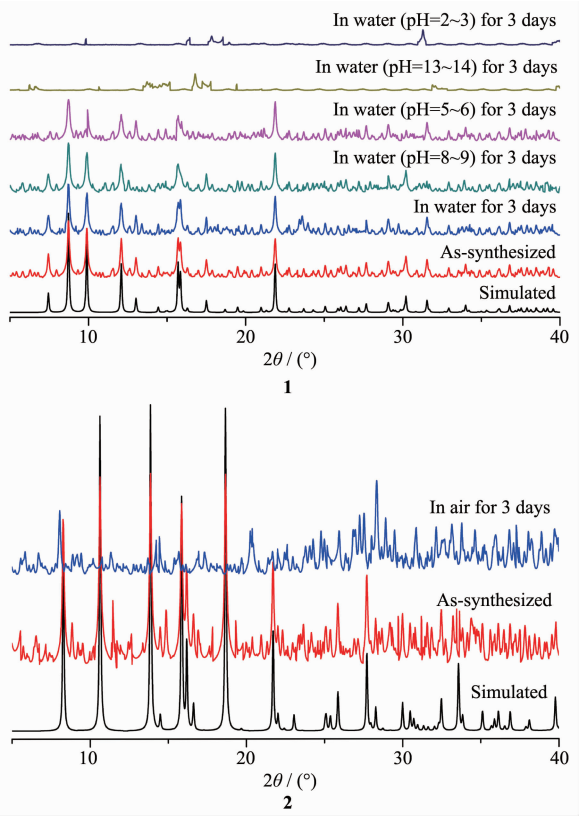



Fig.4　PXRD patterns of **1** and **2**

solutions with different pH values and stirred for 3 day at room temperature. Compared to **1**, compound **2** is unstable even in air (Fig.4). Stirring under more basic (pH=13~14) or acidic (pH=2~3) conditions, **1** shows a complete decomposition of its framework. The stabilities of **1** are similar to several aluminum-isophthalate-based MOFs (CAU-10-X, where X =H, $CH_3$, $OCH_3$, $NO_2$, $NH_2$, OH) [22], but lower than the series of carboxylate-based MOFs involving zirconium ions[23-24]. However, the result is very remarkable, especially when compared with other CPs constructed from aromatic carboxylate, N-containing auxiliary ligands and zinc/cadmium ions[25-27]. Moreover, the peak positions of the experimental patterns are in a good agreement with the simulated patterns, which clearly indicates the good purity of the complexes (Fig.4).

### 2.4 Luminescent properties

Luminescent properties of coordination polymers with $d^{10}$ metal centers have attracted intense interest due to their potential applications[28-29]. The photoluminescenct spectrum of $H_2DBA$ ligand shows emission maxima at 391 nm ($\lambda_{ex}$=314 nm)[30] (Fig.5). The emission band of the free ligand is probably attributable to the $\pi^*$-$\pi$ transition. Upon complexation of the ligands with Zn(Ⅱ) ions, the emission peak occur at 416 nm ($\lambda_{ex}$=322 nm) for **1** and 405 nm ($\lambda_{ex}$=313 nm) for **2**, respectively. The short wavelength band of **1** and **2** are almost same as that of the $H_2DBA$ ligand, which indicates that the emissions of **1** and **2** probably origins from the $H_2DBA$ ligands. The luminescent lifetimes of solid **1** and **2** using an Edinburgh FLS920 phosphorimeter with 450 W xenon lamp as excitation source show lifetimes of 231.4 ns and 2.33 ns, respectively (Fig.6).

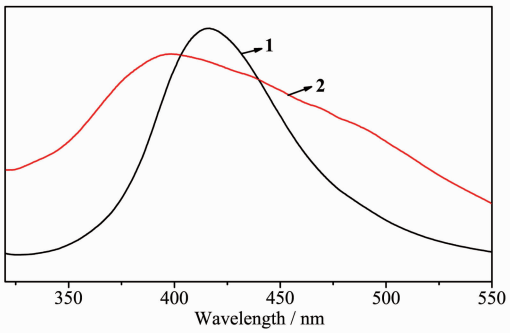



Fig.5 Emission spectra for **1** and **2** in solid state at room temperature

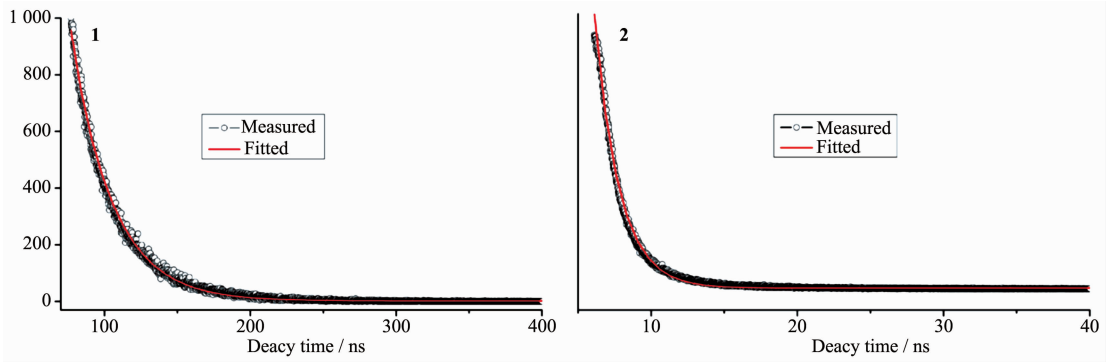



Fig.6 Luminescent lifetimes of **1** and **2** in solid state at room temperature

## 3 Conclusions

In summary, by changing the auxiliary ligands, two zinc-based CPs have been prepared based on the reaction of 4,4′-methylenedibenzoic acid and $Zn(NO_3)_2 \cdot 6H_2O$ under the same reaction conditions. **1** performs a polycatenated layer (2D→2D) structure and is stable in aqueous solutions in the pH value range of 5~9, whereas **2** shows a ribbon-like infinite chain with dabco ligands point alternately up and down with respect to the chain and is unstable in air. Both **1** and **2** emit bright blue fluorescence with the lifetimes of 231.4 ns and 2.33 ns, respectively.

Supporting information is available at http://www.wjhxxb.cn

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