

4'-(3-甲氧基-4-羟基苯基)-2,2':6',2''-三联吡啶及其铜、锌配合物的合成、结构和性质

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摘要: 通过一锅反应制备了 4'-(3-甲氧基-4-羟基苯基)-2,2':6',2''-三联吡啶(L), 通过重结晶的方法得到了其醋酸盐单晶体(HL) (CH₃COO) (**1**), 并以 L 为配体组装了过渡金属配合物[Cu(NO₃)(CH₃OH)L](NO₃) (**2**)和[Zn(NO₃)₂L](C₂H₅OH) (**3**), 通过元素分析、IR 光谱和单晶 X-射线衍射等表征了 3 个化合物的结构。结果表明, 化合物 **1** 中, 三联吡啶环呈反-反构型, 而在 **2** 和 **3** 中, 三联吡啶环呈现出顺-顺构型, 然后采取三齿螯合模式连接金属离子形成单核单元。3 个化合物中, 三联吡啶分子之间以及它们与溶剂分子之间形成的氢键、 π - π 堆积和范德华力等相互作用将化合物 **1~3** 的单核单元连接形成三维网状结构。

关键词: 三联吡啶; 晶体结构; 过渡金属配合物; 氢键

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Syntheses, Structures and Properties of 4'-(3-Methoxy-4-Hydroxyphenyl)-2,2':6',2''-Terpyridine Together with Copper and Zinc Complexes

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Abstract: The ligand 4'-(3-methoxy-4-hydroxyphenyl)-2,2':6',2''-terpyridine (L) and its transition-metal complexes have been prepared. The crystal structures of (HL)(CH₃COO) (**1**), [Cu(NO₃)(CH₃OH)L](NO₃) (**2**) and [Zn(NO₃)₂L](C₂H₅OH) (**3**) have been determined by single crystal X-ray diffraction. The result demonstrated that the three pyridine rings in crystal **1** were *trans-trans* conformation and one pyridine nitrogen atom is protonated, while the three pyridine rings of the ligand in crystal **2** and **3** were *cis-cis* conformation and they adopted tridentate chelating mode to link metal ions into mononuclear units. Further, these mononuclear units are assembled into 3D networks by hydrogen bonds, π - π stacking interactions and Van der Waals force. CCDC: 992747, **1**; 992748, **2**; 992749, **3**.

Key words: terpyridine; crystal structure; transition metal complexes; hydrogen bonds

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0 Introduction

2,2':6',2''-Terpyridine (tpy) and its derivatives as a class of organic heterocyclic tridentate ligands have been well studied for their wide application in supramolecular chemistry and material chemistry^[1-5]. Especially, the ligands containing aromatic substituents at the C(4') position are of great attraction with high chemical and thermal stability along with the large conjugated system to tailor the electronic and photophysical properties in recent years^[6-10]. In addition, 4'-substituted phenyl-2,2':6',2''-terpyridine has strong coordination ability and can act as tridentate chelating ligand to react with transition metals to form stable complexes^[11-14]. The metal-bonded terpyridine complexes have potential applications in photoluminescence, catalysis and new electroluminescent materials^[15-16]. Moreover, the large conjugated aromatic rings and the substituents at C(4') position can control the organization of metal-complexes by non-covalent interactions, such as hydrogen bonds and π - π stacking interactions, which extend the structures into higher dimensionality.

In this paper, tpy-based ligand with aryl-substituted 4'-position: 4'-(3-methoxy-4-hydroxyphenyl)-2,2':6',2''-terpyridine (L), was synthesized by the facile one-step condensation of 2-acetylpyridine with the 3-methoxy-4-hydroxybenzaldehyde. Single crystal (HL)(CH₃COO) (**1**) was obtained by recrystallized ligand L from acetic acid. It is noteworthy that this is the first example of one-step synthesis of tpy-based ligands with hydroxyl and methoxy substituents in the benzene ring, which extended the conjugation π -systems. On the other hand, the hydroxyl is benefit for forming hydrogen bonds than methoxy^[17]. This architectural feature makes this system interesting for the construction of network. So, we also prepared the corresponding complexes of Cu(II) and Zn(II)-based with the functionalized organic ligand L, which are [Cu(NO₃)(CH₃OH)L](NO₃) (**2**) and [Zn(NO₃)₂L](C₂H₅OH) (**3**). Herein, we reported the syntheses, crystal structures of compounds **1**~**3** and the thermal property of **2**.

1 Experimental

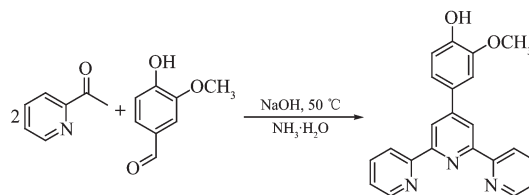
1.1 General methods and materials

All chemicals were commercially available and used without further purification. Elemental analyses (C, H and N) were measured on a Perkin-Elmer auto-analyzer. Infrared spectra were recorded on a BIO-RAD FTS3000 system by using KBr pellets in the range 4 000~400 cm⁻¹. Thermogravimetric analysis (TGA) was carried out on a Netzsch STA 409 PG/PC in a static air atmosphere from room temperature to 800 °C at a ramp rate of 5 °C·min⁻¹. The ¹H NMR spectra was measured on a Bruker AVANCE 300 spectrometer using TMS as internal standard.

1.2 Syntheses

1.2.1 Synthesis of the ligand L

The synthesis route of L is outlined in Scheme 1.



Scheme 1 Synthesis route of L ligand

To a solution of 2-acetylpyridine (2.42 g, 0.02 mol) in ethanol (30 mL) added aqueous NaOH (10 mol·L⁻¹, 10 mL), and stirred at room temperature for 30 min. 4-hydroxy-3-methoxybenzaldehyde (1.52 g, 0.01 mol) was added to the solution. After the mixture was stirred at room temperature for 15 min, 25% ammonia solution (15 mL) was added. The reaction mixture was stirred at 50 °C overnight. The suspension was filtered and the precipitate was washed with 50 mL of water and 30 mL of cold methanol. The crude product was recrystallized from EtOH to yield compound L (C₂₂H₁₇N₃O₂). Yield: 2.42 g, 63.1%. IR (KBr pellet, cm⁻¹): 3 403(m), 3 059(w), 3 009(w), 2 929(w), 2 839(w), 1 581(s), 1 564(m), 1 546(w), 1 518(s), 1 466(s), 1 438(s), 1 392(s), 1 354(m), 1 322(w), 1 266(s), 1 199(m), 1 168(w), 1 122(m), 1 077(m), 1 028(m), 990(w), 857(m), 818(w), 790(s), 758(m), 737(m), 681(m), 660(m), 625(w), 597(m), 562(w). ¹H NMR (300 MHz, DMSO-d₆) δ : 9.49 (d), 8.678 (d), 7.994(s), 7.432(d), 6.970(d), 3.928(d), 3.352(s), 2.496(s). Anal.

Calcd.(%): C: 74.37, H: 4.78, N: 11.83. Found(%): C: 74.07, H: 4.86, N: 11.72.

1.2.2 Synthesis of (HL)(CH₃COO) (**1**)

L (0.36 g, 1 mmol) was dissolved in acetic acid (25 mL) and stirred for 10 min to afford a clear yellow solution. The solution was slowly evaporated and pale yellow crystals of **1** (C₂₄H₂₁N₃O₄) were obtained after 2 d. IR (KBr pellet, cm⁻¹): 3 403(m), 3 059(w), 3 009(w), 2 929(w), 2 839(w), 1 715(m), 1 581(s), 1 564(m), 1 546(w), 1 518(s), 1 466(s), 1 438(s), 1 392(s), 1 354(m), 1 322(w), 1 266(s), 1 199(m), 1 168(w), 1 122(m), 1 077(m), 1 028(m), 990(w), 857(m), 818(w), 790(s), 758(m), 737(m), 681(m), 660(m), 625(w), 597(m), 562(w). Anal. Calcd.(%): C: 69.32, H: 5.05, N: 10.11. Found(%): C: 69.73, H: 5.47, N: 9.84.

1.2.3 {Synthesis of [Cu(NO₃)(CH₃OH)L](NO₃) (**2**)

A solution containing 0.018 g (0.05 mmol) of L in 5 mL CHCl₃ and 5 mL methanol solution containing 0.012 g (0.05 mmol) of Cu(NO₃)₂·3H₂O were placed in a tube. After 5 d, blue crystals of **2** (C₂₃H₂₁N₅O₉Cu) were obtained via liquid-liquid diffusion. Yield: 13.6 mg (47.32% based on Cu). IR(KBr pellet, cm⁻¹): 3 429(s), 3 106(w), 3 016(w), 2 971(w), 1 607(s), 1 548(m), 1 524(m), 1 472(s), 1 436(w), 1 389(s), 1 283(m), 1 207(m), 1 160(m), 1 066(m), 1 025(m), 948(w), 854(s), 795(s). Anal. Calcd.(%): C: 48.00, H: 3.65, N: 12.17. Found(%): C: 48.12, H: 3.45, N: 12.56.

1.2.4 Synthesis of [Zn(NO₃)₂L](C₂H₅OH) (**3**)

A mixture of Zn(NO₃)₂·6H₂O (0.029 8 g, 0.1 mmol) and L (0.041 5 g, 0.1 mmol) in 25 mL EtOH was stirred for 3 h to afford a clear yellow solution. The mixture was filtered and kept for slow evaporation to get single crystals of **3** (C₂₄H₂₃N₅O₉Zn). Yield: 25 mg (42.51% based on Zn). IR(KBr pellet, cm⁻¹): 3 446(s), 3 112(w), 3 016(w), 2 929(w), 1 606(s), 1 572(w), 1 549(m), 1 528(m), 1 507(w), 1 475(s), 1 411(w), 1 363(w), 1 294(s), 1 209(m), 1 164(m), 1 136(m), 1 020(m), 954(w), 858(s), 796(s). Anal. Calcd.(%): C: 48.74, H: 3.89, N: 11.85. Found(%): C: 48.65, H: 3.78, N: 11.93.

1.3 X-ray crystallography

Single-crystal X-ray diffraction measurements for the title compounds were carried out on a Bruker Smart 1000 CCD diffractometer equipped with graphite-monochromatized Mo K α radiation with a wavelength of 0.071 07 nm by using the ω -scan technique. Semi-empirical absorption corrections were applied using the SADABS program^[18]. All the structures were solved by a direct method and refined by full-matrix least squares on F^2 using the SHELXS 97 and SHELXL-97 program^[19-20]. All non-hydrogen atoms were refined anisotropically. Only H3 of **2** was found according to the Fourier transformed maps, other H atoms were placed in their theoretical positions. The crystallographic data for **1**~**3** are listed in Table 1. Selected bond lengths (nm) and angles (°) are listed in Table 2.

CCDC: 992747, **1**; 992748, **2**; 992749, **3**.

Table 1 Crystallographic data and structure refinement parameters for compounds **1**~**3**

Compounds	1	2	3
Chemical formula	C ₂₄ H ₂₁ N ₃ O ₄	C ₂₃ H ₂₁ CuN ₅ O ₉	C ₂₄ H ₂₃ ZnN ₅ O ₉
Formula weight	415.44	574.99	590.84
Temperature / K	293(2)	113(2)	293(2)
Wavelengths / nm	0.071 073	0.071 073	0.071 073
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/c$	$P2_1/c$
a / nm	1.249 5(3)	0.713 6(14)	1.064 6(2)
b / nm	0.861 7(17)	4.141 0(8)	2.659 8(5)
c / nm	1.933 9(4)	0.794 4(16)	0.993 6(2)
β / (°)	91.42(3)	93.10(3)	114.63(3)
Volume / nm ³	2.081 5(7)	2.343 9(8)	2.557 6(9)
Z	4	4	4
Calculated density	1.326	1.629	1.534

Continued Table 1

Absorption coefficient / mm ⁻¹	0.092	0.998	1.023
<i>F</i> (000)	872	1 180	1 216
θ range for data collection / (°)	2.59~25.02	2.61~27.87	1.53~25.02
Limiting indices	$-14 \leq h \leq 10$, $-10 \leq k \leq 10$, $-23 \leq l \leq 23$	$-9 \leq h \leq 8$, $-54 \leq k \leq 54$, $-10 \leq l \leq 10$	$-12 \leq h \leq 11$, $-31 \leq k \leq 29$, $-11 \leq l \leq 11$
Reflections collected / unique	15 912 / 3 640 ($R_{\text{int}}=0.044$ 2)	15 582 / 5 366 ($R_{\text{int}}=0.073$ 7)	18 420 / 4 513 ($R_{\text{int}}=0.057$ 4)
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Goodness-of-fit	1.005	1.025	1.057
Data / restraints / parameters	3 640 / 0 / 284	5 366 / 1 / 350	4 513 / 1 / 357
Final <i>R</i> indices ($I > 2\sigma(I)$)	$R_1=0.076$ 8, $wR_2=0.213$ 3	$R_1=0.043$ 1, $wR_2=0.083$ 4	$R_1=0.062$ 1, $wR_2=0.144$ 6
Largest diff. peak and hole / (e·nm ⁻³)	703 and -538	357 and -535	629 and -399

Table 2 Selected bond lengths (nm) and bond angles (°) for compounds 2 and 3

Compound 2					
Cu(1)-O(3)	0.223 6(18)	Cu(1)-O(4)	0.196 5(17)	Cu(1)-N(1)	0.202 0(19)
Cu(1)-N(2)	0.192 6(19)	Cu(1)-N(3)	0.203 2(19)		
O(4)-Cu(1)-N(1)	101.82(7)	O(4)-Cu(1)-N(3)	96.98(7)	O(4)-Cu(1)-O(3)	89.91(7)
N(1)-Cu(1)-O(3)	96.24(7)	N(1)-Cu(1)-N(3)	159.70(8)	N(2)-Cu(1)-N(3)	80.36(8)
N(2)-Cu(1)-O(3)	96.27(7)	N(2)-Cu(1)-O(4)	173.31(7)	N(2)-Cu(1)-N(1)	80.11(8)
N(3)-Cu(1)-O(3)	91.34(7)				
Compound 3					
Zn(1)-O(3)	0.200 4(4)	Zn(1)-O(6)	0.206 7(4)	Zn(1)-N(1)	0.215 9(4)
Zn(1)-N(2)	0.204 7(3)	Zn(1)-N(3)	0.217 8(3)		
O(3)-Zn(1)-O(6)	113.37(15)	O(3)-Zn(1)-N(1)	102.34(14)	O(3)-Zn(1)-N(2)	137.18(15)
O(3)-Zn(1)-N(3)	94.74(13)	O(6)-Zn(1)-N(1)	103.04(14)	O(6)-Zn(1)-N(3)	89.79(13)
N(1)-Zn(1)-N(3)	152.20(13)	N(2)-Zn(1)-N(1)	76.56(13)	N(2)-Zn(1)-N(3)	76.00(12)
N(2)-Zn(1)-O(6)	108.38(14)				

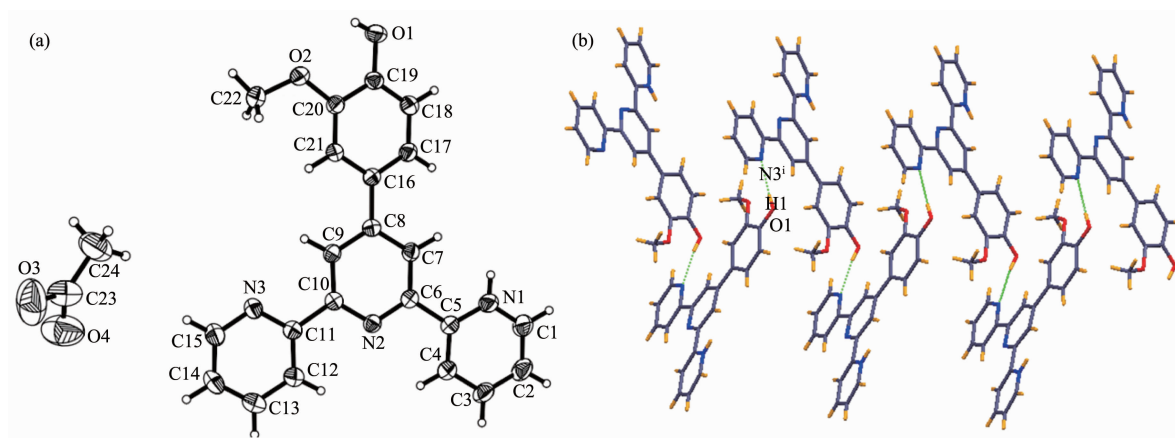
2 Results and discussion

2.1 Structure description of compound 1

Single crystal X-ray diffraction analysis shows that compound **1** is acetate of ligand L. It belongs to monoclinic crystal system and is $P2_1/n$ space group. The L ligand captures a CH₃COOH in a hydrogen-bonded pocket, and one pyridine nitrogen atom (N1) is protonation. In the compound, the pyridine rings reveal a *trans-trans* arrangement to minimize electrostatic interactions between the nitrogen lone pairs. This arrangement is the most stable for terpyridine, which is identical to the literature reported^[21]. In **1**, the three pyridine rings are nearly

coplanar. Two terminal pyridine rings are twisted with dihedral angles 7.3° and 5.5° respect to the central pyridine ring, respectively. The deviation from planarity is obviously due to sterical hindrance. The benzene ring and the central pyridine ring are also twisted with a dihedral angle of 13.3° (Fig.1a).

In the crystal, the protonation terpyridine molecules are linked via hydrogen bonds (O1-H1B⁺⋯N3ⁱ, symmetry code: $i: -x+3/2, y+1/2, -z+1/2$) between the hydroxyl and pyridine nitrogen atoms to give rise to 1D hydrogen-bonded chain along the b-axis (Fig. 1b). These chains are further connected into 3D network by Van der Waals force and π - π stacking interactions between the pyridine rings with the



(a) Displacement ellipsoids are drawn at 30% probability level; (b) Symmetry code: $^i -x+3/2, y+1/2, -z+1/2$

Fig.1 (a) Crystal structure of **1**; (b) 1D hydrogen-bonded chain of **1** along the *b* axis

Table 3 Hydrogen bonds data for compounds **1**, **2** and **3**

D—H...A	<i>d</i> (D—H) / nm	<i>d</i> (H...A) / nm	<i>d</i> (D...A) / nm	∠(DHA) / (°)
Compound 1				
O(1)—H(1B)...N(3) ⁱ	0.082	0.204	0.276 8(3)	148.3
O(1)—H(1B)...O(2)	0.082	0.226	0.268 8(3)	112.6
N(1)—H(1A)...O(4) ⁱⁱ	0.086	0.206	0.272 3(5)	134.0
Compound 2				
O(1)—H(1)...O(8) ⁱ	0.084	0.189	0.272 1(2)	170.5
O(3)—H(3)...O(7) ⁱⁱ	0.084(10)	0.194(12)	0.275 6(3)	165(3)
Compound 3				
O(1)—H(1)...O(9) ⁱ	0.082	0.206	0.279 0(7)	147.9
O(1)—H(1)...O(2)	0.082	0.221	0.265 7(4)	114.8
O(9)—H(9A)...O(3)	0.082	0.219	0.290 6(5)	146.4
O(9)—H(9A)...O(5)	0.082	0.242	0.319 9(7)	158.1

Symmetry codes: ⁱ $-x+3/2, y+1/2, -z+1/2$; ⁱⁱ $x+1, y+1, z$ in **1**; ⁱ $x, -y+1/2, z-1/2$; ⁱⁱ $x+1, y, z$ in **2**; ⁱ $x-1, -y+1/2, z-3/2$ in **3**.

distances of 0.367 1 and 0.380 4 nm. In the compound, the CH_3COO^- takes part in forming hydrogen bonds with protonation nitrogen atoms, which stabilize the structure of **1**. The main hydrogen bonds are listed in Table 3.

2.2 Structure description of complex **2**

Single crystal X-ray diffraction analysis reveals that complex **2** is a monomeric entity. In the asymmetric unit, there is one crystallographically independent Cu(II) ion, one L ligand, one coordinated nitrite ion, one coordinated methanol molecule and one free nitrate ion. The Cu(II) is five-coordinated, three nitrogen atoms from one tridentate chelating L ligand, one oxygen atom from one nitrite ion and another oxygen atom from one methanol molecule. The

Cu—O lengths are 0.196 5(4) and 0.223 6(3) nm, the Cu—N lengths are in the range of 0.192 6(2)~0.203 2(2) nm. Thus, the coordination geometry configuration is a distorted square pyramid (Fig.2).

In complex **2**, the three pyridine rings is *cis-cis* conformation, which is different from **1**. Each L ligand coordinates to Cu (II) with tridentate site. The three pyridine rings are nearly coplanar, two terminal pyridine rings are twisted with dihedral angles 0.8° and 1.0° respect to the central pyridine ring, respectively. The benzene ring and the central pyridine ring are twisted with a dihedral angle of 36.4°. In **2**, the free nitrate ions acting as hydrogen acceptors take part in forming hydrogen bonds (O1—H1...O8ⁱ, O3—H3...O7ⁱⁱ, symmetry code: ⁱ $x, -y+1/2,$

$z-1/2$, $^{\text{ii}} x+1, y, z$) with terpyridine molecules, which link the monomeric units into 1D hydrogen-bonded chain (Fig.3). These chains are further assembled into 3D network by Van der Waals force and π - π stacking interactions between two pyridine rings with the distance of 0.352 1 nm and two benzene rings with the distance of 0.397 5 nm. The detailed hydrogen bonds are summarized in Table 3.

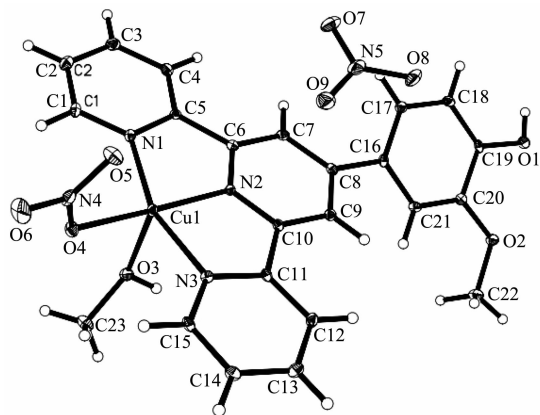
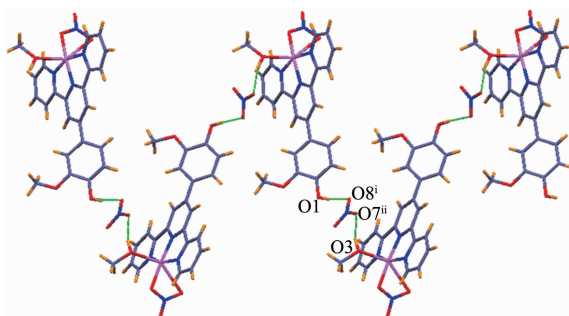


Fig.2 Crystal structure of **2** with 30% probability thermal ellipsoids



Symmetry code: $^i x, -y+1/2, z-1/2$; $^{ii} x+1, y, z$

Fig.3 1D hydrogen-bonded chain of **2** along the c axis

2.3 Structure description of complex **3**

Single crystal X-ray diffraction analysis reveals that complex **3** is also a monomeric entity. It crystallizes in monoclinic crystal system and $P2_1/c$ space group. In the asymmetric unit, there is one crystallographically independent Zn(II) ion, one L ligand, two coordinated nitrate ions and one free ethanol molecules (Fig.4). Zn(II) ion is five-coordinated by three nitrogen atoms from one L ligand, two oxygen atoms from two nitrite ions. The Zn-O lengths are in the range of 0.200 4(4)~0.206 7(4) nm, the Zn-N lengths range from 0.204 7(3) to 0.217 8(3) nm. So,

the Zn(II) ion displays a distorted triangle bipyramidal geometry.

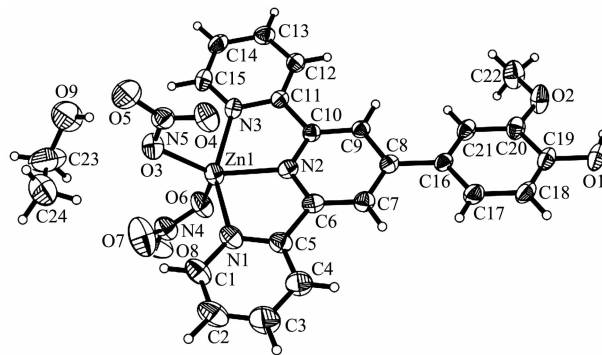
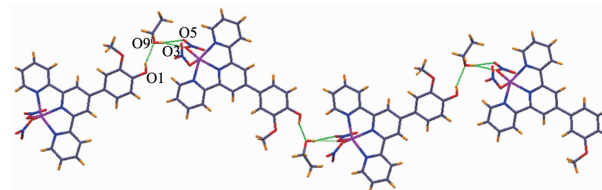


Fig.4 Crystal structure of **3** with 30% probability thermal ellipsoids

In **3**, the three pyridine rings reveal a *cis-cis* arrangement, it is identical to **2**. This arrangement may be related to tridentate chelating mode of L ligand. The three pyridine rings are almost planar with dihedral angles of 2.6° and 2.7° between the central pyridine ring and the adjacent pyridine rings. The benzene ring and the central pyridine ring are also twisted with a dihedral angle of 9.2° . Different from **2**, two nitrate ions are both coordinated. The ethanol molecule is free, which lies in the voids. The free ethanol molecule linked the monomeric Zn(II) units into 1D hydrogen-bonded chain by forming hydrogen bonds ($O1-H1\cdots O9^i$, $O9-H9\cdots O3$ and $O9-H9\cdots O5$, symmetry code: $^i x-1, -y+1/2, z-3/2$) with nitrate ions and terpyridine molecules (Fig.5). Furthermore, these 1D hydrogen-bonded chains are assembled into 3D network by Van der Waals force and π - π stacking interactions. In **3**, there are two kinds of π - π stacking, one is the interaction between two pyridine rings with the distance of 0.374 7 nm, the other is the interaction between the benzene and pyridine with the distance of 0.355 1 nm. The detailed hydrogen bonds are listed in Table 3.



Symmetry code: $^i x-1, -y+1/2, z-3/2$

Fig.5 1D hydrogen-bonded chain of **3** along the c axis

2.4 Thermal properties

The thermal stability of complex **2** was investigated by thermogravimetric analysis. As was observed from the TG curves, the thermal decomposition process of **2** can be described in three steps. The first weight loss of 5.12% occurred between 30 and 188 °C corresponding to the release of coordinated methanol molecule (Calcd. 5.56%). The second step lost the two coordinated and uncoordinated nitrate ions with the mass loss of 22.23% between 188 and 302 °C (Calcd. 21.56%). As temperature rose, no obvious plateau appeared indicating that complex **2** continued to decompose.

3 Conclusions

In summary, we synthesized tpy-based ligand **L** and prepared the complexes of Cu (II) and Zn(II) based on the functional ligand via solution diffusion method. For the title complexes, ligands **L** both adopt tridentate chelating mode to link metal ions into mononuclear units. Compared to the other functional terpyridines, ligand **L** contains a phenol binding site and a methoxy to the periphery of the terpyridine. These functional groups can act as acceptor-donor for hydrogen bonds interactions, which can stabilize the structures of the complexes. Moreover, the different complexes can be obtained by varying the metal ions, mole ratio of the reactants and solvent etc.

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