

以 1-(3-羧苯基)-5-甲基-1 氢-1,2,3-三唑-4-羧酸为配体构筑 的两个配合物的合成、晶体结构及性质

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摘要: 1-(3-羧苯基)-5-甲基-1 氢-1,2,3-三唑-4-羧酸为配体与锰金属盐、锌金属盐在水热合成法得到 2 个配位聚合物: {Mn(cpmtc)₂(H₂O)₂}_n (**1**), {Zn(cpmtc)₃(H₂O)}_n (**2**)。通过单晶衍射仪测得这 2 个晶体结构, 配位聚合物 **1** 为单斜晶系, *P*2₁/*c* 空间群; *a*=1.183 52(19) nm, *b*=1.198 01(19) nm, *c*=0.998 76(16) nm, β =113.201°。配位聚合物 **2** 属于单斜晶系, *P*2₁/*c* 空间群; *a*=1.023 3(3) nm, *b*=1.172 0(4) nm, *c*=1.023 3(3) nm, β =92.8700°。除此之外, 这 2 个配位聚合物通过红外和荧光进行表征。

关键词: 锰(II)配位聚合物; 锌(II)配位聚合物; 晶体结构; 水热合成

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Syntheses, Structures, and Characterization of Two Compounds with 1-(3-Carboxyphenyl)-5-methyl-1*H*-1,2,3-triazole-4-carboxylic Acid

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Abstract: Hydrothermal reaction of Mn(II), Zn(II) ion with 1-(3-carboxyphenyl)-5-methyl-1*H*-1,2,3-triazole-4-carboxylic acid (Hcpmtc) ligand afforded two coordination complexes: {Mn(cpmtc)₂(H₂O)₂}_n (**1**), {Zn(cpmtc)₃(H₂O)}_n (**2**). And the crystal structures were determined via single-crystal X-ray diffraction. Complex **1** crystallizes in monoclinic space group *P*2₁/*c*, *a*=1.183 52(19) nm, *b*=1.198 01(19) nm, *c*=0.998 76(16) nm, β =113.201°, while complex **2** crystallizes in the monoclinic system, space group *P*2₁/*c*, *a*=1.023 3(3) nm, *b*=1.172 0(4) nm, *c*=1.023 3(3) nm, β =92.8700°. In addition, the two compounds are characterized by FT-IR and solid-state fluorescent emission spectroscopy. CCDC: 972330, **1**; 972117, **2**.

Key words: Mn(II) complex; Zn(II) complex; crystal structure; hydrothermal synthesis

0 Introduction

In recent decades, the crystal engineering of metal-organic coordination polymers has attracted enormous interest in synthetic chemistry and material

science, which significantly boosts the understanding of the relationship between molecular structure and material function^[1-4]. Although a variety of coordination polymers which using multidentate ligands such as poly-carboxylates and N-heterocyclic ligand^[5-8] have

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been successfully synthesized with intriguing architectures, topologies^[9] and physical properties^[10-13], rational control in the assembly of these complexes with desired properties still remains a distant prospect in crystal engineering. In particular, it is a great challenge to prepare advanced luminescent materials with predictable structure and properties through the combination of organic ligands with metal ions^[14].

Recently, there is a growing interest in the assembly of metal-organic framework (MOFs) based on triazole, tetrazole and imidazole derivatives^[15-19]. It is well-known that the construction of MOFs is mainly dependent on the combination of several factors, such as the organic ligands, solvents, reaction conditions and metal atoms^[20-23]. 1-(3-carboxyphenyl)-5-methyl-1H-1,2,3-triazole-4-carboxylic acid (Hcpmtc) was chosen for the following reason: the flexible carboxyphenyl and rigid triazolyl group can provide multidentate metal-binding sites and diverse coordinate modes.

In this work, we herein report the synthesis of two novel coordination polymers under hydrothermal conditions: $\{\text{Mn}(\text{cpmtc})_2(\text{H}_2\text{O})_2\}_n$, $\{\text{Zn}(\text{cpmtc})_3(\text{H}_2\text{O})\}_n$, the structures have been determined by single-crystal X-ray diffraction analyses.

1 Experimental

1.1 General procedures

All reagents and solvents were obtained from commercial sources and were used without further purification. Crystal structures were determined with a Xcalibur, Atlas, Gemini ultra diffractometer. IR spectra were obtained with KBr pellets from 4 000 to 400 cm^{-1} using a Nicolet 5700 spectrophotometer. Luminescence spectra for the solid samples were investigated with a Horiba Fluoromax-4 fluorescence spectrophotometer.

1.2 Synthesis of $\{\text{Mn}(\text{cpmtc})_2(\text{H}_2\text{O})_2\}_n$ (1)

$\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ (0.039 5 g, 0.2 mmol) and Hcpmtc (0.049 4 g, 0.2 mmol) were placed in a Teflon-lined stainless container, after the addition of acetonitrile (10 mL) and water (10 mL), then heated to 120 $^\circ\text{C}$, and keep the temperature for 6 d. Then cooled to room temperature at 10 $^\circ\text{C} \cdot \text{h}^{-1}$, colorless block crystal

were obtained in about 39% yield based on MnCl_2 . IR (KBr, cm^{-1}): 3 403(m), 3 033(vs), 1 655(m), 1 618(vs), 1 577(vs), 1 399(vs), 1 381(s), 1 298(vs), 1 242(m), 1 115(m), 819(s), 799(m), 777(s), 748(m), 696(m).

1.3 Synthesis of $\{\text{Zn}(\text{cpmtc})_3(\text{H}_2\text{O})\}_n$ (2)

$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (0.036 6 g, 0.2 mmol) and Hcpmtc (0.049 4 g, 0.2 mmol) were placed in a Teflon-lined stainless container, after the addition of acetonitrile (10 mL) and water (10 mL), then heated to 120 $^\circ\text{C}$, and keep the temperature for 5 d. Then cooled to room temperature at 10 $^\circ\text{C} \cdot \text{h}^{-1}$, colorless block crystal were obtained in about 57% yield based on $\text{Zn}(\text{CH}_3\text{COO})_2$. IR (KBr, cm^{-1}): 3 462(vs), 1 718(vs), 1 638(vs), 1 595(m), 1 466(s), 1 379(vs), 1 320(vs), 1 282(m), 1 251(m), 1 165(m), 1 143(vs), 850(m), 827(s), 759(vs), 692(vs), 644(m).

1.4 X-ray crystal structure determination

The single crystal X-ray diffraction data collection for the complex was performed with a Bruker SMART APEX II diffractometer using $\text{Mo K}\alpha$ radiation (0.071 07 nm). The structure was solved by direct methods with SHELXS-97 and refined by full-matrix least-squares on F^2 using the SHELXL-97 program^[24]. Anisotropic displacement parameters were refined for all nonhydrogen atoms. The hydrogen atoms were added in the riding model. Detailed data collection and refinements of **1**, **2** are summarized in table 1. Selected bond lengths and angles are listed in table 2. Relevant hydrogen bonding parameters of **1**, **2** are summarized in table 3.

CCDC: 972330, **1**; 972117, **2**.

2 Results and discussion

2.1 Structure description of $\{\text{Mn}(\text{cpmtc})_2(\text{H}_2\text{O})_2\}_n$ (1)

The crystal structure shows that complex **1** crystallises in the monoclinic space group $P2_1/c$ and all atoms are situated in general positions. The asymmetric unit comprises two ipmtca, one Mn ion, and two water molecules. The Mn(II) are six coordinated by two water molecules, three carboxylic acid oxygen atoms and one triazole nitrogen atom. Two adjacent Mn are bridged through two oxygen atoms

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

	1	2
Empirical formular	C ₁₁ H ₁₁ MnN ₃ O ₆	C ₁₁ H ₉ ZnN ₃ O ₅
Formula weight	336.17	328.58
Crystal size / mm	0.11×0.15×0.22	0.16×0.19×0.26
Temperature / K	293	293
Crystal system	Monoclinic	Monoclinic
Space group	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> 2 ₁ / <i>c</i>
<i>a</i> / nm	1.183 52(19)	1.023 3(3)
<i>b</i> / nm	1.198 01(19)	1.172 0(4)
<i>c</i> / nm	0.998 76(16)	1.023 3(3)
β / (°)	113.201(2)	92.870 0
Volume / nm ³	1.301 6(8)	1.225 7(7)
<i>Z</i>	4	4
<i>D_c</i> / (g·cm ⁻³)	1.715	1.780
μ / mm ⁻¹	1.05	2.03
θ range / (°)	3.0~27.5	1.74~25.48
Reflections collected	9 413	8 401
Independent reflections	2 509	2 144
Observed reflections	2 193	1 932
<i>F</i> (000)	684	660
Goodness-of-fit on <i>F</i> ²	1.058	1.068
<i>R</i> (all data) ^a	<i>R</i> ₁ =0.036 6; <i>wR</i> ₂ =0.080 0	<i>R</i> ₁ =0.037 0; <i>wR</i> ₂ =0.098 4
<i>R</i> indexes (<i>I</i> >2 σ (<i>I</i>)) ^a	<i>R</i> ₁ =0.028 6; <i>wR</i> ₂ =0.077 6	<i>R</i> ₁ =0.031 8; <i>wR</i> ₂ =0.095 5

Table 2 Selected bond lengths (nm) and angles (°) for the complexes **1** and **2**

1					
Mn2-O4	0.214 05(16)	Mn2-O6	0.213 80(18)	Mn2-N7#2	0.224 22(18)
Mn2-O5	0.224 31(15)	Mn2-O4#2	0.234 32(15)	Mn2-O1#1	0.210 96(18)
O4-Mn2-O5	89.37(6)	O1#1-Mn2-O4#2	105.17	O4B-Mn2-O6	162.63(6)
O4-Mn2-O6	108.66(6)	O1#1-Mn2-N7#2	86.51(6)	O6-Mn2-N7#2	100.92(7)
O1#1-Mn2-O4#2	104.69(6)	O4-Mn2-N7#2	148.28(6)	Mn2-O4-Mn2#2	102.68(6)
O4-Mn2-O4#2	77.32(5)	O5-Mn2-O6	81.45(6)	O4#2-Mn2-N7#2	71.08(6)
O5-Mn2-N7#2	83.84(6)	O1A-Mn2-O5	165.13(6)		
O1#1-Mn2-O6	89.33(7)	O4#2-Mn2-O5	82.34(6)		
2					
Zn1-O1	0.256 8(2)	Zn1-O8	0.204 2(2)	Zn1-N3#3	0.214 7(2)
Zn1-O2	0.202 3(2)	Zn1-O3#3	0.217 8(2)	Zn1-O4#4	0.200 5(2)
O1-Zn1-O2	55.80(7)	O2-Zn1-O8	99.56(8)	O8-Zn1-N3#3	118.16(8)
O1-Zn1-O8	154.73(7)	O2-Zn1-O3#3	85.98(8)	O4#4-Zn1-O8	91.61(9)
O1-Zn1-O3#3	91.19(7)	O2-Zn1-N3#3	133.98(9)	O3#3-Zn1-N3#3	76.04(8)
O1-Zn1-N3#3	82.32(7)	O2-Zn1-O4#4	111.85(8)	O3#3-Zn1-O4#4	161.60(8)
O1-Zn1-O4#4	102.47(8)	O3#3-Zn1-O8	80.53(8)	O4#4-Zn1-N3#3	93.46(8)

Symmetry transformations used to generate equivalent atoms: Compound **1**: #1: $-1+x, y, z$; #2: $-x, 1-y, 1-z$;
Compound **2**: #3: $1+x, y, z$; #4: $1+x, 1/2-y, 1/2+z$

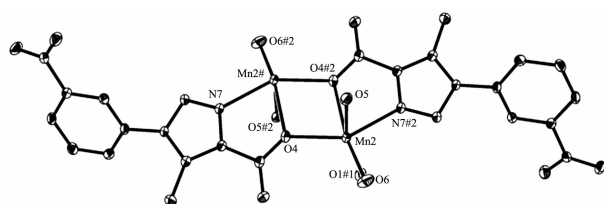
Table 3 Geometrical parameters of hydrogen bonds in 1 and 2

D-H...A	$d(\text{D-H}) / \text{nm}$	$d(\text{H}\cdots\text{A}) / \text{nm}$	$d(\text{D}\cdots\text{A}) / \text{nm}$	$\angle \text{DHA} / (^{\circ})$
1				
O5-H5B...O2#5	0.096 00	0.182 0	0.269 6(2)	150.00
O5-H5C...O3#6	0.096 00	0.188 0	0.281 9(3)	166.00
O6-H6A...O2#7	0.096 00	0.176 0	0.265 1(3)	153.00
O6-H6B...O3#8	0.096 00	0.226 0	0.294 4(2)	127.00
2				
O8-H8...O1#9	0.082 00	0.19 00	0.267 0(3)	158.00
O8-H8W...O1#10	0.089(5)	0.199(5)	0.286 9(3)	172(4)

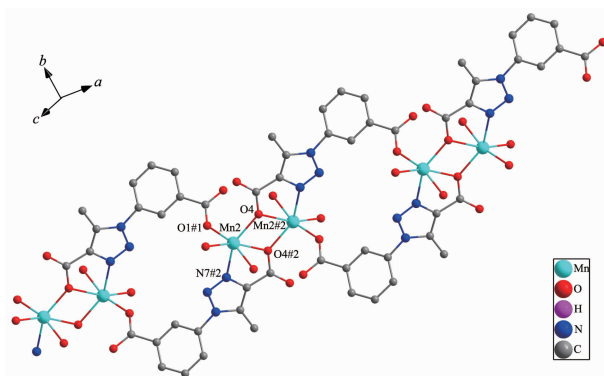
Symmetry transformations used to generate equivalent atoms: Compound **1**: #5: $1-x, 1/2-y, 1-z$; #6: $-x, -1/2+y, 3/2-z$;

#7: $-1+x, 3/2-y, 1/2+z$; #8: $x, 3/2-y, 1/2+z$. Compound **2**: #9: $2-x, 1/2+y, 1/2-z$; #10: $x, 1/2-y, -1/2+z$.

from carboxylic group, forming a parallelogram (Fig.1). The distance of Mn2-O are 0.234 32, 0.214 05 nm, respectively. The angles of parallelogram are 77.32° , 102.68° , respectively. Complex **1** forms an infinite zigzag chain by two different coordinated modes. One oxygen atom of carboxyphenyl group functions as a monoatomic bridge. And the other is functioning as a bridging bidentate ligand by one triazole nitrogen atom and one carboxylic acid oxygen atom between two symmetry Mn2 atoms (Fig.2).



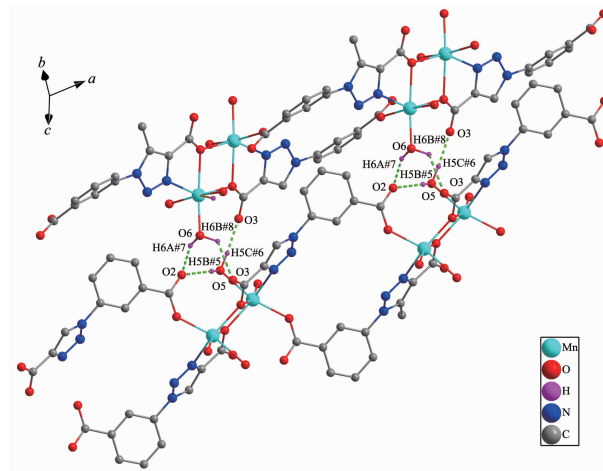
H atoms are omitted for clarity; Displacement ellipsoids are drawn at the 50% probability; Symmetry code: #1: $-1+x, y, z$; #2: $-x, 1-y, 1-z$

Fig.1 Coordination environment of Mn(II) ion


All of H atoms are omitted for clarity; Symmetry code: #1: $-1+x, y, z$; #2: $-x, 1-y, 1-z$

Fig.2 1D coordination network of 1

In the crystal structure of **1**, the two coordinated waters form four hydrogen bonds. One is strong intramolecular hydrogen bond O5-H5B...O2#5 with the H5B to O2 distance of 0.1820 nm. and the other three strongly hydrogen bonding interactions, including O5-H5C...O3#6, O6-H6A...O2#7, O6-H6B...O3#8, are found in the structure as the main action force to form a 2D structure supermolecular network (Fig.3).



Some carbon and hydrogen atoms are omitted for clarity. Symmetry code: #5: $1-x, 1/2-y, 1-z$; #6: $-x, -1/2+y, 3/2-z$; #7: $-1+x, 3/2-y, 1/2+z$; #8: $x, 3/2-y, 1/2+z$

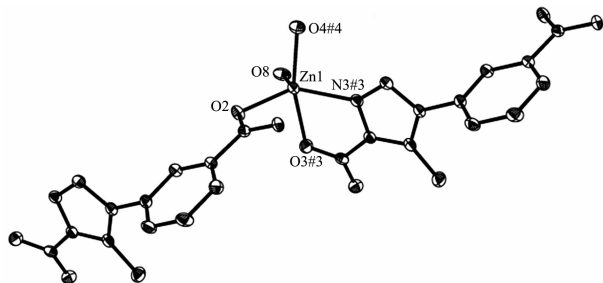
Fig.3 Two-dimensional layer constructed by hydrogen bonds in 1

2.2 Structure description of $\{\text{Zn}(\text{cpmtc})_3(\text{H}_2\text{O})\}_n$ (2)

The asymmetric unit of **2** includes a Zn(II) ion, three cpmtc⁻ and a coordinated water molecule. Zn(II) ion is six-coordinated with four oxygen atoms from the carboxylate group and one nitrogen atoms from the

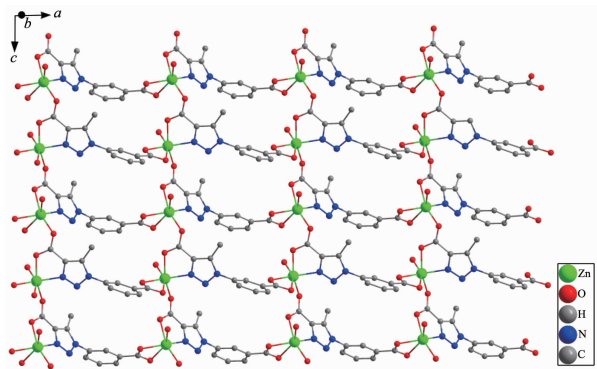
triazole ring, forming a distorted octahedral coordination polyhedron (Fig.4), the angle of O1-Zn1-O8, O2-Zn1-N3#3, O1-Zn1-O3#3, O4#4-Zn1-O8 O1-Zn1-O2 O3#3-Zn1-N3#3 are 154.73° , 133.98° , 91.19° , 91.61° , 55.80° , 76.04° , respectively. And the Zn1-O3#3, Zn1-N3#3 distances are 0.217 8, 0.214 7 nm, respectively. This is similar to the previously reported bond separation of 0.208 6, 0.218 9 nm among the triazole-carboxylate oxygen atom and the 1,2,3-triazole nitrogen atom coordinated to Zn(II)^[25], respectively. In complex **2**, the deprotonated Hcpmtc is bidentate with one oxygen atom of triazole-carboxylate group and one nitrogen atom of 1,2,3-triazole ring chelating to Zn1 atom, and another deprotonated Hcpmtc is bidentate with two oxygen atoms of carboxyphenyl group chelating to the Zn1 atom from the opposite direction, while the other oxygen atom of triazole-carboxylate monodentately to the adjacent Zn1 atom, forming 2D packing network (Fig.5).

In the crystal structure of **2**, several strongly hydrogen-bonding interactions, including O8-H8...



H atoms are omitted for clarity. Displacement ellipsoids are drawn at the 50% probability; Symmetry code: #3: $1+x, y, z$; #4: $1+x, 1/2-y, 1/2+y$

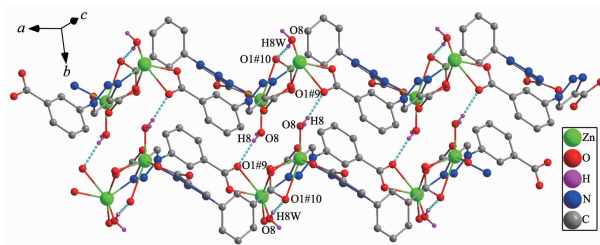
Fig.4 Coordination environment of Zn(II) ion



All of H atoms are omitted for clarity

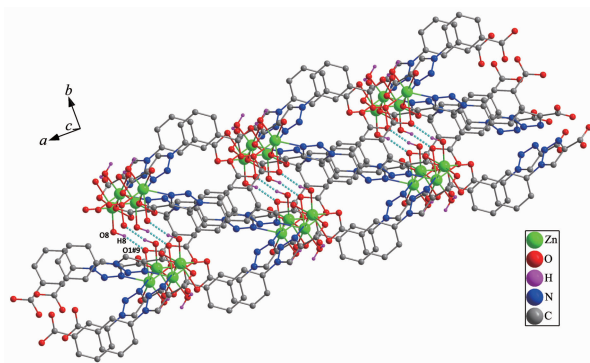
Fig.5 2D coordination network of **1**

O1#9 ($2-x, 1/2+y, 1/2-z$), O8-H8W...O1#10 ($x, 1/2-y, -1/2+z$), are found in the structure as the main action force to form a 3D structure supermolecular network (Fig.6, Fig.7). These layers are further interconnected by π - π interactions of the adjacent phenyl ring, Cg is the centroid of the phenyl rings (C6, C7, C8, C13, C14, C30). The distances of Cg-Cg#3 is 0.361 15 nm (Fig.8).



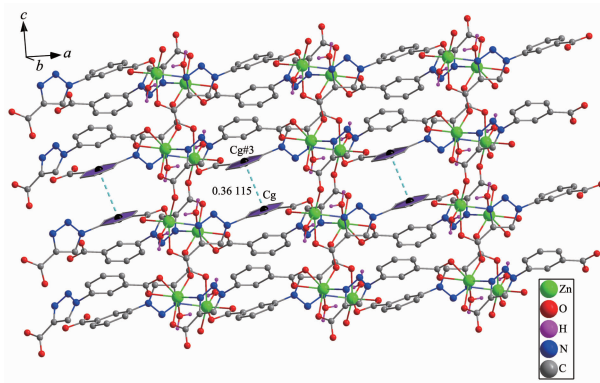
Some H atoms are omitted for clarity; Symmetry code: #9: $2-x, 1/2+y, 1/2-z$; #10: $x, 1/2-y, -1/2+z$

Fig.6 Hydrogen-bonding interactions by a coordinated water



Symmetry code: #9: $2-x, 1/2+y, 1/2-z$

Fig.7 Two neighboring layers interconnected by hydrogen-bonding (O8-H8...O1#9) constructed 3D structure in **1**



Symmetry code: #3: $1+x, y, z$

Fig.8 π ... π interactions between two neighboring layers are further constructed 3D structure

2.3 Luminescent properties

Luminescent properties of complexes with d^{10} metal centers have attracted much interest due to their potential applications in electroluminescent display, chemical sensors, photochemistry^[26-31]. Therefore, the luminescence of **1** and **2** as well as the free Hmptc ligand was investigated in the solid state at room temperature (Fig.9), since two compounds and the free Hmptc ligand are virtually insoluble in most common solvents such as ethanol, acetone, chloroform, benzene, water, etc. At the room temperature, the free Hmptc ligand exhibits a narrowly emission with maximum at 359 nm upon excitation at 280 nm in the solid state, On comparison with the free ligand, the emission bands for **1** and **2** are similar to those of the ligand, compound **1** shows emission peaks at 349 nm (λ_{ex} =290 nm) and compound **2** shows emission peaks at 349 nm (λ_{ex} =295 nm), the emission Strength of **1** is weaker to the emission of compound **2**. The varying coordination modes of Hmptc have great potential for synthesizing novel frameworks with intriguing structure and unique properties.

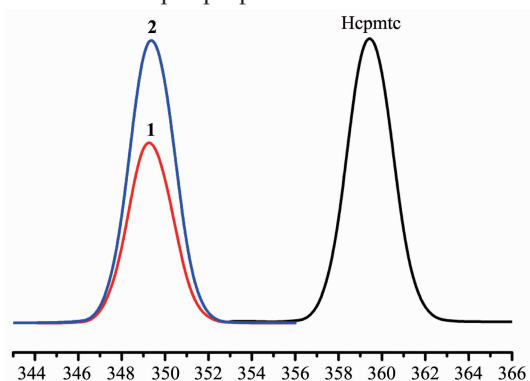


Fig.9 Solid-state fluorescence spectra of **1**, **2** and the free Hmptc ligand at room temperature

3 Conclusions

In conclusion, this study demonstrates the synthesis of two complexes under hydrothermal reaction. The complexes prove that the triazole-carboxylate group and the carboxyphenyl group are valuable multidentate chelating ligands. Although more and more complexes containing triazole ligands have been found in the recent literature^[32-35], in which most contain 1,2,4-triazole derivatives, the complexes

with 1-(3-carboxyphenyl)-5-methyl-1H-1,2,3-triazole-4-carboxylic acid ligand are firstly reported.

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