第 30 卷第 6 期
2014 年 6 月

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CHINESE JOURNAL OF INORGANIC CHEMISTRY

Vol.30 No.6
1451-1458

5-异烟酰胺异钛酸构筑的 Cu(Ⅰ)-Eu 和 Ni(Ⅱ)-Eu 化合物的合成、晶体结构和性质研究

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摘要: 利用不同过渡金属盐在溶剂热中合成了 2 个杂核化合物{[$CuEu_2(INAIP)_3(HCOO)(H_2O)_3]\cdot 3H_2O$}$_n$(**1**)和{[$NiEu_2(INAIP)_4(H_2O)_4]\cdot 4H_2O$}$_n$(**2**)(INAIP=异烟酰胺吡啶基异酞酸根),并对其进行了元素分析、IR 及 X-射线衍射法表征。晶体结构研究表明:配合物 **1** 和 **2** 都属于三斜晶系,$P\bar{1}$ 空间群。晶胞参数:配合物 **1** a=1.088 7(3) nm, b=1.515 8 (4) nm, c=1.564 4 (2) nm, V=2.333 2 (10) nm^3, Z=2, D_c=1.955 g·cm^{-3}, μ=3.203 mm^{-1}, F(000)=1352, R_{int}=0.027, R_1=0.0505, wR_2=0.1309。配合物 **2** a=1.013 4(3) nm, b=1.083 6 (6) nm, c=1.374 1 (2) nm, V=1.453 0 (9) nm^3, Z=1, D_c=1.878 g·cm^{-3}, μ=2.554 mm^{-1}, F(000)=818, R_{int}=0.045 8, R_1=0.034 1, wR_2=0.086 9。配合物 **1** 是由配体异烟酰胺吡啶基异酞酸连接而成的三维二重贯穿 **sqc27** 拓扑结构, 而配合物 **2** 是由配体异烟酰胺吡啶基异酞酸连接稀土铕离子形成二维层状结构,该二维层通过 Ni-O 和 Ni-N 键连接成三维非贯穿网络结构。配合物 **1** 和 **2** 具有典型的稀土铕离子红色荧光和高的热稳定性。

关键词: 3*d*-4*f* 配合物; 晶体结构; 荧光性质; 稳定性

中图分类号: O641.4 **文献标识码**: A **文章编号**: 1001-4861(2014)06-1451-08

DOI: 10.11862/CJIC.2014.192

Syntheses, Crystal Structures and Properties of Cu(Ⅰ)-Eu and Ni(Ⅱ)-Eu Coordination Polymers Constructed from 5-(Isonicotinamido)isophthalic Acid

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Abstract: Two 3*d*-4*f* heteronuclear coordination polymers {[$CuEu_2(INAIP)_3(HCOO)(H_2O)_3]\cdot 3H_2O$}$_n$ (**1**) {[$NiEu_2(INAIP)_4(H_2O)_4]\cdot 4H_2O$}$_n$(**2**) were obtained by solvothermal assembly of $CuCl/NiSO_4\cdot 7H_2O$ and $Eu(NO_3)_3\cdot 6H_2O$ with the H_2INAIP (5-(isonicotinamido)isophthalic acid) ligand. Both complex **1** and **2** crystallize in triclinic, space group $P\bar{1}$. For **1**: a=1.088 7(3) nm, b=1.515 8 (4) nm, c=1.564 4 (2) nm, V=2.333 2 (10) nm^3, Z=2, D_c=1.955 g·cm^{-3}, μ=3.203 mm^{-1}, F(000)=1 352, R_{int}=0.027, R_1=0.050 5, wR_2=0.130 9. **2**: a=1.013 4(3) nm, b=1.083 6 (6) nm, c=1.374 1 (2) nm, V=1.453 0 (9) nm^3, Z=1, D_c=1.878 g·cm^{-3}, μ=2.554 mm^{-1}, F(000)=818, R_{int}=0.045 8, R_1=0.034 1, wR_2=0.086 9 for **2**. Single-crystal X-ray diffraction analysis revealed that each $INAIP^{2-}$ ligand uses its two carboxyl groups to connect two or three Eu(Ⅲ) ions into 2D lanthanide bi-layer structure. Then the 2D layers are further connected by pyridyl groups to give a three-dimensional two-fold interpenetrated pillared-layer structure in **1** with **sqc27** topology. While in complex **2**, the Ni(Ⅱ) bonds to the pyridyl group (N) and carboxylate group (O) linking the 2D lanthanide layer into a 3D framework. In addition, the luminescent properties of complexes **1** and **2** have been investigated, which showed the obviously red photoluminescence in them. CCDC:943863, **1**; 966618, **2**.

Key words: 3*d*-4*f* complex; crystal structure; luminescent property; thermal stabity

收稿日期:2013-10-15。收修改稿日期:2014-01-25。

湖南省自然科学基金(No.13JJ6069),湖南省教育厅创新平台开放基金(No.11K009)和功能金属有机材料湖南省普通高等学校重点实验室(No.13K06)和湖南省重点学科基金资助项目。

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0 Introduction

The design and synthesis of higher-dimensional heterometallic metal-organic frameworks (HMOFs) have been studied for many years, owing to their intriguing architectures and topologies and potential applications in catalysts, luminescent probes, magnetic materials and so on [1-7]. As we know, many multi-carboxylate or heterocylic carboxylic acids have been used for satisfied the purpose. Furthermore, 3- and 4-pyridinecarboxylic and pyridinedicarboxylate anions have recently been found to act as excellent building blocks with multi-connecting ability in the construction of heterometllic coordination polymers with photoluminescence or magnetic properties [8-11]. To the best knowledge, 5-(isonicotinamido)isophthalic acid (H_2INAIP) can show richer coordination modes due to its two carboxylate groups and one pyridyl group. Therefore, it is an excellent candidate for the construction of metal-organic frameworks [12-15]. In this paper, we present the use of 5-(isonicotinamido) isophthalic acid as a multifunctional bridging ligand which possesses of oxygen and nitrogen donors as a potential linker between the lanthanide and transition metal centers. Herein, we report on the syntheses, crystal structures and luminescence properties of a new 3*d*-4*f* heterometallic coordination polymer through solovthermal reaction, $[CuEu_2(INAIP)_3(HCOO)(H_2O)_3]\cdot 3H_2O$ (**1**) and $\{[NiEu_2(INAIP)_4(H_2O)_4]\cdot 4H_2O\}_n$ (**2**).

1 Experimental

1.1 Materials and instruments

The regents were used as commercial sources without further purification. Elemental analyses were performed on a Perkin-Elmer 240C elemental analyzer. The IR spectra were recorded on Bruker Vector22 FTIR spectrophotometer using KBr discs. Thermogravimetric analyses were performed on a simultaneous SDT 2960 thermal analyzer under nitrogen with a heating rate of 10 ℃·min^{-1}. Powder X-ray diffraction (PXRD) patterns were measured on a Shimadzu XRD-6000 X-ray diffractometer with Cu $K\alpha$(λ=0.154 18 nm) radiation at room temperature. The luminescent spectra for the solid powdered samples were recorded at room temperature on an Aminco Bowman Series **2** spectrophotometer with xenon arc lamp as the light source. In the measurements of the emission and excitation spectra, the pass width was 4.0 nm. All the measurements were carried out under the same conditions.

1.2 Synthesis of the complexes 1 and 2

Complex **1** was synthesized by solvothermal method in a 16 mL Teflon-lined autoclave by heating a mixture of $Eu(NO_3)\cdot 6H_2O$ (0.05 mmol, 22.5 mg), CuCl (0.05 mmol, 5.2 mg), H_2INAIP (0.1 mmol, 28.7 mg), NaOH (0.15 mmol, 6.0 mg), DMF (4mL) and ethanol (4 mL) at 150 ℃ for 3 d under autogenous pressure. Cooling the reactor subsequently to room temperature at a rate of 10 ℃·h^{-1}, yellow crystals of **1** were obtained. IR (cm^{-1}): 3 423(s), 2 936 (w), 1 659 (m), 1 548(s), 1 526 (s), 1 445(s), 1 380(s), 1 237(m), 1 109(m), 886(s), 783 (s), 600 (w). Anal. Calcd. for $C_{43}H_{37}CuEu_2N_6O_{23}$ (%): C, 37.58, H, 2.69, N, 6.12. Found (%): C, 37.53, H, 2.73, N, 6.10. Complex **2** was synthesized by similar method with $NiSO_4\cdot 7H_2O$ (28.2 mg, 0.1 mmol), instead of CuCl (0.05 mmol, 5.2 mg). Block green single crystals of 2 were collected by filtration and washed by water and ethanol for several times with a yield of 34% (based on H_2INAIP). Anal. Calcd. for $C_{56}H_{48}NiEu_2N_8O_{28}$ (%): C 40.88; H 2.92; N 6.81; Found (%): C 40.94; H 2.86; N 6.85%. IR (KBr pellet, cm^{-1}): 3 410 (s), 1 665 (m), 1 619 (m), 1 548 (s), 1 439 (m), 1 378 (s), 1 287 (m), 889 (w), 785 (w), 726 (m), 618(m), 596 (w).

1.3 X-ray crystallography

The X-ray diffraction measurement for **1** and **2** were performed on the Bruker Smart Apex-II CCD diffractometer with graphite-monochromated Mo $K\alpha$ radiation (λ=0.071 073 nm) at room temperature. The data were integrated by using the SAINT program [16], which also did the intensity corrections for Lorentz and polarization effect. An empirical absorption correction was applied using the SADABS program [17]. The structures were solved by direct methods using the program SHELXS-97 and all the non-hydrogen atoms were refined anisotropically on F^2 by the full-matrix least-squares technique using the SHELXL-97 crystallographic software package[18-19]. Crystal data and

structure refinement parameters are listed in Table 1. The selected bond lengths and bond angles are given in Table 2.

CCDC:943863, **1**; 966618, **2**.

Table 1 Crystal data and structure parameters for complex 1 and 2

Complexes	**1**	**2**
Empirical formula	$C_{43}H_{37}CuEu_2N_6O_{23}$	$C_{56}H_{48}NiEu_2N_8O_{28}$
Formula weight	1 373.25	1 643.65
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
a / nm	1.088 7(3)	1.013 4(3)
b / nm	1.515 8(4)	1.083 6(6)
c / nm	1.564 4(2)	1.374 1(2)
V / nm^3	2.333 2(10)	1.453(9)
Z	2	1
Absorption coefficient / mm^{-1}	3.203	2.554
F(000)	1 352	1 764
Reflections collected / unique	11 721/8 084 (R_{int}=0.027 0)	7 297/5 040 (R_{int}=0.045 8)
Data / restraints / parameters	8 084/0/676	5 040/0/430
Final R indices ($I>2\sigma(I)$)	R_1=0.050 5, wR_2=0.130 9	R_1=0.034 1, wR_2=0.086 9
Largest diff. peak and hole / (e·nm^{-3})	1 664 and −1 122	1 661 and −1 180

Table 2 Selected bond lengths (nm) for complexes 1 and 2

			1		
Cu(1)-N(3)	0.191 9(7)	Cu(1)-N(1E)	0.189 3(8)	Eu(1)-O(11)	0.248 2(7)
Eu(1)-O(10)	0.243 1(7)	Eu(1))-O(2W)	0.241 7(6)	Eu(1)-O(3W)	0.235 2(7)
Eu(1)-O(15)	0.242 0(6)	Eu(1)-O(16)	0.245 4(6)	Eu(1)-O(17A)	0.227 8(7)
Eu(1)-O(18B)	0.230 5(7)	Eu(2)-O(2)	0.228 2(7)	Eu(2)-O(6)	0.227 8(6)
Eu(2)-O(8)	0.252 6(6)	Eu(2)-O(9)	0.243 7(7)	Eu(2)-O(3C)	0.233 2(5)
Eu(2)-O(1W)	0.2445(7)	Eu(2)-O(4D)	0.258 1(6)	Eu(2)-O(5D)	0.235 9(6)
			2		
Ni(1)-O(1W)	0.217 4(4)	Ni(1)-O(1WA)	0.217 4(4)	Ni(1)-O(1A)	0.209 1(3)
Ni(1)-N(4)	0.215 0(4)	Ni(1)-O(1)	0.209 1(3)	Ni(1)-N(4A)	0.215 0(4)
Eu(1)-O(2W)	0.233 3(4)	Eu(1)-O(2)	0.223 4(4)	Eu(1)-O(9D)	0.238 7(3)
Eu(1)-O(3B)	0.242 7(4)	Eu(1)-O(6C)	0.246 4(4)	Eu(1)-O(8D)	0.248 1(3)
Eu(1)-O(7C)	0.236 6(3)	Eu(1)-O(4B)	0.241 4(4)		

Symmetry code for **1**: A: 1+x, y, z, B: −x, 2−y, 1−z, C: 1−x, 1−y, 2−z, D: −1+x, y, z, E: 1−x, −y, 1−z. For **2**: A: 2−x, 1−y, 1−z, B: x, −1+y, z, C: −1+x, y, 1+z, D: x, y, 1+z

2 Results and discussion

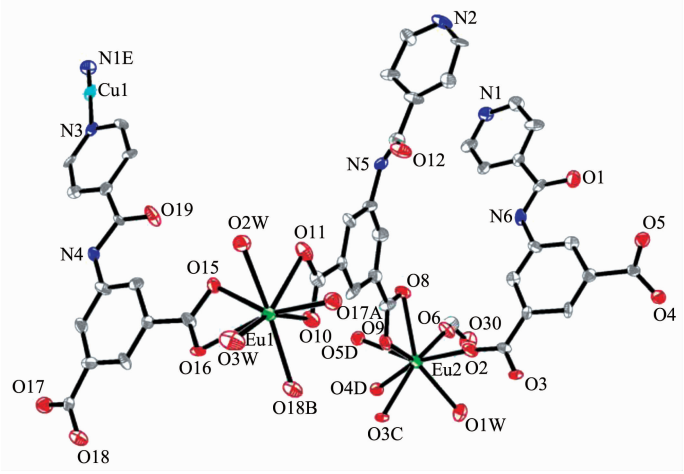
2.1 Structure description

The results of the structure analysis revealed that complex **1** crystallizes in the triclinic space group $P\bar{1}$ and exhibits a novel 3D heterometallic coordination framework. The formate anion is derived from the hydrolyzed *N*,*N*-dimethylformamide (DMF) molecules. The asymmetric unit contains one Cu (I), two Eu (Ⅲ), three ligands, one formate anion, three coordinated and three free water molecule as shown in Fig.1. Both Eu(Ⅲ) atoms are eight-coordinated in distorted square antiprismatic arrangement with different coordinated environment. Eu (1) is eight coordinated with six

carboxylate groups O atoms (O10, O11, O15, O16, O17A and O18B) from three different $INAIP^{2-}$ ligands and two O atoms (O2W, O3W) from two water molecules. However, Eu (2) is eight coordinated with seven carboxylate groups O atoms (O2, O8, O9, O3C, O4D, O5D and O6) from three different $INAIP^{2-}$ ligands and one formate, the other O atom is (O1W) from one water molecule. The coordination bond lengths1around the Eu(Ⅲ) atom are in the range of 0.227 8(6)~0.258 1(6) nm, as listed in Table 2. If the weak coordination interactions between the carboxylate group and Cu(I) are omitted, the Cu center has a distorted linear coordination environment made up of two N atoms from two bridging $INAIP^{2-}$ ligands, which the average Cu-N bond lengths are of 0.190 6(7) nm. It is note that the unique $INAIP^{2-}$ ligands exhibit two coordination modes, namely one ligand coordinate to three Eu(Ⅲ) atoms using its two carboxylate groups in μ_2-η^1:η^1-bismonodentate and μ_1-η^1:η^1-chelate modes and one Cu(I) atom through its pyridyl nitrogen atom. However, the other ligand only coordinate to two Eu (Ⅲ) atoms using its two carboxylate groups in the μ_1-η^1:η^1-chelate mode with free coordination of the pyridyl group, which is different from the complexes [LnAg (INAIP)$_2$]·3H$_2$O[15]. If the coordination interactions between the Cu-N and Cu-O are neglected, the neighboring binuclear subunits are connected via the $INAIP^{2-}$ ligands to form a two-dimensional (2D) layer network lying in the ac plane. Such 2D nets are further connected together via the Cu-N coordination interactions between the pyridyl group and Cu (I) to generate a 3D pillared framework as illustrated in Fig.2. It is clear that there is a large 1D channel along the *a*-axis, in order to minimize the hollow cavities and stabilize the framework, the potential voids formed via a single 3D framework show combination with another identical one, giving a two-fold interpenetrated structure of **1**.

To better identify the nature of the 3D framework of **1**, suitable connectors can be defined by using a topological approach. As described above, each Eu_2 is surrounded by six adjacent $INAIP^{2-}$ ligands, so each Eu_2 subunit is a six-connected node. Likely, since it links two Eu_2 subunits and one Cu(I) atom, the $INAIP^{2-}$ ligand can be considered as a three-connecting node. Consequently, according to the calculation of TOPOS[20], the final framework of **1** belongs to a binodal (3, 6)-connected **sqc27** type of topology net, with Schläfli symbol of $(4\cdot6^2)_2(4^4\cdot6^{10}\cdot8^3)$ (Fig.3).

When $NiSO_4\cdot7H_2O$ was used, instead of CuCl, to react with ligand H_2INAIP, and the complex **2** was isolated. As illustrated in Fig.4, the asymmetric unit of **2** consists of one Eu (Ⅲ) atom, half Ni (Ⅱ) atom, two $INAIP^{2-}$ ligands, two coordinated (Fig.4) and two lattice water molecules. The Ni (Ⅱ) cation is located on an inversion center and is coordinated by two pyridine N atoms, two carboxylate O atoms and two water



All hydrogen atoms and free water molecules are omitted for clarity; Symmetry code: A: 1+*x*, *y*, *z*, B: −*x*, 2−*y*, 1−*z*, C: 1−*x*, 1−*y*, 2−*z*, D: −1+*x*, *y*, *z*, E: 1−*x*, −*y*, 1−*z*

Fig.1 Coordination enviroment of Cu(I) and Tb(Ⅲ) in complex **1** with 30% thermal ellipsoids

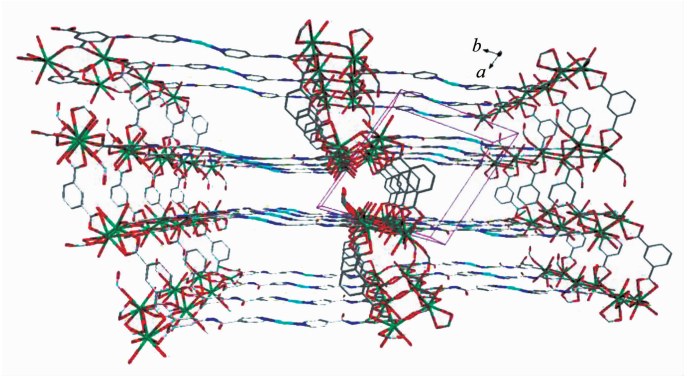


Fig.2 3D pillared-layer framework of **1** viewed along the a-axis without uncoordinated pyridyl groups and free water molecules

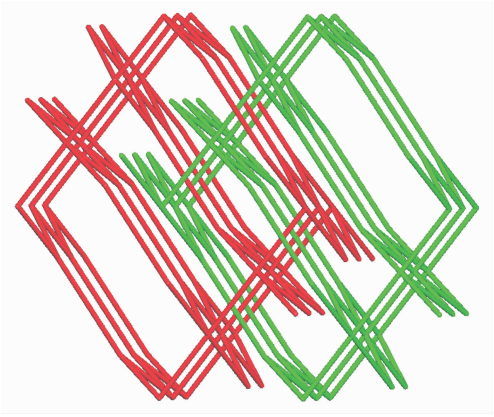
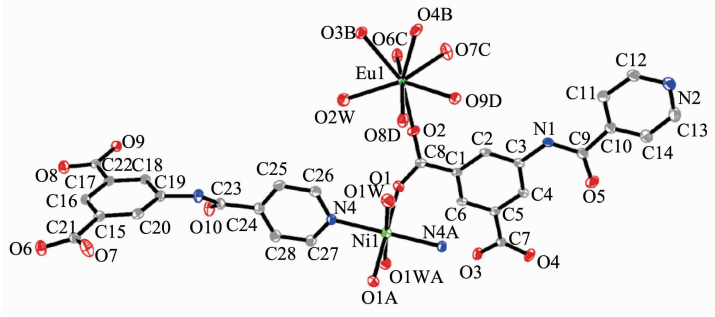


Fig.3 Toplogically representation of the two-fold interpenetrated **sqc27** structure of **1**



All hydrogen atoms and free water molecules are omitted for clarity; Symmetry code: A: 2–*x*, 1–*y*, 1–*z*, B: *x*, –1+*y*, *z*, C: –1+*x*, *y*, 1+*z*, D: *x*, *y*, 1+*z*

Fig.4 Molecular structure of complex **2**

molecules in a distorted octahedral geometry. Each Eu(Ⅲ) ion is eight-coordinated by seven carboxylate oxygen atoms from four different $INAIP^{2-}$ ligands and one from a coordinated water molecule. The Eu center can be described as having a distorted square antiprismatic coordination geometry with Eu-O bond distances ranging from 0.223 4 (4) to 0.248 1 (4) nm, which is in agreement to those observed in other Ln-INAIP complexes [16]. In the structure of **2**, the two $INAIP^{2-}$ ligands have two different kinds of coordination modes. One coordinated to two Eu(Ⅲ) and one Ni(Ⅱ) atoms using its two carboxylate groups with μ_1-η^1:η^1-chelate and μ_2-η^1:η^1 bis-monodentate coordination modes while the pyridyl group is free of coordination, and the other one coordinated to two Eu(Ⅲ) through the carboxylate groups with μ_1-η^1:η^1-chelate coordination

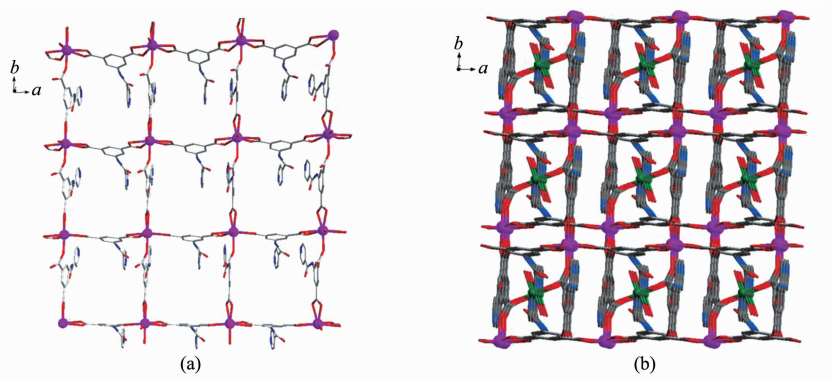


Fig.5 (a) 2D layer structure linked by $INAIP^{2-}$ ligands and Eu(Ⅲ) centersand (b) projection along *c* axis showing the three-dimensional structure of **2**

mode and one Ni(Ⅱ) via the pyridyl group. Firstly, one-dimensional (1D) chain is formed by the connections between the μ_1-η^1:η^1-chelate carboxylate groups and Eu(Ⅲ) atoms neglecting the coordination of pyridyl group along b-axis. Then the 1D chains are further joined together by another μ_2-η^1:η^1 bis-monodentate carboxylate groups coordination modes to result in the formation of the (4,4) 2D Eu(Ⅲ)-carboxylate network as shown in Fig.5a, omitting the coordination of the Ni-N and Ni-O. Finally, the 2D networks are further linked together by Ni-N and Ni-O coordination interactions to form a non-interpenetrated 3D framework (Fig.5b), which is entirely different from the two-fold interpenetrated **1**. The different structures of complexes **1** and **2** showed that the reaction material plays crucial role in the formation of the frameworks **1** and **2**.

2.2 IR and photoluminescence property

The infrared spectra of the title complexes have been recorded and some important assignments are shown above. No strong IR band from -COOH appeared at nearly 1 700 cm^{-1}, indicating that the H_2INAIP ligands are entirely protonated in it, and peaks at 3 423 cm^{-1} (3 410 cm^{-1}) could be assigned to characteristic peaks of the ν(O-H) absorptions of water molecules. These IR results are coincident with the crystallographic structural analyses.

Due to the excellent luminescent properties of Eu(Ⅲ) ions, the room temperature photoluminescence in the solid state of **1** and **2** were investigated. Red luminescence (Fig.6) appearing upon excitation at 360 nm has emissions at 576 (577), 591 (592), 615 (618), 649 (650), and 701 (702) nm attributed to the characteristic transitions $^5D_0 \rightarrow {}^7F_0$, $^5D_0 \rightarrow {}^7F_1$, $^5D_0 \rightarrow {}^7F_2$, $^5D_0 \rightarrow {}^7F_3$, and $^5D_0 \rightarrow {}^7F_4$ of the Eu(Ⅲ) ion [21-22]. Moreover, $^5D_0 \rightarrow {}^7F_2$ is an electric dipole transition and is extremely sensitive to chemical bonds in the vicinity of the Eu(Ⅲ) ion. The intensity ratio $I(^5D_0 \rightarrow {}^7F_2)/I(^5D_0 \rightarrow {}^7F_1)$ is equal to ca. 3.8 ~5.2, which suggests a non-centrosymmetric coordination environment of the Eu(Ⅲ) ions [23-24]. The most intense transition is $^5D_0 \rightarrow {}^7F_2$ at 615 (618) nm, implying the intense red luminescence of **1** and **2**.

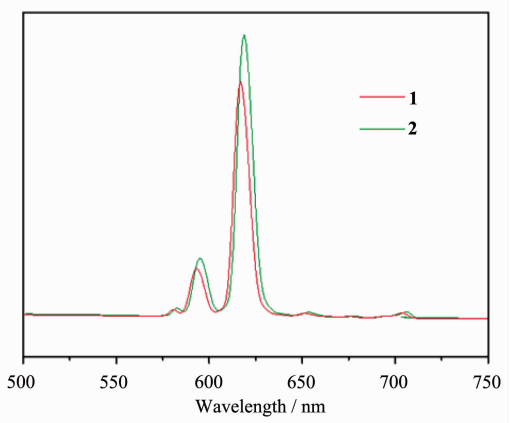


Fig.6 Solid-state emission spectrum of the title complexes **1** and **2**

2.3 Thermogravimetric analyses and PXRD patterns

The results of TGA indicate that the two complexes have high stability. Therefore, complex **1** is taken as an example to describe its TGA and PXRD here. The thermogravimetric analysis (TGA) of complex **1** reveals

that there are three stages of weight loss in the temperature range from 25~700 ℃ (Fig.7). The first stage, occurring between 25 and 105 ℃, is attributed to the loss of three free water molecules per formula (Observed weight loss, 3.91%; Calcd. 3.93%). The second stage, occurring from 95 to 160 ℃, is attributed to the loss of three coordinated water molecules per formula (Observed weight loss, 3.94%; Calcd. 3.93%). After the loss of all the water molecules, the supramolecular framework is stable up to 320 ℃, followed by another weight loss at high temperature. This means that complex **1** is stable up to the high temperature of 320 ℃. We have also carried out variable-temperature XRPD for checking the stability. The diffraction pattern of **1** after heating at 160 ℃ is similar to that of the as-synthesized complex, which indicates that the host framework remains intact after removal of the guest and coordinated water molecules, as shown in Fig.4. But after the complex as heated at 350 ℃, the main diffraction peaks were absent in the pattern, in good agreement with the TG curve of **1**.

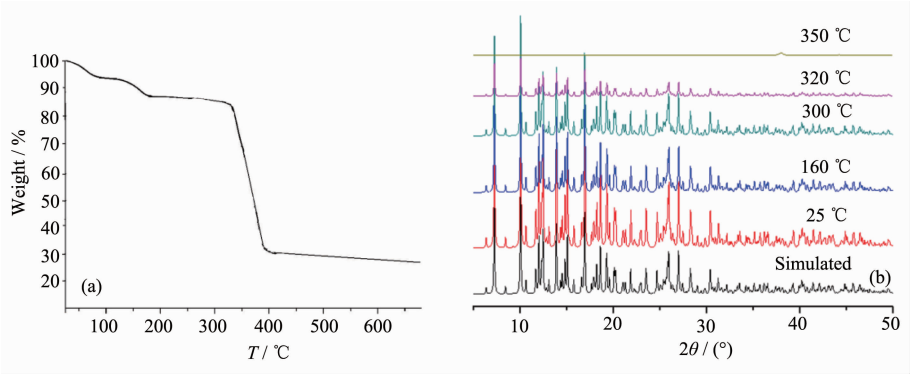


Fig.7 (a) TGA curve of complex **1** and (b) temperature dependent powder X-ray diffraction patterns of complex **1**: simulated; as-synthesized at 25 ℃; 160 ℃; 300 ℃; 320 ℃; 350 ℃

3 Conclusion

Two new 3*d*-4*f* coordination polymers with varied structures were successfully synthesized by solvothermal reactions of different transition metal salts with carboxamide containing ligand 5-(isonicotinamido) isophthalic acid. Complex **1** is two-fold interpenetrated 3D framework with **sqc27** type of topology, while **2** is a complicated 3D non-interpenetrated network. The results revealed that the transition metal ion and solvent media play important roles in determining the structure and topology of coordination polyners.

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