

两个由 2-(4'-氯-苯甲酰基)苯甲酸和双咪唑基配体 构筑的锰、镉配合物的合成及晶体结构

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摘要: 通过水热法合成了 2 个配位聚合物 $[\text{Mn}(\text{cbba})_2(\text{bimb})]_n$ (**1**) 和 $[\text{Cd}(\text{cbba})_2(\text{mbix})]_{2n}$ (**2**) (Hcbba =2-(4'-氯-苯甲酰基)苯甲酸, bimb =1,4-双(咪唑基-1-基)丁烷, mbix =1,3-双(咪唑基-1-基)苯)。并对其进行了元素分析、红外光谱、热重和 X-射线单晶衍射测定。这两个配合物通过氢键或 π - π 相互作用形成了三维超分子网状结构。此外, 还研究了配合物 **1** 的紫外光谱和配合物 **2** 的荧光性质。

关键词: 水热合成; 晶体结构; 锰配合物; 镉配合物

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Synthesis and Crystal Structure of Two Complexes of Manganese, Cadmium Assembled by 2-(4'-Chlorine-benzoyl)-benzoic Acid and Bis(imidazol) Ligands

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Abstract: Two coordination polymers $[\text{Mn}(\text{cbba})_2(\text{bimb})]_n$ (**1**) and $[\text{Cd}(\text{cbba})_2(\text{mbix})]_{2n}$ (**2**) (Hcbba =2-(4'-chlorine-benzoyl)-benzoic acid, bimb =1,4-bis(imidazol-1-yl)-butane, mbix =1,3-bis(imidazol-1-ylmethyl)-benzene) have been hydrothermally synthesized and structurally characterized by elemental analysis, IR spectrum, TG and single-crystal X-ray diffraction. They are further extended into a three-dimensional supramolecular network structure through hydrogen-bonding or π - π interactions. Moreover, the UV spectrum of complex **1** and luminescent property of complex **2** have been investigated in the solid state. CCDC: 936604, **1**; 928554, **2**.

Key words: hydrothermal synthesis; crystal structure; manganese complex; cadmium complex

0 Introduction

Recently, there has been much interest in the construction of metal-organic frameworks (MOFs) due

to their versatile structures and interesting topologies^[1-4] as well as their potential applications as functional materials in the fields of molecular magnetism, catalysis, gas sorption and optoelectronic devices^[5-9].

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The most useful building blocks for constructing organic-inorganic hybrid MOFs are carboxylate and N-donor ligands. Aromatic carboxylate ligands have been extensively employed in the construction of various dimensional MOFs because of their abundant coordination modes and high structural stability for functional materials applications^[10-16].

Imidazole and its derivatives are typically heterocyclic ligands with nitrogen as the donor atom. It exhibits a wide variety of pharmacological activities as antihelmintics, anticancer, antifungal and anti-inflammatory agent^[17]. Because of this, the coordination chemistry of related ligands has been the subject of numerous investigations. As a good candidate, the conformational flexible ligand 1,4-bis(imidazol-1-yl)butane and 1,3-bis(imidazol-1-ylmethyl)benzene have been used as auxiliary ligand because of the free rotation of the two imidazole planes, which result in *cis* or *trans* conformations as required by the metal coordination geometry in the assembly process^[18-21].

To explore the combination effects of this neutral di-imidazole ligand (bimb, mbix) and anionic squarate ligands, we synthesize coordination polymers of Mn(II), Cd(II) containing these ligands. Herein, we report the synthesis and characterization of two coordination polymers, $[\text{Mn}(\text{cbba})_2(\text{bimb})]_n$ (**1**) and $[\text{Cd}(\text{cbba})_2(\text{mbix})]_{2n}$ (**2**) under hydrothermal conditions.

1 Experimental

1.1 General procedures

All materials were commercially purchased and used without further purification. Infrared spectra (KBr pellets) were taken on a Perkin-Elmer 2400LS II spectrometer and elemental analyses for C, H and N were performed on a Perkin-Elmer 240C analyzer. The TG studies were performed on a Perkin-Elmer TGA7 analyzer. The fluorescent studies were carried out on a computer-controlled JY Fluoro-Max-3 spectrometer at room temperature. The UV spectrum was obtained on a Shimzu UV-250 spectrometer in the 200~400 nm range.

1.2 Synthesis

$[\text{Mn}(\text{cbba})_2(\text{bimb})]_n$ (**1**). A mixture of $\text{Mn}(\text{OAc})_2 \cdot$

$4\text{H}_2\text{O}$ (0.2 mmol, 0.05 g), Hcbba (0.2 mmol, 0.05 g), bimb (0.2 mmol, 0.038 g) and H_2O (18 mL) was sealed in a 30 mL Teflon-lined autoclave under autogenous pressure at 140 °C for seven days. After cooling to room temperature, pale yellow block crystals were collected by filtration and washed with distilled water in 38% yield (based on Mn). Anal. Calcd.(%) for $\text{C}_{38}\text{H}_{30}\text{Cl}_2\text{MnN}_4\text{O}_6$: C, 56.70; H, 3.96; N, 7.33. Found(%): C, 56.43; H, 3.66; N, 7.05. IR (KBr, cm^{-1}): 3 448w, 3 121w, 2 929w, 1 677m, 1 637m, 1 589m, 1 572w, 1 519w, 1 482w, 1 446w, 1 400s, 1 309w, 1 288w, 1 274w, 1 250w, 1 230w, 1 166w, 1 150w, 1 116w, 1 092s, 1 037w, 1 016m, 933s, 891w, 890w, 817w, 775s, 748s, 714m, 744m, 679w, 654w, 624w, 566m, 533w, 478w, 456w.

$[\text{Cd}(\text{cbba})_2(\text{mbix})]_{2n}$ (**2**). A mixture of $\text{Cd}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$ (0.2 mmol, 0.053 g), Hcbba (0.4 mmol, 0.10 g), mbix (0.2 mmol, 0.048 g) and H_2O (18 mL) was sealed in a 30 mL Teflon-lined autoclave under autogenous pressure at 140 °C for seven days. After cooling to room temperature, colorless block crystals were collected by filtration and washed with distilled water in 47% yield (based on Cd). Anal. Calcd.(%) for $\text{C}_{84}\text{H}_{60}\text{Cd}_2\text{Cl}_4\text{N}_8\text{O}_{12}$: C, 57.98; H, 3.48; N, 6.44. Found(%): C, 57.71; H, 3.26; N, 6.28. IR (KBr, cm^{-1}): 3 454w, 3 129w, 1 663m, 1 584m, 1 557w, 1 483w, 1 443w, 1 397s, 1 288s, 1 248w, 1 236w, 1 174w, 1 148w, 1 112w, 1 089s, 1 034w, 1 012m, 932s, 888w, 841m, 826w, 814w, 798w, 769w, 744m, 717w, 698w, 671w, 655m, 632w, 553w, 525w, 477w, 435w.

1.3 Structure determination

Single crystal diffraction data of **1** and **2** were respectively collected on a Bruker SMART APEX-CCD diffractometer equipped with a graphite-monochromatic $\text{Mo } K\alpha$ ($\lambda=0.071\ 073\ \text{nm}$) radiation using an ω scan mode at 292(2) K. The correction for Lp factors was applied. The structure was solved by direct methods with SHELXS-97 program^[22] and refined by full-matrix least-squares techniques on F^2 with SHELXL-97^[23]. All non-hydrogen atoms were refined anisotropically and the hydrogen atoms of organic ligands were generated geometrically. The selected bond parameters are given in Table 1.

Crystal data for **1**: $\text{C}_{38}\text{H}_{30}\text{Cl}_2\text{MnN}_4\text{O}_6$, monoclinic,

Table 1 Selected bond lengths (nm) and bond angles ($^{\circ}$) for complexes **1** and **2**

1					
Mn(1)-O(1)	0.230 5(2)	Mn(1)-O(2)	0.239 19(19)	Mn(1)-O(4)	0.212 42(17)
Mn(1)-O(5A)	0.216 88(19)	Mn(1)-N(1)	0.223 2(2)	Mn(1)-N(3)	0.223 3(2)
O(1)-Mn(1)-O(5A)	109.19(7)	O(4)-Mn(1)-N(1)	91.82(8)	O(5A)-Mn(1)-N(1)	93.41(7)
O(4)-Mn(1)-N(3)	90.66(7)	O(5A)-Mn(1)-N(3)	85.46(7)	N(1)-Mn(1)-N(3)	177.50(7)
O(4)-Mn(1)-O(1)	92.21(7)	O(5A)-Mn(1)-O(1)	158.29(7)	N(1)-Mn(1)-O(1)	89.45(7)
N(3)-Mn(1)-O(1)	90.79(7)	O(4)-Mn(1)-O(2)	147.95(7)	O(5A)-Mn(1)-O(2)	102.39(7)
N(1)-Mn(1)-O(2)	91.60(8)	N(3)-Mn(1)-O(2)	86.46(8)	O(1)-Mn(1)-O(2)	55.98(7)
2					
Cd(1)-O(1)	0.230 1(2)	Cd(1)-O(2)	0.243 1(2)	Cd(1)-O(4)	0.248 8(2)
Cd(1)-O(5)	0.225 9(2)	Cd(1)-N(1)	0.224 3(2)	Cd(1)-N(4A)	0.226 9(2)
N(1)-Cd(1)-O(5)	103.58(8)	N(1)-Cd(1)-N(4A)	88.83(8)	O(5)-Cd(1)-N(4A)	109.56(8)
N(1)-Cd(1)-O(1)	109.95(8)	O(5)-Cd(1)-O(1)	138.64(8)	N(4A)-Cd(1)-O(1)	94.89(8)
N(1)-Cd(1)-O(2)	88.61(8)	O(5)-Cd(1)-O(2)	103.55(8)	N(4A)-Cd(1)-O(2)	146.45(8)
O(1)-Cd(1)-O(2)	55.02(8)	N(1)-Cd(1)-O(4)	149.92(9)	O(5)-Cd(1)-O(1)	54.53(7)
N(4A)-Cd(1)-O(4)	81.43(8)	O(1)-Cd(1)-O(4)	99.24(8)	O(2)-Cd(1)-O(4)	114.87(8)

Symmetry transformations used to generate the equivalent atoms: **1**: A: 2-x, -y, 2-z; **2**: A: 1-x, -y, 1-z

space group $P2_1/c$, Formula weight 765.50, $a=1.536\ 5(7)$, $b=1.140\ 4\ (5)$, $c=2.034\ 6\ (9)$ nm, $\beta=94.763\ (8)^{\circ}$, $V=3.553(3)$ nm³, $Z=4$, $F(000)=1\ 572$, $\mu(\text{Mo } K\alpha)=0.575$ mm⁻¹, $D_c=1.429$ g·cm⁻³, 18 831 reflections measured, 6 918 unique ($R_{\text{int}}=0.041\ 6$), 4 542 observed reflections with $I>2\sigma(I)$, $R=0.041\ 2$, $wR=0.085\ 2$, $S=1.012$.

Crystal data for **2**: C₈₄H₆₀Cd₂Cl₄N₈O₁₂, triclinic, space group $P\bar{1}$, Formula weight 1 740.00, $a=1.176\ 87(13)$, $b=1.284\ 29(15)$, $c=1.410\ 35(16)$ nm, $\alpha=98.108(2)$, $\beta=111.702(2)$, $\gamma=96.849(2)^{\circ}$, $V=1.926\ 4(4)$ nm³, $Z=1$, $F(000)=880$, $\mu(\text{Mo } K\alpha)=0.759$ mm⁻¹, $D_c=1.500$ g·cm⁻³, 10 670 reflections measured, 7 552 unique ($R_{\text{int}}=0.011\ 1$), 6 529 observed reflections with $I>2\sigma(I)$, $R=0.033\ 8$, $wR=0.086\ 4$, $S=1.042$.

CCDC: 936604, **1**; 928554, **2**.

2 Results and discussion

2.1 IR spectrum

Complex **1**: The COO⁻ is coordinated with its asymmetric and symmetric stretching appearing at 1 589 cm⁻¹ ($\nu(\text{OCO})_{\text{asym}}$) and 1 400 cm⁻¹ ($\nu(\text{OCO})_{\text{sym}}$)^[24], respectively. The $\Delta\nu(\nu(\text{OCO})_{\text{asym}}-\nu(\text{OCO})_{\text{sym}})$ is 189 cm⁻¹ (<200), showing the presence of bidentate linkage of carboxylates in the dianions. Thus the carboxylates

coordinate to the metal as bidentate ligands via the carboxylate groups^[25].

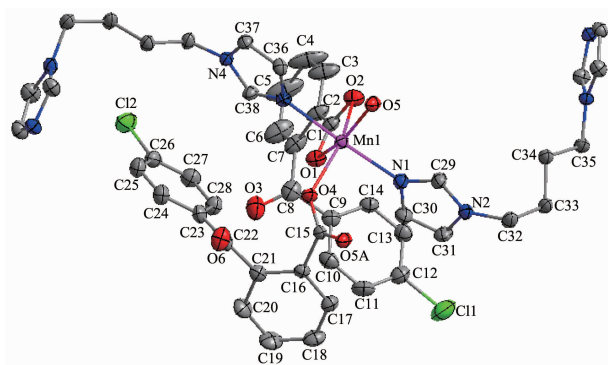
Complex **2**: The COO⁻ is coordinated with its asymmetric and symmetric stretching appearing at 1 584 cm⁻¹ ($\nu(\text{OCO})_{\text{asym}}$) and 1 397 cm⁻¹ ($\nu(\text{OCO})_{\text{sym}}$)^[24], respectively. The $\Delta\nu(\nu(\text{OCO})_{\text{asym}}-\nu(\text{OCO})_{\text{sym}})$ is 187 cm⁻¹ (<200), showing the presence of bidentate linkages of carboxylates in the dianions. Thus, the carboxylates also coordinate to the metal as bidentate ligands via the carboxylate groups^[25].

In addition, X-ray diffraction analysis further indicates the bidentate coordination manners of carboxylate groups in **1** and **2**.

2.2 Description of the structure

Complex **1**: A single-crystal X-ray diffraction study reveals that complex **1** crystallizes in the monoclinic space group $P2_1/c$ and displays a 2D network framework. As shown in Fig.1, the asymmetric unit of complex **1** consists of one Mn(II) atom, one bimb ligand and two cbba ligands. The Mn(II) atom is hexa-coordinated with four oxygen atoms (O(1), O(2), O(4A), O(5)) from three different cbba ligands and two nitrogen donors (N(1) and N(3)) from two different bimb molecules, forming a distorted octahedral geometry.

The bond distances of Mn-O in compound **1** fall in the 0.212 42(17)~0.239 19(19) nm range, and Mn-N bond length fall in the 0.223 2(2)~0.223 3(2) nm, which are in the normal range and the coordination angles around Mn atom are in the range 55.98(7)°~177.50(7)°. In the coordination environment, the four carboxylate oxygen atoms (O(1), O(2), O(4A), O(5)) are located in the basal plane, whereas two imidazole nitrogen atoms (N(1), N(3)) occupy the axial positions from the opposite direction. In the crystal structure of complex **1**, the completely deprotonated cbba ligands display two kinds of coordination modes, namely bidentate bridging mode and bidentate chelating mode, and linked Mn(II) ions to form dinuclear subunits. The bimb ligands adopts *cis*-conformation bridging modes with a dihedral angle between the two imidazole rings of 0° to link two metal Mn(II) atoms (Fig.2), and this lead to the formation of an infinite $\{Mn_2(bimb)_2\}_\infty$ double chains along the *a* axis. The Mn...Mn separation through the bimb bridges are 1.042 7 and



Hydrogen atoms are omitted for clarity; Symmetry code: A: $2-x, -y, 2-z$

Fig.1 Coordination environment (at 30% probability level) of the Mn(II) center of **1**

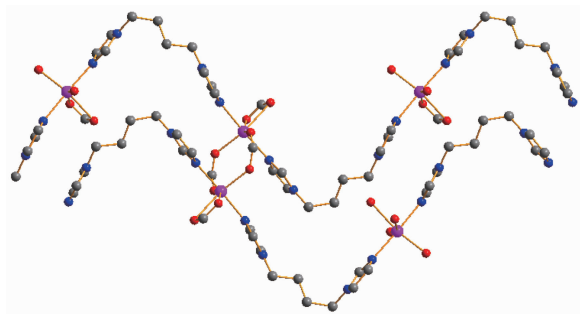
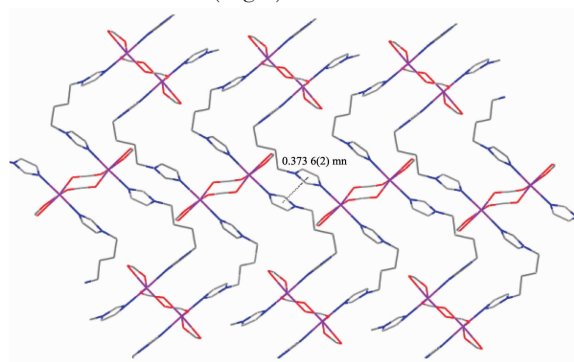


Fig.2 View of the infinite $\{Mn_2(bimb)_2\}_\infty$ double chains along the *a* axis in **1**

1.366 0 nm, the neighboring 1D $\{Mn_2(bimb)_2\}_\infty$ infinite chains are linked via bimb ligands to develop into 2D network framework (Fig.3).

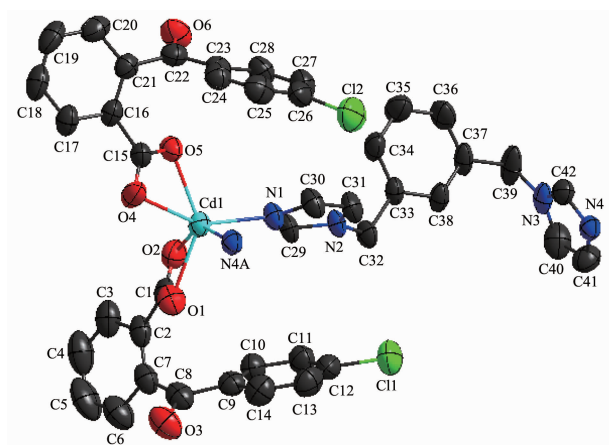


Hydrogen atoms and part of the carbon atoms of cbba ligands are omitted for clarity

Fig.3 View of the 2D network structure of the complex **1**

Moreover, there are π - π interactions in complex **1** between imidazole rings of bimb ligands. The centroid-to-centroid distance between adjacent ring is 0.373 6(2) nm for N3C36C37N4C38 and N3'C36'C37'N4'C38' ($2-x, 1-y, 2-z$) imidazole rings. The perpendicular distance is 0.343 55(10) nm for N3C36C37N4C38 and N3'C36'C37'N4'C38' ($2-x, 1-y, 2-z$) imidazole rings. These supramolecular interactions together with the coordinate-covalent interactions between metal ions and organic ligands strengthen the stability of the network structure.

Complex 2: A single crystal X-ray diffraction study reveals that complex **2** crystallizes in triclinic system with space group $P\bar{1}$ and the asymmetric unit contains one Cd(II) atom, one mbix ligand and two cbba ligands, as shown in Fig.4. Cd(II) is six coordinated with distorted octahedral coordination geometry defined by two nitrogen donors (N(1), N(4A)) from two different mbix ligands and four carboxylate oxygen atoms (O(1), O(2), O(4), O(5)) from two different cbba ligands. Three carboxylate oxygen (O(1), O(2), O(5)) atoms, one nitrogen (N(4A)) atoms define an equatorial plane, while the axial coordination sites are occupied by another nitrogen (N(1)) atom and carboxylate oxygen (O(4)) atom. The bond distances of Cd-O in complex **2** fall in the 0.225 9(2)~0.248 8(2) nm range, and Cd-N bond length is 0.224 3(2)~0.226 9(2) nm. The coordination angles around Cd atom are in the range



Hydrogen atoms are omitted for clarity; Symmetry code: A: $1-x$, $-y$, $1-z$

Fig.4 Coordination environment (at 30% probability level) of the Cd(II) center of **2**

$54.53(7)^\circ \sim 149.92(9)^\circ$.

The completely deprotonated cbba ligands display one kind of coordination mode, namely bidentate bridging mode and the mbix ligand adopts *trans*-conformation bridging mode with a dihedral angle between the two imidazole rings of 62.92° . As a result, Cd(II) atoms are linked by mbix ligands to form binuclear subunits with thirty numbered ring. From the packing diagram, we can see that the cbba and mbix ligands are also linked by means of C—H \cdots O (Table 2) hydrogen-bonding interactions which are known to be important in the synthesis of supramolecular architectures^[26]. Moreover, there are π - π interactions in complex **2**, namely, within the same direction between pyridine rings. The centroid-to-centroid distances between adjacent aromatic rings is 0.404 0(2) nm for C23C24C25C26C27C28 and C23'C24'C25'C26'C27'C28' ($2-x$, $1-y$, $1-z$) aromatic rings. The perpendicular distances is 0.369 78(15) nm for C23C24C25C26C27C28 and C23'C24'C25'C26'C27'C28' ($2-x$, $1-y$, $1-z$) aromatic rings. Therefore, through hydrogen bonds and π - π interactions, the binuclear subunits

are further extended into a three-dimensional supramolecular framework (Fig.5).

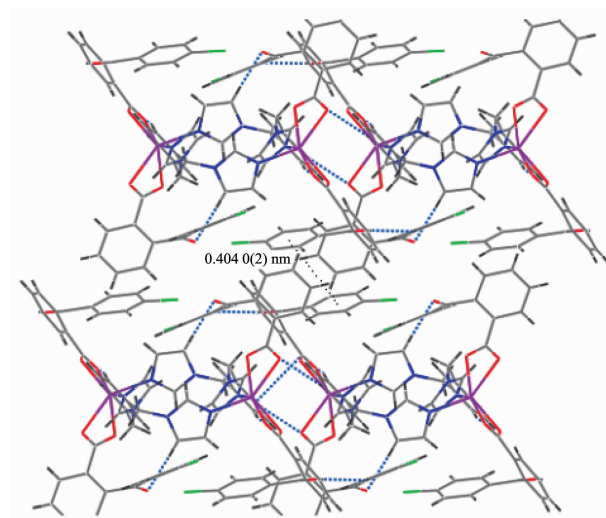


Fig.5 View of the 3D supramolecular architecture of **2** formed by hydrogen-bonding and π - π interactions

2.3 Thermal analysis

TG curve of **1** (Fig.6) show that the first weight loss of 24.7% from 280 to 365 $^\circ\text{C}$ corresponds to the removal of bimb ligands (Calcd. 24.9%). Upon further heating, an obvious weight loss (64.4%) occurs in the temperature range of 365~901 $^\circ\text{C}$, corresponding to the release of cbba ligands (Calcd. 67.9%). Above

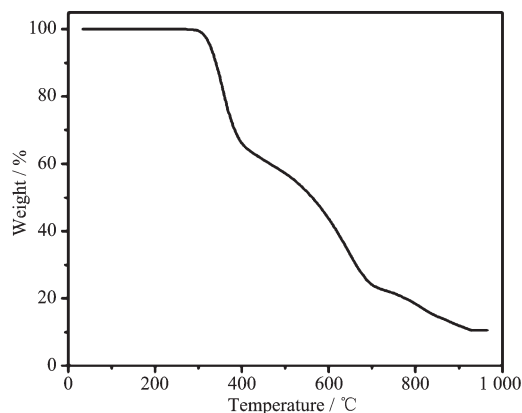


Fig.6 TG curve of the complex **1**

Table 2 Hydrogen bonds for compound **2**

D—H \cdots A	$d(\text{D—H})$ / nm	$d(\text{H}\cdots\text{A})$ / nm	$d(\text{D}\cdots\text{A})$ / nm	$\angle \text{DHA}$ / ($^\circ$)
C(11)—H(11A) \cdots O(6)	0.093	0.252	0.318 0(5)	127
C(34)—H(34A) \cdots O(2)	0.093	0.250	0.326 2(5)	139
C(36)—H(36A) \cdots O(4)	0.093	0.249	0.35 7(3)	155
C(40)—H(40A) \cdots O(3)	0.093	0.232	0.322 0(5)	161

901 °C no weight loss is observed, which means the complete decomposition of **1**. The residual should be MnO.

TG curve of **2** (Fig.7) show that the first weight loss of 57.1% from 156 to 523 °C corresponds to the removal of cbba ligands (Calcd. 59.7%). Upon further heating, an obvious weight loss (32.6%) occurs in the temperature range of 523~671 °C, corresponding to the release of mbix ligand (Calcd. 27.4%). Above 671 °C no weight loss is observed, which means the complete decomposition of **2**. The residual should be CdO.

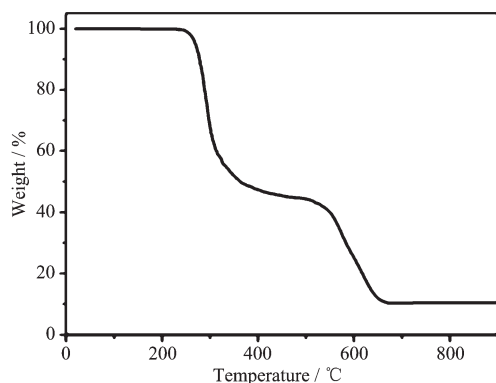


Fig.7 TG curve of the complex **2**

2.4 UV Spectrum Analysis

The UV spectra for the compound **1**, Hcbba and bimb ligands have been investigated in the solution state. For bimb ligand, there is no absorption band, Hcbba ligand has one absorption band at about 270 nm, while the compound **1** has one absorption band at about 231 nm (Fig.8), which should be assigned to the $n \rightarrow \pi^*$ transition of Hcbba. However, after Hcbba coordinating to the Mn^{2+} ion, the absorption intensity

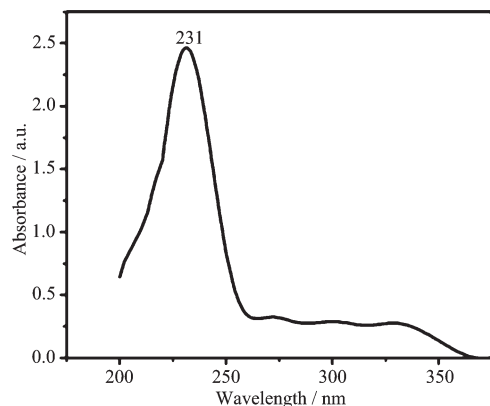


Fig.8 UV spectrum of the complex **1**

decreases, showing that they are affected by the metal coordination.

2.5 Luminescence property

The solid-state photoluminescence spectra of **2**, free Hcbba and mbix ligands were investigated at room temperature. Excited by 325 nm, coordination polymer **2** gives wide green emission with the maximum peak at 547 nm (Fig.9). The main emission peak of ligand mbix is at 440 nm. However, no obvious emission bands are observed for the free Hcbba ligand in the range of 400~800 nm under the same experimental conditions. The significant phenomenon of the fluorescent emission of **2** here could be tentatively assigned to the ligand-to-metal charge transfer (LMCT)^[28]. For possesses strong fluorescent intensity, it appears to be good candidates for novel hybrid inorganic-organic photoactive materials.

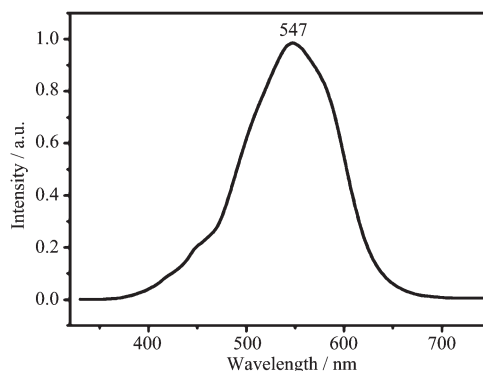


Fig.9 Solid-state emission spectrum of **2** at room temperature

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