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pH Dependent Synthesis of Two Manganese Compounds based on 5-(2-Pyrimidyl)tetrazole-1-acetic Acid

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Keywords: pH value; Manganese; 5-(2-Pyrimidyl)tetrazole-1-acetic acid; Crystal structure; Luminescence

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Abstract. Reactions of Hpymtza [Hpymtza = 5-(2-pyrimidyl)tetrazole-1-acetic acid] with $MnCl_2{\cdot}4H_2O$ under different pH conditions, afforded the complexes $[Mn(pymtza)_2(H_2O)_4]$ (**1**) and $[Mn_2(pymtza)_2Cl_2(EtOH)]{\cdot}H_2O$ (**2**). The compounds were structurally characterized by elemental analysis, IR spectroscopy and single-crystal X-ray diffraction. Compound **1** shows a mononuclear structure, whereas complex **2** has a 1D chain structure. In compound **1**, the pymtza ligand only acts in a monodentate manner to coordinate to one central Mn^{II} atom by one carboxylate atom, In **2**, pymtza acts as tetradentate ligand to connect three Mn^{II} ions. Compounds **1** and **2** display 3D networks by hydrogen bonding interactions. Furthermore, the luminescence properties of Hpymtza as well as compounds **1** and **2** were investigated at room temperature in the solid state.

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1 Introduction

Over the past decade, coordination compounds based on tetrazole-carboxylate ligands have attracted intense attention from chemists, due to their fascinating structural topologies and potential applications as functional materials in the fields of chemistry.[1–9] Numerous tetrazole-carboxylate ligands such as tetrazolate-5-acetic acid, tetrazolate-5-formic acid, tetrazole-1-acetic acid, 5-(2-pyridyl) tetrazole-2-acetic acid have already been studied, and a number of their complexes have been reported.[10–13] The selection of these ligand arises from several considerations as follows: Tetrazole-carboxylate ligands includes both nitrogen atoms and carboxylate oxygen atoms are very likely to display a variety of coordination modes; the abundant nitrogen and oxygen atoms can participate in hydrogen bonds, stabilizing the supramolecular assembly. However, it would still be a challenge for a long time to design and construct coordination compounds with desired topologies and properties, because many factors can affect the formation of the targeted molecules, such as ligand-to-metal ratio, solvent system, pH value, and temperature etc.[14–16] As is well known, the pH value of the reaction system can affect not only the coordination ability of the multicarboxylate ligand but also the related connection modes, therefore it is particularly important in the formation of the resulting structures.[17–19]

Taking all the observations into consideration, in this work, 5-(2-pyrimidyl)tetrazole-1-acetic acid was chosen to react with $MnCl_2{\cdot}4H_2O$. Notably, [Mn(pyrtza)Cl], which is derived from 5-(2-pyrimidyl)tetrazole-2-acetic aicd (Hpyrtza, Scheme 1), the isomer of 5-(2-pyrimidyl)tetrazole-1-acetic aicd (Hpymtza, Scheme 1), has been reported earlier.[4] The different position of the carboxylate group of the tetrazole ring is likely to have a great impact on the coordination modes of such ligands, thus leading to structure variation. By reaction of Hpymtza and $MnCl_2{\cdot}4H_2O$ at low pH value, $[Mn(pymtza)_2(H_2O)_4]$ (**1**) was constructed under solvothermal conditions, whereas at a higher pH value, $[Mn_2(pymtza)_2Cl_2(EtOH)]{\cdot}H_2O$ (**2**) was obtained. Herein, the structures and the luminescence properties of these compounds are discussed in detail.

Scheme 1. Structures of Hpymtza and Hpyrtza.

2 Experimental Section

2.1 Materials and Instrumentation

General chemicals were commercially available reagents of analytical grade and used without further purification. IR spectra were recorded with a Thermo NICOLET-380 instrument as KBr disks (4000–400 cm^{-1}). Photoluminescent analyses were performed with an F-4600 fluorescence spectrometer. Single crystal X-ray diffraction was carried out

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with a Rigaku SCXmini-CCD diffractometer. The ^{1}H NMR spectroscopic measurement was performed with a Bruker DPX300 instrument. The elemental analysis was performed with a PE-2400 instrument.

2.2 Synthesis of Hpymtza

5-(2-Pyrimidyl)tetrazole (Hpymtz) was prepared by a [2+3] cycloaddition reaction, involving a treatment of 2-cyanopyrimidyl with NaN_3 in toluene in the presence of triethylammonium chloride. A mixture of chloroacetic acid (4.73 g, 0.05 mol), Hpymtz (7.40 g, 0.05 mol), and potassium hydroxide (5.61 g, 0.10 mol) in ethanol (100 mL) were refluxed for 24 h at 90 °C. After cooling to room temperature, the precipitate was filtered off, washed with 2 × 30 mL of ethanol, and dissolved in distilled water (30 mL). The pH of the solution was adjusted to 2 with concentrated HCl (12 mol·L^{-1}), giving 5-(2-pyrimidyl)tetrazole-1-acetic acid (Hpymtza) as a powder. Yield: 51 % (based on Hpymtz). $C_7H_6N_6O_2$: calcd. C 40.8; H 2.9; N 40.8 %; found: C 40.5; H 3.0; N 40.9 %. **IR** (KBr): $\tilde{\nu}$ = 3494 (w), 1634 (s), 1581 (s), 1533 (m), 1395 (s), 1352 (m), 1277 (w), 1043 (s), 1031 (w), 1010 (m), 994 (m), 825 (m) cm^{-1}. **^{1}H NMR** (400 MHz, in D_2O): δ = 11.96 (1H), 7.66(1H), 8.95(2H), 5.20 (2H) ppm.

2.3 Synthesis of [Mn(pymtza)$_2$(H$_2$O)$_4$] (1)

Hpymtza (0.0412 g, 0.2 mmol) was dissolved in a mixture of distilled water (2 mL) and ethanol (3 mL), and the pH value of the solution was adjusted to 4 with KOH (0.2 mol·L^{-1}). $MnCl_2·4H_2O$ (0.0197 g, 0.1 mmoL) was added to this solution. The mixture was sealed in a 25 mL Teflon-lined stainless steel container, which was heated at 120 °C for 48 h and finally cooled to room temperature. Colorless crystals of **1** were obtained. Yield: 44 % based on Mn^{2+}. $C_{14}H_{18}MnN_{12}O_8$: calcd. C 31.29; H 3.38; N 31.28 %; found: C 31.51; H 3.35; N 31.08 %. **IR** (KBr): $\tilde{\nu}$ = 3432 (s), 1651 (s), 1617 (s),1578 (s), 1437 (s), 1414 (s), 1325 (m), 1304 (w), 1270 (w), 1061 (w), 823 (s), 739 (s), 708 (m), 684 (m), 640 (m), 593 (m) cm^{-1}.

2.4 Synthesis of [Mn$_2$(pymtza)$_2$Cl$_2$(EtOH)]·H$_2$O (2)

An identical procedure to that of **1** was followed to prepare **2** except that the pH value of the solution was adjusted to 6 with KOH (0.2 mol·L^{-1}). Colorless crystals of **2** were obtained. Yield: 48 % based on Mn. $C_{16}H_{18}Cl_2Mn_2N_{12}O_6$: calcd. C 29.33; H 2.77; N 25.65 %; found: C 29.11; H 2.75; N 25.85 %. **IR** (KBr): $\tilde{\nu}$ = 3425 (s), 1654 (s), 1618 (s), 1579 (s), 1481 (w), 1435 (m), 1409 (s), 1322 (w), 1307 (w), 1270 (w), 824 (s), 743 (m), 707 (m), 680 (m), 641 (m), 592 (m) cm^{-1}.

2.5 X-ray Data Collection and Structure Determinations

Suitable single crystals of complexes **1** and **2** were mounted on a Rigaku SCXmini-CCD diffractometer equipped with a graphite-monochromated Mo-K_α radiation (λ = 0.71073 Å) at 291 K. All absorption corrections were performed using the CrystalClear programs. The crystal structures of **1** and **2** were solved by direct methods and refined on F^2 by full-matrix least-squares using anisotropic displacement parameters for all non-hydrogen atoms.[20] For **1** and **2**, important crystal data and collection and refinement parameters are summarized in Table 1, and selected bond lengths and angles are given in Table 2. Hydrogen-bonding parameters are given in Table S1 (Supporting Information).

Table 1. Selected crystallographic data and structure refinement for **1** and **2**.

	1	**2**
Empirical formula	$C_{14}H_{18}MnN_{12}O_8$	$C_{16}H_{18}Cl_2Mn_2N_{12}O_6$
Formula mass	537.34	655.20
Crystal system	monoclinic	triclinic
Space group	$P2_1/c$	$P\bar{1}$
a /Å	5.2653(11)	8.9624(18)
b /Å	10.953(2)	8.9998(18)
c /Å	19.147(5)	17.720(4)
α /°		100.16(3)
β /°	103.72(3)	96.70(3)
γ /°		104.51(3)
V /Å^3	1072.7(4)	1342.5(5)
Z	2	2
T /K	291(2)	291(2)
$D_{calcd.}$ /g·cm^{-3}	1.664	1.621
μ /mm^{-1}	0.687	1.195
Reflections collected	10772	14102
Unique reflections (R_{int})	2454 (0.1031)	6135 (0.0459)
No. observations [$I > 2.00\ \sigma$ (I)]	1567	4258
No. variables	154	349
$R^{a)}$, $wR^{b)}$	0.0716, 0.1227	0.0523, 0.1016
GOF$^{c)}$	1.074	1.046
Δ/ρ_{max} /e·Å^{-3}	0.341	0.557
Δ/ρ_{min} /e·Å^{-3}	–0.389	–0.424

a) $R = \Sigma||F_o|-|F_c|/\Sigma|F_o|$. b) $Rw = \{\Sigma w(F_o^2-F_c^2)^2/\Sigma w(F_o^2)^2\}^{1/2}$. c) GOF $= \{\Sigma[w((F_o^2-F_c^2)^2)/(n-p)\}^{1/2}$, where n = number of reflections and p = total numbers of parameters refined.

Table 2. Selected bond lengths /Å and angles /° for **1** and **2**.

[Mn(pymtza)$_2$(H$_2$O)$_4$] (**1**)			
Mn(1)–O(3)	2.1563	Mn(1)–O(1)	2.213(3)
Mn(1)–O(4)	2.226(2)		
O(3A)–Mn(1)–O(1)	91.65(7)	O(3)–Mn(1)–O(1)	88.35(7)
O(3)–Mn(1)–O(4)	92.59(6)	O(1A)–Mn(1)–O(1)	180.00(13)
O(3A)–Mn(1)–O(4)	87.41(6)	O(1A)–Mn(1)–O(4)	89.73(9)
O(1)–Mn(1)–O(4)	90.27(9)	O(4A)–Mn(1)–O(4)	180.00
O(3A)–Mn(1)–O(3)	180.00		
[Mn$_2$(pymtza)$_2$Cl$_2$(EtOH)]·H$_2$O (**2**)			
Mn(1)–O(2A)	2.131(2)	Mn(1)–O(3B)	2.159(3)
Mn(1)–N(7)	2.343(3)	Mn(1)–N(11)	2.393(3);
Mn(1)–Cl(2)	2.4525(13)	Mn(1)–Cl(1)	2.5285(12)
Mn(2)–O(4B)	2.105(2)	Mn(2)–O(1A)	2.141(3)
Mn(2)–O(5)	2.219(2)	Mn(2)–N(1)	2.319(3)
Mn(2)–N(5)	2.373(3)	Mn(2)–Cl(1)	2.4713(11)
O(2A)–Mn(1)–O(3B)	88.30(11)	O(2A)–Mn(1)–N(7)	154.21(10)
O(3B)–Mn(1)–N(7)	85.94(10)	O(2A)–Mn(1)–N(11)	84.20(10)
O(3B)–Mn(1)–N(11)	81.00(10)	N(7)–Mn(1)–N(11)	70.08(10)
O(2A)–Mn(1)–Cl(2)	94.88(9)	O(3B)–Mn(1)–Cl(2)	171.53(7)
N(7)–Mn(1)–Cl(2)	87.82(8)	N(11)–Mn(1)–Cl(2)	91.50(8)
O(2A)–Mn(1)–Cl(1)	104.46(7)	O(3B)–Mn(1)–Cl(1)	92.45(8)
N(7)–Mn(1)–Cl(1)	100.89(7)	N(11)–Mn(1)–Cl(1)	169.08(8)
Cl(2)–Mn(1)–Cl(1)	94.34(5)	O(4B)–Mn(2)–O(1)	93.51(11)
O(4B)–Mn(2)–O(5)	91.34(11)	O(1A)–Mn(2)–O(5)	168.41(9)
O(4B)–Mn(2)–N(1)	159.72(10)	O(1A)–Mn(2)–N(1)	87.35(10)
O(5)–Mn(2)–N(1)	84.48(10)	O(4B)–Mn(2)–N(5)	88.85(10)
O(1A)–Mn(2)–N(5)	85.85(10)	O(5)–Mn(2)–N(5)	83.72(11)
N(1)–Mn(2)– N(5)	70.99(10)	O(4B)–Mn(2)–Cl(1)	99.72(7)
O(1A)–Mn(2)–Cl(1)	99.37(8)	O(5)–Mn(2)–Cl(1)	90.17(8)
N(1)–Mn(2)–Cl(1)	100.13(7)	N(5)–Mn(2)–Cl(1)	169.62(8)

Symmetry code: For **1** A: $-x$, $1-y$, $1-z$; For **2**. A: $-x$, $-y$, $1-z$. B: $-x$, $-y$, $-z$.

Crystallographic data (excluding structure factors) for the structures in this paper have been deposited with the Cambridge Crystallographic Data Centre, CCDC, 12 Union Road, Cambridge CB21EZ, UK. Copies of the data can be obtained free of charge on quoting the depository numbers CCDC-893413 (**1**) and CCDC-893414 (**2**) (Fax: +44-1223-336-033; E-Mail: deposit@ccdc.cam.ac.uk, http://www.ccdc.cam.ac.uk)

Supporting Information (see footnote on the first page of this article): Depictions of the packing of compounds **1** and **2**.

3 Results and Discussion

3.1 General Characterizations of 1 and 2

In this investigation, we have selected Hpymtza and $\mathrm{Mn^{II}}$ to construct metal coordination architectures in order to explore whether or not the pH value will have an effect on the formation of such compounds and the structure variation caused by the different positions of the carboxylate group. Compound **1**, with mononuclear structure, was obtained when the pH value was 4, whereas complex **2**, which has a 1D chain structure, was synthesized at a pH value of 6. Compounds **1** and **2** are stable towards oxygen and moisture. The elemental analyses show that the components of these complexes are in accordance with the results of the structural analysis. IR spectra of the products show typical bands (1304–1604 $\mathrm{cm^{-1}}$) of the tetrazolyl and pyrimidyl groups. The band at 3432 $\mathrm{cm^{-1}}$ for compound **1** is attributed to the O–H stretching vibration of coordinated water, and the band at 3425 $\mathrm{cm^{-1}}$ for compound **2** is attributed to the O–H stretching vibration of both guest water and coordinated ethanol. The structures of **1** and **2** are confirmed by X-ray crystallography.

3.2 Crystal Structure of [Mn(pymtza)$_2$(H$_2$O)$_4$] (1)

X-ray diffraction demonstrates that compound **1** crystallizes in monoclinic lattice space group $P2_1/n$ and the asymmetric unit contains half of the $[\mathrm{Mn(pymtza)_2(H_2O)_4}]$ molecule. As shown in Figure 1, the $\mathrm{Mn^{II}}$ ion lies on an inversion center and is six-coordinate by two oxygen (O1, O1A) atoms from the carboxylate group of two pymtza ligands and four oxygen (O3, O3A, O4, O4A) atoms from four water molecules, forming a distorted $\mathrm{MnO_6}$ octahedral arrangement. The Mn–O distances [2.1563–2.226(2) Å] are similar to those of previously reported $\mathrm{Mn^{II}}$ complexes.[21] Compared to the previously reported 2D complex $[\mathrm{Mn(pztza)_2(H_2O)_2}]$,[9] in which the pztza ligand acts as a bidentate ligand bridging coordination mode by the pyrazinyl ring and the oxygen of the carboxylate group, the pymtza anion in **1** acts as a monodentate ligand to connect one $\mathrm{Mn^{II}}$ ion by one carboxylate oxygen atom, resulting in a mononuclear structure. Adjacent $[\mathrm{Mn(pymtza)_2(H_2O)_4}]$ molecules are linked together through five kinds of hydrogen bonding interactions between the coordinated water and the nitrogen atom of the tetrazole ring [O(3)–H(3A)···N(4), 2.8211 Å/142°], between the coordinated water and the oxygen atom of the carboxylate group [O(3)–H(3B)···O(2), 2.7070 Å/145°], between the coordinated water and the nitrogen atom of the tetrazole ring [O(4)–H(4A)···N(3), 2.933(4) Å/139°], between the coordinated water and the oxygen atom of the carboxylate group [O(4)–H(4B)···O(1), 2.772(4) Å/164°], and between the C–H group of the carboxylate group and the oxygen atom of the carboxylate group [C(2)–H(2A)···O(2), 3.479(5) Å/170°], generating a 3D supramolecular network (Table S1, Figure S1, Supporting Information).

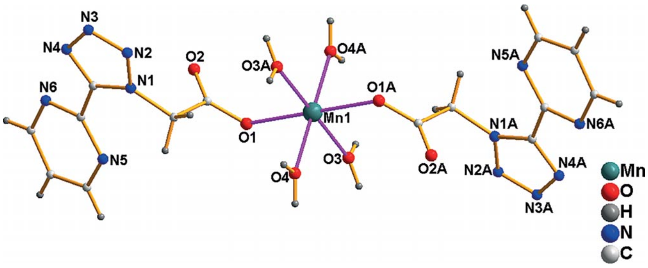

Figure 1. Coordination environment of the $\mathrm{Mn^{II}}$ atoms in compound **1**.

3.3 Crystal Structure of [Mn$_2$(pymtza)$_2$Cl$_2$(EtOH)]·H$_2$O (2)

Compound **2** crystallizes in the triclinic lattice space group $P\bar{1}$ and the asymmetric unit contains one $[\mathrm{Mn_2(pymtza)_2Cl_2(EtOH)}]\cdot\mathrm{H_2O}$ molecule. Two crystallographically independent $\mathrm{Mn^{II}}$ atoms [Mn(1) and Mn(2)] are observed. As shown in Figure 2, both central $\mathrm{Mn^{II}}$(1) or $\mathrm{Mn^{II}}$(2) atoms are surrounded by six atoms, forming a distorted octahedral coordination arrangement, respectively. The Mn(1) atom is coordinated by two O(O2B, O3A) atoms from two pymtza ligands, and two N(N7, N11) atoms from one pymtza ligand and two Cl anions. The Mn(2) atom is coordinated by two O(O1B, O4A) atoms from two pymtza ligands, and two nitrogen (N1, N5) atoms from one pymtza ligand, and one chloride anion and one oxygen(O5) atom from one ethanol molecule. Compared to compound **1**, in which pymtza adopts a monodentate mode, neighboring $\mathrm{Mn^{II}}$ atoms are doubly bridged by two carboxylate groups from two pymtza ligands in a $\mu_{1,3}$-COO bridging mode and one chloride ligand in a $\mu_{1,1}$-Cl bridging mode with the Mn(1)···Mn(2) distance of 3.6890 Å, forming a one-dimensional structure stretching along the *c* axis with the Mn···Mn···Mn bite angle of 154.953°. Each pymtza ligand acts as a tetradentate ligand to connect three $\mathrm{Mn^{II}}$ by

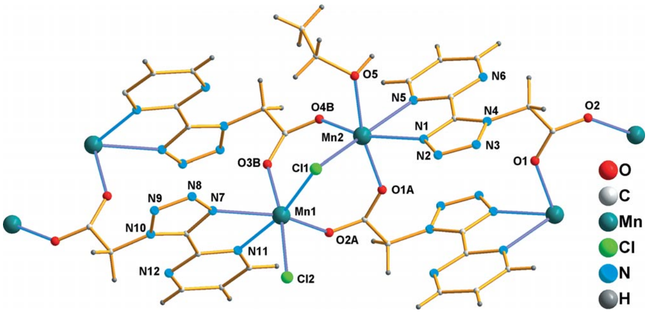

Figure 2. 1D chain structure extending along the *c* axis of compound **2**, showing the coordination environment of $\mathrm{Mn^{II}}$.

two nitrogen atoms from the tetrazole and the pyrimidine to link the same Mn^{II} atom and two oxygen atoms from the same carboxylate group to connect two Mn^{II} atoms, whereas [Mn(pyrtza)Cl] contains neither ethanol nor water molecules.[4] The pyrtza ligand adopts a tetradentate coordination mode to connect to three Mn^{II} ions, showing a 2D network. Adjacent one-dimensional chains are held together through four kinds of hydrogen bonding interactions between the ethanol and the solvent water [O(5)–H(5A)···O(6), 2.720(4) Å/173(4)°], between the guest water and the Cl atom [O(6)–H(6B)···Cl(2), 3.116(3) Å/164°], between the C–H group of the carboxylate group and the Cl atom [C(9)–H(9A)···Cl(2), 3.675(4) Å/163°], and between the C–H group of the pyrimidine and the nitrogen atom from tetrazole [C(14)–H(14)···N(8), 3.234(5) Å/126°], to generate a three-dimensional network structure (Table S1, Figure S2, Supporting Information).

3.4 Luminescence Properties and Thermogravimetric Analyses (TGA)

The luminescence properties of **1** and **2** and the free ligand were investigated at room temperature in the solid state. As is shown in Figure 3, the free ligand Hpymtza exhibits emission with maximum intensity at 442 nm and upon excitation at 375 nm. Compound **2** exhibits fluorescent emission bands at 460 nm with λ_{ex} = 375 nm. This luminescence could be tentatively assigned to the intraligand luminescence, because similar emission was also observed for the free ligand Hpymtza (442 nm). Generally speaking, the intraligand wavelength is determined by the energy gap between the π and π* molecular orbitals of the free ligand, which is related to the extent of π conjugation.[9] Complex **1** does not exhibit detectable emission, and this can be explained by the fact that this complex contains manganese, whose low d-d transition quenches luminescence, which is similar to that of $[Mn(pztza)_2(4,4'\text{-}bipy)(H_2O)_2]\cdot 2H_2O$.[7] To characterize the compounds in terms of thermal stability, thermal gravimetric analysis (TGA) of **1** and **2** were carried out under nitrogen atmosphere (Figure 4). From the TG curves, Complexes **1** and **2** are stable up to 230 °C and 275 °C, respectively, and above the temperature, the decomposition of the complexes start. For complexes **1** and **2** the remaining weight of 16.50 %, 26.27 % corresponds to the final product MnO_2 (calcd. 16.18 %, 900 °C; 26.53 %, 900 °C, respectively).

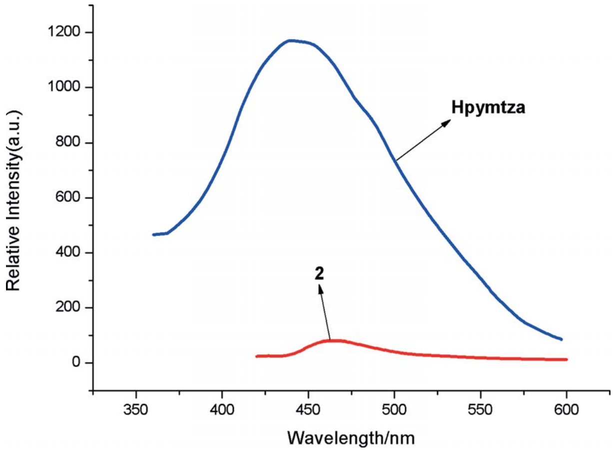

Figure 3. Emission spectra of compound **2** and Hpymtza at room temperature in the solid state.

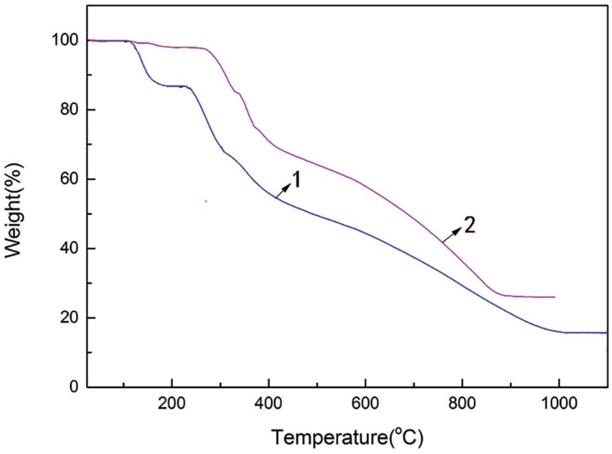

Figure 4. TG curves of compounds **1** and **2**.

4 Conclusions

Different positions of the carboxylate group of the tetrazole ring have a substantial effect on the crystal structures of complexes like those investigated in this paper. The luminescence of compound **2** shows intraligand emission. The experimental results show that the pH value and the position of the carboxylate group have a significant influence on the structures. Further exploration of other metal ions compounds with Hpymtza is underway.

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