Journal of Molecular Structure 992 (2011) 106–110

Contents lists available at ScienceDirect

Journal of Molecular Structure

journal homepage: www.elsevier.com/locate/molstruc

Synthesis, structure and properties of one novel 2D Mn-heterocyclic carboxylic acid complex $[Mn(TPA)Cl(H_2O)]_n$

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ARTICLE INFO

Article history:
Received 27 January 2011
Received in revised form 25 February 2011
Accepted 25 February 2011
Available online 4 March 2011

Keywords:
Mn(II) complex
Crystal structure
Hydrothermal condition
Fluorescence

ABSTRACT

A novel 2D layer complex $[Mn(TPA)Cl(H_2O)]_n$ (**1**) has been synthesized by two methods through the reaction of $MnCl_2$ and TPC or TPA under hydrothermal conditions and characterized by single crystal X-ray diffraction, elemental analysis, infrared spectrometry (IR), powder X-ray diffraction (XRD) and thermogravimetric analysis (TGA), where heterocyclic carboxylic acid ligand TPA = 2-(5-(pyridin-2-yl)-2H-tetrazol-2-yl)acetic acid, TPC = 2-(5-(pyridin-2-yl)-2H-tetrazol-2-yl)acetonitrile. The distorted octahedral Mn(II) centers are bridged by carboxylic O atoms resulting in the formation of a 1D chain. Then the 1D chains are connected with each other through TPA ligands into a 2D (3,3)-connected topology framework. The H-bonding interactions extend the complex into a three-dimensional network, and such weak interactions further stabilized the complex. Furthermore, solid-state fluorescence spectrum of complex **1** exhibits intense broad emissions at 396 nm at room temperature, which is red-shifted by 21 nm relative to that of free ligand TPA.

1. Introduction

Self-assembled metal–organic frameworks (MOFs) containing central metal ions and polydentate organic ligands have been attracting considerable attention in recent years owing to their novel and diverse crystal structures, topologies [1–6] and potential applications in the areas of luminescence, nonlinear optics, magnetism, catalysis, ferroelectric, electrical conductivity and gas storage [7–11]. To get anticipant and interesting structures and desired properties, an enormous amount of researches have being focused on constructing novel MOFs by choosing functional metal ions and versatile binding-ligands through hydrothermal methods. Heterocyclic carboxylic acids as multifunctional building blocks have attracted great attention, such as pyridine-, tetrazole-, imidazole-, and pyrazole-carboxylic acids [12–19]. The heterocyclic N atoms can coordinate to the metal centers, while carboxylate groups can coordinate in monodentate, chelate or bridged modes resulting in the formations of various MOFs with novel structures and useful properties [20–28]. Literatures showed that pyridine acid and tetrazole-5-carboxylic acid and their derivatives can behave as multifunctional ligands, which improve the stability of its metal polymers with the flexibility of the coordination modes and stereo crystal packing forces, thus result in the tremendous network diversity [29–34].

As a continuation of those work, we focus our attention here on the pyridine-tetrazole-heterocyclic nitrile (TPC) and pyridine-tetrazole-heterocyclic acid (TPA), (Scheme 1). Considering that the heterocyclic N atoms and carboxylic acid functional group may act as useful coordination sites, employment of the two ligands to react with metal ions can produce intriguing structures. In this work, the novel 2D MOF $[Mn(TPA)Cl(H_2O)]_n$ (**1**) has been isolated under hydrothermal condition. Then, its crystal structure, solid state luminescence and thermogravimetric analysis were reported.

2. Experimental section

2.1. Material and instrument

Chemicals and solvents in this work were commercially obtained and used without any further purification. Elemental analysis for carbon, hydrogen and nitrogen were performed on a Vario EL III elemental analyzer. The infrared spectra (4000–500 cm^{-1}) were recorded by using KBr pellet on a Shimadzu IRprestige-21 FTIR-8400S spectrometer. The X-ray powder diffraction (XRD) data were collected with a Siemens D5005 diffractometer with Cu $K\alpha$ radiation ($\lambda = 1.5418$ Å). Thermogravimetric analyses were performed on a simultaneous SDT 2960 thermal analyzer under flowing N_2 with a heating rate of 20 °C/min from ambient temperature to 750 °C. The fluorescent spectra were recorded on a Shimadzu Instrument FL/FS-920 fluorescent spectrometer.

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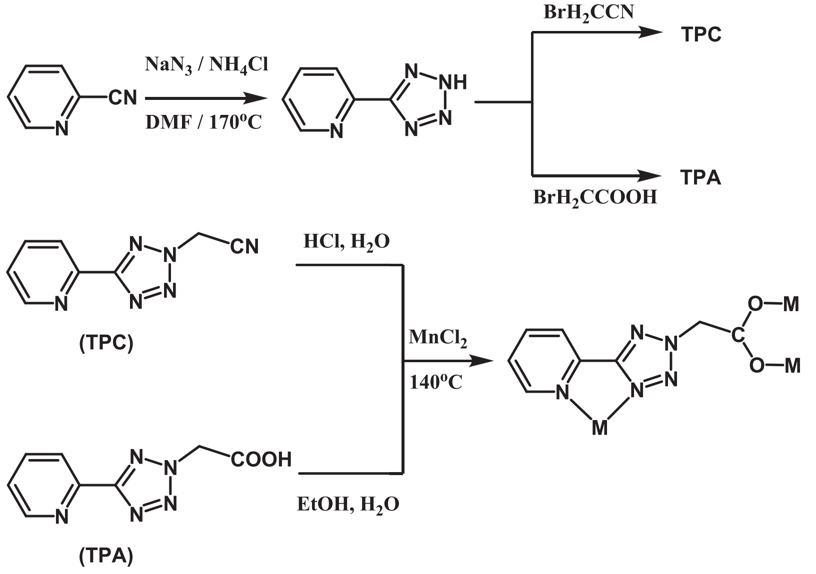
doi:10.1016/j.molstruc.2011.02.054



Scheme 1. The synthesis of ligands and complex **1**, and the coordination modes of TPA.

2.2. Preparation of the ligands TPC and TPA

The ligands, TPC and TPA, were prepared according to the literature methods [35] from picolinonitrile through two steps (Scheme 1).

TPC: Anal. Calcd. for $C_8H_6N_6$: C, 51.61; H, 3.23; N, 45.16. Found: C, 51.49; H, 3.18; N, 45.02. ^{1}H NMR (300 MHz, CD_3OD): δ 4.88 (s, 2H), 7.28 (t, 1H), 7.74 (t, 1H), 8.29 (d, 1H), 8.51 (d, 1H). ESI-MS *m/z* (%): [M–H] = –185.29; [M+H] = +187.28. Main IR (cm^{-1}, KBr): 3086 (m), 2971 (m), 2280 (m), 15,718 (w), 1560 (w), 1481 (w), 1402 (s), 1323 (w), 1251 (w), 1125 (w), 1049 (w), 935 (w), 860 (m), 779 (w), 754 (s), 705 (w), 675 (w), 555 (w), 511 (w), 472 (w), 422 (w).

TPA: Anal. Calcd. for $C_8H_7N_5O_2$: C, 46.83; H, 3.41; N, 34.15. Found: C, 46.71; H, 3.35; N, 34.02. 1HNMR (300 MHz, CD_3OD): δ 4.75 (s, 2H), 7.51 (t, 1H), 7.92 (t, 1H), 8.42 (d, 1H), 8.63 (d, 1H), 10.53 (s, 1H). ESI-MS *m/z* (%): [M–H] = –204.33; [M + H] = +206.32. Main IR (cm^{-1}, KBr): 3435 (s), 3138 (ms), 2988 (w), 1680 (s), 1607 (m), 1561 (w), 1480 (w), 1405 (s), 1327 (w), 1289 (m), 1247 (w), 1181 (w), 1163 (w), 1119 (w), 1051 (w), 1008 (w), 932 (w), 860 (m), 779 (w), 754 (s), 705 (w), 675 (w), 555 (w), 511 (w), 472 (w), 422 (w).

2.3. Preparation of complex $[Mn(TPA)Cl(H_2O)]_n$(**1**)

The title complex has been synthesized by the following two methods from different starting materials under hydrothermal synthesis conditions.

2.3.1. Method A

A mixture of $MnCl_2$ (10 mmol, 1.98 g), TPC (10 mmol, 1.80 g), HCl (1.5 ml, 36%) and distilled water (18.0 ml) were placed in a 25 ml Teflon-Lined stainless steel vessel, which was heated to 140 °C for 7 days. Then the reaction was slowly cooled to room temperature at a rate of 5 °C/h. Colorless block crystals suitable for X-ray diffraction were obtained in 53% yield (based on TPC).

2.3.2. Method B

A mixture of $MnCl_2$ (1.0 mmol, 0.198 g), TPA (1.0 mmol, 0.206 g), ethanol (5.0 ml) and distilled water (15.0 ml) were placed in a 25 ml Teflon-Lined stainless steel vessel, which was heated to 140 °C for 5 days. Then the reaction was slowly cooled to room temperature at a rate of 5 °C/h. The resultant white precipitate was filtered off and the colorless solution was left to stand at room temperature. Colorless block crystals suitable for X-ray diffraction were harvested by slow evaporation of the filtrate within one month (Yield: 34%, based on TPA).

Elemental analysis for **1**, $C_8H_8ClMnN_5O_3$ (312.58): Anal. (%) calcd. C 30.71, H 2.56, N 22.39; found C 30.59, H 2.48, N 22.23. IR spectrum (KBr): ν (cm^{-1}): 3345 (s), 3275 (w), 3041 (m), 2921 (m), 1601 (s), 1570 (w), 1547 (w), 1477 (m), 1430 (m), 1405 (s), 1304 (m), 1213 (m), 1203 (m), 845 (m), 778 (m), 731 (m), 705 (m), 668 (m).

2.4. Crystal structure determination

A single crystal of the title complex with approximate dimensions 0.40 mm × 0.35 mm × 0.25 mm was selected for data collection at 293(2) K, using a Rigaku Saturn diffractionmeter by the ω scan technique at room temperature with graphite monochromated Mo Kα radiation (λ = 0.071073 nm). The structure parameters were obtained by CrystalClear [36] with the θ range for data collection from 3.11 to 27.45°. Of the 12,457 reflections collected, there were 2866 unique reflections (R_{int} = 0.0244). The absorption correction was carried out by multi-scan method. The structure was solved by direct methods with SHELXS-97 and refined by full matrix least squares on F^2 with SHELXL-97 [37]. All non-hydrogen atoms were refined with anisotropic thermal parameters. Hydrogen atoms bonded to the carbon atoms were placed in calculated positions and refined as riding mode, with C–H = 0.93 Å (aromatic) and 0.97 Å (methylene) with $U_{iso}(H) = 1.2U_{eq}(C)$. The water hydrogen atoms were located in the difference Fourier maps and refined with an O–H distance restraint [0.85(2) Å]. Detailed data collection and refinement of the compound **1** are summarized in Table 1, and the selected bond distances, angles and H-bonding are listed in Table 2. CCDC: 807,071 **1** contains the supplementary crystallographic data for this paper. Copies of this information may be obtained from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44 1233 336,033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

3. Results and discussion

3.1. IR, UV/Vis spectrua and powder XRD

The IR spectrum shows a series of peaks at 1570, 1477, 1203, 778 and 705 cm^{-1} are the characteristic of aromatic ring. The

Table 1
Crystal data and structure refinement parameters for **1**.

Empirical formula	$C_8H_8ClMnN_5O_3$
Formula weight	312.58
Temperature (K)	293 (2)
Crystal size (mm)	0.20 × 0.10 × 0.10
Wavelength (Å)	0.71073
Crystal system	monoclinic
Space group	*C*2/*c*
a (Å)	25.471 (5)
b (Å)	10.246 (2)
c (Å)	9.7147 (19)
β (°)	99.33 (3)
V (Å)^3	2501.7 (9)
Z	8
$D_{calc.}$ (mg m^{-3})	1.66
μ (mm^{-1})	1.276
F(0 0 0)	1256
θ range (°)	3.11–27.45
Index ranges	$-32 \leqslant h \leqslant 32$, $-13 \leqslant k \leqslant 13$, $-12 \leqslant l \leqslant 12$
Reflections collected/unique	12,457/2866 [*R*(int) = 0.0244]
Completeness to *θ* = 27.45°	99.8%
Data/restraints/parameters	2866/2/163
Goodness-of-fit on F^2	1.094
Final *R* indices [$I > 2$sigma(I)]	$R_1 = 0.0260$, $wR_2 = 0.0641$
R indices (all data)	$R_1 = 0.0293$, $wR_2 = 0.0656$
Largest diff. peak and hole	0.322 and −0.348 e Å^{-3}

[a] $R_1 = \Sigma||F_o| - |F_c|/\Sigma|F_o|$.
[b] $wR_2 = \left\{\sum\left[w(F_o^2 - F_c^2)^2\right]/\sum w\left[(F_o^2)^2\right]\right\}^{1/2}$.

Table 2
Selected bond-length (Å), bond-angle (°) and hydrogen-bonding (Å, °) of compound **1**.

Mn(1)–O(1W)	2.1352(14)	Mn(1)–O(1)	2.1452(12)
Mn(1)–O(2)	2.1542(13)	Mn(1)–N(2)	2.3204(14)
Mn(1)–N(1)	2.3548(15)	Mn(1)–Cl(2)	2.4961(7)
O(1W)–Mn(1)–O(1)	97.38(6)	O(1W)–Mn(1)–O(2)	92.35(5)
O(1)–Mn(1)–O(2)	95.72(5)	O(1W)–Mn(1)–N(2)	175.68(6)
O(1)–Mn(1)–N(2)	86.91(5)	O(2)–Mn(1)–N(2)	86.69(5)
O(1W)–Mn(1)–N(1)	104.91(6)	O(1)–Mn(1)–N(1)	157.09(5)
O(2)–Mn(1)–N(1)	88.71(5)	N(2)–Mn(1)–N(1)	70.87(5)
O(1W)–Mn(1)–Cl(2)	87.96(4)	O(1)–Mn(1)–Cl(2)	89.09(4)
O(2)–Mn(1)–Cl(2)	175.10(4)	N(2)–Mn(1)–Cl(2)	92.63(4)
N(1)–Mn(1)–Cl(2)	86.48(4)		

D–H···A	*d*(D–H)	*d*(H···A)	*d*(D···A)	∠(DHA)
O1W–H1WA···Cl2	0.75	2.48	3.200(15)	163.7
O1W–H1WB···Cl2	0.69	2.54	3.171(16)	153.7
O1W–H1WB···O1	0.69	2.55	2.973(2)	122.8

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

bands at 1547, 1430 cm^{-1} are attributed to the tetrazole group [35]. To compare with the IR spectrum of raw material TPC, the absence of a strong peak at 2280 cm^{-1} for **1** indicates that the cyano group has been completely hydrolyzed. Moreover, the new double peaks at 1601 cm^{-1} and 1405 cm^{-1} showed that the carboxyl was deprotonated to generate COO$^-$ anion upon reaction with the central metal ion, which is agreement with its X-ray single structure. The double peaks are the characteristics of ν_{as}(COO$^-$) and ν_s(COO$^-$), respectively. And the difference between the asymmetric stretching and symmetric stretching bands of the carboxyl group ($\Delta\nu = \nu_{as}$(C=O) − ν_s(C=O)) is 196 cm^{-1}, which indicates strong coordination of the carboxylate oxygen to the mental center through 1,3-bidentate bridging fashion. The broad band around 3345 cm^{-1} indicates the presence of ν(O–H) stretching frequency of coordinated water molecule.

The UV/Vis spectrum of the ligand and complex **1** was measured in the solid state at room temperature (Fig. 1). It shows that both, the ligand and the complex, exhibit absorption bands at the range of 200–400 nm. After coordination, the absorption bands underwent a slightly redshift, owing to the formation of the more large conjugated system after coordination, which is consisted of pyridine and tetrazole rings. The above spectral analyses are in agreement with the determined crystal structure.

Powder XRD analysis: The agreement between the experimental and simulated XRD patterns indicated the phase purity of **1** (Fig. 2). The difference in reflection intensities between the simulated and experimental patterns was due to the variation in preferred orientation for the powder sample during collection of the experimental XRD data.

3.2. Description of the crystal structure

The complex **1** has been synthesized by the reaction of heterocyclic ligands TPC or TPA with $MnCl_2$ under hydrothermal conditions. Single-crystal X-ray analysis reveals that complex **1** crystallized in the monoclinic space group C2/c. The asymmetrical unit consists of one Mn(II) ion, one TPA anion, one chloride anion and one coordinated water molecule. The carboxyl group was deprotonated to maintain the charge balance. The coordination environment of complex **1** with the atomic labeling is shown in Fig. 3. The Mn1 sits in an irregular six-coordinate (MnN_2O_3Cl) environment composed of two chelate nitrogen donors from one TPA ligand, two bridging-bidentate carboxyl oxygen atoms (O1A and

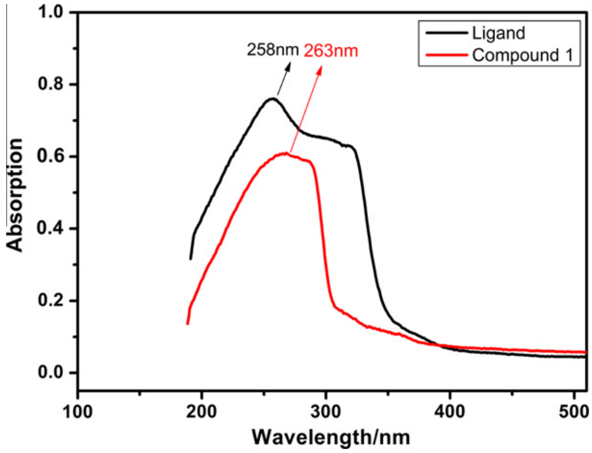


Fig. 1. UV spectra of the ligand and compound **1** in the solid state at room temperature.

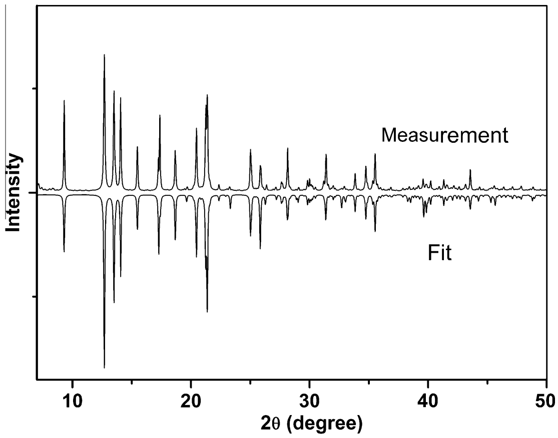


Fig. 2. Simulated and experimental powder X-ray diffraction patterns of **1**.

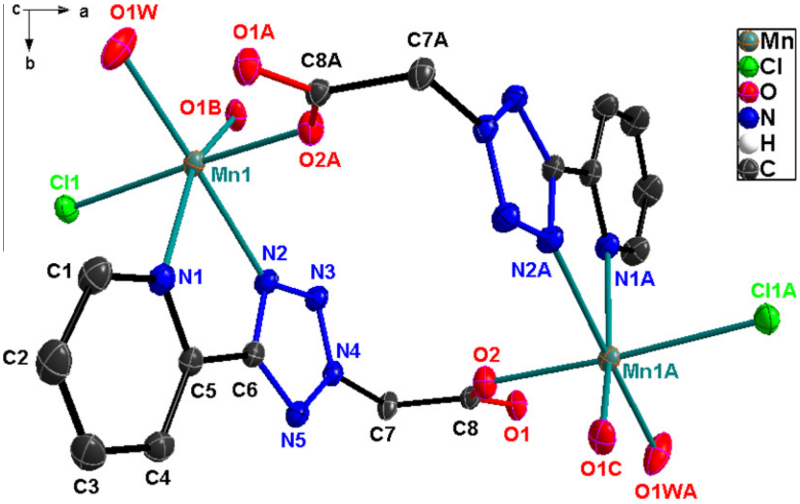


Fig. 3. View of the coordination environment of Mn(II) and TPA in complex **1**. Hydrogen atoms have been omitted for clarity, displacement ellipsoids are drawn at the 30% probability level. Symmetry codes: A: $-x+1/2$, $y-1/2$, $-z+1/2$; $-x+1/2$, $-y+1/2$, $-z$; $-x+1/2$, $y+1/2$, $-z+1/2$.

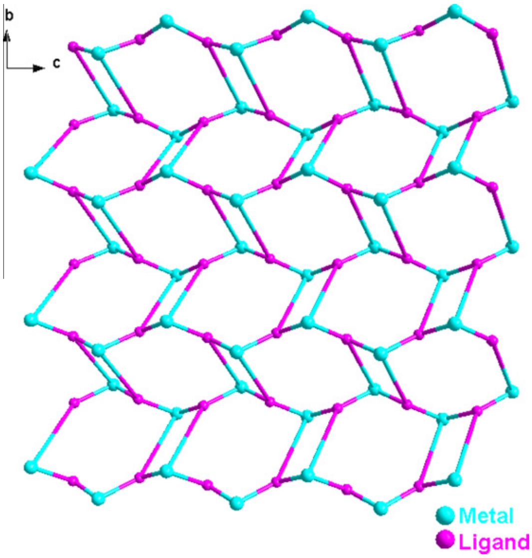


Fig. 4. The 2D (3,3)-connected topologic structure of **1**.

O2A) from two different TPA ligands and the another oxygen atom from the coordinated water molecule (O1W), and a terminal Cl1 atom. That is to say, each Mn(II) atom adopts a slightly distorted octahedral geometry with the small O(N)–Mn–O(N, Cl) angles range from 70.87(5)° to 104.91 (6)° and the Mn–O(N) distances range from 2.1352(1) Å to 2.3548(1) Å, deviating from the values of an ideal octahedron. The Mn1–N1 = 2.3548(1) Å and Mn1–N2 = 2.3204 (1) Å bond length are a little bit longer than the normal range. Along c-axis direction, the neighboring metal centers Mn1 and Mn1B ions are bridged by carboxylic O1A and O2A atoms resulting in the formation of $[\mathrm{Mn{-}O{-}C{-}O{-}Mn}]_n$ 1D-chain with a Mn···Mn distance of 5.2705(9) Å. The 1D chains are parallel and linked with each other through TPA ligands into a 2-D sheet parallel to the (1 0 0) plane. To get better insight into the framework structure, its topological analysis was carried out. As discussed above, every Mn(II) center can be seen as one nod, each Mn(II) nod coordinated with three TPA ligands, thus can be defined as 3-connector. And each ligand links three Mn(II) nod, it can be considered as a 3-connecting node. According to the simplification principle of the TOPOS software [38], the overall network topology was described as a (3,3)-connected framework (Fig. 4). The O1W aqua molecule was involved in the O–H···O and O–H···Cl H-bonds

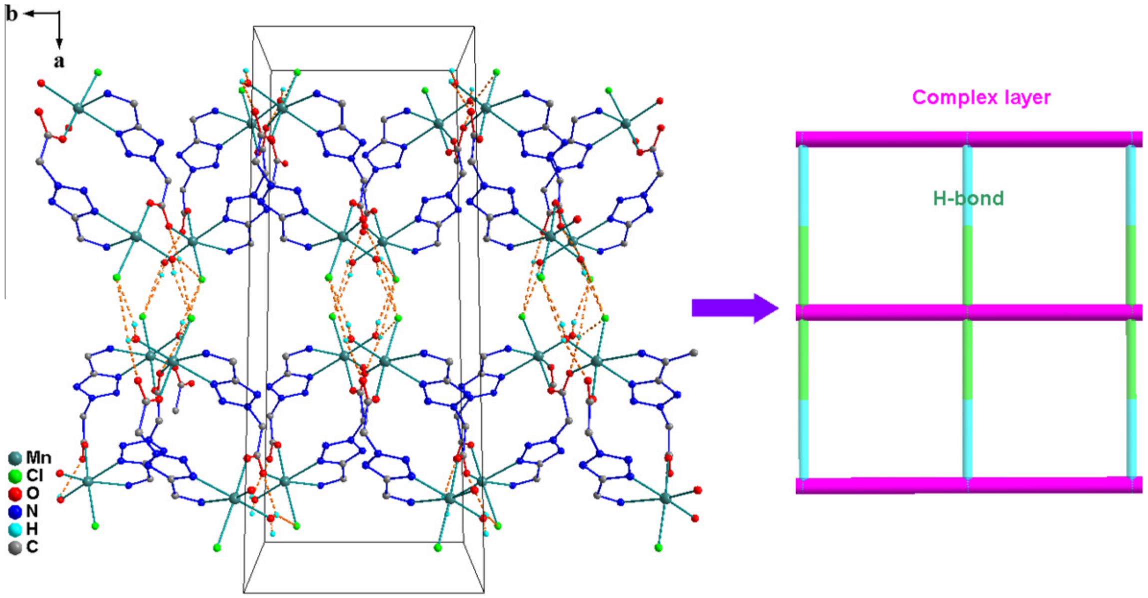


Fig. 5. The simplified O–H···O and O–H···Cl H-bonds network of **1** viewed along the *c*-axis in which pyridine rings are omitted for clarity.

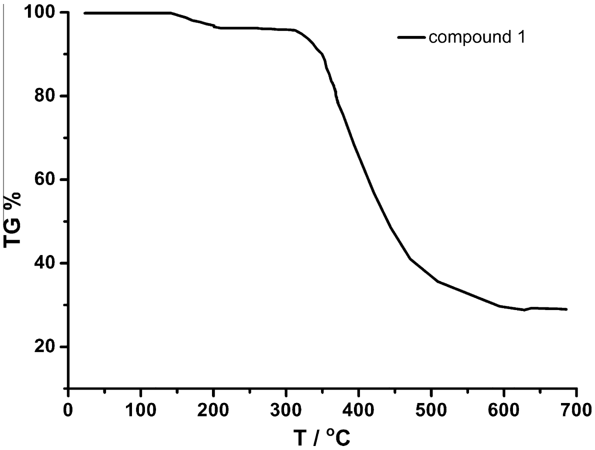


Fig. 6. The TGA curve for complex **1**.

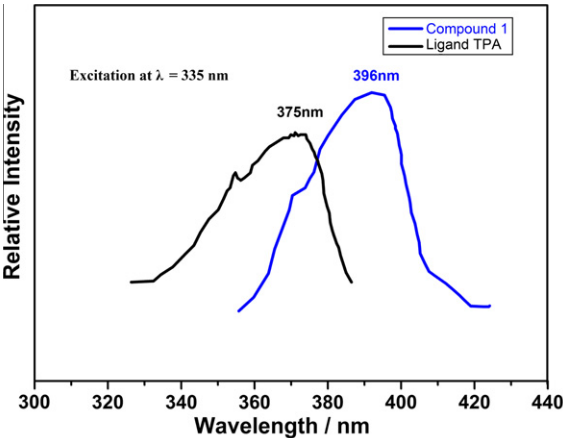


Fig. 7. Fluorescent emission spectra of the compound **1** and free TPA ligand.

with the carboxyl O1A atom and terminal Cl1 atom. Thus the 3-D network structure was obtained by those H-bonds to bridge the 2D layer networks (Fig. 5).

3.3. Thermogravimetric analysis

The TGA curve shows that the complex **1** begins to decompose at 135 °C displaying two stages of mass loss (Fig. 6). The first weight loss of 5.89% (calc 5.76%) from 135 to 240 °C is consistent with the release of a coordinated water molecule. The degradation of the TPA occurs in the second step above 335 °C. Due to decomposition of the network, the total observed weight loss at 750 °C is 76.12%, and the final presumably residual is Mn_3O_4. It is concluded that the framework is very stable and worthy of further investigation as candidate of thermally stable material.

3.4. Photoluminescent properties

The solid-state fluorescent property of the complex was investigated at room temperature. As illustrated in Fig. 7, upon excitation of the solid sample at $\lambda = 335$ nm, the intense broad emission bands at 396 nm was observed. Moreover, for excitation wavelength between 280 and 380 nm, the free TPA ligand presents a weak photoluminescence emission at 375 nm under the same experimental conditions. The emission of complex is red-shifted about 21 nm compared to that of free ligand TPA. Obviously, this strong emission come from the heterocyclic carboxylic acid ligand, and the large red-shift effect may be attributed to the coordination or an excited state of a metal-perturbing intraligand. That is, both the rigidity of conjugated ring and the conjugation effect of ligand were increased after coordination. It plays the main role to luminescence intensity and red-shift effect. Complex **1** is air-stable and insoluble in water and common organic solvents. This coordination polymer may be excellent candidate for potential photoluminescence material because they are highly thermally stable.

4. Conclusions

In conclusion, a novel 2D layer complex $[Mn(TPA)Cl(H_2O)]_n$ (**1**) was prepared by two methods through employing heterocyclic nitrile or heterocyclic carboxylic acid ligands under hydrothermal conditions. We studied here the structure, TGA and fluorescence properties of the coordination polymer. This material provides a new impetus to construction of novel functional material with potentially useful physical properties.

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