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Four Novel Metal–Organic Frameworks Based on 3,4,7,8-Tetramethyl-1,10-Phenanthroline: Syntheses, Structures, and Thermal Properties[1]

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Abstract—Four novel metal–organic frameworks, $[Cu(Tmp)_2(H_2O)] \cdot NO_3$ (**I**) (Tmp = 3,4,7,8-tetramethyl-1,10-phenanthroline), $[Mn(Tmp)_2(H_2O)_2] \cdot 2NO_3 \cdot H_2O$ (**II**), $[Pb(Tmp)(CH_3COO)_2] \cdot 3H_2O$ (**III**) and $[Zn(Tmp)_2(H_2O)_2] \cdot 2NO_3 \cdot 2H_2O$ (**IV**), have been synthesized and characterized by single crystal X-ray diffraction (CIF files CCDC nos. 88362–88365 for **I**–**IV**, respectively), IR spectroscopy, elemental analysis and thermogravimetric analysis. Both **I** and **II** complexes are crystallized in monoclinic system with space groups $C2/c$, $P2_1/c$, respectively, while **III** and **IV** complexes are crystallized in triclinic system with space groups $P\bar{1}$. Generally, these crystal structures are stabilized by O–H···O hydrogen bonds and π–π interactions between the phenanthroline rings of neighboring molecules. Thermogravimetric analyses of compounds **I**–**IV** display considerable thermal stability.

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INTRODUCTION

Metal-organic frameworks (MOFs) represent a relatively new class of hybrid inorganic-organic materials that have attracted much attention for their extremely wide-open structure, in which the free space is available for host molecules [1–4] and beneficial properties such as porosity, catalysis, luminescence, photoactivity and magnetism [5–14]. 1,10-Phenanthroline (Phen) also has pharmacological importance as a vital compound in drug industry [15]. Due to its coordinating ability and large conjugated system, Phen often was widely used to build coordination polymers. Additionally, functionalized Phen derivatives have been flourishing in recent years, they generally dominate in magnetism and play an essential role in optical activity, luminescence and photochemistry. 3,4,7,8-Tetramethyl-1,10-phenanthrolin (Tmp) with a noticeable coordination ability for metal cations has caused great concern in the coordination chemistry [16–23].

In this paper, we present the synthesis and structural characterization of the four novel MOFs, $[Cu(Tmp)_2(H_2O)] \cdot NO_3$ (**I**), $[Mn(Tmp)_2(H_2O)_2] \cdot 2NO_3 \cdot H_2O$ (**II**), $[Pb(Tmp)(CH_3COO)_2] \cdot 3H_2O$ (**III**) and $[Zn(Tmp)_2(H_2O)_2] \cdot 2NO_3 \cdot 2H_2O$ (**IV**). All of them were constructed from Tmp and corresponding metal ions.

EXPERIMENTAL

Materials and characterizations. All commercially available chemicals are reagent grade and used without further purification. Elemental analysis of C, H and N for the compounds was performed on a Carlo Erba 1106 elemental analyzer. Infrared (IR) spectra, in the region from 400 to 4000 cm^{-1}, were recorded on a Nicolet nexus FT–IR spectroscopy. Thermogravimetric analysis was performed from room temperature to 1000°C in a nitrogen stream at a rate of 10°C min^{-1} on a Netzsch STA 449 C system using α-Al_2O_3 as a reference material.

Synthesis of complex I. A methanol solution containing 0.0483 g (0.2 mmol) of copper nitrate trihydrate and 0.0945 g (0.4 mmol) of Tmp was refluxed for 5 h and filtered, a green solution was obtained. After standing it at ambient temperature for three weeks, blue bulk crystals of **I** were formed.

For $C_{32}H_{34}N_5O_4Cu$ (M = 615.54)

anal. calcd., %:	C, 62.38;	H, 5.52;	N, 11.37.
Found, %:	C, 62.40;	H, 5.49;	N, 11.39.

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Table 1. Crystallographic data and structure refinement for complexes **I**–**IV**

Parameter	Value			
	I	**II**	**III**	**IV**
Crystal system	Monoclinic	Monoclinic	Triclinic	Triclinic
Crystal size, mm	0.43 × 0.37 × 0.29	0.23 × 0.21 × 0.05	0.25 × 0.15 × 0.13	0.26 × 0.17 × 0.14
Space group	$C2/c$	$P2_1/c$	$P\bar{1}$	$P\bar{1}$
a, Å	24.870(6)	11.6682(15)	8.9731(9)	9.2680(13)
b, Å	7.6469(18)	28.096(4)	10.6101(10)	10.9533(2)
c, Å	17.699(4)	10.7556(14)	12.7679(12)	18.464(3)
α, deg	90	90	108.575(1)	102.794(2)
β, deg	115.833(2)	108.597(2)	100.042(1)	102.123(2)
γ, deg	90	90	96.520(1)	102.550(2)
V, Å^3	3029.7(12)	3341.9(7)	1115.8(2)	1717.3(4)
Fw	615.54	705.62	615.63	734.07
Z	4	4	2	2
T, K	1408	1476	600	768
Total reflections/	294	294	296	296
unique	2824/2824	6205/6205	4121/4121	6334/6334
R_{int}	0.027	0.099	0.015	0.043
Refinement parameter	213	420	277	450
GOOF on F^2	1.048	1.008	1.041	1.026
R_1, wR_2 ($I > 2\sigma(I)$)	0.0357, 0.0964	0.0627, 0.1288	0.0169, 0.0397	0.0627, 0.1542
R_1, wR_2 (all data)	0.0441, 0.1033	0.1455, 0.1653	0.0194, 0.0402	0.1112, 0.1832
Large diff. peak and hole, e Å^{-3}	0.350 and –0.373	0.342 and –0.319	0.422 and –0.563	0.769 and –0.353

Synthesis of complex II. Manganese nitrate (0.0405 g, 0.1 mmol) was added to the solution of Tmp (0.0473 g, 0.2 mmol) in 10 mL of methanol. The mixture was refluxed for 3 h and filtered to give a yellow-green solution. The suitable crystals of **II** were obtained by slow evaporation of the filtrate at room temperature after 4 weeks.

For $C_{32}H_{38}N_6O_9Mn$ (M = 705.62)

anal. calcd., %:	C, 54.42;	H, 5.39;	N, 11.90.
Found, %:	C, 54.39;	H, 5.43;	N, 11.87.

Synthesis of complex III. To a suspension containing lead acetate trihydrate (0.0569 g, 0.4 mmol) and Tmp (0.0366 g, 0.15 mmol) in ethanol (10 mL) was added water (5 mL). The mixture was refluxed for 3 h and filtered to give a colorless solution. After standing it at ambient temperature for ten weeks, the colorless crystals of **III** were formed.

For $C_{20}H_{28}N_2O_7Pb$ (M = 615.63)

anal. calcd., %:	C, 38.98;	H, 4.55;	N, 4.55.
Found, %:	C, 38.97;	H, 4.52;	N, 4.58.

Synthesis of complex IV. An aqueous solution (1 mL) containing zinc nitrate hexahydrate (0.1487 g, 0.5 mmol) was added slowly to the solution of Tmp (0.2361 g, 0.1 mmol) in 10 mL of methanol. The mixture was refluxed for 9 h and filtered to give a colorless solution. After standing it at ambient temperature for 4 weeks, colorless bulk crystals of **IV** were collected.

For $C_{32}H_{40}N_6O_{10}Zn$ (M = 734.07)

anal. calcd., %:	C, 52.31;	H, 5.45;	N, 11.44.
Found, %:	C, 52.36;	H, 5.43;	N, 11.43.

X-ray crystallography. The single-crystal X-ray data were collection on a Bruker APEX-II CCDC diffractometer with graphite-monochro-matized MoK_α radiation (λ = 0.71073 Å). Absorption corrections were applied using the SADABS program [24]. The crystal structure was solved by direct methods and refined through the full-matrix least-squares method using the SHELXL-97 [25]. Non-hydrogen atoms were refined anisotropically while hydrogen atoms were generated geometrically and treated by a mixture of independent and constrained refinement. Crystallographic data and structural refinements for compound **I**–**IV** are summarized in Table 1, and the selected bond lengths for all compounds are listed in Table 2.

Table 2. Selected bond lengths (Å) for complexes **I**–**IV**

Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å
I		**II**		**III**		**IV**	
Cu(1)–N(1)	1.9974(25)	Mn(1)–N(1)	2.2578(20)	Pb(1)–N(1)	2.5379(26)	Zn(1)–N(1)	2.1561(43)
Cu(1)–N(2)	2.0473(16)	Mn(1)–N(2)	2.2517(17)	Pb(1)–N(2)	2.6510(18)	Zn(1)–N(2)	2.1461(42)
Cu(1)–O(1)	2.1671(31)	Mn(1)–N(3)	2.2629(15)	Pb(1)–O(1)	2.7255(26)	Zn(1)–N(3)	2.1559(29)
		Mn(1)–N(4)	2.2369(15)	Pb(1)–O(2)	2.5530(23)	Zn(1)–N(4)	2.1419(39)
		Mn(1)–O(1)	2.1515(32)	Pb(1)–O(3)	2.6418(31)	Zn(1)–O(1)	2.1440(38)
		Mn(1)–O(2)	2.1784(38)	Pb(1)–O(4)	2.5319(27)	Zn(1)–O(2)	2.1125(31)

Supplementary material for structures **I**–**IV** has been deposited with the Cambridge Crystallographic Data Centre (nos. 88362–88365 for **I**–**IV**, respectively; deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

In order to study the IR bands of complexes **I**–**IV**, the characteristic bands of Tmp, nitrate and acetate anions were applied to fingerprint the functional groups. In the Tmp ligand, the strong bands in the region of 1426–1432 cm^{-1} are assigned to the benzene ring deformation modes and the ν(C=C) in the phenanthroline ligand, and the sharp bands in the region of 1520–1529 cm^{-1} to the ν(C=C–C=N) bands of the phenanthroline ligand, which is in good agreement with the positions reported in the literature [26]. In the IR spectra of the complexes, These bands were observed with an upshift of 6–10 cm^{-1}. At the same time, the weak bands at about 730 cm^{-1} for all of the complexes are ascribed to the ν(C–H) of the phenanthroline ligand. The sharp ν(O–H) stretching vibration bands in the region of 3352–3422 cm^{-1} suggest the presence of water molecules in **I**–**IV**. The presence of nitrate anion can be identified by the sharp ν(O–N) bands in the region 1384–1393 cm^{-1} for **I**, **II**, and **IV**. For complex **III**, the strong ν(C=O) stretching band at 1576 cm^{-1} suggests the presence of acetate anion.

The single-crystal X-ray diffraction analysis revealed that the asymmetrical unit of compound **I** consists of a distorted tetrahedron structure (Fig. 1).

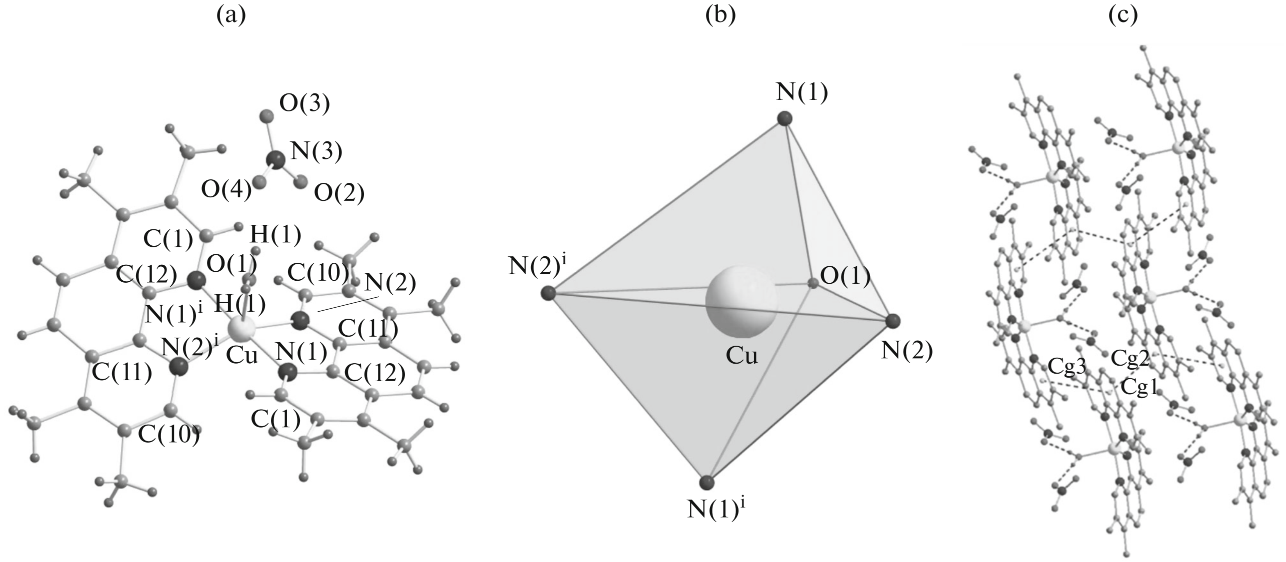

Fig. 1. The perspective view of the structure of **I** (a); highlight of the coordination polyhedron for the crystallographically independent Cu^{2+} ion (b); part of the crystal structure of **I**, supporting by O–H···O hydrogen bonds and week π–π packing interactions. The hydrogen atoms are omitted for clarity (c).

Table 3. Geometric parameters of hydrogen bonds for complexes **I–IV**

D–H···A	Distance, Å			Angle DHA, deg	Coordinates of A atom
	D–H	H···A	D···A		
I					
O(1)–H(1)···O(4)	0.82	1.91	2.716(3)	164	x, y, z
II					
O(1)–H(1*w*)···O(5)	0.82	1.99	2.673(5)	141	$1 - x, -y, 1 - z$
O(1)–H(2*w*)···O(5)	0.83	1.87	2.691(5)	169	$x, y, 1 + z$
O(2)–H(3*w*)···O(4)	0.82	2.06	2.850(6)	162	$1 - x, -y, 1 - z$
O(2)–H(4*w*)···O(6)	0.83	1.89	2.694(6)	165	
O(9)–H(5*w*)···O(8)	0.94	2.20	3.010(5)	145	
O(9)–H(6*w*)···O(7)	0.85	2.34	3.185(5)	180	$x, 1/2 - y, 1/2 + z$
III					
O(5)–H(1*w*)···O(1)	0.83	2.01	2.838(4)	177	x, y, z
O(5)–H(2*w*)···O(4)	0.84	2.02	2.802(4)	155	$1 + x, y, z$
O(6)–H(3*w*)···O(2)	0.83	1.96	2.786(3)	177	$-x, -y, 1 - z$
O(6)–H(4*w*)···O(5)	0.83	2.03	2.827(4)	159	$1 - x, -y, 1- z$
O(7)–H(5*w*)···O(6)	0.84	1.96	2.763(4)	159	x, y, z
O(7)–H(6*w*)···O(3)	0.84	2.07	2.884(4)	164	$-x, 1 - y, 1 - z$
IV					
O(1)–H(1*w*)···O(7)	0.82	1.97	2.749(8)	160	$1 + x, y, z$
O(1)–H(2*w*)···O(3)	0.84	2.01	2.840(6)	171	x, y, z
O(2)–H(3*w*)···O(10)	0.82	2.15	2.711(5)	126	$1 + x, 1 + y, z$
O(2)–H(4*w*)···O(8)	0.82	1.91	2.700(8)	161	$1 + x, y, z$
O(9)–H(5*w*)···O(6)	0.85	1.97	2.823(10)	178	x, y, z
O(9)–H(6*w*)···O(10)	0.82	2.35	3.029(9)	140	x, y, z
O(10)–H(7*w*)···O(5)	0.83	2.28	3.000(8)	146	$-1 + x, y, z$
O(10)–H(8*w*)···O(4)	0.83	2.52	3.067(7)	125	$-1 + x, y, z$

The crystallographic analysis confirmed that the Cu^{2+} ion is coordinated by one oxygen atom of water molecule and four nitrogen atoms from Tmp ligand. The axial position is occupied by N(1) and N(1)^{i} (^{i} $-x$, y, $1/2 - z$), while Cu, N(2), N(2)^{i}, and O(1) atoms lie in one equatorial plane. Two Cu–N bond lengths of complex **I** are, respectively, 1.9974(25) and 2.0473(16) Å. The other bond lengths are listed in Table 2, and these bond lengths are comparable to those found in the reported structures [27]. In complex **I**, the crystal structure is stabilized by intermolecular O–H···O hydrogen bonds (Table 3 and Fig. 1). The coordinated water molecules also participate in hydrogen bonding with oxygen atoms of the nitrate. The crystal packing is further stabilized by π–π stacking interactions between adjacent Tmp molecules. The Cg1···Cg2^{ii} separation is 3.717 Å (Cg1 and Cg2^{ii} are the centroids of the C(4)–C(7)/C(11)–C(12) and C(4)–C(7)/C(11)–C(12) rings, respectively; symmetry code: ^{ii} $-x$, $1 - y$, $1 - z$); the Cg1···Cg3^{iii} separation is 4.234 Å (Cg1 and Cg3^{iii} are the centroid of the C(4)–C(7)/C(11)–C(12) and N(1)/C(1)–C(4)/C(12) rings, respectively; symmetry code: ^{iii} $-x$, $2 - y$, $1 - z$).

The crystal structure of **II** is shown in Fig. 2. The asymmetric unit of compound **II** is formed by $[Mn(Tmp)_2(H_2O)_2]^{2+}$ monomer with two additional nitrate counter-ions balancing the charge and one water molecule. The coordination geometry about the Mn^{2+} ion is six-coordinated by four pyridine ring N atoms and two oxygen atoms of two water molecules in a slightly distorted octahedron arrangement. The measured Mn–N bond lengths, ranging from 2.2369(15) to 2.2629(15) Å, are listed in Table 2. In this case, the crystal structure of the complex is stabilized by an extended network of strong, intermolecular O–H···O hydrogen bonds (Fig. 2). The donors in this interaction are oxygen atoms of coordinated and uncoordinated water molecules; the acceptors are oxygen atoms of nitrate ions. Additionally, the adjacent layers are stacked in a staggered fashion through weak π–π intermolecular interactions between Tmp rings of the neighboring sheets. The Cg1···Cg2^{i} and

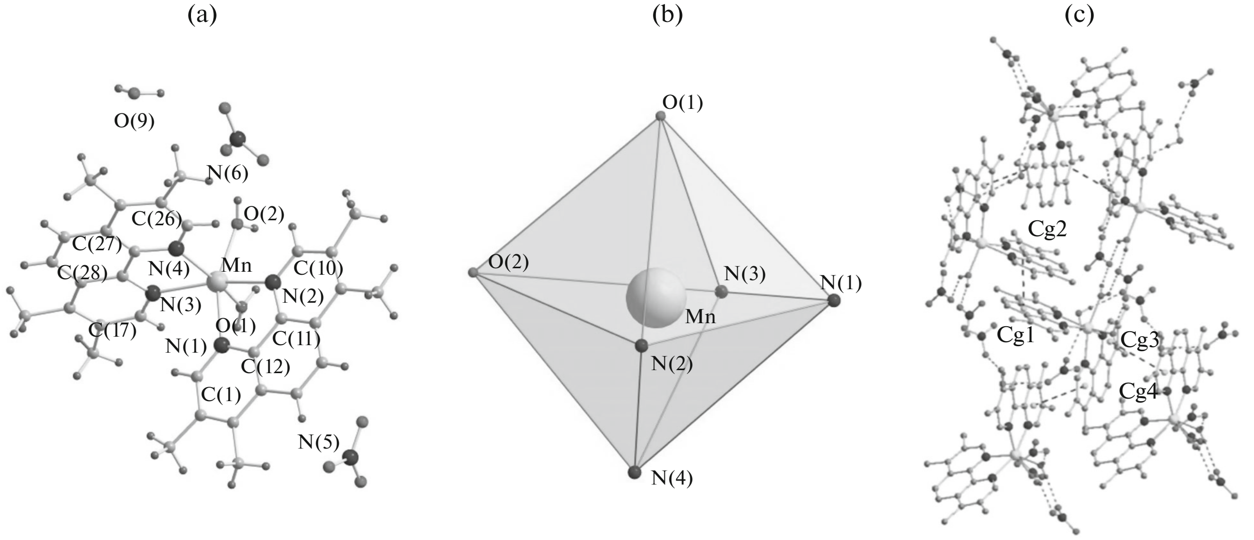

Fig. 2. The perspective view of the structure of **II** (a); highlight of the coordination polyhedron for the crystallographically independent Mn^{2+} ion (b); part of the crystal structure of **II**, supporting by O–H···O hydrogen bonds and week π–π packing. The hydrogen atoms are omitted for clarity (c).

Cg3···Cg4^{ii} distances are 3.786 and is 3.936 Å. Among them, Cg1, Cg2^{i}, Cg3, and Cg4 ^{ii} are the centroid of the C(4)–C(7)/C(11)–C(12), N(2)/C(7)–C(11), N(3)/C(17)–C(20)/C(28) and N(4)/C(23)–C(27) rings, respectively (symmetry codes: ^{i} $1 - x$, $-y$, $-z$; ^{ii} x, $1/2 - y$, $1/2 + z$).

Single-crystal X-ray analysis revealed that this compound consists of one crystallographic independent lead cation, one bidentated Tmp molecule, two uncoordinated water molecules and two coordinated acetate anions as illustrated in Fig. 3. The coordination geometry about the center atom is a six-coordinated distorted monocapped tapered pentagonal cluster with two nitrogen atoms and four oxygen atoms from two coordinated acetate anions. As given in Table 2, two Pb–N bond lengths of the complex are 2.5379(26) and 2.6510(18) Å, respectively. Intermolecular O–H···O hydrogen bonds further stabilize this crystal structure (Table 3 and Fig. 3). Especially, the oxygen atoms of coordinated and uncoordinated water molecules are involved in hydrogen bonds with oxygen atoms of acetate anions. Weak π–π stacking interactions between adjacent Tmp molecules were found as shown in Fig. 3. The Cg1···Cg2^{i} separation is 3.665 Å (Cg1 and Cg2^{i} are the centroid of the N(1)/C(1)–

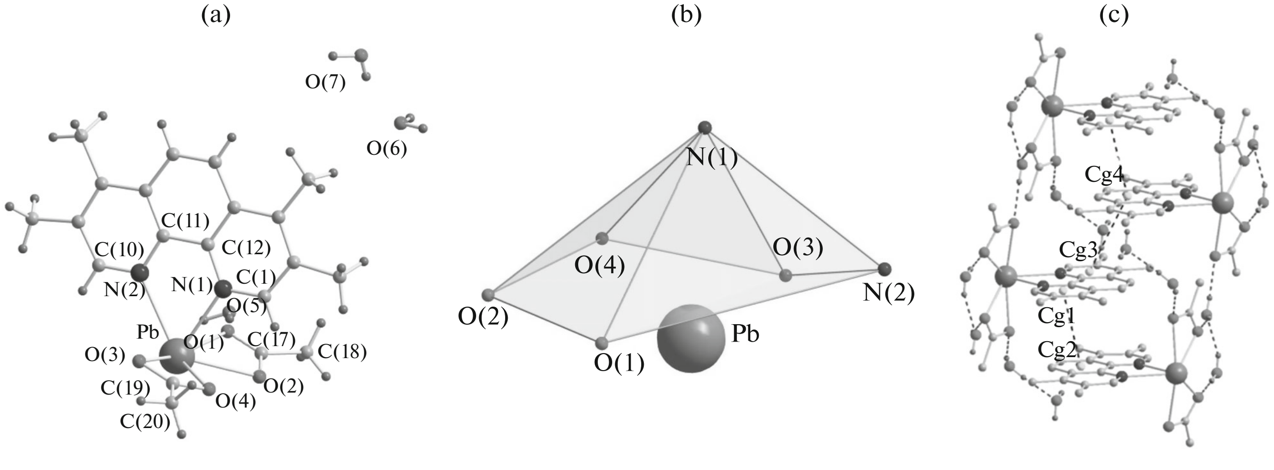

Fig. 3. The perspective view of the structure of **III** (a); highlight of the coordination polyhedron for the crystallographically independent Pb^{2+} ion (b); part of the crystal structure of **III**, supporting by O–H···O hydrogen bonds and week π–π packing. The hydrogen atoms are omitted for clarity (c).

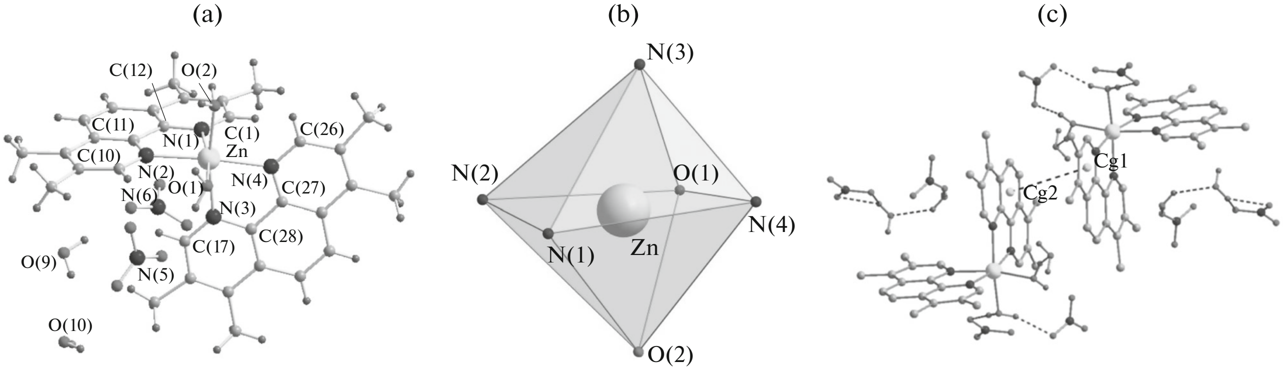

Fig. 4. The perspective view of the structure of **IV** (a); highlight of the coordination polyhedron for the crystallographically independent Zn^{2+} ion (b); part of the crystal structure of **IV**, supporting by O–H···O hydrogen bonds and week π–π packing. The hydrogen atoms are omitted for clarity (c).

C(4)/C(12) and C(4)–C(7)/C(11)–C(12) rings, respectively; symmetry code: ^{i} $-x$, $1-y$, $1-z$); the Cg3···Cg4^{ii} separation is 4.005 Å (Cg3 and Cg4^{ii} are the centroid of the N(2)/C(7)–C(11) and C(4)–C(7)/C(11)–C(12) rings, respectively; symmetry code: ^{ii} $1-x$, $1-y$, $1-z$).

The structure of **IV** is similar to **II** in coordination mode except that this complex consists of two uncoordinated water molecules (Fig. 4). In this case, the Zn^{2+} ion has octahedral coordination environment, and it is built up by four nitrogen atoms of Tmp molecules and two oxygen atoms from two coordinated water molecules. The important Zn–N bond lengths range from 2.1419(39) to 2.1561(43) Å. The crystal structure of this complex is mainly stabilized by strong, intermolecular hydrogen bondings. It can be seen from Fig. 4 and Table 3 that the coordinated water molecules are involved in hydrogen bondings with oxygen atoms of the nitrate [28]. There are also intermolecular π–π stacking interactions responsible for the crystal packing, whereas this interaction occurs between the pyridine and benzene rings (Cg–Cg distance is 3.799 Å) of neighboring molecules.

The loss of coordinated and uncoordinated water molecules of compounds **I**–**IV** was observed with their dehydration temperatures in the range of 80–100°C, and further decomposition began at 322, 316–322, 251–338, and 330°C, respectively. Among them, the

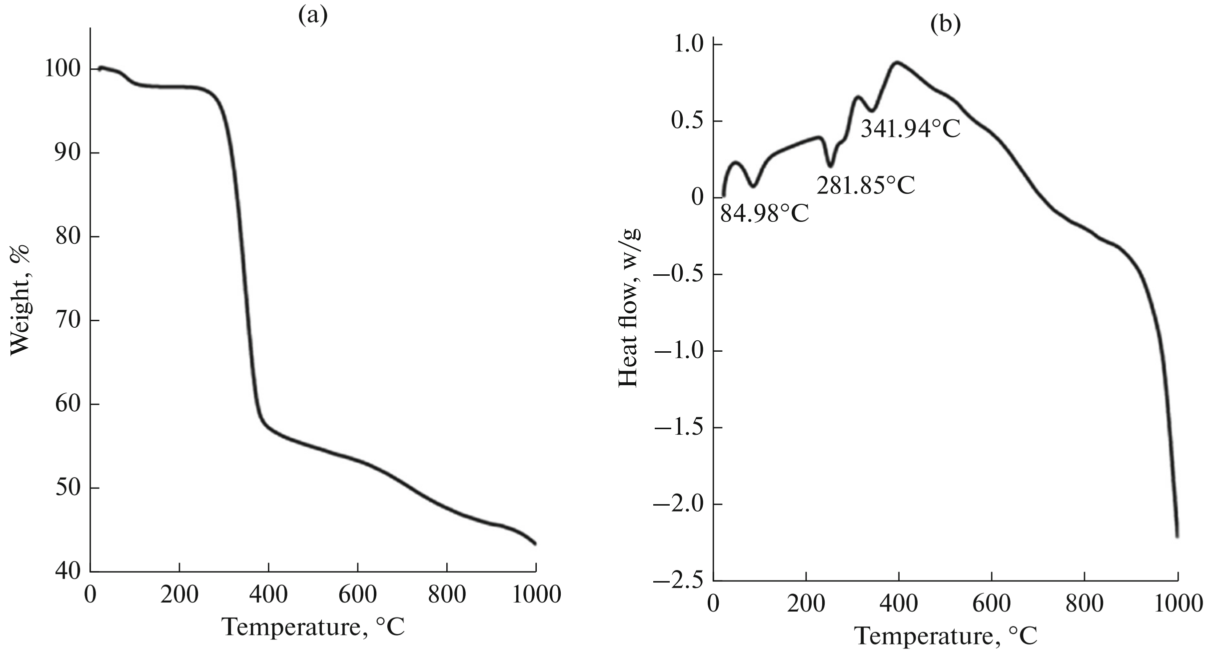

Fig. 5. The TGA (a) and DSC (b) curves of the compound **III**.

typical TG–DSC curves of the compound **III** under nitrogen gas are shown in Fig. 5, which give a relative different decomposition pattern due to the different structure. The first loss of weight starts at ~85°C. The experimental mass loss of 1.62% assigns to the loss of non-coordinated water molecules, agreeing with the calculated value of 1.80%. The second stage from 251 to 338°C is due to the Tmp ligand decomposition, showing a weight loss of 54.91% (calcd. 54.54%). The percentage weight of the final experimental residual PbO_2 is 43.47% (calcd. 38.88%).

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