ISSN 1070-3284, Russian Journal of Coordination Chemistry, 2013, Vol. 39, No. 6, pp. 450–457. © Pleiades Publishing, Ltd., 2013.

Hydrothermal Synthesis, Crystal Structure of Two New Coordination Polymer $\{[Ni_2(Imazameth)_2(4,4'\text{-}Bipy)] \cdot (ClO_4) \cdot CH_2OH \cdot H_2O\}_n$ and $[Cu_2(PDA)_{1.5}(2,2\text{-}Bipy)_2](H_2O)_5(NO_3)$[1]

Z. Zhang and X. H. Yin*

College of Chemistry and Ecological Engineering, Guangxi University for Nationalities, Nanning, 530006 P.R. China

**e-mail: yxhphd@163.com*

Received September 9, 2011

Abstract—Two new metal–organic coordination polymers framework formulated as $[Cu_2(PDA)_{1.5}(2,2'\text{-}Bipy)_2](H_2O)_5(NO_3)$ (**I**) and $\{[Ni_2(Imazameth)_2(4,4'\text{-}Bipy)] \cdot (ClO_4) \cdot CH_2OH \cdot H_2O\}_n$ (**II**), where H_2PDA = 1,4-phenylenediacetic acid, 2,2'-Bipy = 2,2'-bipyridine, Imazameth = (+/–)2-(4,5-dihydro-4-methyl-4-(1-methylethyl)-5-oxo-1H-imidazol-2-yl)-5-methyl-3-pyridinecarboxylic acid, 4,4'-Bipy = 4,4'-bipyridine, have been prepared and characterized by spectral method (IR), elemental analysis and single crystal X-ray diffraction techniques. Complex **I** consists of one-dimensional ladder-like chains featuring binuclear unit in which the two Cu^{2+} ions have different coordination geometry: one is five-coordinated, the other is six-coordinated. Complex **II** is a new two-dimensional copper complex with the peculiarity of having the 4,4'-Bipy ligand and Imazameth ligand acting as bridge to form planar network polymer. Both **I** and **II** exist abundant hydrogen bonds. It is result in the formation of a supermolecular crystal, in which they seem to be effective in the stabilization of the structure.

DOI: 10.1134/S1070328413050114

INTRODUCTION

The self-assembled construction of coordination polymers is of current interest in the field of supramolecular chemistry and crystal engineering, due to their intriguing network topologies and potential applications in functional materials, such as gas storage, selective catalysis, molecular separation and magnetism [1–5]. Construction of coordination polymers using metal ions and rigid organic ligands characterized a fruitful subclass of materials applicable in both the academic and business areas. As far as we know, selecting appropriate ligands is the most effective strategy in obtaining coordination polymers. As bridging ligands, carboxylates, especially multi-carboxylates, are of immense interest in the construction of polymeric coordination architectures not only because the fact that these polymers have a wide range of structural diversities and potential applications as porous materials and magnetic materials, but also because the multi-carboxylates are capable of functioning as hydrogen bond donors and/or acceptors [6]. 1,4-Phenylenediacetic acid (**H_2PDA**) is a typical dicarboxylate. It has two flexible acetates, resulting in *trans*- and *cis* configuration [7].

Much effort has been devoted to the synthesis [8, 9], crystal structure [10, 12] and catalysis [13, 14] of the compounds containing imidazolidinone during the last few years. One of them is (+/–)-2-(4,5-Dihydro-4-methyl-4-(1-methylethyl)-5-oxo-1H-Imidazol-2-yl)-5-methyl-3-pyridinecarboxylic acid (Imazameth), which provides with efficient metal-chelating ability. The Imazameth contains a pyridine carboxylic acid and an imidazole ring, which are well-known versatile ligands. The pyridine carboxylic acid and its anion have been extensively used in the design of coordination compounds, due to a variety of its bonding ability and exhibiting strong hydrogen bonds [15, 17]. Imidazole ring, which is one of the polydentate amine ligands, generally coordinates to metal ions using the N atoms as donors. Hydrogen bonding can play an important role in the stabilization of supramolecular systems both in solution and in the solid state; sometimes metal ions in such structure can also act as glues in holding the water cluster.

The reaction of H_2PDA, 2,2'-Bipy with $Cu(NO_3)_2 \cdot 3H_2O$ was carried out under open-air mild reaction conditions to obtain **I**. And the reaction of Imazameth, 4,4'-Bipy with $Ni(ClO_4)_2 \cdot 6H_2O$ was carried out under hydrothermal conditions to obtain **II**. In this work, we reports synthesis, crystal structure, spectral analysis (IR), elemental analysis, thermal gravimetric analysis (TG).

EXPERIMENTAL

Materials and methods. All reagents and solvents employed were commercially available and used as received without further purication. Elemental analysis

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for C, H, and N was carried out on a PerkinElmer 2400 II elemental analyzer. The FT–IR spectrum was obtained on a PE Spectrum One FT–IR Spectrometer Fourier transform infrared spectroscopy in the 4000–400 cm^{-1} regions, using KBr pellets. PerkinElmer Diamond TG/DTA thermal analyzer was used to record simultaneous TG curves in the static air atmosphere at a heating rate of 10 K min^{-1} in the temperature range 0–800°C using platinum crucibles.

Synthesis of complex I. H_2PDA (0.09705 g, 0.5 mmol) and 2,2'-Bipy (0.07807 g, 0.5 mmol) were dissolved in the mixture of 15 mL N,N-dimethylformamide and 10 mL H_2O. Then an aqueous solution of sodium hydroxide was added dropwise with stirring to adjust the pH value of the solution being 6. At last, 10 mL aqueous solution of $Cu(NO_3)_2 \cdot 3H_2O$ (0.1206 g, 0.5 mmol) was input. The mixture was kept stirring for 6 h at 70°C and then filtered. The filtrate was kept at room temperature and a few days later X-ray quality blue block single crystals were obtained. The crystals were isolated, washed with ethanol and dried at room temperature. The yield was 70% based on Cu).

For $C_{70}H_{76}N_{10}O_{28}Cu_4$

anal. calcd., %:	C, 47.78;	H, 4.35;	N, 7.96.
Found, %:	C, 47.71;	H, 4.37;	N, 7.93.

IR spectrum (ν, cm^{-1}): 3480 br, 3076 w, 1601 s, 1519 m, 1500 m, 1471 m, 1454 m, 1390 s, 1149 w, 1097 s, 851 m, 772 m, 736 m, 619 w.

Synthesis of complex II. A solution of Imazameth (0.3912 g, 1.5 mmol) in 10 mL ethanol was added dropwise with stirring at room temperature to a solution of $Ni(ClO_4)_2 \cdot 6H_2O$ (0.1328 g, 0.5 mmol) in 5 mL water. The mixture was stirred at room temperature until it was homogeneous, and then added to a solution of 4,4'-Bipy (0.1608 g, 1 mmol) in 5 mL ethanol. Then the mixture was sealed in a 25 mL Teflon-lined stainless reactor, kept under autogenous pressure at 100°C for 48 h, and then slowly cooled to room temperature at a rate of 5°C per hour. After several days, green block crystals suitable for X-ray diffraction were separated and washed with distill water and dried at ambient temperature. The yield was 65% based on Ni.

For $C_{21}H_{30}N_4O_{10}ClNi$

anal. calcd., %:	C, 42.56;	H, 5.10;	N, 9.45.
Found, %:	C, 42.49;	H, 5.06;	N, 9.47.

IR spectrum (ν, cm^{-1}): 3392 m, 3063 w, 2973 m, 1755 s, 1608 m, 1526 v.s, 1481 s, 1107 s, 815 w, 622 w.

X-ray structure determination. Crystallographic data of complexes **I** and **II** were collected on a Bruker SMART CCD area detector at 298 K using graphite mono-chromated MoK_α radiation ($\lambda = 0.71073$ Å). Empirical absorption corrections were applied using the SADABS program [18]. The structure was solved by the direct method and refined by full-matrix least squares on F^2 using the SHELXTL program [19]. All non hydrogen atoms were refined anisotronically. A summary of the crystallographic data and structure refinement of complexes **I** and **II** are shown in Table 1, selected bond lengths and angles are listed in Table 2 and hydrogen bonging geometry are given in Table 3.

Supplementary material has been deposited with the Cambridge Crystallographic Data Center (no. 1.822338 (**I**), no. 2.824696 **II**); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

The X-ray crystal structure determination of complex **I** reveals that the asymmetric unit contains two kinds of metal environments (Fig. 1). Cu(1) ion is five-coordinated by two nitrogen atoms N(1), N(2) from one chelating 2,2'-Bipy ligand and three oxygen atoms O(1), O(3), O(5) from three PDA ions. Cu^{2+} ion is six-coordinated by two nitrogen atoms N(3), N(4) from one chelating 2,2'-Bipy ligand and four oxygen atoms O(1), O(2), O(3), O(6) from three PDA ions. The Cu–N bond distances change from 1.966(4) to 2.004(3) Å, which is in agreement with those found in the previously published literatures [20–22]. All of the Cu–O bond lengths are in the normal range, except for Cu(1)–O(1) 2.310(2) Å and Cu(2)–$O(3)^{\#1}$ 2.342(2) Å (symmetry codes: $^{\#1}x - 1, y, z$), which may rise from the Jahn-Teller effect [22].

In complex **I**, PDA ions display *trans* configuration and adopt two kinds of coordination modes which play different roles in the formation of the coordination polymers. The first type is that two oxygen atoms of one carboxyl group chelating coordinate with one metal ion, but one oxygen atom of the two coordinates with another metal ion too; in the other carboxyl group, one oxygen atom coordinates with two metal ions, but the other oxygen atom is free. The other type is each PDA anion acts as a bridge and coordinates to Cu^{2+} ions with all four oxygen atoms. As shown in Fig. 2, the PDA anions adopting the a coordination mode connect Cu^{2+} ions into one-dimensional chains, and the PDA anions adopting the coordination mode connect two chains together forming a one-dimensional infinite ladder-like chain.

In the crystal lattice of complex **I**, five crystallographically independent water molecules O(7), O(8), O(9), O(10) and O(11) and a free NO_3^- anion are observed. There are abundant hydrogen bonds between them; the details of hydrogen bonds are listed in Table 3. As shown in Fig. 3, we can see that O(8), O(10), O(8a), and O(10a) form a tetramer water cluster, at the same time, O(8), O(8a), O(10), O(10a), O(11), O(11a), O(12A), O(12Aa), O(14A), and O(14Aa) form a 10-membered hydrogen bonds ring. The tetramer water clusters and the 10-membered hydrogen bonds rings connect with each other alternate-

Table 1. Crystallographic data and experimental details for complexes **I** and **II**

Parameter	Value	
	I	**II**
Color/shape	Blue/block	Green/block
Formula weight	1759.57	592.65
Crystal system, space group	Triclinic, $P\bar{1}$	Monoclinic, $P2_1/c$
Unit cell dimensions		
a, Å	10.051(4)	10.655(2)
b, Å	13.344(5)	21.227(5)
c, Å	15.453(5)	13.573(3)
α, deg	81.380(5)	90
β, deg	86.974(5)	111.157(3)
γ, deg	69.073(5)	90
Volume, Å^3	1913.9(12)	2862.9(11)
Z	1	4
ρ_{calcd}, g cm^{-3}	1.527	1.375
Absorption coefficient, mm^{-1}	1.185	0.826
F(000)	906	1236
Crystal size, mm	0.41 × 0.39 × 0.37	0.41 × 0.40 × 0.35
θ range for data collection, deg	1.33–26.50	1.87–25.10
Reflections collected	11775	16260
Unique reflections (R_{int})	7757 (0.0369)	5085 (0.0294)
Completeness to θ = 25.10, %	97.8	99.6
Absorption correction	Semi-empirical	Semi-empirical
Parameters	541	369
Goodness-of-fit on F^2	1.097	1.073
R indices ($I > 2\sigma(I)$)	$R_1 = 0.0556$, $wR_2 = 0.1640$	$R_1 = 0.0595$, $wR_2 = 0.1686$
R indices (all data)	$R_1 = 0.0760$, $wR_2 = 0.1916$	$R_1 = 0.0769$, $wR_2 = 0.1864$
Largest diff. features, e Å^{-3}	0.711 and –0.512	0.961 and –0.682

ly forming a one-dimensional hydrogen-bonding chain. A serious view of the water cluster finds that each water monomer acts as both H-bond donor and H-bond acceptor. We can also find that π–π stacking interactions exist between two 2,2'-Bipy molecules from adjacent complex chains. These complicated hydrogen bonds and π–π stacking interactions result in the formation of a 3D supramolecular structure.

The thermogravimetric analyses were carried out in flowing N_2 with a heating rate of 10°C min^{-1} in the temperature range 0–800°C for complex **I**. Two distinct weight-loss stages are observed on the TGA curve of **I**. The first stage, which occurs from 30 to 80°C, is attributed to the loss of five water molecules; the observed weight loss (9.8%) is in agreement with the calculated value (10.23%). The second stage occurs between 203 and 340°C and is due to the decomposition of the uncoordinated NO_3^- anion, two 2,2'-Bipy molecules and half a PDA molecule (exp. 70.66%, calcd. 71.79%). The residual percentage weight (observed 19.01%) at the end of the decomposition of the complex is consistent with the formation of CuO (expected 18.19%).

Table 2. Selected bond lengths and angles for complexes **I*** and **II****

I

Bond	*d*, Å	Bond	*d*, Å
Cu(1)–O(5)	1.947(3)	Cu(1)–O(3)$^{\#1}$	1.968(3)
Cu(1)–N(1)	1.987(3)	Cu(1)–N(2)	1.988(4)
Cu(1)–O(1)	2.311(2)	Cu(2)–O(6)	1.916(3)
Cu(2)–N(4)	1.966(4)	Cu(2)–O(1)	1.999(3)
Cu(2)–N(3)	2.004(3)	Cu(2)–O(3)$^{\#1}$	2.342(2)
Angle	**ω, deg**	**Angle**	**ω, deg**
O(5)Cu(1)O(3)$^{\#1}$	93.39(12)	O(5)Cu(1)N(1)	170.13(13)
O(3)$^{\#1}$Cu(1)N(1)	94.05(14)	O(5)Cu(1)N(2)	90.63(14)
O(3)$^{\#1}$Cu(1)N(2)	171.75(11)	N(1)Cu(1)N(2)	81.20(15)
O(5)Cu(1)O(1)	92.21(11)	N(1)Cu(1)O(1)	95.51(11)
O(3)$^{\#1}$Cu(1)O(1)	79.58(10)	N(2)Cu(1)O(1)	107.48(12)
O(6)Cu(2)N(4)	171.29(15)	O(6)Cu(2)O(1)	92.48(12)
N(4)Cu(2)O(1)	96.23(14)	O(6)Cu(2)N(3)	90.26(14)
N(4)Cu(2)N(3)	81.56(15)	O(1)Cu(2)N(3)	161.31(12)
O(1)Cu(2)O(3)$^{\#1}$	78.23(9)	N(3)Cu(2)O(3)$^{\#1}$	120.38(12)
O(6)Cu(2)O(3)$^{\#1}$	87.34(11)	N(4)Cu(2)O(3)$^{\#1}$	94.18(12)

* Symmetry transformations used to generate equivalent atoms: $^{\#1}$ $x - 1, y, z$.

II

Bond	*d*, Å	Bond	*d*, Å
Ni(1)–N(3)	1.957(4)	N(3)–C(9)	1.372(6)
Ni(1)–N(1)	2.018(3)	N(2)–C(5)	1.337(5)
Ni(1)–N(2)	2.046(3)	N(2)–C(6)	1.348(5)
Ni(1)–O(4)	2.226(4)	N(1)–C(15)	1.310(6)
Cl(1)–O(6)	1.350(15)	N(1)–C(19)	1.342(6)
Cl(1)–O(8)	1.356(15)	N(4)–C(8)	1.306(6)
Ni(1)–O(1)$^{\#1}$	1.947(3)	N(3)–C(8)	1.343(6)
Angle	**ω, deg**	**Angle**	**ω, deg**
N(3)Ni(1)N(2)	80.08(14)	N(2)Ni(1)O(4)	101.82(15)
O(1)$^{\#1}$Ni(1)N(3)	172.54(13)	N(1)Ni(1)N(2)	158.33(15)
O(1)$^{\#1}$Ni(1)N(1)	88.26(14)	O(1)$^{\#1}$Ni(1)O(4)	86.77(14)
N(3)Ni(1)N(1)	96.99(15)	N(3)Ni(1)O(4)	97.50(17)
O(1)$^{\#1}$Ni(1)N(2)	93.09(12)	N(1)Ni(1)O(4)	99.85(15)

** Symmetry transformations used to generate equivalent atoms: $^{\#1}$ $x, -y + 1/2, z - 1/2$.

Table 3. Geometric parameters of hydrogen bonds for complexes **I** and **II***

Contact D–H···A	Distance, Å		Angle D–H···A, deg
	H···A	D···A	
I			
O(7)–H(7B)···O(4)	2.08	2.802(5)	143
O(7)–H(7C)···O(11)	2.3180	2.772(8)	115
O(8)–H(8B)···O(11)	1.82	2.671(9)	179
O(8)–H(8C)···O(10)	1.93	2.764(9)	167
O(10)–H(10C)···O(14A)	2.23	2.98(3)	150
O(10)–H(10C)···O(14Aa)	2.62	3.00(5)	157
O(9)–H(9C)···O(13A)	2.18	2.87(2)	170
O(9)–H(9C)···O(13Aa)	2.36	2.94(3)	126
O(11)–H(11C)···O(12A)	2.13	2.93(5)	112
O(11)–H(11C)···O(12Aa)	2.51	3.00(2)	121
O(11)–H(11D)···O(7)	2.33	2.772(8)	113
II			
O(10)–H(10D)···O(3)	2.28	3.130(8)	176
N(4)–H(4)···O(2)	1.82	2.570(5)	144
O(4)–H(4C)···O(7)$^{\#1}$	2.06	2.898(18)	167
O(4)–H(4C)···O(8')$^{\#1}$	2.09	2.84(4)	140
O(4)–H(4D)···O(10)$^{\#2}$	2.10	2.925(7)	164
O(10)–H(10C)···O(9)$^{\#3}$	1.96	2.80(2)	177

Notes: * Symmetry codes: $^{\#1}$ $-x + 1, -y + 1, -z + 1$; $^{\#2}$ $-x + 1, -y + 1, -z$; $^{\#3}$ $x, -y + 3/2, z - 1/2$.
** D–H is 0.85.

In the IR spectrum of complex **I**, there is a strong broadband centered at 3500 cm^{-1} which can be assigned to the characteristic band of water molecules. The strong bands of the antisymmetric and symmetric stretching vibrations of the carboxylate group are observed around at 1500–1601 and 1390–1471 cm^{-1} for complex **I**. In addition, there are no bands corresponding to stretching of OH of carboxyl groups in complex **I** which is in accordance with full deprotonated PDA ligands.

The X-ray crystal structure determination of complex **II** (Fig. 4) reveals that the local coordination environment around the Ni ion can be best described as a slightly distorted pyramid (5-coordinated) with three N atoms (two N atoms from pyridyl rings and the oxoimidazole rings of Imazethapyr ions, respectively, and one N atom from 4,4'-Bipy) and two O atoms (one O atom from a carboxylate group and the other from H_2O) to complete such a 5-coordinate mode. Thus, each Imazethapyr ion links two Ni ions through one of the two O atoms of the carboxylate moiety and two N atoms of pyridyl and oxoimidazole rings to complex each Ni ion with a stable planar five-membered chelating ring (1Ni2N2C, as shown in Fig. 4). A comparison of the bond lengths in the chelated ring reveals that the pyridine N(2)–Ni bond is significantly longer than the imidazole N(3)–Ni bond. Most probably, this is a consequence of the different basicity of the N(2) and N(3) donors. As usual in such systems, the NNiN angle of the chelated ring (80.08°) is significantly contracted relative to that for the regular pyramid geometry. In fact, complex **II** is a new one-dimensional copper complex with the peculiarity of having the Imazethapyr ligand very unusually acting as bridges, which form polymeric chains along the crystallographic z axis (Fig. 5a). Furthermore, each 4,4'-Bipy ligand links two different Ni atoms from contiguous chains, which forms a two-dimensional condensed framework as depicted in Fig. 5b. In addition, the intramolecular N–H···O and intermolecular O–H···O hydrogen bonds seem to be effective in the stabilization of the structure, resulting in the formation of a supramolecular structure (Fig. 5c).

The strong and broad absorption bands at about 3660–3450 and 1615 cm^{-1} are attributed to the symmetric O–H stretching modes and O–H bending modes of crystal water molecules in the crystal, respectively. Single crystal X-ray data support the presence of water in the complex **I**. The shift to lower frequencies of these stretching modes and the shift to higher frequencies of the accompanying O–H bending modes may be attributed to hydrogen bonding. Other peculiar feature is observed, which exhibits a band at 1369 cm^{-1}. An intense absorption of similar wavenumber was observed previously for a Co(III) complex of Imazapyr coordinating through the deprotonated lactam group [10]. Compared with complex [Mn(Imz)$_2$] [10], complex **II** exhibits the similar wave-

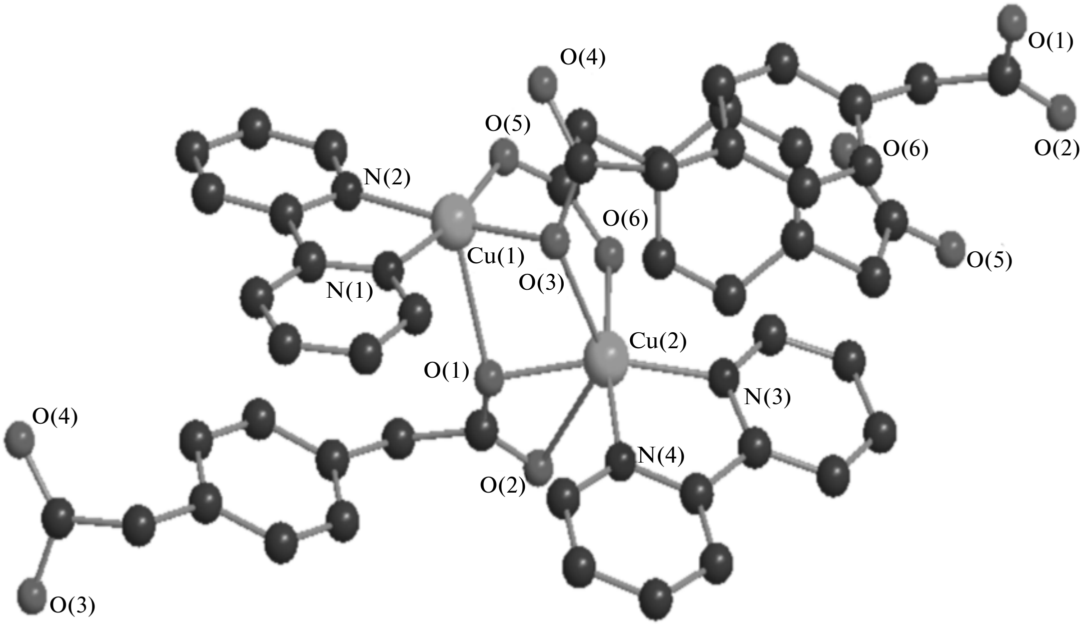

Fig. 1. Coordination environment of Cu^{2+} ions in complex **I**. All H atoms are omitted for clarity.

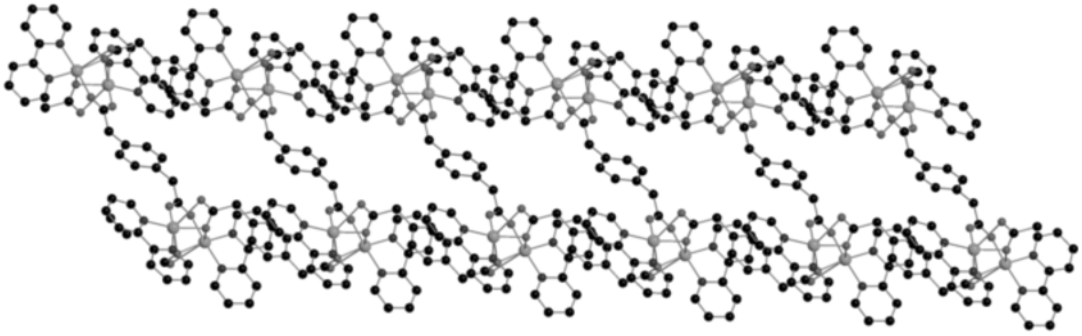

Fig. 2. The 1D ladder-like chain of complex **I**. All H atoms are omitted for clarity.

number values for the carboxylate stretches. The COO groups of the title complex are ν_{as}(COO) 1610 cm^{-1}, ν_s(COO) 1481 cm^{-1}, $\Delta\nu$ = 129 cm^{-1} and the [Mn($Imz)_2$] are 1559, 1416, $\Delta\nu$ = 143 cm^{-1}, respectively. While in complex [Co($Imz)_3$] · $3H_2O$, the COO groups are ν_{as}(COO) 1628, ν_s(COO) 1401, $\Delta\nu$ = 227 cm^{-1}. The difference results form the O atoms of COO groups are engaged in the metal coordination in the Mn(II) and Ni(II) complexes, except for the O···H–N hydrogen bonding, while there is only

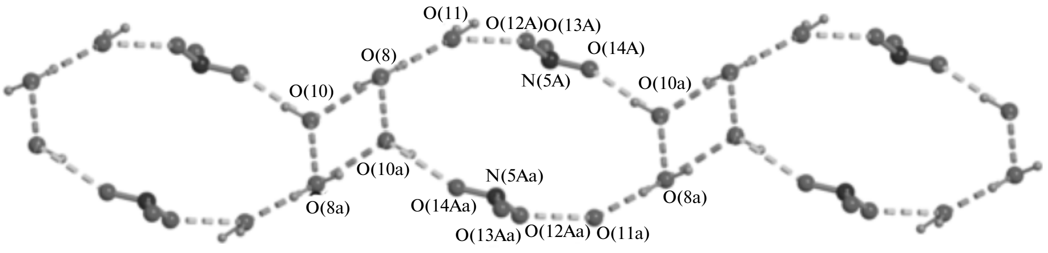

Fig. 3. A view of the one-dimensional hydrogen-bonding chain in complex **I**.

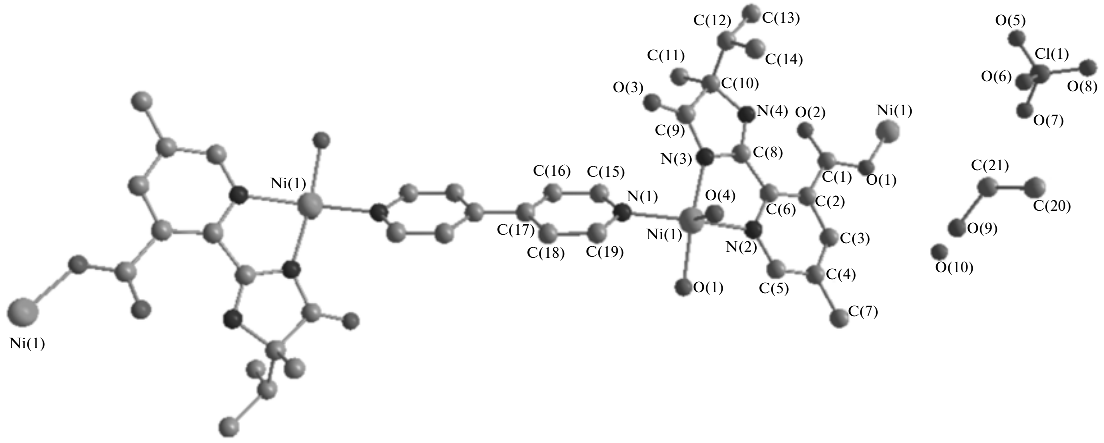

Fig. 4. The coordination environments of Ni^{2+} ions in the asymmetric unit of complex **I**.

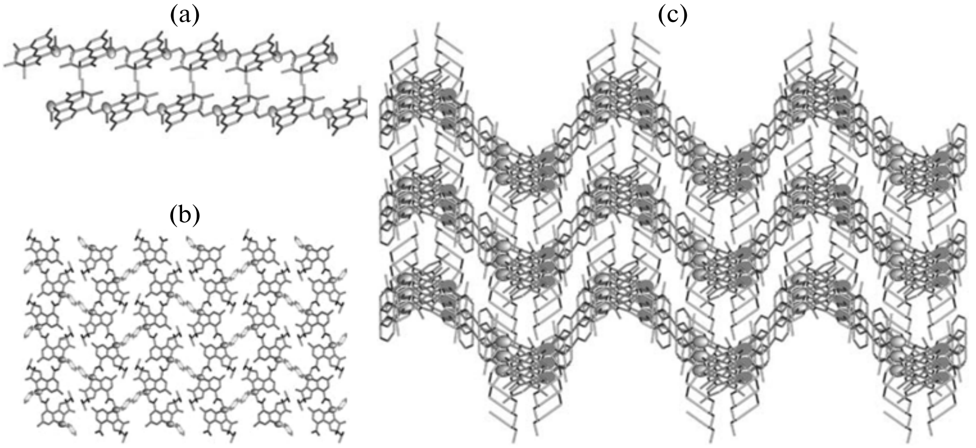

Fig. 5. 1D (a), 2D (b), 3D (c) view of the complex with H atoms are omitted for clarity.

O···H–N hydrogen bonding in the Co(III) complex. As regards the ν(C=N) modes, these are distinguished at 1695 cm^{-1} in Imazameth and at 1608 cm^{-1} in the Ni(II) complex, which is consistent with the $[Mn(Imz)_2]$ complex, 1627 cm^{-1}. In comparison, the $[Co(Imz)_3] \cdot 3H_2O$ derivative exhibits a high wavenumber value (1661 cm^{-1}) consistent with the decreased length of the C=N bond (1.29 Å).

ACKNOWLEDGMENTS

The authors gratefully acknowledge the financial support provided by the National Natural Science Foundation of China (no. 20761002), PR China and Innovation Project of Guangxi University for Nationalities (no. gxun-chx2011084).

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