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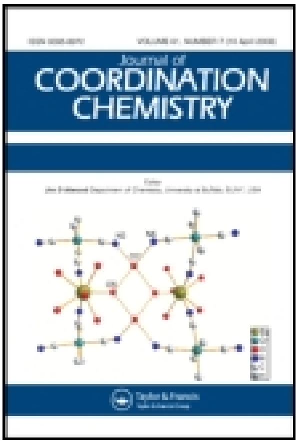

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Two cobalt(II) complexes based on 2-propyl-4,5-imidazoledicarboxylic acid: syntheses, crystal structures, and properties

Zhi-Rong Luo [a], Ji-Chang Zhuang [a], Qiao-Lan Wu [a], Xianhong Yin [a], Sheng-Wei Tan [a] & Ji-Zhong Liu [a]

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

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Two cobalt(II) complexes based on 2-propyl-4,5-imidazoledicarboxylic acid: syntheses, crystal structures, and properties

ZHI-RONG LUO, JI-CHANG ZHUANG, QIAO-LAN WU, XIANHONG YIN*, SHENG-WEI TAN and JI-ZHONG LIU

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

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Two complexes, $[Co(H_2pimdc)_2(H_2O)_2]\cdot 2DMF$ (**1**) and $[Co(H_2pimca)_2(H_2O)_2]\cdot 4H_2O$ (**2**), have been designed, synthesized, and characterized (X-ray single crystal analysis, thermal analysis, electrochemical analysis, and infrared spectrum) based on the imidazole ligand [$H_3pimdc =$ 2-propyl-4,5-imidazoledicarboxylic acid]. Both compounds have 3-D hydrogen-bonding networks. Because of different coordination of water, **1** forms a layer-like geometry *via* hydrogen bonds, but **2** forms a fishingnet-like latticed geometry *via* hydrogen bonds.

Keywords: 2-Propyl-4,5-imidazoledicarboxylic acid; Crystal structure; Thermal and electrochemical property; Electrochemical analysis

1. Introduction

Supramolecular chemistry and metal–organic polymeric networks *via* crystal engineering have produced many new complexes with unique structures [1–11]. Engineering of metal–organic network structures relies on judicious selections of metal and organic ligands that afford desirable covalent dative bonds, hydrogen-bonding linkages, or π–π interactions for the construction of new supramolecular or polymeric complexes. Network structures utilizing both covalent and hydrogen bonds have attracted interest due to their flexible structural features [12].

Imidazole-derived ligands have attracted scientists for bioactivities, materials, catalysis, etc. It is essential to study the syntheses and crystal structures of complexes formed by imidazole systematically to understand the factors that influence the formation and structures of such complexes. We will explore studies that may lead to functional materials and also provide theoretical foundations for supramolecular chemistry and crystal engineering [13–15]. The role of hydrogen bonding in metal-coordinated network structures results in a large number of coordination polymers.

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*Corresponding author. Email: yxhphd@163.com

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Imidazoledicarboxylic acid is a bis-bidentate bridging ligand, having two nitrogens and two carboxyl groups, and can act both as a multiple proton donor and acceptor, and highly accessible to metal ions, to build interesting crystal structures. Water is important for hydrogen bond networks, especially the morphologies of water clusters in diverse chemical environments. Water clusters including tetramers [16], hexamers [17], octamers [18], and decamers [19] have been structurally characterized, with hydrogen-bonding interactions key to the formation of water clusters [20–23].

2. Experimental

2.1. *Materials and analyses*

All reagents and solvents were used as supplied commercially. Elemental analyses (C, H, N, and O) were determined with a Perkin-Elmer model 240°C automatic instrument. Infrared (IR) spectra on KBr pellets were performed on a BRUKER EQUINOX-55 spectrometer from 4000 to 400 cm^{-1}. Perkin-Elmer Diamond TG/DTA thermal analyzer was used to record simultaneous TG and DTG curves in static air at a heating rate of 10 $K\,min^{-1}$ from 25°C to 1000°C using platinum crucibles. Cyclic voltammetry was performed on a CHI 660C electrochemical workstation. The three-electrode system consisted of a platinum wire as a counter electrode, a saturated calomel as a reference electrode, and a modified glassy carbon electrode (3.0 mm diameter) as a working electrode. All electrochemical experiments were carried out in a conventional electrochemical cell holding water/ethanol solution at room temperature.

2.2. *Syntheses of the ligand and complexes*

2.2.1. $[Co(H_2pimca)_2(H_2O)_2]\cdot 2DMF$ (1). A solution of H_3pimdc (0.1982 g, 1 mmol) was dissolved in 20 mL distilled water at room temperature. The pH of the solution was adjusted to 6 with triethylamine, $Co(CH_3COO)_2 \cdot 4H_2O$ (0.1246 g, 0.5 mmol) was dissolved in 20 mL *N*,*N*-dimethylformamide, the two solutions were mixed, and refluxed for 8 h. Then the filtered solution was evaporated at room temperature for 15 days, giving highly pure and well-shaped red block crystals which were washed with distilled water (yield: 78%). Elemental analysis Calcd (%) for $C_{22}H_{36}CoN_6O_{12}$: C, 41.16; H, 5.71; N, 13.22; O, 30.21. Found (%): C, 41.61; H, 5.75; N, 13.19; O, 30.23. IR spectrum (cm^{-1}): 3498(m), 3136(m), 1715(m), 1652(vs), 1578(vs), 1541(vs), 1482(vs), 1393(s), 1273(m), 1117(m), 1054(m), 963(m), 864(m), 780(m), 678(m), 508(w), 493(w).

2.2.2. $[Co(H_2pimca)_2(H_2O)_2]\cdot 4H_2O$ (2). A solution of H_3pimdc (0.1982 g, 1 mmol) was dissolved in 20 mL distilled water at room temperature. The pH was adjusted to 6 with sodium hydroxide (1 $mol\,L^{-1}$), $CoCl_2 \cdot 6H_2O$ (0.1190 g, 0.5 mmol) was dissolved in 20 mL ethanol, the two solutions were mixed, and heated to 70°C for 8 h. Then the filtered solution was evaporated at room temperature for 5 days and highly pure and well-shaped red block crystals were obtained and washed with distilled water (yield: 81.3%). Elemental analysis Calcd (%) for $C_{32}H_{60}CoN_8O_{28}$: C, 36.13; H, 5.69; N, 10.53; O, 42.11. Found (%): C, 36.10; H, 5.72; N, 10.51; O, 42.13.

IR spectrum (cm^{-1}): 3432(s), 3249(s), 1708(s), 1582(vs), 1538(vs), 1489(vs), 1399(s), 1273(s), 1232(w), 1124(m), 1093(w), 1054(m), 1025(m), 951(m), 870(m), 774(m), 660(m), 511(m).

2.3. *X-ray crystallography*

Diffraction experiments for **1** and **2** were carried out with Mo-Kα radiation using a BRUKER SMART APEX CCD diffractometer at 296 K. The structures were solved by direct methods and refined with full-matrix least-squares on F^2 using SHELXS-97 and SHELXL-97 [24]. All non-hydrogen atoms were refined anisotropically. A summary of the crystallographic data and structure refinements is shown in table 1, selected bond lengths and angles of the complexes are listed in table 2, and hydrogen bond geometries are given in table 3.

3. Results and discussion

3.1. *Crystal structures of 1 and 2*

H_3pimdc is a tetradentate ligand with two carboxyl groups and two nitrogens. Cobalt(II) in **1** and **2** are coordinated by two water molecules, two nitrogens, and two $[H_2pimdc]^-$ groups. The N,O chelates form two five-membered chelate rings.

Table 1. Crystal data and structure refinements for **1** and **2**.

Complexes	1	2
Empirical formula	$C_{22}H_{36}CoN_6O_{12}$	$C_{16}H_{30}CoN_4O_{14}$
Formula weight	635.50	561.37
Temperature (K)	296(2)	296(2)
Crystal system	Triclinic	Triclinic
Space group	*P-1*	*P-1*
Unit cell dimensions (Å, °)		
a	7.3280(19)	10.4493(16)
b	9.335(2)	10.6354(19)
c	11.222(3)	11.2827(19)
α	76.889(3)	82.618(8)
β	87.555(3)	83.611(7)
γ	68.781(3)	87.516(8)
Volume (Å^3), *Z*	696.3(3), 1	1235.2(4), 2
Calculated density ($g\,cm^{-3}$)	1.516	1.509
Absorption coefficient (mm^{-1})	0.688	0.768
F(000)	333	586
θ range for data collection (°)	1.87–25.01	1.93–25.01
Data/restraints/parameters	2428/0/191	4298/12/336
Goodness-of-fit on F^2	1.173	1.072
Final *R* indices $[I > 2\sigma(I)]^{a,b}$	$R_1 = 0.0351$, $wR_2 = 0.0968$	$R_1 = 0.0587$, $wR_2 = 0.1830$
R indices (all data)	$R_1 = 0.0365$, $wR_2 = 0.0979$	$R_1 = 0.0660$, $wR_2 = 0.1900$

[a] $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; [b] $wR_2 = [\Sigma[\omega(F_o^2 - F_c^2)^2]/\Sigma[\omega(F_o^2)^2]]^{1/2}$.

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

1			
Co(1)–N(2)	2.1023(15)	O(5)#1–Co(1)–O(5)	180.0
Co(1)–N(2)#1	2.1023(15)	N(2)–Co(1)–O(1)#1	101.81(5)
Co(1)–O(5)#1	2.1078(15)	N(2)#1–Co(1)–O(1)#1	78.19(5)
Co(1)–O(5)	2.1078(15)	O(5)#1–Co(1)–O(1)#1	88.64(6)
Co(1)–O(1)#1	2.1669(15)	O(5)–Co(1)–O(1)#1	91.36(6)
Co(1)–O(1)	2.1669(15)	N(2)–Co(1)–O(1)	78.19(5)
N(2)–Co(1)–N(2)#1	180.0	N(2)#1–Co(1)–O(1)	101.81(5)
N(2)–Co(1)–O(5)#1	87.83(6)	O(5)#1–Co(1)–O(1)	91.36(6)
N(2)#1–Co(1)–O(5)#1	92.17(6)	O(5)–Co(1)–O(1)	88.64(6)
N(2)–Co(1)–O(5)	92.17(6)	O(1)#1–Co(1)–O(1)	180.0
N(2)#1–Co(1)–O(5)	87.83(6)	–	–
2			
Co(1)–O(1)	2.140(3)	N(1)–Co(1)–O(1)	77.67(12)
Co(1)–O(5)	2.168(3)	O(9)–Co(1)–N(3)	89.09(15)
Co(1)–O(9)	2.045(3)	O(10)–Co(1)–N(3)	170.50(14)
Co(1)–O(10)	2.092(3)	N(1)–Co(1)–N(3)	94.12(13)
Co(1)–N(1)	2.118(4)	O(1)–Co(1)–N(3)	97.93(13)
Co(1)–N(3)	2.144(3)	O(9)–Co(1)–O(5)	89.46(14)
O(9)–Co(1)–O(10)	90.60(16)	O(10)–Co(1)–O(5)	93.20(14)
O(9)–Co(1)–N(1)	169.46(14)	N(1)–Co(1)–O(5)	101.04(13)
O(10)–Co(1)–N(1)	87.87(14)	O(1)–Co(1)–O(5)	175.01(11)
O(9)–Co(1)–O(1)	91.95(14)	N(3)–Co(1)–O(5)	77.30(12)
O(10)–Co(1)–O(1)	91.57(14)	–	–

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

Table 3. Hydrogen-bonding geometries for **1** and **2**.

D–H···A	Symmetry code	D–H (Å)	H···A (Å)	D···A (Å)	D–H···A (°)
Compound **1**					
N(1)–H(1)···O(6)	$x-1$, $y+1$, z	0.86	1.84	2.679(2)	166.2
O(5)–H(5A)···O(4)	$-x+1$, $-y+1$, $-z+1$	0.89	1.89	2.776(2)	173.9
O(5)–H(5B)···O(4)	$x+1$, $y-1$, z	0.86	2.12	2.942(2)	161.7
Compound **2**					
N(2)–H(2)···O(14)	–	0.86	1.90	2.751(6)	171.5
O(3)–H(3)···O(2)	–	0.82	1.75	2.521(5)	155.4
O(7)–H(7)···O(6)	–	0.82	1.67	2.469(5)	163.9
O(14)–H(14G)···O(11)	–	0.85	1.84	2.673(8)	165.4
N(4)–H(4)···O(13)	$-x+2$, $-y+1$, $-z+1$	0.86	1.90	2.728(6)	160.5
O(9)–H(9A)···O(8)	$-x+2$, $-y+1$, $-z+2$	0.85	1.89	2.740(4)	177.3
O(9)–H(9B)···O(12)	x, y, $z+1$	0.85	1.82	2.668(5)	177.1
O(10)–H(10A)···O(4)	$-x+1$, $-y+2$, $-z+1$	0.85	1.96	2.807(5)	174.3
O(10)–H(10B)···O(8)	$x-1$, y, z	0.85	2.06	2.906(4)	174.6
O(11)–H(11C)···O(1)	x, y, $z-1$	0.85	1.91	2.740(7)	163.8
O(11)–H(11D)···O(1)	$-x+1$, $-y+2$, $-z+1$	0.85	2.30	2.805(7)	117.9
O(11)–H(11D)···O(2)	$-x+1$, $-y+2$, $-z+1$	0.85	2.52	3.350(8)	164.4
O(12)–H(12A)···O(2)	$-x+1$, $-y+2$, $-z+1$	0.85	1.96	2.806(5)	172.2
O(12)–H(12B)···O(5)	$-x+1$, $-y+1$, $-z+1$	0.85	2.06	2.900(5)	172.5
O(12)–H(12B)···O(6)	$-x+1$, $-y+1$, $-z+1$	0.85	2.63	3.201(5)	126.1
O(13)–H(13C)···O(11)	x, $y-1$, z	0.85	1.78	2.581(9)	156.5
O(13)–H(13D)···O(12)	$-x+1$, $-y+1$, $-z$	0.85	2.07	2.871(7)	158.1
O(14)–H(14H)···O(7)	$-x+2$, $-y+1$, $-z+1$	0.85	2.07	2.904(5)	166.6

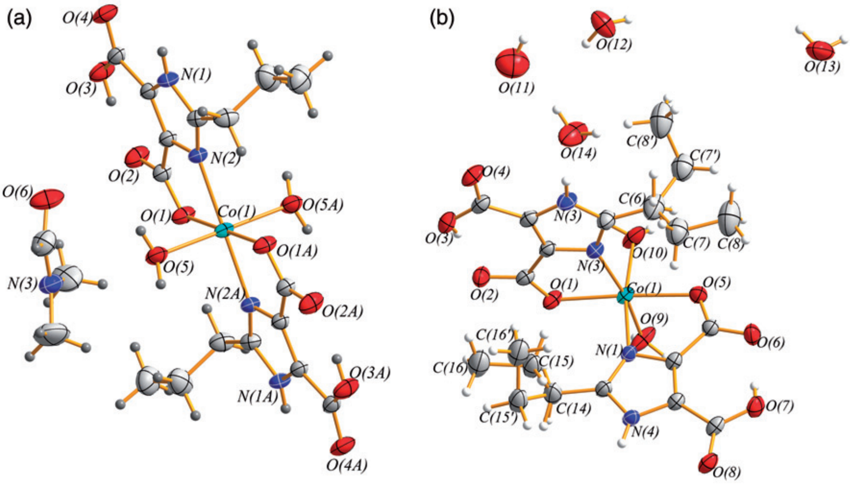

Figure 1. The ORTEP of coordination environments of **1** and **2** with thermal ellipsoids at 30% probability.

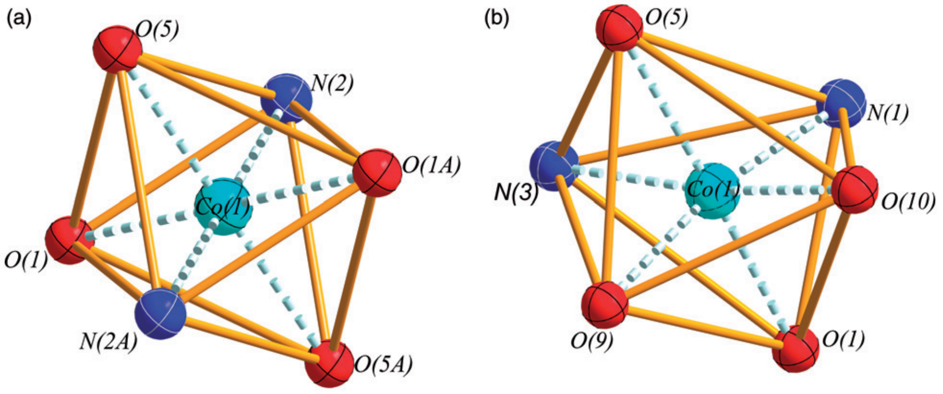

Figure 2. The coordination polyhedra of **1** and **2**.

Each Co(II) is six-coordinate by two nitrogens and two oxygens of carboxylate from two H_3pimdc ligands and two water molecules.

The molecular structure of **1**, depicted in figure 1(a), has two symmetrical coordination water molecules, an interstitial *N*,*N*-dimethylformamide, and two imidazoledicarboxylates. Two nitrogens and four oxygens form octahedral geometry, the Co(1)–L (L = O, N) distances are shown in table 2, and its structure is shown in figure 2(a). Complex **2** forms a stratiform coordination polymer *via* hydrogen bonding (figure 3a) between O5 and O4, the *N*,*N*-dimethylformamide exists *via* hydrogen bonding between the N1 and O6, the distances are shown in table 3. The two O5 and two O4-atoms form a parallelogram geometry (figure 4a). There are $\pi\cdots\pi$ stacking interactions between one imidazole ring and the other imidazole ring in the complex **1**

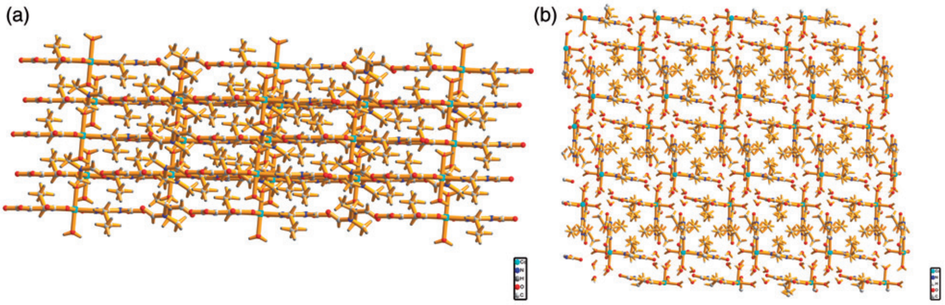

Figure 3. The packing diagrams of **1** and **2**.

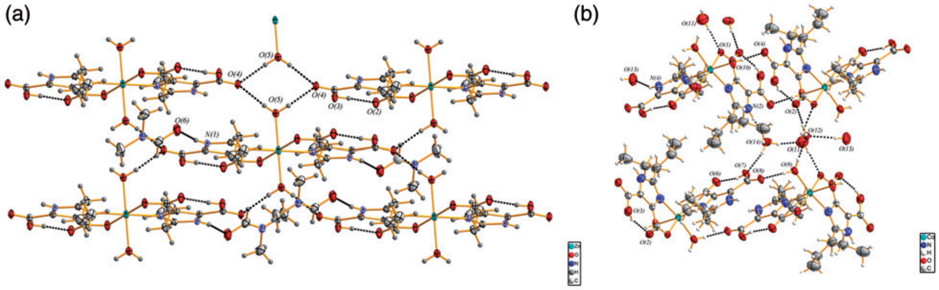

Figure 4. A view of the crystal packing of **1** and **2** showing O–H···O hydrogen bonds. Displacement ellipsoids are drawn at the 30% probability level and hydrogen bonds are indicated by dashed lines.

(figure 5a); the plane distance and dihedral angle is 3.414(10) Å and 0° between one plane (N1/C4/C3/N2/C5) and the other plane (N1/C4/C3/N2/C5).

Complex **2** has the same metal center as **1** (cobalt(II) ion), but the coordination environment of Co(II) is different. The molecular structure of **2** is depicted in figure 1(b) and selected molecular geometry parameters are listed in table 2. The geometry around this cobalt(II) can be described as distorted octahedral in which the two nitrogens are ipsilateral in the distorted octahedron (figure 2b). Analysis of the crystal packing of **2** reveals the existence of multiple intermolecular O–H···O hydrogen bonds between mononuclear subunits and nomadic water (figure 3b) forming a fishingnet-like latticed structure (from *b*-axis to observe it). In this mode, the interstitial water molecules connect complex molecules (figure 4b). The center of the latticed structure has four disordered propyl groups (C7, C8 disordered over two distinct sites with occupancy factors of 0.718 and 0.282, respectively. C15 and C16 are disordered over two distinct sites with occupancy factors of 0.640 and 0.360, respectively). The structure down the *b*-axis looks like a butterfly (figure 5b). Two imidazole rings are also assembled in the (010) direction by $\pi\cdots\pi$ stacking interaction between one molecule and the other molecule defined by N1/C2/C3/N2/C5 (plane–plane distance 3.271 Å, dihedral angle 0°). There is one more $\pi\cdots\pi$ stacking interaction between one molecule and the

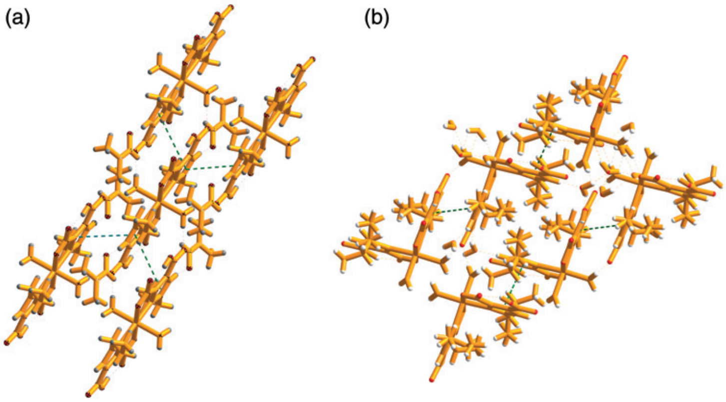

Figure 5. The π–π stacking interactions of **1** and **2**.

other molecule defined by N3/C13/N4/C11/C10 (plane–plane distance 3.318 Å, dihedral angle 0°).

3.2. *Vibrational spectroscopy*

Complexes **1** and **2** show very similar IR spectra. Strong and broad absorptions from 3400 to 3500 cm^{-1} in **1** and **2** indicate the presence of –OH. Strong absorptions of –NH in **1** and **2** occur at 3100–3400 cm^{-1}. For **1**, the absorptions at 1715(1652) and 1482(1393) cm^{-1} correspond to ν_{as}(OCO) and ν_s(OCO), respectively. For **2**, very strong absorptions at 1708(1582) cm^{-1} can be assigned to ν_{as}(OCO) and the bands at 1489(1399) cm^{-1} can be attributed to ν_s(OCO). The differences between ν_{as}(OCO) and ν_s(OCO) for **1** are 233(249) cm^{-1} and for **2** are 219(183) cm^{-1}, indicative of monodentate carboxylate [25], consistent with the crystal structures.

3.3. *Electrochemical studies*

Complexes **1** and **2** were dissolved in distilled water and ethanol at room temperature and electrochemical studies with modified glassy carbon electrodes were carried out in 0.1 $mol\,L^{-1}$ lithium perchlorate aqueous solution. As shown in "Supplementary material", **1** showed a quasi-reversible redox couple in aqueous medium [26], attributed to redox of Co(II)/Co(I), and the mean peak potential $E_{1/2} = (E_{pa} + E_{pc})/2$ was 395 mV. While for **2**, a quasi-reversible redox peak attributed to redox of Co(II)/Co(I) was observed. The mean peak potential $E_{1/2} = (E_{pa} + E_{pc})/2$ was 384 mV for **2**.

3.4. *Thermal analysis*

TG–DTG curves of **1** with a heating rate of 10°C min^{-1} are shown in "Supplementary material". The DTG indicates that **1** undergoes decomposition in four stages. The first stage from 101°C to 177.7°C corresponds to release of two DMF molecules from

$[Co(H_2pimca)_2(H_2O)_2]\cdot 2DMF$ with weight loss of 22.82% (calculated 22.93%). The second stage occurs from 177.7°C to 238.3°C with a mass loss of 6.05% (calculated 5.65%) that correlate with the elimination of two coordinated water molecules for **1**. The third stage occurs from 238.3°C to 319.4°C corresponding to the loss of a $[H_2pimdc]^-$ ($C_8H_9N_2O_4$) for **1** with 30.7% (actual value: 30.9%). The fourth stage takes place at 319.4–471.6°C corresponding to the release of $C_6H_{10}N_2$ from **1** with a weight loss of 17.6% (calculated 17.28%). Finally, the compound becomes CoC_2O_4 at 471.6°C.

The TG–DTG curves of **2** (Supplementary material) indicate that **2** undergoes decomposition in five stages. The first stage at 44.5–73.6°C corresponds to the release of two water molecules ($H_2O(13)$ and $H_2O(14)$) from $[Co(H_2pimca)_2(H_2O)_2]\cdot 4H_2O$ with a weight loss of 6.43% (calculated 6.42%). The second stage at 73.6–104.3°C with a mass loss of 6.52% (calculated 6.42%) correlates with the elimination of two free water molecules ($H_2O(11)$ and $H_2O(12)$) for **2**. The third stage occurs from 104.3°C to 135.3°C with a mass loss of 6.04% (calculated 6.42%) that correlates with the elimination of two coordinated water molecules. Because of hydrogen bonds of four free water molecules, they decompose in different temperature ranges. The fourth stage takes place at 248–327°C corresponding to the release of a $[H_2pimdc]^-$ ($C_8H_9N_2O_4$) with a weight loss of 35.28% (calculated 35.12%). The fifth stage takes place from 327°C to 478.3°C corresponding to the release of $C_6H_{10}N_2$ from **2** with a weight loss of 20.19% (calculated 19.62%). The resulting compound becomes CoC_2O_4 at 478.3°C.

4. Conclusion

We report synthesis and characterization of $[Co(H_2pimca)_2(H_2O)_2]\cdot 2DMF$ and $[Co(H_2pimca)_2(H_2O)_2]\cdot 4H_2O$. The two complexes both crystallize in the triclinic space group *P-1* with imidazoledicarboxylate a bidentate N, O chelate. The symmetric unit of **1** contains a DMF and coordinated water with a layer-like topology *via* hydrogen bonds. Two O4 and two O5 form a parallelogram *via* hydrogen bonds. These chains of **1** join by intermolecular interactions, hydrogen bonds, and $\pi\cdots\pi$ stacking interactions forming a 3-D solid-state structure. Complex **2** contains four free water molecules and two coordinated water molecules, which form a window-like latticed structure by intermolecular interactions, hydrogen bonds, and $\pi\cdots\pi$ stacking interactions. This study helps to explore the structural variations of 2-propyl-4,5-imidazoledicarboxylic acid with different metals and auxiliary ligands.

Supplementary material

CCDC 769658 and CCDC 777320 contain the supplementary crystallographic data for this article. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

Acknowledgments

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