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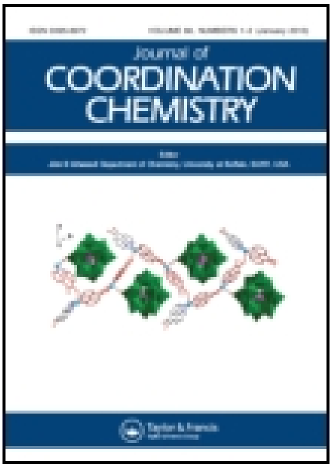

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Cobalt(II) and copper(II)-diphenate complexes with 4-methylimidazole: linkage isomerism and weak C-H···Cu interaction

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Cobalt(II) and copper(II)-diphenate complexes with 4-methylimidazole: linkage isomerism and weak C–H···Cu interaction

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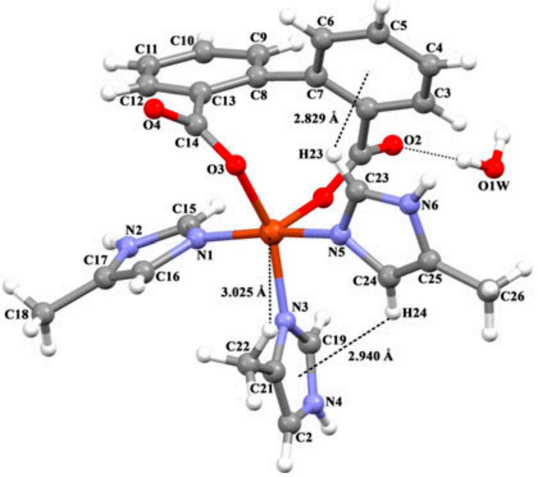

The molecular structure of **2** with intramolecular C−H···Cu, C−H···π and hydrogen bonding interactions.

New Co(II) and Cu(II) diphenate complexes with 4-methylimidazole were synthesized and characterized through elemental analysis, magnetic susceptibilities, and IR spectroscopic studies. The crystal structures of [Co(dpa)(5-meim)$_4$]·2H$_2$O (**1**) and [Cu(dpa)(4-meim)(5-meim)$_2$]·H$_2$O (**2**) were determined by single-crystal X-ray diffraction (H$_2$dpa = diphenic acid, 4-meim = 4-methylimidazole and 5-meim = 5-methylimidazole). In **1**, Co(II) has distorted octahedral geometry with four 5-meim and one diphenate ligands. Complex **2** has distorted trigonal bipyramidal geometry with one 4-methylimidazole, two 5-methylimidazole, and one diphenate ligands. In the complexes, the diphenate is coordinated to the metal(II) ions via the deprotonated oxygens of carboxylate as a bidentate ligand.

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The 4-meim and 5-meim linkage isomers within the same complex are found, and this complex is an unusual example. Moreover, another interesting feature of **2** is the presence of C–H···Cu weak hydrogen-bonding interactions.

Keywords: Linkage isomerism; Mixed ligand complex; Cobalt(II) complex; Copper(II) complex; Diphenate complexes

1. Introduction

Coordination compounds have attracted interest due to their chemical, biological, environmental, catalytic, and ion-exchange importance. Several strategies have been developed to synthesize mixed ligand complexes of transition metal(II) ions containing nitrogen donor ligands [1].

Selection of ligand and metal ion is important in the construction of coordination compounds. The coordination geometry of the central atom, the structure of the organic ligand, the pH of the reaction solution, reaction time, and temperature can also affect the final structure [2, 3]. Various coordination modes and linkage of metal ions in different ways by the ligand play crucial roles in constructing coordination compounds. Aromatic carboxylates as a multifunctional ligand play an important role in coordination chemistry. Especially, aromatic polycarboxylate ligands, such as diphenic acid (H_2dpa) have been extensively used in the synthesis of various complexes due to their rich coordination modes (monodentate, chelating, and/or bridging), which give functional characteristics [4, 5].

We selected H_2dpa as the first ligand based on the following considerations. First, H_2dpa has two carboxylate groups that can be completely or partially deprotonated and may serve as potential anions [6]. This makes it very desirable for the design of coordination polymers with interesting structures and properties [7–10]. Second, in deprotonated H_2dpa, two phenyl rings are not coplanar with each other, owing to the steric hindrance of carboxylate groups in the coordination process. The distortion of the diphenyl spacer about the central bond allows dpa to link metal ions into helical chains or 1-D chains [11–13]. Based on these considerations, constructing coordination compounds from carboxylates and nitrogen-containing mixed ligands has become our current interest. For this reason, 4-methylimidazole was selected as the secondary ligand. The 4-methylimidazole ligand can exhibit linkage isomerism that is typical for the range of ambidentate ligands [14–19]. The preference of different coordination modes depends on the relative basicity of the nitrogen in the imidazole ring and steric effect imposed by the methyl group.

In this study, we report the synthesis, crystal structures, spectroscopic (IR), and thermal properties of cobalt(II) and copper(II)-diphenate complexes with 4-methylimidazole.

2. Experimental

2.1. *Materials and instrumentation*

All chemicals were of analytical grade and were purchased commercially. Elemental analyses for C, H, and N were carried out by standard methods (TUBITAK Marmara Research Center, Turkey). IR spectra of the complexes were determined as KBr disks using a Bruker Tensor 27 FT-IR spectrometer from 4000 to 400 cm^{-1}; magnetic susceptibility measurements were performed using a Sherwood Scientific MXI model Gouy magnetic balance at room temperature. A Perkin Elmer Diamond TG/DTA Thermal Analyzer was used to record simultaneous TG, DTG, and DTA curves in static air at a heating rate of 10 °C min^{-1} from 30 to 1000 °C using platinum crucibles. The decomposition enthalpies (ΔH, kJ/mol) of each stage were examined by differential scanning calorimetry (DSC) at a heating rate of 10 °C min^{-1} in a Seiko DSC 6200 (Exstar 6000, Seiko Instruments Inc.).

2.2. *Crystallographic analyses*

Crystallographic data for **1** and **2** were recorded on a Bruker Kappa APEXII CCD area-detector diffractometer using Mo Kα radiation ($\lambda = 0.71073$ Å). Absorption corrections by multi-scan were applied. The structures were solved by direct methods using Olex2 [20] and SHELXS97 [21] with anisotropic thermal parameters for all non-hydrogen atoms. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares methods (SHELXL-97) [21]. Molecular drawings were obtained using Mercury [22].

2.3. *Synthesis of complexes*

2.3.1. [Co(dpa)(5-meim)$_4$]·2H$_2$O (1). A solution of diphenic acid (0.5 g, 2 mmol) in methanol (15 mL) was added dropwise with stirring at 50 °C to a solution of Co $(CH_3COO)_2 \cdot 4H_2O$ (0.52 g, 2 mmol) in water (15 mL). The solution immediately precipitated and was stirred for 1 h at 50 °C. The addition of 4-meim (0.781 g, 0.59 mmol) in water (10 mL) to the precipitated mixture resulted in a clear solution. The mixture was stirred for 2 h at 50 °C and then cooled to room temperature. The crystal growth of **1** was observed after six days. The crystals formed were filtered and washed with 10 mL of water and dried in air.

Elemental analysis and IR spectrum confirmed the formula of **1**. Elemental Anal. Calcd for $C_{30}H_{36}CoN_8O_6$ (%): C, 55.81; H, 5.31; N, 17.36. Found (%): C, 53.98; H, 5.27; N, 17.24.

Main IR features (cm^{-1}, KBr pellet): 3506(s, br), 3332(s), 3120(vs), 3003(s), 2868(s), 1560(vs), 1438(s), 1440(m), 1390(vs), 1238(m), 1155(m), 1107(w), 952(w), 819(m), 756 (m), 721(m) and 663(s).

2.3.2. [Cu(dpa)(4-meim)(5-meim)$_2$]·H$_2$O (2). Complex **2** was synthesized by a similar method as that described for **1**, with $Co(CH_3COO)_2 \cdot 4H_2O$ replaced by $Cu(CH_3COO)_2 \cdot H_2O$ (0.41 g, 2 mmol). *N,N*-Dimethylformamide was also used in **2** as a solvent (10 mL).

Elemental analysis and IR spectrum confirmed the formula of **2**. Elemental Anal. Calcd for $C_{26}H_{28}CuN_6O_5$: C, 54.97; H, 4.97; N, 14.79(%). Found: C, 55.58; H, 4.92(%); N, 14.90.

Main IR features (KBr pellet, cm^{-1}): 3456(s, br), 3141(s), 3012(s), 2916(s), 2638(m), 1556(vs), 1496(m), 1440(m), 1390(vs), 1271(m), 1236(m), 1159(w), 1099(m), 1014(w), 968(w), 827(m), 752(m), 659(m), 621(m) and 534(m).

3. Results and discussion

The crystal data and refinement details of the complexes are given in table 1. Selected bond distances, bond angles, and hydrogen-bonding geometries of **1** and **2** are listed in tables 2 and 3, respectively.

Table 1. Crystal data and structure refinement parameters for **1** and **2**.

	1	**2**
Empirical formula	$C_{30}H_{36}CoN_8O_6$	$C_{26}H_{28}CuN_6O_5$
Formula weight	663.60	568.08
Temperature (K)	100	
Wavelength (Å)	Mo Kα/ 0.71073	
Crystal system	Orthorhombic	Triclinic
Space group	*Pnna*	*P1*
a (Å)	21.4561(4)	7.8483(3)
b (Å)	19.1348(4)	9.6899(5)
c (Å)	7.6485(1)	10.3186(5)
α (°)	90	114.043(5)
β (°)	90	102.829(4)
γ (°)	90	101.923(4)
V (Å^3)	3140.15(10)	658.72(5)
Z	4	1
Absorption coefficient (mm^{-1})	0.60	0.88
D_{Calcd} (Mg m^{-3})	1.404	1.432
Crystal size (mm)	0.25 × 0.10 × 0.10	0.30 × 0.25 × 0.20
θ range for data collection (°)	2.3–26.3	2.3–29.3
Measured reflections	9877	5179
Independent reflections	3223	3305
Absorption correction	Integration Stoe X–RED (Stoe and Cie, 2001)	
Refinement method	Full-matrix least-squares on F^2	
R_{int}	0.025	0.044
$R[F^2 > 2\sigma(F^2)]$	0.030	0.069
$wR(F^2)$	0.079	0.223
Goodness-of-fit on F^2	1.06	1.04
Flack parameter	–	−0.01(3)
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å^{-3})	0.32, −0.34	1.43, −0.59

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

Bond lengths (Å)			
Co1−N1	2.116(1)	Co1−O1	2.171(1)
Co1−N3	2.150(1)		
Angles (°)			
N1−Co1−N1^{i}	166.60(7)	N3−Co1−O1	84.70(4)
N1−Co1−N3	95.01(5)	N1−Co1−O1^{i}	83.27(4)
N1−Co1−N3^{i}	94.78(5)	N3−Co1−O1^{i}	170.43(4)
N3−Co1−N3^{i}	86.07(7)	O1−Co1−O1^{i}	104.63(5)
N1−Co1−O1	88.54(4)		
D−H···A	d(H···A) (Å)	d(D···A) (Å)	<(DHA) (°)
N2−H2···O2^{ii}	2.02(1)	2.8944(16)	173(2)
N4−H4···O1W	1.92(1)	2.7905(17)	168(2)
O1W−H11···O2^{iii}	2.07(2)	2.9037(15)	167(2)
O1W−H12···O2^{iv}	1.88(2)	2.7410(16)	175(2)

Note: Symmetry codes: (i) $x, -y + 1/2, -z + 1/2$; (ii) $x, y, z - 1$; (iii) $-x + 1, -y, -z + 1$; (iv) $x - 1/2, y, -z + 1$.

3.1. *Crystal structure*

The X-ray crystallographic analysis shows that **1** crystallizes in the monoclinic space group *P2$_1$/c*. As shown in figure 1, the molecular structure of **1** consists of one Co(II), one dpa, four 5-meim ligands, and two crystal water molecules. The Co1 ion is six-coordinate in a

Table 3. Selected bond distances (Å) and angles (°) for **2**.

Bond lengths (Å)			
Cu1−O1	2.210(5)	Cu1−O3	2.002(5)
Cu1−N1	1.918(12)	Cu1−N3	2.007(7)
Cu1−N5	1.948(10)		
Angles (°)			
N1−Cu1−N5	169.9(5)	O3−Cu1−N3	152.6(3)
N1−Cu1−O3	90.6(4)	N1−Cu1−O1	91.9(4)
N5−Cu1−O3	87.4(3)	N5−Cu1−O1	98.0(3)
N1−Cu1−N3	86.3(4)	O3−Cu1−O1	113.6(2)
N5−Cu1−N3	90.9(3)	N3−Cu1−O1	93.8(2)
D–H···A	d(H···A) (Å)	d(D···A) (Å)	<(DHA) (°)
N2−H2···O1W^{i}	1.82	2.662(16)	159
N4−H4···O4^{ii}	1.91	2.713(9)	152
N6−H6···O2^{iii}	1.89	2.767(11)	178
O1W−H11···O2	1.97	2.806(10)	178
O1W−H12···O4^{iv}	1.91	2.752(12)	178

Note: Symmetry codes: (i) x, $y-1$, z; (ii) $x+1$, $y+1$, $z+1$; (iii) $x-1$, y, z; (iv) $x+1$, $y+1$, z.

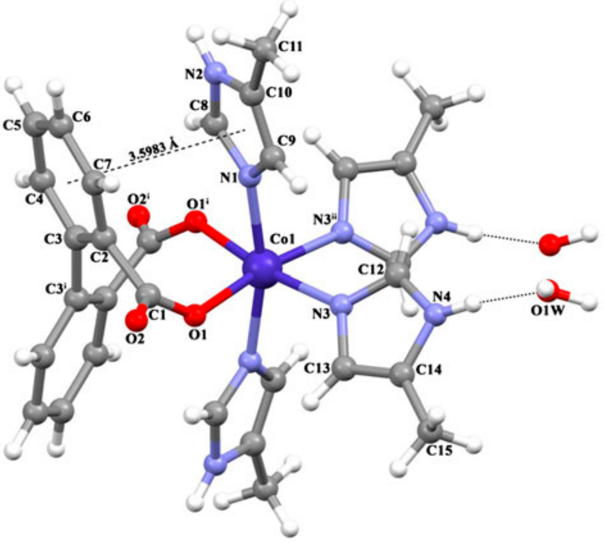

Figure 1. The molecular structure of **1** with intramolecular $\pi\cdots\pi$ and hydrogen-bonding interaction.

distorted octahedral geometry, coordinated by four nitrogens of four 5-meim ligands and two carboxyl oxygens of dpa. The basal plane is defined by O1, O1^{i}, N3, and N3^{i} from the one dpa and two 5-meim ligands, respectively. The apical position is also occupied by N1 and N1^{i} of 5-meim ligands ((i) x, $1/2-y$, $1/2-z$). The Co−O1 bond distance of 2.171(1) Å is in the normal range of related Co(II) complexes [23] and also somewhat longer than reported for [$Co_2(dpa)_2(H_2O)_8$] (2.0462(12) and 2.0632(11) Å) [24], {[$Co_2(dpa)_2(bipy)_2(H_2O)_2$]·$2H_2dpa$}$_n$ (2.067(2) and 2.107(2) Å) [25] and [$Co(Hdpa)_2(bipy)_2$]$_n$ (2.037(4) Å) [26].

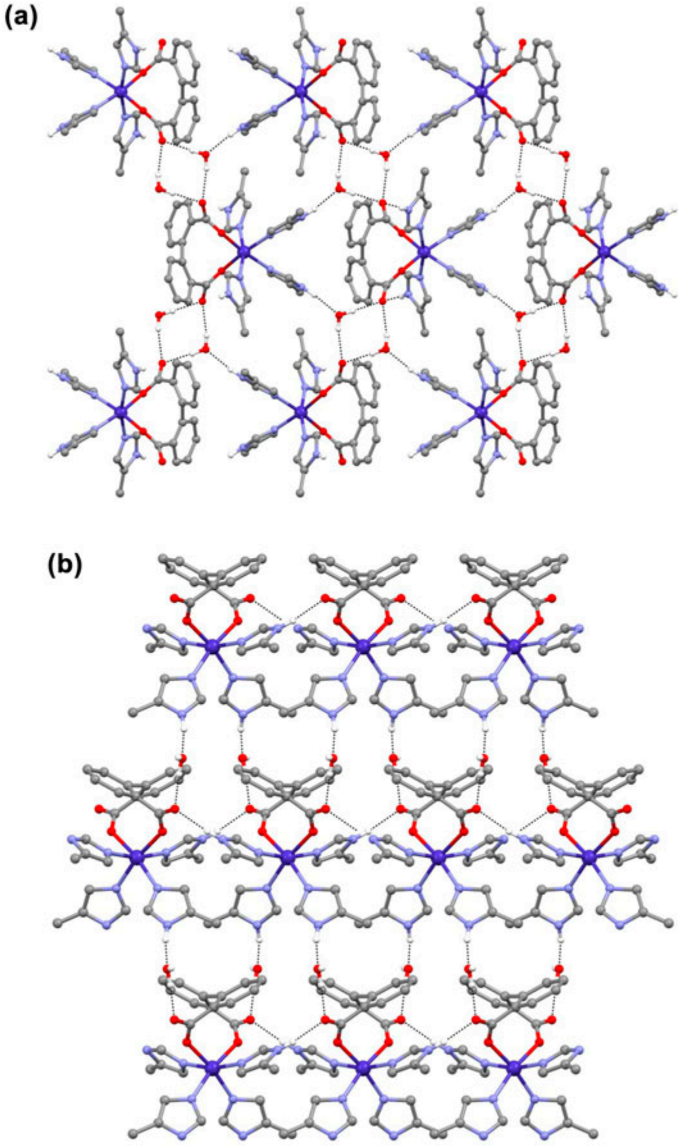

Figure 2. View of the 2-D network formed by hydrogen bonding interactions: (a) *ab* plane and (b) *c*-axis.

The crystal packing is mainly stabilized by hydrogen bonds. Adjacent complex units are connected by hydrogen bonds between uncoordinated carboxyl oxygens of dpa and crystal waters with a pattern $R_4^4(8)$ to form a 1-D chain (figure 2). The N–H···O type hydrogen bonds were also observed between adjacent chains, resulting in a 2-D layer. Adjacent 2-D layers are connected by hydrogen bonds between the amine hydrogens of 5-meim and the oxygens of dpa to form a 3-D network. The 3-D network contains a 1-D channel occupied by water molecules (figure 3). There are also $\pi\cdots\pi$ interactions between the phenyl (Cg1)

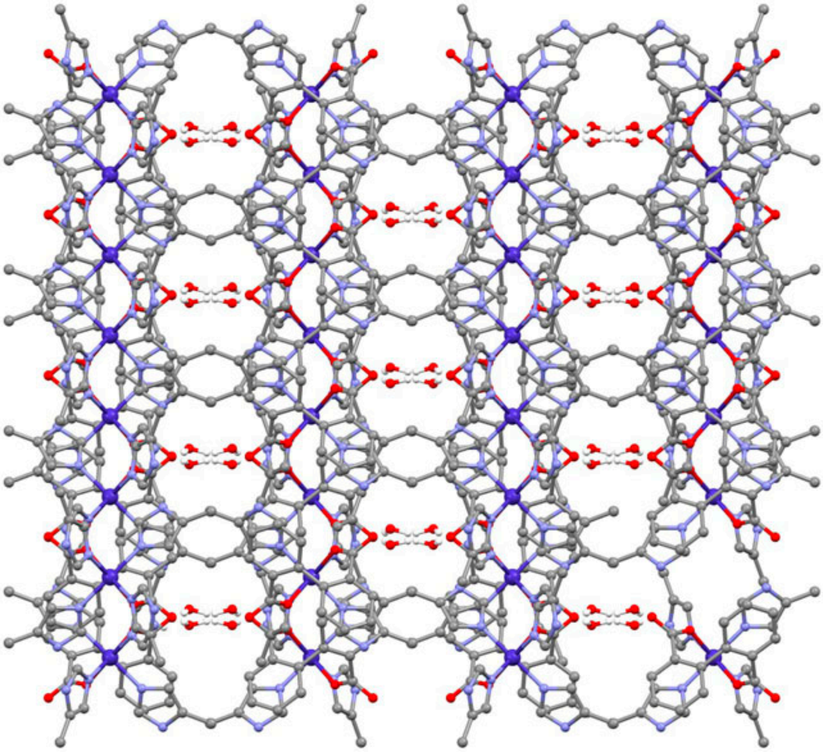

Figure 3. View of the 3-D network containing the 1-D channel occupied by water molecules.

and imidazole (Cg2) rings (Cg1: C2–C7 and Cg2=N1–C8–N2–C9–C10, Cg1···Cg2=3.5983 Å) (figure 1).

When the Co(II) ion was replaced by Cu(II), we obtained **2** containing a linkage isomerism of the 4(5)-methylimidazole ligand. The single-crystal X-ray structural analysis reveals that **2** crystallizes in the triclinic space group *P1* and the molecular structure of **2** consists of one Cu(II), one dpa, and one 4-meim and two 5-meim ligands. As shown in figure 4, Cu(II) is a distorted square pyramidal geometry with a τ_4 value of 0.29 [$\tau = 169.9 - 152.6)/60 = 0.29$] [27] completed by three nitrogens from two 5-meim, one 4-meim ligands and two oxygens from bidentate dpa. N1, N3, N5, and O3 form the basal plane, while O1 occupies the apical position. The Cu–N and Cu–O bond distances [Cu1–N1 = 1.918(12), Cu1–N3 = 2.007(7), Cu1–N5 = 1.948(10), Cu1–O1 = 2.210(5), and Cu1–O3 = 2.002(5) Å] are similar to the corresponding ones in known copper complexes [28–33].

The most striking feature is that the structure contains two coordination modes of the 4(5)-meim ligand in the same coordination sphere. In our previous studies of [Zn(4-meim)$_2$(5-meim)$_2$]sq·3H$_2$O [14] and {[Cd(pzdc)(4-meim)(5-meim)$_2$]·1/2H$_2$O}$_n$ [15], it was determined that the 4(5)-meim ligand exhibited linkage isomerism. In these complexes, C–H···π interaction between the methyl group of the 4-meim ligand and adjacent 5-meim ligand plays an important role to coordinate to the metal as 4-meim. Another interesting feature of this complex is the presence of C–H···Cu weak hydrogen-bonding interactions between the Cu(II) and H22C of the methyl group (figure 4). This weak C–H···Cu interaction

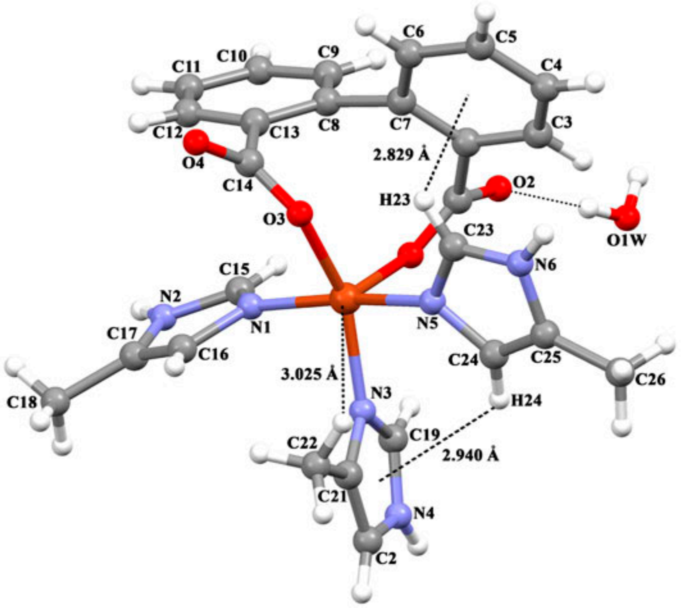

Figure 4. The molecular structure of **2** with intramolecular C−H···Cu, C−H···π and hydrogen-bonding interactions.

has facilitated coordination of the 4-meim to the Cu(II). The bond distance (H22C···Cu1) and angle (C22–H22C···Cu1) are 3.025 Å and 101.63°, respectively [34, 35].

Crystal packing is achieved by intermolecular hydrogen bonds which play an important role in formation of the 3-D supramolecular network of **2**. Molecules of **2** are bridged by N–H···O hydrogen bonds, forming a hydrogen-bonded chain (figure 5). Adjacent chains are extended to a 2-D layer by hydrogen bonds between the water hydrogens and the carboxyl oxygens of dpa. These 2-D layers are also connected by hydrogen bonds between the amine hydrogens of 5-meim and the carboxyl oxygens of dpa to form a 3-D framework (figures 5 and 6). Furthermore, there are C–H···π interactions between the C–H groups of 4(5)-meim ligands (C22–H22A, C23–H23, C24–H24, and C26–H26A) and phenyl (Cg1 and Cg2), or imidazole (Cg3) rings (Cg1: C2–C3–C4–C5–C6–C7, Cg2: C8–C9–C10–C11–C12–C13, Cg3: N3-C19–N4–C20–C21; C22···Cg1^{i} = 3.5066 Å, C22-H22A···Cg1^{i} = 161°, C26···Cg2^{ii} = 3.6186 Å, C26-H26A···Cg2^{ii} = 173°, C23···Cg1 = 3.2076 Å, C23–H23···Cg1 = 105°, C24···Cg3 = 3.5862 Å, C24–H24···Cg3 = 126°, (i) *x*, *y*, 1 + *z*; (ii) *x*, 1 + *y*, 1 + *z*) (figure 5).

3.2. *Thermal analyses*

To examine the thermal stability of the two compounds, thermal analyses were carried out for **1** and **2** between 30 and 1000 °C.

The complexes exhibit three decomposition stages. Loss of lattice water in **1** was completed under 94 °C (observed 5.53%, Calcd 5.42%; $\text{DTA}_{\text{max}} = 129$ °C, $\Delta H = +54.38$ kJ /mol).

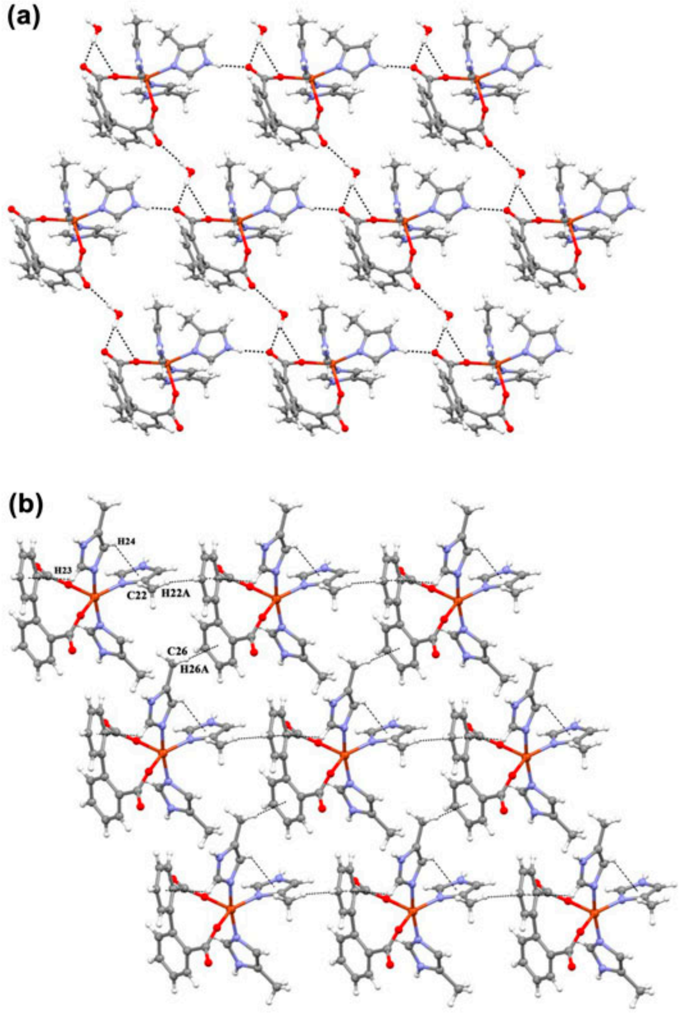

Figure 5. View of (a) the hydrogen-bonding interactions and (b) C−H···π interactions.

Beyond this step, two indiscernible processes with a total weight loss of 83.63% corresponding to the combustion of 4-meim (Calcd 85.99%) and dpa (Calcd 15.72%; DTA_{max} = 424 °C, ΔH = –1650 kJ /mol) were observed, which resulted in the collapse of the supramolecular framework. The remaining weight of 9.87% corresponds to the percentage (11.29%) of Co and O components, indicating that the final product is CoO (figure 7). For **2**, a total weight loss of 3.61% occurred from 100 to 240 °C, corresponding to loss of lattice water (Calcd 3.16%; DTA_{max} = 197 °C, ΔH = +20.65 kJ /mol). Beyond this step, two indiscernible processes with a total weight loss of 83.05% corresponding to the removal of 4(5)-meim (Calcd 40.87%) and dpa (Calcd 85.99%; DTA_{max} = 478 °C, ΔH = –912 kJ/mol) were observed which resulted in the collapse of the supramolecular framework. The remaining weight of 12.75% corresponds to the percentage (14.00%) of Cu and O components, indicating that the final product is CuO (figure 8).

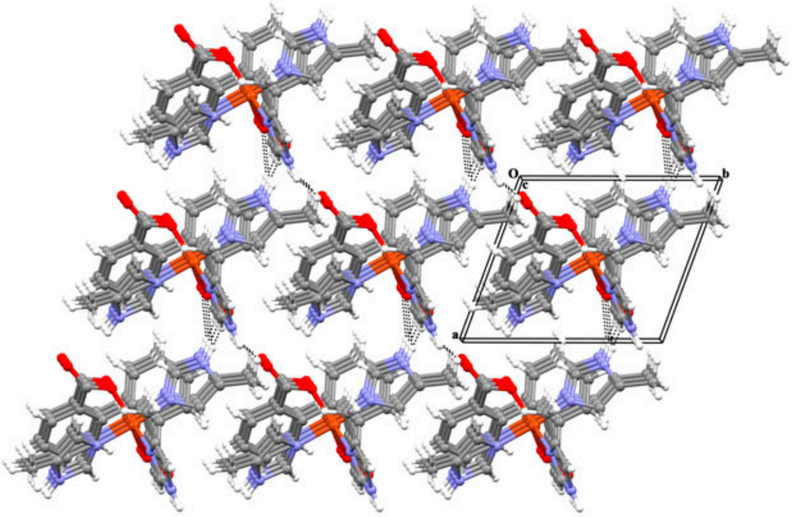

Figure 6. View of the 3-D network of **2**.

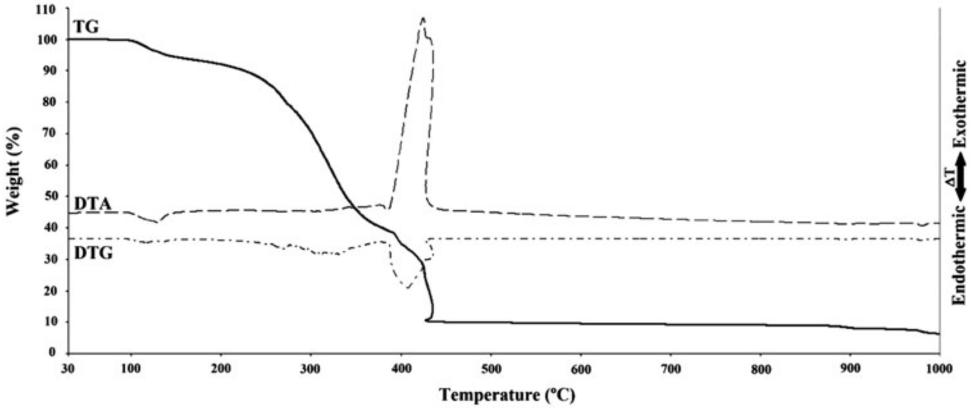

Figure 7. TG, DTG, and DTA curves of **1**.

3.3. *Magnetic susceptibilities*

The magnetic susceptibilities of $[Co(dpa)(5\text{-}meim)_4]\cdot 2H_2O$ (**1**) and $[Cu(dpa)(4\text{-}meim)(5\text{-}meim)_2]\cdot H_2O$ (**2**) were measured at room temperature. Complex **1**, exhibits a magnetic moment of 4.56 BM, which corresponds to three unpaired electrons. These values are higher than the spin-only value of 3.87. Not only the spin magnetic moment contributes to the total magnetic moment, but also the orbital magnetic moment in Co(II) complexes. Complex **2**, exhibits magnetic moment of 1.59 BM, which corresponds to one of the unpaired electrons [36].

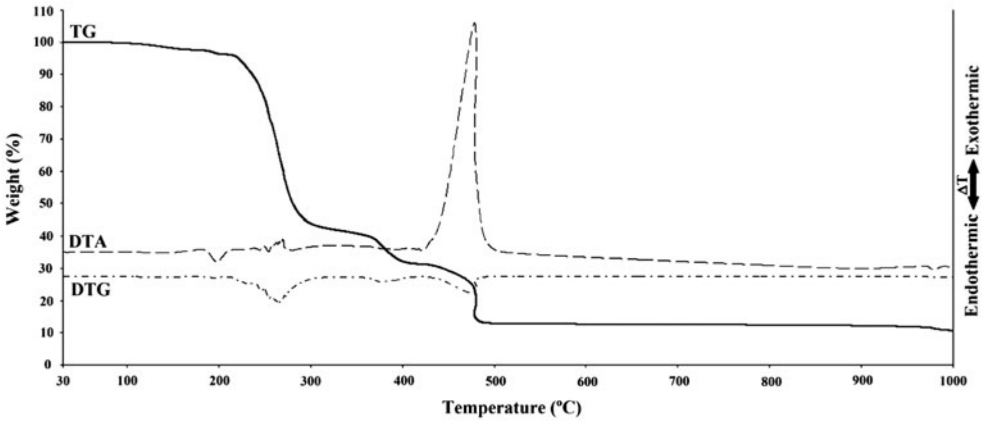

Figure 8. TG, DTG, and DTA curves of **2**.

4. Conclusion

Co(II) and Cu(II) complexes with diphenate (dpa) and 4-methylimidazole have been prepared and structurally characterized. We observe linkage isomerism with the 4-methylimidazole in the same complex. The dpa is a bidentate ligand through two oxygens from different carboxylates. The most interesting features of **2** are the presence of C–H•••Cu interaction and linkage isomerization.

Supplementary material

CCDC 886550 and 886551 contain supplementary crystallographic data for **1** and **2**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: (+44) 1223 336 033; or E-mail: deposit@ccdc.cam.ac.uk.

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