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Three Co^{II}/Co^{III} complexes with a 1-substituted tetrazole-5-thiol ligand

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1-Methyl-5-mercapto-tetrazole (mmtz) reacts with Co(II) salts in the presence of N-containing ligands and three Co^{II}/Co^{III} complexes, $[Co^{II}(mmtz)_2(4,4'\text{-bipy})(H_2O)_2]_n$ (**1**), $[Co^{III}(mmtz)_2(1,10\text{-phen})_2]NO_3$ (**2**), and $[Co_2^{III}(mmtz)_2(2,2'\text{-bipy})_2(\mu\text{-OH})_2]Cl_2$ (**3**), have been characterized using single-crystal X-ray diffraction structure determination, IR, and elemental analysis. Complex **1** has a 1-D polymeric structure with neutral $[Co(mmtz)_2(4,4'\text{-bipy})(H_2O)_2]$ as repeating unit. These 1-D infinite chains are further connected into a 3-D supramolecular framework through intermolecular hydrogen bonds. In **2**, mmtz is an S-bonding ligand to form a mononuclear structure. In **3**, mmtz as a μ_2 bridging ligand links Co(III) ions to form a dimeric structure. Thermostability of **2** and **3** and electrochemical properties of **1**–**3** are discussed.

Keywords: Cobalt complexes; Crystal structures; Thermostability; Cyclic voltammetry

1. Introduction

N-heterocyclic compounds have received attention because of their intriguing architectures and topologies and their functional applications in gas absorption, luminescence, catalysis, magnetic materials, nonlinear optics, etc. [1–6]. N-heterocycles with sulfhydryl groups have the transformation, –N(H)–C(=S) ↔ –N=C(–SH) [7–9]. Sulfurs outside the ring and the heterocyclic nitrogen both can participate in coordination. The soft sulfur reacts with soft acids, for example, Cu(I) and Ag(I) [7, 10, 11]. Sulfur has strong coordination ability and also an empty 3d orbital can accept electrons from the metal. Research about heterocyclic nitrogen rings with sulfhydryl mainly focuses on sulfhydryl imidazole, sulfhydryl (1,2,4) triazole, and sulfhydryl tetrazole [12–15].

1-Methyl-5-mercapto-tetrazole (mmtz) can bridge several metals to afford a coordination polymer or metal clusters [16]. Here three complexes, $[Co^{II}(mmtz)_2$

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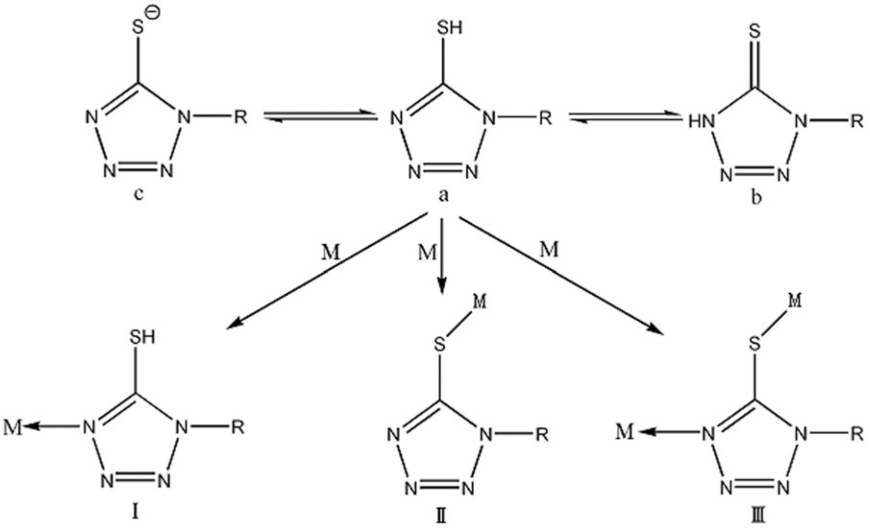

Scheme 1. The three coordination modes of mmtz in **1**–**3**.

(4,4′-bipy)$(H_2O)_2]_n$ (**1**), $[Co^{III}(mmtz)_2(1,10\text{-phen})_2]NO_3$ (**2**), and $[Co_2^{III}(mmtz)_2(2,2'\text{-bipy})_2(\mu\text{-}OH)_2]Cl_2$ (**3**), were synthesized using three different auxiliary N-containing ligands including 4,4′-bipyridine, 1,10-phenanthroline, and 2,2′-bipyridine. According to previously published papers, mmtz is invariably coordinated through its sulfur [17–20]. However in these complexes, with the influence of auxiliary N-containing ligands, mmtz has three coordination modes (scheme 1). All these complexes further extend through intermolecular hydrogen bonds to form 3-D supramolecular frameworks. Thermostability of **2** and **3** and electrochemical properties of **1**–**3** are presented.

2. Experimental

2.1. *Materials and instruments*

All reagents and solvents were of analytical grade and used without purification. Elemental analyses for C, H, and N were carried out with a Perkin Elmer 2400II elemental analyzer. IR spectra were recorded on a Perkin Elmer Spectrum One FT-IR spectrometer with KBr pellets from 400 to 4000 cm^{-1}. Thermogravimetric (TG) analyses were conducted on a Perkin Elmer Pyris Diamond TG/DTA thermogravimetric analyzer in flowing N_2 with a heating rate of 10°C min^{-1}. Electrochemical measurements were performed with a BAS Epsilon Electrochemical workstation. A conventional three-electrode system consisting of a glassy carbon working, a platinum wire auxiliary, and a saturated calomel reference electrode was used. KCl (0.1 mol L^{-1}) was used as supporting electrolyte in aqueous solution.

Table 1. Crystallographic data and structure refinement details for **1**–**3**.

Complex	**1**	**2**	**3**
Empirical formula	$C_{14}H_{18}CoN_{10}O_2S_2$	$C_{28}H_{22}CoN_{13}O_3S_2$	$C_{24}H_{24}Cl_2Co_2N_{12}O_2S_2$
Formula weight	481.43	711.64	765.43
Temperature (K)	298(2)	298(2)	298(2)
Wavelength (Å)	0.71073	0.71073	0.71073
Crystal system	Tetragonal	Monoclinic	Monoclinic
Space group	$I4(1)/acd$	$P2(1)/n$	$C2/c$
a	23.208(3)	15.6448(16)	14.0925(14)
b	23.208(3)	9.3280(11)	16.968(2)
c	14.5814(17)	20.674(3)	13.9007(12)
α	90	90	90
β	90	103.107(2)	117.298(2)
γ	90	90	90
Volume (Å^3), Z	7853.9(15), 16	2938.4(6), 4	2953.8, 4
Calculated density (Mg m^{-3})	1.629	1.609	1.721
Absorption coefficient (mm^{-1})	1.12	0.78	1.49
$F(000)$	3952	1456	1552
Crystal size (mm^3)	$0.22 \times 0.16 \times 0.13$	$0.38 \times 0.13 \times 0.04$	$0.21 \times 0.09 \times 0.07$
θ range	1.75–25.01	1.48–25.01	2.02–25.00
Reflections collected	17,782	14,068	7593
Independent reflection	1740 [$R_{(int)} = 0.070$]	4985 [$R_{(int)} = 0.1308$]	2593 [$R_{(int)} = 0.0473$]
Max. and min. transmission	0.7904 and 0.8679	0.7548 and 0.9693	0.7444 and 0.9026
Data/restraints/parameters	1740/0/181	4985/0/426	2593/0/257
Goodness-of-fit on F^2	1.039	0.995	1.026
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0637$ $wR_2 = 0.1659$	$R_1 = 0.0987$ $wR_2 = 0.2282$	$R_1 = 0.0313$ $wR_2 = 0.0609$
R indices (all data)	$R_1 = 0.1006$ $wR_2 = 0.1936$	$R_1 = 0.1652$ $wR_2 = 0.2691$	$R_1 = 0.0909$ $wR_2 = 0.0759$
Largest difference peak and hole (e Å^{-3})	0.75 and −1.02	2.89 and −0.50	0.33 and −0.21

2.2. *X-ray diffraction crystallography*

All measurements were recorded on a Bruker Smart CCD apparatus at 298 K using graphite-monochromated Mo-Kα radiation ($\lambda = 0.710730$ Å). The primitive structures were solved by direct methods and refined using full-matrix least-squares on F^2 employing SHELXL-97 [21]. Anisotropic thermal parameters were assigned to all non-hydrogen atoms. Organic hydrogen atoms were generated geometrically. Hydrogen atoms of water were located from difference maps and refined. Crystallographic data and experimental details for structure analyses are summarized in table 1. Selected bond lengths and angles are listed in table 2.

2.3. *Syntheses of the complexes*

2.3.1. Synthesis of [Co^{II}(mmtz)$_2$(4,4′-bipy)(H$_2$O)$_2$]$_n$ (1). A mixture of mmtz (116.2 mg, 1.00 mmol), $CoCl_2 \cdot 6H_2O$ (237.9 mg, 1.00 mmol), 4,4′-bipy (112 mg, 0.50 mmol), NaOH (40 mg, 1.00 mmol), and distilled water (10 mL) was stirred under reflux for 3 h and then cooled and filtered. About 30 days later, pink needle-like crystals were obtained. Yield: 70%. Anal. Calcd for $C_{14}H_{18}CoN_{10}O_2S_2$ (%): C, 34.89; H, 3.73; N, 29.08; S, 13.29. Found (%): C, 34.92; H, 3.84; N, 29.01; S, 13.13. IR (KBr, cm^{-1}): 3430.75m, 1600.59s,

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

1			
Co1–O1	2.065(5)	Co1–N5	2.223(6)
Co1–N4	2.14(11)	Co1–N6B	2.257(7)
O1A–Co1–O1	179.0(2)	N4–Co1–N5	90(2)
O1–Co1–N4	95.4(15)	O1–Co1–N6B	89.52(12)
O1–Co1–N4A	84.6(15)	N4–Co1–N6B	90(2)
N4A–Co1–N4	179(5)	O1–Co1–N5	90.48(12)
2			
Co1–N9	1.946(7)	Co1–N11	1.957(6)
Co1–N12	1.955(7)	Co1–S1	2.266(2)
Co1–N10	1.956(6)	Co1–S2	2.270(2)
N9–Co1–N12	174.9(3)	N10–Co1–S1	171.73(19)
N9–Co1–N10	83.5(3)	N11–Co1–S1	94.6(2)
N12–Co1–N10	93.0(3)	N9–Co1–S2	89.5(2)
N9–Co1–N11	92.5(3)	N12–Co1–S2	94.4(2)
N12–Co1–N11	84.1(3)	N10–Co1–S2	93.86(19)
N10–Co1–N11	93.5(3)	N11–Co1–S2	172.6(2)
N9–Co1–S1	94.3(2)	S1–Co1–S2	78.11(9)
N12–Co1–S1	89.7(2)		
3			
Co1–O1	1.885(7)	Co2–N4	1.89(4)
Co1–N5	1.927(10)	Co2–O1	1.899(7)
Co1–S1	2.22(3)	Co2–N6	1.923(10)
O1–Co1–O1A	84.1(4)	N4A–Co2–N4	169(3)
O1–Co1–N5A	179.4(3)	N4–Co2–O1	84.3(15)
O1–Co1–N5	96.0(3)	N4–Co2–O1A	87.2(14)
N5A–Co1–N5	83.9(6)	O1A–Co2–O1	83.3(4)
O1–Co1–S1A	94.6(5)	N4A–Co2–N6	95.6(15)
O1–Co1–S1	90.2(5)	N4–Co2–N6	92.9(14)
N5–Co1–S1	86.0(6)	O1A–Co2–N6	179.7(4)
N5–Co1–S1A	89.2(6)	O1A–Co2–N6A	96.9(4)
S1A–Co1–S1	173.6(9)	N6–Co2–N6A	82.8(6)

Symmetry codes: for **1**, A: $-x+1/2$, y, $-z$; B: x, $y+1/2$, $-z$ and for **3**, A: $-x+2$, y, $-z+1/2$.

1363.26s, 1294.37s, 1172.91m, 1081.08w, 1004.91w, 815.23w, 715.81w, 627.71w, 522.31w.

2.3.2. Synthesis of [$Co^{III}(mmtz)_2(1,10\text{-}phen)_2]NO_3$ (2). Complex **2** was prepared by a similar procedure as described for **1**. mmtz (116.2 mg, 1.00 mmol), $Co(NO_3)_2 \cdot 6H_2O$ (291.1 mg, 1.00 mmol), 1,10-phen (99.11 mg, 0.50 mmol), NaOH (40 mg, 1.00 mmol), and distilled water (10 mL) were used for **2**. About 60 days later, green needle-like crystals were obtained. Yield: 60%. Anal. Calcd for $C_{28}H_{22}CoN_{13}O_3S_2$ (%): C, 37.62; H, 3.13; N, 21.94; S, 8.36. Found (%): C, 37.71; H, 3.24; N, 21.86; S, 8.29. IR (KBr, cm^{-1}): 3435.35w, 1630.60w, 1423.14w, 1384.28s, 1349.27m, 1167.75w, 852.23w, 718.81w, 696.90w.

2.3.3. Synthesis of [$Co_2^{III}(mmtz)_2(2,2'\text{-}bipy)_2(\mu\text{-}OH)_2]Cl_2$ (3). The preparation of **3** is similar to **1** except that 4,4′-bipy was replaced by 2,2′-bipy giving red needle-like, X-ray-quality crystals of **3**. Yield: 70%. Anal. Calcd for $C_{24}H_{24}C_{12}Co_2N_{12}O_2S_2$ (%): C, 47.21; H, 3.09; N, 25.57; S, 8.99. Found (%): C, 47.33; H, 3.13; N, 25.43; S, 8.74. IR

(KBr, cm^{-1}): 3428.57m, 2991.70s, 1603.84m, 1469.64m, 1446.50s, 1309.14s, 1240.78w, 1111.09w, 927.34w, 772.19s, 722.78m, 574.37w, 477.43w.

3. Results and discussion

3.1. *Description of crystal structures*

3.1.1. $[Co^{II}(mmtz)_2(4,4'\text{-bipy})(H_2O)_2]_n$ (1). X-ray analysis reveals that **1** has an infinite, linear 1-D polymeric structure (figure 1b) with neutral $[Co(mmtz)_2(4,4'\text{-bipy})(H_2O)_2]$ as repeating unit. 4,4′-Bipyridine is an effective linker between Co's for propagation of coordination network. Co is in a distorted octahedral geometry, by N4 and N4A from two mmtzs, N5 and N6B from two 4,4′-bipy and two water molecules (figure 1a). The mmtz is an N-bonding monodentate ligand (scheme 1-I). The axial position of the octahedron is occupied by O1 and O1A with an O1–Co1–O1A bond angle of 179.0 (2)°. The equatorial plane is provided by N4, N4A, N5, and N6B. Co–O bond lengths are 2.065(5) Å, similar to $\{[Co(ClO_4)(H_2O)(L)][Co(H_2O)_2(L)]\}(ClO_4)_3$ [L = 3,11-dithia-7,17-diazabicyclo[11.3.1]-heptadeca-1(17)-13,15-triene] [22]. The Co–N bond lengths range from 2.14(11)–2.257(7) Å, consistent with those observed in $\{[Co(L_1)(MeCN)_2](ClO_4)_2\}_\infty$ [L_1 = bis(2-pyrazylmethyl)sulfide] [23]. These 1-D infinite chains are further connected into a 3-D supramolecular framework through hydrogen bonds among coordinated water molecules, the tetrazolyl 3-N, and sulfur, indicated by dashed lines in figure 1(c).

3.1.2. $[Co^{III}(mmtz)_2(1,10\text{-phen})_2]NO_3$ (2). The structure of **2** contains $[Co(mmtz)_2(1,10\text{-phen})_2]^+$, and an NO_3^-. A perspective view of the molecular structure is shown in figure 2(a). The coordination geometry of Co1 is distorted octahedral with four nitrogen atoms (N9, N10, N11, and N12) from two 1,10-phen and S1 and S2 from two mmtz. The Co–N bond lengths are 1.946(7)–1.957(6) Å, consistent with those observed in $[LCo^{III}(\mu\text{-}O_2CMe)(\mu\text{-}OMe)_2Co^{III}L](ClO_4)_3$ [L = N-methyl-N,N-bis(2-pyridylmethyl)amine] [24]. In **2**, two bidentate 1,10-phen are large, so mmtz is S-bonded monodentate (scheme 1-II) and the tetrazole ring is on the outside. The coordination environment of **2** exhibits a butterfly-like structure, linked to another unit through intermolecular hydrogen bonds among free NO_3^-, the tetrazolyl (N, S, and C) and N and C from 1,10-phen to form a 3-D supramolecular framework (figure 2b).

3.1.3. $[Co_2^{III}(mmtz)_2(2,2'\text{-bipy})_2(\mu\text{-}OH)_2]Cl_2$ (3). As shown in figure 3(a), Co1 is in a distorted octahedral coordination environment with S1 and S1A from two different mmtz, N5 and N5A from 2,2′-bipy and two μ_2-OH. The axial position is occupied by S1 and S1A, with an S1–Co1–S1A angle of 173.6 (9)°. The equatorial plane is provided by O1, O1A, N5, and N5A. The coordination of Co2 is similar to that of Co1. N4 and N4A occupy axial positions, with N4–Co2–N4A = 169 (3)°, while N6, N6A, O1, and O1A occupy the equatorial plane. Co–N and Co–O bond lengths are similar to $[L_3Co_3^{III}(\mu\text{-}OH)_4(OH)](ClO_4)_4$ [L = N-methyl-N,N-bis(2-pyridylmethyl)amine] [24]. Two Co(III) ions are bridged by a pair of μ_2-O1 yielding a Co_2O_2 binuclear parallelogram. Complex **3** exhibits a dimeric structure with two Co(III) ions bridged by

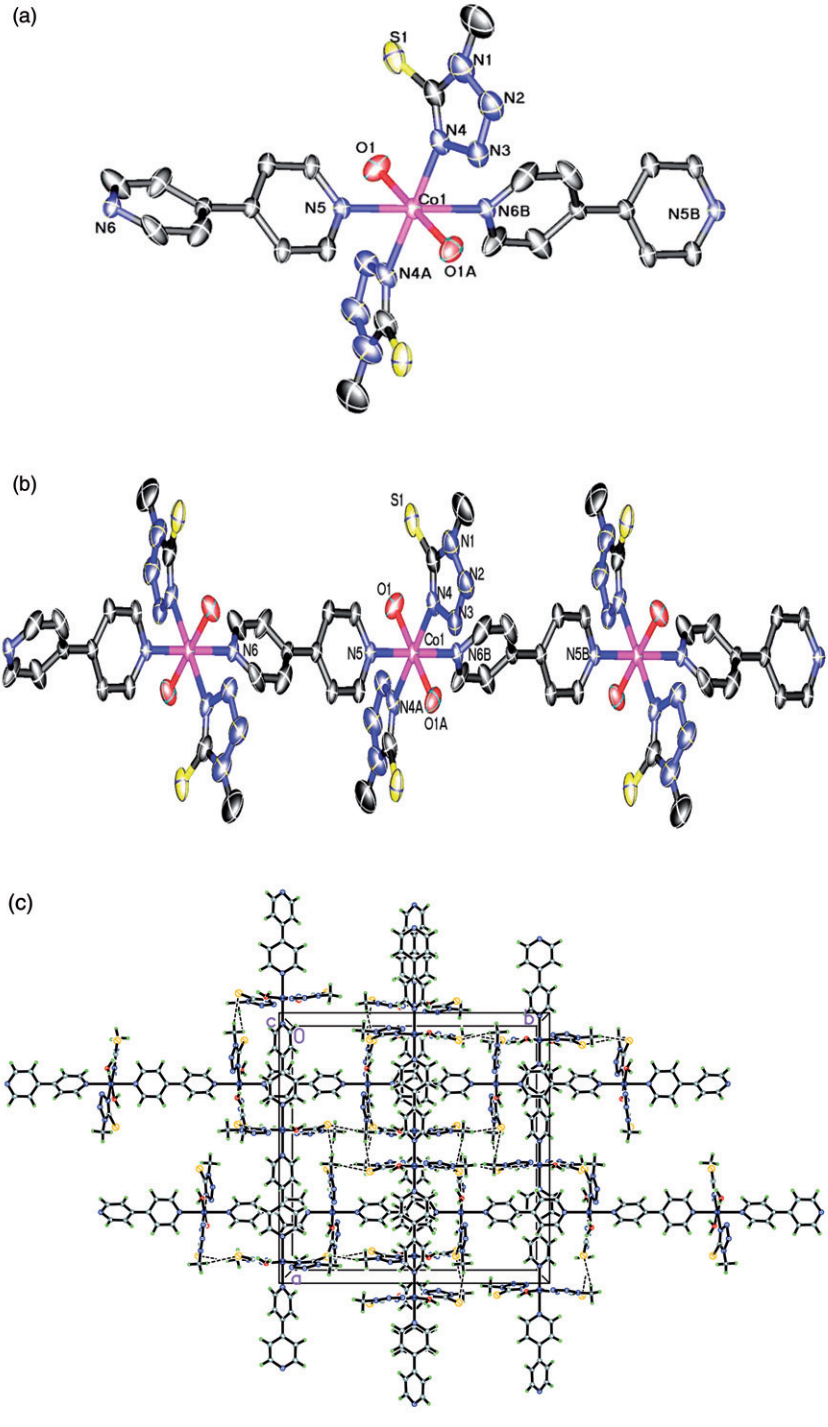

Figure 1. (a) A view of the coordination environment of **1**. Symmetry codes: A: $-x+1/2$, y, $-z$; B: x, $y+1/2$, $-z$. (b) A view of the 1-D infinite chain of **1**. Symmetry codes: A: $-x+1/2$, y, $-z$; B: x, $y+1/2$, $-z$. (c) 3-D packing drawing of **1**. Hydrogen-bond geometry for **1**: O1–H1*C*···N3#3: 0.85 Å, 1.94 Å, 2.783(19) Å, 170°; O1–H1*B*···S1: 0.85 Å, 2.62 Å, 3.309(9) Å, 138° (symmetry code: #3 $-x$, $-y$, $-z$).

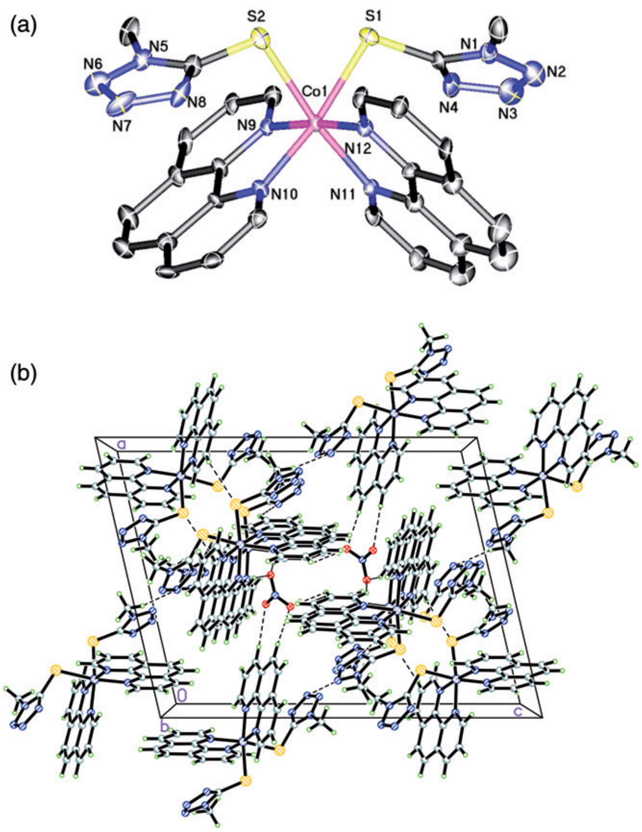

Figure 2. (a) A view of the coordination environment of **2.** (b) 3-D packing drawing of **2**.

two bidentate bridging mmtz (scheme 1-III) forming an eight-membered ring. The Co1···Co2 distance is 2.8180(3) Å. These dinuclear structures are stacked through intermolecular hydrogen bonds to form a 3-D supramolecular framework (figure 3b).

3.2. *Cyclic voltammetry discussion*

The cyclic voltammogram of **1** in the potential range +1.50 to −1.00 V and for scan rate of 100 mV s^{-1} is shown in "Supplementary material" section. Complex **1** displays a pair of oxidation–reduction peaks at −0.5415 V and −0.2932 V. The redox peak potential separation (ΔE_P) of 248.3 mV suggests that the Co(II)/Co(I) redox couple was quasireversible [25]. For **2** and **3**, cyclic voltammetry was performed by scanning from +1.20 V to −0.40 V at a scan rate of 80 mV s^{-1}. For **2**, an anodic peak located at −0.035 V and a cathodic peak at 0.038 V, the ΔE_P of 73 mV, shows a quasireversible electrochemical process of Co(III)/Co(II) redox [26]. For **3**, an anodic peak appears at −0.2217 V while there is no corresponding cathodic wave, indicating that the electrochemical behavior of the Co(III)/Co(II) redox couple is irreversible [27].

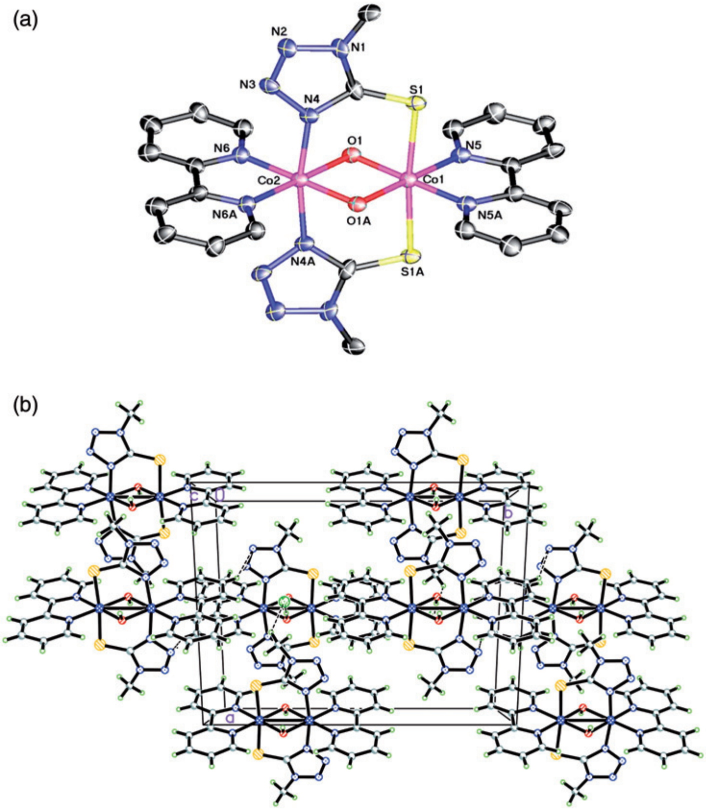

Figure 3. (a) Coordination environment of **3**. Symmetry codes: A: $-x+2$, y, $-z+1/2$. (b) 3-D packing of **3**. Hydrogen-bond geometry for **3**: O1–H1···Cl1#2: 0.85 Å, 2.25 Å, 3.089(2) Å, 171° (symmetry code: #2 $-x+2$, $-y$, $-z$).

3.3. *TG analyses*

The thermogravimetric analysis (TGA) curve ("Supplementary material" section) shows that **2** is stable to 214°C and then collapsed due to decomposition of ligand. The subsequent steps are decomposition of 1,10-phen and nitrate and a residue of 13.08% at 991°C is consistent with the calculated value (11.65%) assuming Co_2O_3 as the final product.

The TGA of **3** is similar to that of **2** ("Supplementary material" section). Complex **3** is stable to 200°C and then collapsed due to decomposition of ligand. Upon further heating, 2,2′-bipy, hydroxyl, and chloride show three steps of weight loss. The residue of **3** is 20.04%, consistent with the calculated value (21.66%) considering Co_2O_3 as the final product.

4. Conclusion

A 1-D complex $[Co(mmtz)_2(4,4'\text{-bipy})(H_2O)_2]_n$ (**1**), mononuclear complex $[Co(mmtz)_2(1,10\text{-phen})_2]NO_3$ (**2**), and binuclear complex $[Co_2(mmtz)_2(2,2'\text{-bipy})_2(\mu\text{-OH})_2]Cl_2$ (**3**) bearing mmtz have been synthesized in the presence of 4,4′-bipy/1,10-phen/2,2′-bipy. They further extend through intermolecular hydrogen bonds to form 3-D supramolecular frameworks. These different N-containing ligands and diverse coordination modes of mmtz influence architectures of the complexes. Thermostability of **2** and **3** and electrochemical properties of **1**–**3** have been discussed.

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