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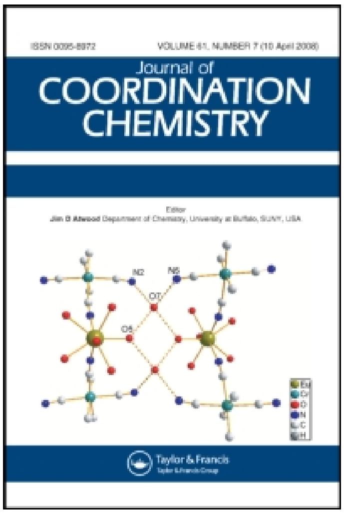

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Synthesis, structures, and characterization of cadmium(II), cobalt(II), and copper(II) metal polymers with tetrazole-1-acetate

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Synthesis, structures, and characterization of cadmium(II), cobalt(II), and copper(II) metal polymers with tetrazole-1-acetate

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Three new coordination polymers $\{[Cd(tza)(2,2'\text{-bpy})(H_2O)](ClO_4)\}_n$ **1**, $\{[Co(tza)(2,2'\text{-bpy})(H_2O)](ClO_4)\}_n$ **2**, and $\{[Cu(tza)(phen)](ClO_4)\}_n$ **3** (Htza = tetrazole-1-acetic acid, 2,2′-bpy = 2,2′-bipyridyl, phen = 1,10-phenanthroline) were synthesized and characterized by X-ray single-crystal diffraction, elemental analysis, and IR spectra. Complexes **1** and **2** exhibit 3-D architectures formed by π–π interaction of 2,2′-bpy ligands interlinking to the adjacent 2-D layers. Complex **3** is a 1-D zigzag double chain and the 3-D structure is formed by π–π stacking interaction of phen and nonclassical hydrogen bonding.

Keywords: Coordination polymers; Crystal structures; Tetrazole-1-acetic acid

1. Introduction

Coordination polymers have potential applications such as gas adsorption [1], photoluminescence [2], electrical conductivity [3], biology [4], magnetism [5], and catalysis [6]. Most coordination polymers are constructed by using appropriate organic ligand, especially bi- or multi-dentate bridging ligands containing O- or N-donors [7]. Tetrazole-1-acetic acid (Htza) with both carboxylate and tetrazole ends could bind to metals via N and/or O atoms to form multi-dimensional coordination polymers. To date, seven coordination modes of Htza have been reported (scheme 1) [8]. 2,2′-Bipyridine (2,2′-bpy) and 1,10-phenanthroline (phen) are bidentate chelating ligand which can provide high-dimensional coordination polymers via noncovalent intermolecular forces such as hydrogen-bonding and π–π stacking [9]. In this method, Htza was employed to synthesize three complexes $\{[Cd(tza)(2,2'\text{-bpy})(H_2O)](ClO_4)\}_n$ **1**, $\{[Co(tza)(2,2'\text{-bpy})(H_2O)](ClO_4)\}_n$ **2**, and $\{[Cu(tza)(phen)](ClO_4)\}_n$ **3**, which

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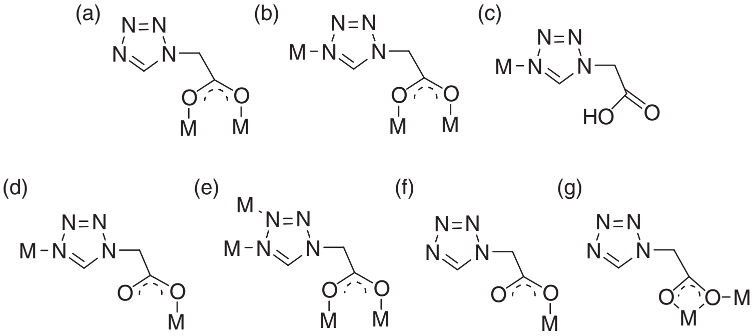

Scheme 1. Coordination modes of Htza.

were characterized by X-ray single-crystal diffraction, elemental analysis, and IR spectra.

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2. Experimental

2.1. *Materials and measurements*

All chemicals were commercially available reagents of analytical grade and were used without purification except for perchlorate salts, which were prepared by us. Elemental analyses (C, H, and N) were performed on a Perkin–Elmer 2400II elemental analyzer. FT-IR spectra were recorded on KBr pellets from 4000 to 450 cm^{-1} on a PE Spectrum One spectrometer.

Caution! Perchlorate salts may be explosive when heated.

2.2. *Preparation of the complexes*

2.2.1. Synthesis of {[Cd(tza)(2,2′-bpy)(H_2O)](ClO_4)}$_n$, 1. The Htza (0.1280 g, 1.0 mmol) and 2,2′-bpy (0.0312 g, 0.2 mmol) were mixed in methanol (5 mL) and ethanol (5 mL), and the mixture was stirred for 0.5 h. Then, $Cd(ClO_4)_2 \cdot 6H_2O$ (0.2099 g, 0.5 mmol) dissolved in ethanol (5 mL) was added slowly to the mixture. The mixture was stirred at 70°C for 3 h and then cooled to room temperature and filtered. The filtrate was allowed to slowly concentrate by evaporation at room temperature. Seven days later, colorless transparent crystals were obtained. Yield: 67% on the basis of Htza. Anal. Calcd for $C_{13}H_{13}CdClN_6O_7$ (%): C, 30.43; H, 2.55; N, 16.38. Found: C, 30.55; H, 2.69; N, 16.21.

2.2.2. Synthesis of {[Co(tza)(2,2′-bpy)(H_2O)](ClO_4)}$_n$, 2. Complex **2** was prepared in a manner similar to **1** using $Co(ClO_4)_2 \cdot 6H_2O$. The yield was 53% on the basis of Htza. Anal. Calcd for $C_{13}H_{13}CoClN_6O_7$ (%): C, 33.97; H, 2.85; N, 18.28. Found: C, 34.89; H, 3.34; N, 17.85.

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Table 1. Crystal data and structure refinement for **1**–**3**.

Complexes	**1**	**2**	**3**
Empirical formula	$C_{13}H_{13}CdClN_6O_7$	$C_{13}H_{13}CoClN_6O_7$	$C_{15}H_{11}ClCuN_6O_6$
Formula weight	513.14	459.67	470.29
Temperature (K)	293(2)	294(2)	293(2)
Wavelength (Å)	0.71073	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	*P*2(1)/*c*	*P*2(1)/*c*	*C*2/*c*
Units of dimensions (Å, °)			
a	11.9478(15)	12.077(3)	22.677(2)
b	13.8416(17)	13.803(3)	11.7223(7)
c	11.2453(14)	10.245(3)	13.8514(10)
β	99.140(2)	93.609(4)	108.963(2)
V (Å^3)	1836.1(4)	1704.5(7)	3482.3(4)
Z	4	4	8
D_{Calcd} (mg m^{-3})	1.856	1.791	1.794
Absorption coefficient (mm^{-1})	1.386	1.218	1.459
Crystal size (mm^3)	$0.42 \times 0.37 \times 0.34$	$0.10 \times 0.08 \times 0.04$	$0.60 \times 0.43 \times 0.35$
θ range for data collection (°)	2.27–27.05	1.69–25.00	3.11–27.48
Index ranges	$-15 \le h \le 15$, $-17 \le k \le 17$, $-14 \le l \le 13$	$-14 \le h \le 13$, $-16 \le k \le 9$, $-12 \le l \le 12$	$-24 \le h \le 29$, $-15 \le k \le 15$, $-17 \le l \le 17$
$F(000)$	1016	932	1896
Reflections collected	10,931	8260	13,197
Independent reflections (R_{int})	3990 (0.0182)	2946 (0.0419)	3980 (0.0191)
Goodness-of-fit on F^2	1.056	1.029	1.034
R_1 [$I > 2\sigma(I)$]	0.0313	0.0683	0.0435
wR_2 [$I > 2\sigma(I)$]	0.0878	0.1748	0.1227
R_1 (all data)	0.0370	0.0893	0.0479
wR_2 (all data)	0.0922	0.1927	0.1269
Largest difference in peak and hole (e Å^{-3})	1.084 and −0.639	1.853 and −0.638	0.822 and −0.616

2.2.3. Synthesis of $\{[Cu(tza)(phen)](ClO_4)\}_n$, 3. The Htza (0.1280 g, 1.0 mmol) and $Cu(ClO_4)_2 \cdot 6H_2O$ (0.1852 g, 0.5 mmol) were mixed in distilled water (10 mL) and the mixture was stirred for 1 h. Then phen (0.0360 g, 0.2 mmol) dissolved in ethanol (5 mL) was added slowly to the mixture. The mixture was stirred for 2 h and then cooled to room temperature and filtered. The filtrate was allowed to slowly concentrate by evaporation at room temperature. Two weeks later, blue block crystals were obtained. Yield: 60% on the basis of Htza. Anal. Calcd for $C_{15}H_{11}CuClN_6O_6$ (%): C, 38.31; H, 2.36; N, 17.87. Found: C, 38.55; H, 2.59; N, 16.81.

2.3. *Crystal structure determination*

Structural measurements of all complexes were performed on a computer controlled Bruker SMART 1000 CCD diffractometer equipped with graphite-monochromated Mo-Kα radiation with radiation wavelength 0.71073 Å by using the ω–φ scan technique. The structures were solved by direct methods and refined with the full-matrix least-squares using the SHELXS-97 and SHELXL-97 programs [10]. The crystallographic data for the complexes are summarized in table 1. CCDC Nos. 672068 for **1**, 672067 for **2**, and 672066 for **3**. The data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12 Union

Road, Cambridge CB2 1EZ, UK; Fax: +44-1223-336-033; or Email: deposit@ccdc.cam.ac.uk).

3. Results and discussion

3.1. *Synthesis*

The reaction of $M(ClO_4)_2 \cdot 6H_2O$ (M = Cd, **1**; Co, **2**; and Cu, **3**) with Htza and 2,2′-bpy or phen in solution afforded the 2-D (for **1** and **2**) or 1-D chain (for **3**) structures. In previous work, four different Cu(II) polymers were synthesized under different pH conditions [8(b)]. Five other 3-D polymers have been obtained by the substitution reactions of $M(CH_3COO)_2$ (M = Zn, Cd, Mn, Co) with Htza [8(d)], which is similar to our method but without additional ligands. In this work, 2,2′-bpy or phen was used to construct multi-dimensional structures by π–π interactions and hydrogen bonding.

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3.2. *Description of the crystal structures*

3.2.1. Structure (1) and (2). Complexes **1** and **2** are isostructural, therefore only the structure of **1** is shown in figure 1. Selected bond lengths and angles for **1** and **2** are summarized in tables 2 and 3, respectively. The central Cd(II) has a distorted octahedral coordination environment with two N atoms from 2,2′-bpy, one N and two O atoms from three different tza ligands and one water. O1A/O2B/N5/N6 (O1A/O2B/N5/N6 for **2**) forms the equatorial plane. O7W and N1 (O3 and N1 for **2**) occupy the axial sites.

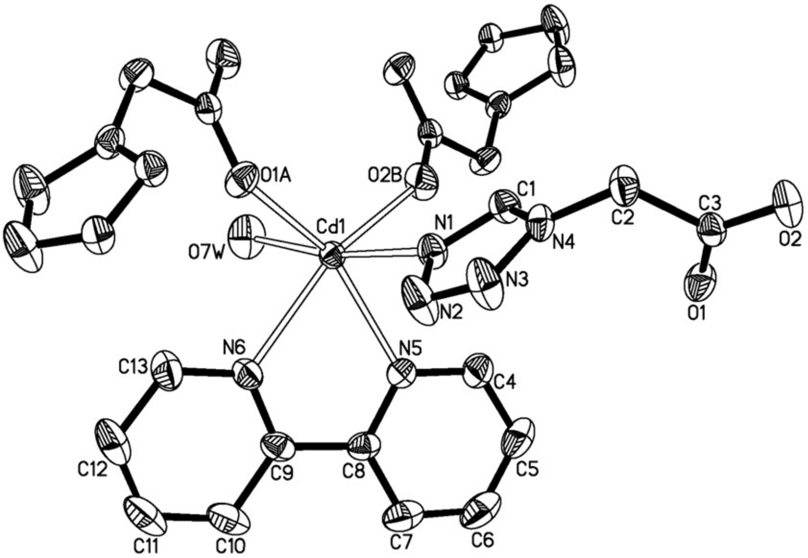

Figure 1. Perspective view of **1**, showing 30% probability ellipsoids for the nonhydrogen atoms and the atom-labeling scheme; all hydrogen atoms have been omitted for clarity.

In **1**, each tza is a μ_3-bridging ligand (mode **b** in scheme 1), which is the same as the coordination mode (II) in tetranuclear Cu(II) polymers [8(b)]. One N and two carboxylate O atoms link Cd(II)'s into a 2-D layer structure (figure 2). There are two types of rings in the layer: $\{Cd(tza)\}_4$ and $\{Cd(O–C–O)\}_2$ rings. The 2,2′-bpy are orientated outwards of the layer and thus play an important role in packing adjacent layers into a three-dimensional network through π–π stacking (figure 3). The face-to-face distance of adjacent planes is 3.510 Å (3.3907 Å for **2**), and the dihedral angle is 0.0° (0.0° for **2**) [11]. Hydrogen bonds between the H of 2,2′-bpy and perchlorate (C6 ⋯ O4, 3.3188 Å) play an important role in constructing the 3-D structure (table 4 and ST1).

3.2.2. Structure (3). In **3**, the Cu(II) is coordinated by three N atoms from one phen, one tza, and two O atoms from two tza ligands. The coordination geometry around Cu(II) is a distorted square pyramid as described by the τ parameter 0.39 [12]. The equatorial plane is formed by N1/N2/N4/O2A, and O1B occupies the axial position (figure 4). The Cu(II) lies at 0.2829 Å from the equatorial plane. The Cu–O bond distances (table 5) are reasonable for complexes of this type [13].

Each tza is also a μ_3-bridging ligand (mode **b** in scheme 1) as in **1** and **2**. One N and two carboxylate O atoms link the Cu(II) ions into a 1-D zigzag double chain structure. The 3-D structure is formed by π–π stacking (face-to-face distance 3.626 Å, dihedral angle 0.365°) and C–H ⋯ O (C9 ⋯ O4, 3.3154 Å, C10 ⋯ O6, 3.3914 Å, C14 ⋯ O6, 3.1351 Å) nonclassical hydrogen bonds (figure 5, table 6) [11].

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

Cd(1)–O(1A)	2.227(2)	Cd(1)–N(5)	2.341(3)
Cd(1)–O(2B)	2.234(2)	Cd(1)–N(1)	2.342(3)
Cd(1)–O(7W)	2.338(3)	Cd(1)–N(6)	2.353(3)
O(1A)–Cd(1)–O(2B)	115.12(10)	O(7W)–Cd(1)–N(1)	170.74(12)
O(1A)–Cd(1)–O(7W)	89.59(14)	N(5)–Cd(1)–N(1)	85.37(10)
O(2B)–Cd(1)–O(7W)	86.55(13)	O(1A)–Cd(1)–N(6)	87.75(10)
O(1A)–Cd(1)–N(5)	154.21(11)	O(2B)–Cd(1)–N(6)	155.00(11)
O(2B)–Cd(1)–N(5)	89.23(11)	O(7W)–Cd(1)–N(6)	83.52(13)
O(7W)–Cd(1)–N(5)	100.82(14)	N(5)–Cd(1)–N(6)	70.31(11)
O(1A)–Cd(1)–N(1)	87.60(10)	N(1)–Cd(1)–N(6)	105.17(10)
O(2B)–Cd(1)–N(1)	86.66(10)		

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

Co(1)–O(1A)	2.052(5)	Co(1)–N(5)	2.138(6)
Co(1)–O(2B)	2.039(4)	Co(1)–N(1)	2.149(5)
Co(1)–O(3)	2.160(5)	Co(1)–N(6)	2.146(5)
O(1A)–Co(1)–O(2B)	101.78(19)	N(1)–Co(1)–O(3)	177.2(2)
O(1A)–Co(1)–O(3)	90.7(2)	N(5)–Co(1)–N(1)	91.4(2)
O(2B)–Co(1)–O(3)	90.6(2)	O(1A)–Co(1)–N(6)	89.2(2)
O(1A)–Co(1)–N(5)	164.9(2)	O(2B)–Co(1)–N(6)	168.8(2)
O(2B)–Co(1)–N(5)	93.2(2)	O(3)–Co(1)–N(6)	87.1(2)
O(3)–Co(1)–N(5)	91.4(2)	N(5)–Co(1)–N(6)	75.9(2)
O(1A)–Co(1)–N(1)	86.7(2)	N(1)–Co(1)–N(6)	93.9(2)
O(2B)–Co(1)–N(1)	88.9(2)		

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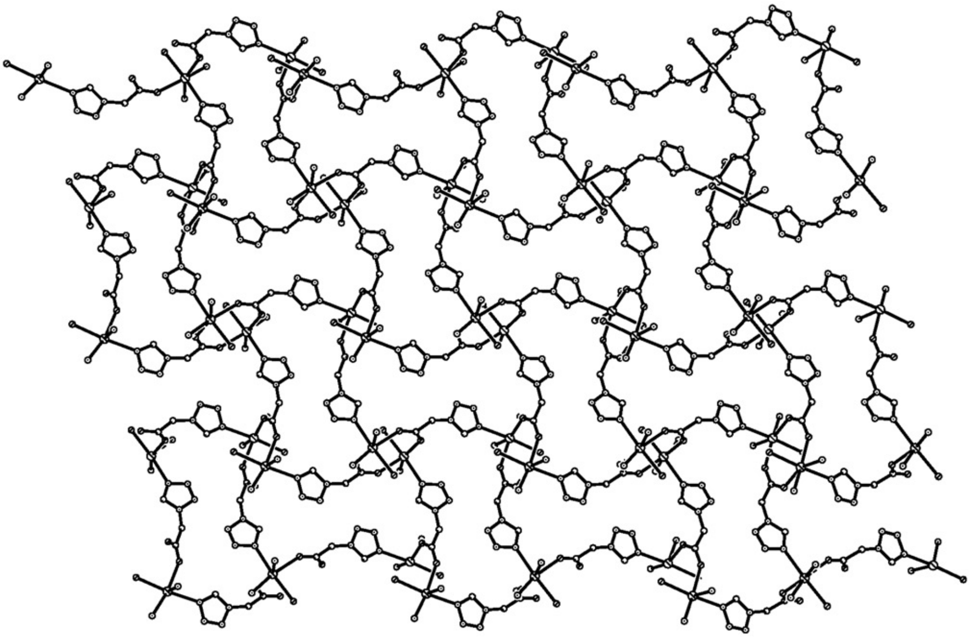

Figure 2. The layer structure of **1** (2,2′-bpy, ClO_4^-, and H atoms are omitted for clarity).

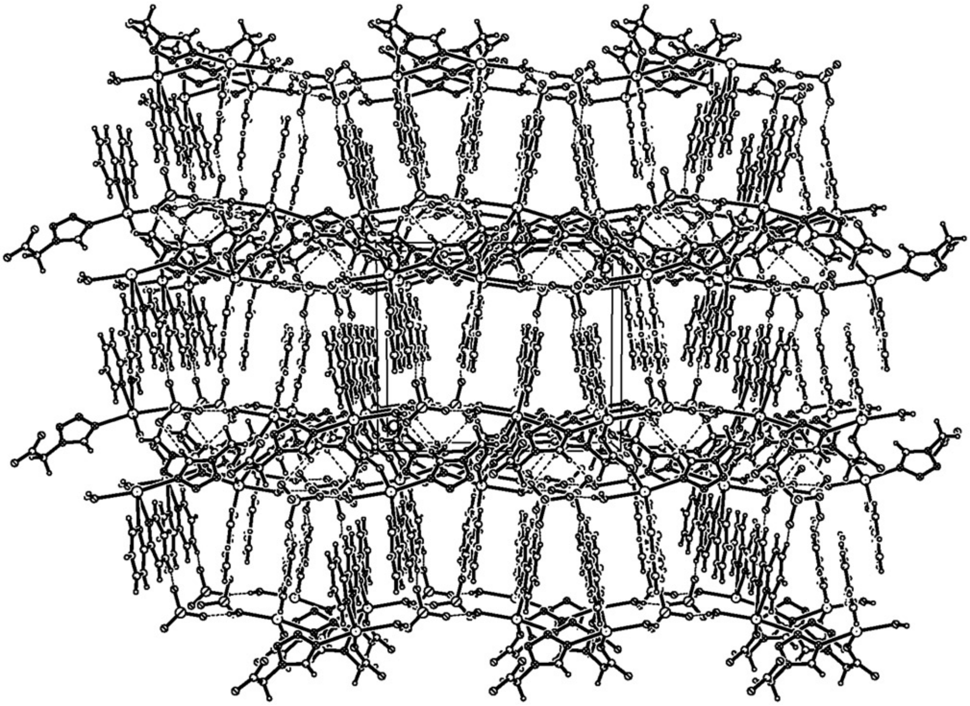

Figure 3. The 3-D supramolecular network of **1**.

Table 4. Hydrogen-bonding geometry (Å, °) for **1**.

D–H···A	*d*(D–H) (Å)	*d*(H···A) (Å)	*d*(D···A) (Å)	∠DHA (°)
O(7W)–H(7A)···O(5)	0.7582	2.2152	2.9676	171.83
O(7W)–H(7B)···O(6)	0.8203	2.1734	2.9728	164.82
C(2)–H(2A)···N(3)	0.9707	2.5035	3.4466	163.91
C(2)–H(2B)···O(5)	0.9694	2.5996	3.2632	125.84
C(1)–H(1)···O(6)	0.9297	2.4840	3.4039	170.23
C(6)–H(6)···O(4)	0.9295	2.4584	3.3188	153.98

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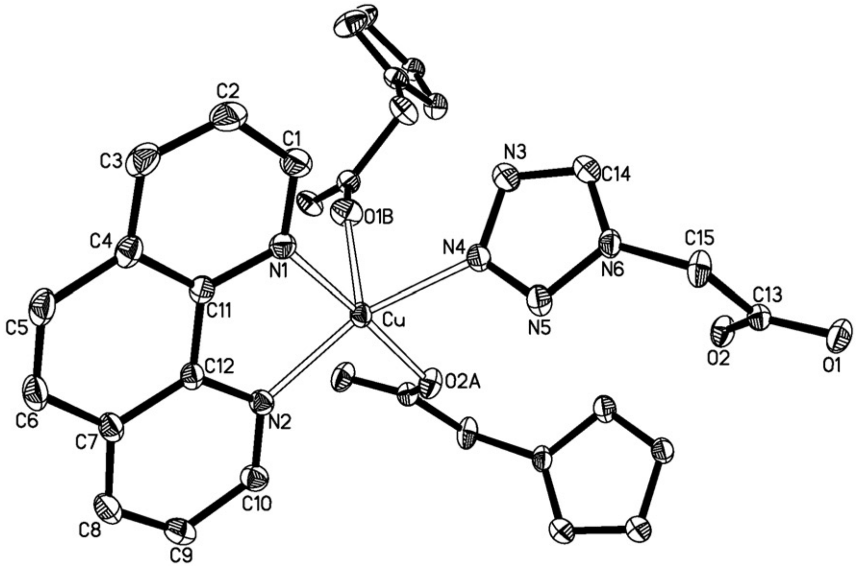

Figure 4. Perspective view of **3**, showing 30% probability ellipsoids for the atoms and the atom-labeling scheme (ClO_4^- is omitted for clarity).

Table 5. Selected bond lengths (Å) and angles (°) for **3**.

Cu–O(2A)	1.9352(17)	Cu–N(1)	2.003(2)
Cu–N(2)	2.027(2)	Cu–N(4)	2.088(2)
Cu–O(1B)	1.497(18)		
O(2A)–Cu–N(1)	175.55(8)	O(2A)–Cu–N(2)	95.53(8)
N(1)–Cu–N(2)	81.76(8)	O(2A)–Cu–N(4)	86.60(8)
N(1)–Cu–N(4)	94.14(8)	N(2)–Cu–N(4)	151.93(8)
O(2A)–Cu–O(1B)	96.53(8)	N(1)–Cu–O(1B)	87.87(8)
N(2)–Cu–O(1B)	118.08(8)	N(4)–Cu–O(1B)	89.32(8)

3.3. *IR spectrum*

Two very strong bands in the IR spectra of **1** and **2**, centered at 1631 and 1673 cm^{-1}, respectively, were assigned to the carboxylate. The absorption at 3446 cm^{-1} for **1** and 3447 cm^{-1} for **2** are attributed to coordinated H_2O. The absorptions at 1475, 1439,

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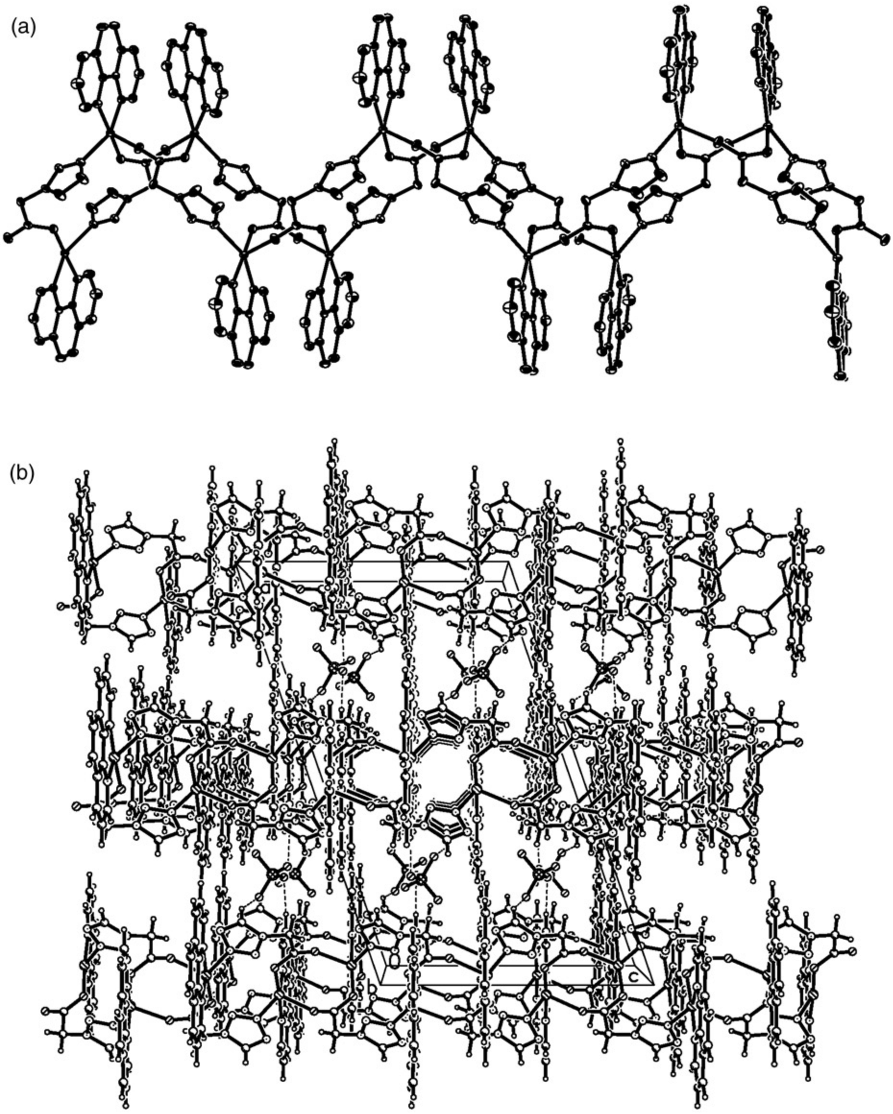

Figure 5. (a) 1-D zigzag double chain of **3**; (b) Perspective view of stacking array of **3** along *b*-axis.

Table 6. Hydrogen-bonding geometry (Å, °) for **3**.

D–H ⋯ A	*d*(D–H) (Å)	*d*(H ⋯ A) (Å)	*d*(D ⋯ A) (Å)	∠DHA (°)
C(9)–H(9A) ⋯ O(4)	0.9301	2.5563	3.3154	139.05
C(10)–H(10A) ⋯ O(6)	0.9299	2.5820	3.3914	145.77
C(14)–H(14A) ⋯ O(6)	0.9300	2.3490	3.1351	142.08

and 1384 $\mathrm{cm^{-1}}$ for **1** and 1473, 1444, and 1403 $\mathrm{cm^{-1}}$ for **2** are assigned to stretching of –N=C– and –N=N– in the ligands, similar to $[\mathrm{Cd(tza)_2}]$ and $[\mathrm{Zn(tza)_2}]$ [8(d)].

The IR spectrum of **3** shows $\mathrm{COO^-}$ at 1650 $\mathrm{cm^{-1}}$. The absorptions at 1522, 1428, and 1393 $\mathrm{cm^{-1}}$ are attributed to stretching of –N=C– and –N=N– in the ligands, similar to other Cu(II) complexes with Htza [8(b)].

The IR spectra show strong absorptions at 1121 and 1087 $\mathrm{cm^{-1}}$ for **1**, 1121 and 1100 $\mathrm{cm^{-1}}$ for **2**, and 1120 and 1091 $\mathrm{cm^{-1}}$ for **3**, for $\mathrm{ClO_4^-}$.

4. Conclusions

In summary, three new tetrazole-1-acetate polymers were synthesized and characterized by X-ray single-crystal diffraction. Complexes **1** and **2** consist of a 2-D layer which is linked by the π–π interaction of 2,2′-bpy into 3-D architectures. Complex **3** is a 1-D zigzag double chain structure with the 3-D structure formed by π–π stacking interaction of phen and nonclassical hydrogen bonds.

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