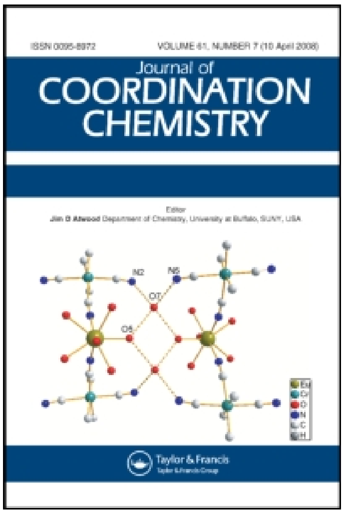

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Syntheses, structures, and fluorescent properties of three Zn(II) and Cu(II) complexes of different ligands derived from 3,6-*bis*(2-pyridyl)-1,2-dihydro-1,2,4,5-tetrazine

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Syntheses, structures, and fluorescent properties of three Zn(II) and Cu(II) complexes of different ligands derived from 3,6-*bis*(2-pyridyl)-1,2-dihydro-1,2,4,5-tetrazine

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Three supramolecular complexes $[Zn(H\mathbf{L_1})_2(H_2O)_2(ZnCl_4)_2]$ (**1**), $[Cu(\mathbf{L_2})_2Cl_2]$ (**2**), and $[Zn(\mathbf{L_3})Cl_2]$ (**3**) have been synthesized and characterized by single crystal X-ray diffraction analysis ($\mathbf{L_1}$ = 3,5-di(2-pyridyl)-4-amino-1,2,4-triazole, $\mathbf{L_2}$ = 3,5-di(2-pyridyl)-1,2,4-triazole, and $\mathbf{L_3}$ = 2-pyridinecarboxylic acid (pyridin-2-ylmethylene)-hydrazide). In **1**, anion–π interactions between Cl^- and the π-systems of $\mathbf{L_1}$ are observed and anion–π, hydrogen bonding and π–π stacking interactions link the two complex units of $[Zn(H\mathbf{L_1})_2(H_2O)_2]^{4+}$ and $[ZnCl_4]^{2-}$ to form a 3-D supramolecular network. In **2**, π–π stacking interactions between aromatic rings of 1,2,4-triazole and pyridine rings are observed; in **3**, hydrogen bonding of $Cl\cdots H–N$ and π–π stacking interactions between parallel pyridine rings of L_3 are observed. The mechanisms of rearrangement reactions of **L** to $\mathbf{L_1}$–$\mathbf{L_3}$ are discussed. The fluorescent properties for solid **1** and **3** are also investigated.

Keywords: Zn(II) and Cu(II) complexes; Anion–π interactions; Crystal structure; Fluorescent properties

1. Introduction

Design and construction of metal supramolecular complexes with specific network has become an active area [1, 2]. A successful approach to build supramolecular frameworks is to select suitable multidentate ligands as building blocks [3]. Among them, heterocyclic tetrazine ligands are candidates for their versatile coordination modes [4–6]. The ligand **L** can transform to other forms (scheme 1) and form crystal structures not only using different coordination modes to transition metal ions but also formation of complementary intermolecular hydrogen bonding and the π–π stacking interactions [7–11]. The π–π stacking and hydrogen bonding interactions organize molecular components in the crystalline state [12–14]. The anion–π interactions are observed between π-acidic systems as diverse as tetrazines, triazines, cyanuric acids, and halides, or multi-atomic anions such as NO_3^- [15–18]. However, anion–π interactions

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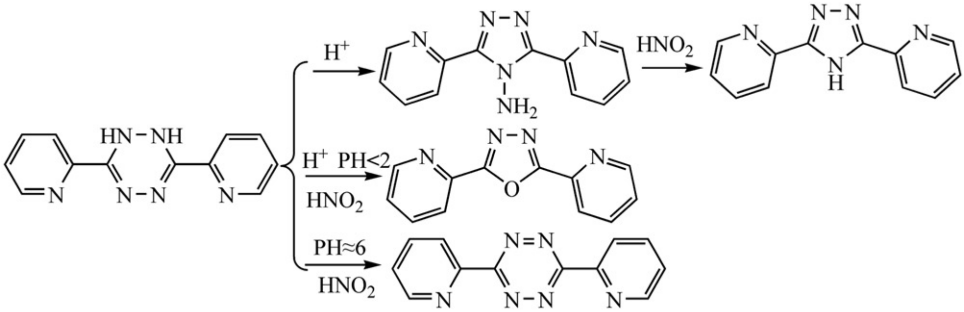


Scheme 1. Possible formation of three different compounds from **L**.

between Cl^- and 3,5-di(2-pyridyl)-4-amino-1,2,4-triazole were not reported previously. Here, three supramolecular complexes **1**–**3** are reported.

2. Experimental

2.1. *Materials and physical measurements*

All reagents were purchased from commercial sources without purification. Deionized water was used for conventional synthesis. Elemental analyses (C, H, and N) were carried out using a Perkin Elmer analyzer model 240. IR (KBr pellets) spectra were recorded from 400–4000 cm^{-1} using a Bruker Tensor 27 spectrophotometer. UV spectra were measured on a UV-2550 UV-Vis spectrophotometer. Excitation and emission spectra were recorded on a F-4500 fluorescence spectrophotometer.

2.2. *Preparation of complexes*

2.2.1. Synthesis of L. **L** was synthesized according to the literature (25% yield) [11].

2.2.2. Synthesis of [Zn(HL$_1$)$_2$(H$_2$O)$_2$(ZnCl$_4$)$_2$] (1). A solution of **L** (47.2 mg, 0.2 mmol) and HCl (0.5 mL, 2 mol L^{-1}) in methanol (20 mL) was slowly added to the stirred solution of $ZnCl_2 \cdot 4H_2O$ (41.6 mg, 0.2 mmol) in water (20 mL). After the solution was stirred for 40 min, the reaction mixture was filtered. After a few days of slow evaporation of the solvent, light yellow crystals suitable for X-ray crystallography were collected and dried in air (45% yield based on **L$_1$**). Elemental Anal. Calcd for $Zn_3(C_{12}H_{11}N_6)_2Cl_8 \cdot 2H_2O$ (%): C, 29.02; H, 2.43; and N, 16.93. found: C, 29.12; H, 2.58; and N, 16.73.

2.2.3. Synthesis of [Cu(L$_2$)$_2$Cl$_2$] (2). A mixture of **L** (47.2 mg, 0.2 mmol) and $CuCl_2 \cdot 2H_2O$ (34.2 mg, 0.2 mmol), $NaNO_2$ (30 mg), HCl (0.5 mL, 2 mol L^{-1}), methanol (10 mL), and water (10 mL) was heated in a 25 mL Teflon-lined autoclave at 160°C for

3 days, followed by slowly cooling (5°C h^{-1}) to room temperature. The resulting mixture was filtered and washed with 95% methanol, and blue crystals of **2** were collected and dried in air (55% yield based on $\mathbf{L_2}$). Elemental Anal. Calcd for $Cu(C_{12}H_9N_5)_2Cl_2$ (%): C, 49.58; H, 3.10; and N, 24.10. Found: C, 49.26; H, 3.38; and N, 24.03.

2.2.4. Synthesis of [Zn($\mathbf{L_3}$)$\mathbf{Cl_2}$] (3). A solution of $ZnCl_2 \cdot 4H_2O$ (102 mg, 0.8 mmol) in water (20 mL) was slowly added to the stirred solution of **L** (23.6 mg, 0.1 mmol), HCl (0.5 mL, 2 mol L^{-1}), and $NaNO_2$ (20.7 mg, 0.3 mg) in methanol (20 mL). After the solution was stirred for 40 min, the reaction mixture was filtered. A few days of slow evaporation of the solvent gave red crystals suitable for X-ray crystallography in 43% yield based on $\mathbf{L_3}$. Elemental Anal. Calcd for $Zn(C_{12}H_{10}N_4O)Cl_2$ (%): C, 39.72; H, 2.76; and N, 15.45. Found: C, 40.19; H, 2.78; and N, 15.38.

2.3. *X-ray single crystal structure determination*

Diffraction data collection was performed on a Bruker Smart APEX-II CCD at 296(2) K, using graphite monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). Empirical absorption corrections were applied by using SADABS. The structures of **1–3** were solved by direct methods and refined by full-matrix least-squares on F^2 in SHELXL-97 program. All non-hydrogen atoms were refined anisotropically and hydrogens were placed in calculated positions and refined isotropically. The crystal data, details on data collection and refinement, are summarized in table 1 and selected bond lengths and angles are given in table 2.

3. Results and discussion

3.1. *Reaction mechanisms*

Initially, we chose ligand **L** to react with 3-D metal salts. We find **L** decomposes to give three different ligands $\mathbf{L_1}$–$\mathbf{L_3}$ under different conditions. The reaction mechanisms of **L** to $\mathbf{L_1}$–$\mathbf{L_3}$ are proposed, as shown in scheme 2. **L** rearranges into $\mathbf{L_1}$ under acid environment as well as metal assistance. Chelating to metal center can further delocalize the π electron density of the imino group and enhance the nucleophilic reactivity of carbon [19], resulting in opening of the tetrazine ring and rearrangement to a 4-amino-1,2,4-triazole complex. $\mathbf{L_2}$ is afforded after deamination of $\mathbf{L_1}$. In this case, deamination occurs under high pressure and high temperature as well as metal assistance. In the synthesis of **3**, $\mathbf{L_3}$ is obtained. It is reasonable to postulate the conversion of **L** to $\mathbf{L_3}$ *via* opening of the tetrazine ring, simultaneous attack by water on the activated carbon, and oxidation by nitrous acid with elimination of a molecule of hydrazine, then rearranging into $\mathbf{L_3}$. The synthesis of $\mathbf{L_3}$ is different from what is reported in the literature [20].

3.2. *The crystal structure of 1*

X-ray structural analysis reveals that **1** crystallizes in the monoclinic system, space group $P2_1/c$. Each unit of **1** consists of a Zn^{2+}, two $\mathbf{L_1}$ ligands, two coordinated waters,

Table 1. Crystallographic data and refinement parameters for **1**–**3**.

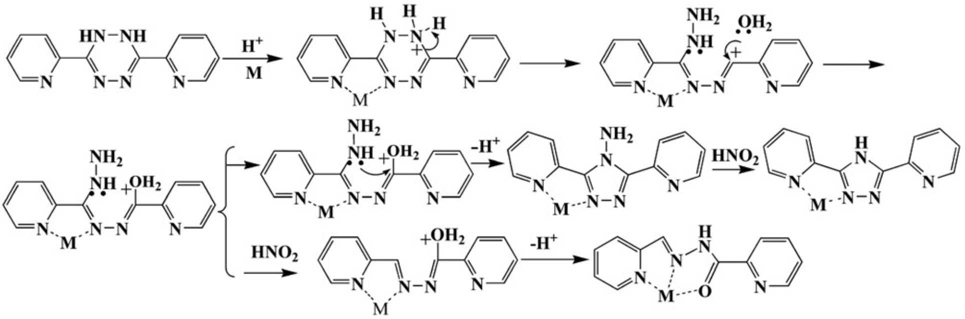
Compound	**1**	**2**	**3**
Empirical formula	$Zn_3(C_{12}H_{11}N_6)_2Cl_8 \cdot 2H_2O$	$Cu(C_{12}H_9N_5)_2Cl_2$	$Zn(C_{12}H_{10}N_4O)Cl_2$
Formula weight	992.26	580.93	362.53
Color	Light yellow	Blue	Red
Crystal system	Monoclinic	Triclinic	Monoclinic
Space group	$P2_1/c$	P-1	$P2_1/c$
Unit cell dimensions (nm, °)			
a	9.7711(2)	8.0238(2)	14.0006(3)
b	15.3660(3)	9.1026(2)	13.8280(2)
c	15.2748(4)	9.4284(2)	7.47680(10)
α	90.00	116.446(2)	90.00
β	127.842(2)	90.628(2)	101.4300(10)
γ	90.00	102.832(2)	90.00
Volume (nm^3), Z	1811.11(7), 2	596.39(3), 1	1418.80(4), 4
Calculated density (g cm^{-3})	1.820	1.617	1.697
F(000)	988.0	295.0	728.0
μ(Mo-Kα) (mm^{-1})	2.606	1.177	2.106
Reflections collected (R_{int})	15,811 (0.0494)	9781 (0.0315)	12,080 (0.0282)
Data/restraints/parameters	4169/0/223	2741/0/170	3263/0/222
Goodness-of-fit on F^2	1.028	0.971	1.055
Final R indices [$I > 2\sigma(I)$]	$R_1{}^a = 0.0403$, $wR_2{}^b = 0.1066$	$R_1{}^a = 0.0416$, $wR_2{}^b = 0.1074$	$R_1{}^a = 0.0338$, $wR_2{}^b = 0.0823$
R indices (all data)	$R_1{}^a = 0.0647$, $wR_2{}^b = 0.1276$	$R_1{}^a = 0.0537$, $wR_2{}^b = 0.1157$	$R_1{}^a = 0.0532$, $wR_2{}^b = 0.0907$

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

Table 2. Selected bond lengths (Å) and angles (°) for **1**–**3**.

1			
Zn(1)–N(2)	2.074(5)	Zn(1)–N(5)	2.145(4)
Zn(1)–O(1)	2.221(3)	Zn(1)–N(2)^{i}	2.074(5)
Zn(1)–N(5)^{i}	2.145(4)	Zn(1)–O(1)^{i}	2.221(3)
N(2)–Zn(1)–N(2)^{i}	179.99(12)	N(2)–Zn(1)–N(5)	77.51(10)
N(2)^{i}–Zn(1)–N(5)	102.49(10)	N(2)–Zn(1)–N(5)^{i}	102.49(10)
N(2)^{i}–Zn(1)–N(5)^{i}	77.51(10)	N(5)–Zn(1)–N(5)^{i}	180.00(11)
N(2)–Zn(1)–O(1)	90.78(11)	N(2)^{i}–Zn(1)–O(1)	89.22(11)
N(5)–Zn(1)–O(1)	87.07(11)	N(5)^{i}–Zn(1)–O(1)	92.93(11)
N(2)–Zn(1)–O(1)^{i}	89.22(11)	N(2)^{i}–Zn(1)–O(1)^{i}	90.78(11)
N(5)–Zn(1)–O(1)^{i}	92.93(11)	N(5)^{i}–Zn(1)–O(1)^{i}	87.07(11)
O(1)–Zn(1)–O(1)^{i}	180.00(11)		
2			
Cu(1)–N(3)	2.5224(25)	Cu(1)–N(3)^{ii}	2.5224(25)
Cu(1)–N(5)^{ii}	1.987(3)	Cu(1)–N(5)	1.987(3)
Cu(1)–Cl(1)^{ii}	2.307(2)	Cu(1)–Cl(1)	2.307(2)
N(5)^{i}–Cu(1)–N(5)	180.00(11)	N(5)^{ii}–Cu(1)–Cl(1)^{ii}	89.88(8)
N(5)–Cu(1)–Cl(1)^{ii}	90.12(8)	N(5)^{ii}–Cu(1)–Cl(1)	90.12(8)
N(5)–Cu(1)–Cl(1)	89.88(8)	Cl(1)^{ii}–Cu(1)–Cl(1)	179.99(3)
3			
Zn(1)–N(2)	2.109(2)	Zn(1)–N(1)	2.187(2)
Zn(1)–Cl(2)	2.215(1)	Zn(1)–Cl(1)	2.234(1)
Zn(1)–O(1)	2.265(2)		
N(2)–Zn(1)–Cl(2)	125.32(6)	N(2)–Zn(1)–N(1)	73.97(8)
N(2)–Zn(1)–Cl(1)	113.10(6)	N(1)–Zn(1)–Cl(2)	100.31(6)
Cl(2)–Zn(1)–Cl(1)	121.31(3)	N(2)–Zn(1)–O(1)	72.06(7)
Cl(2)–Zn(1)–O(1)	95.50(6)	N(1)–Zn(1)–Cl(1)	100.23(6)
N(1)–Zn(1)–O(1)	145.72(8)		

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



Scheme 2. Proposed mechanisms for the rearrangement reactions of ligands $\mathbf{L_1}$–$\mathbf{L_3}$ from **L**.

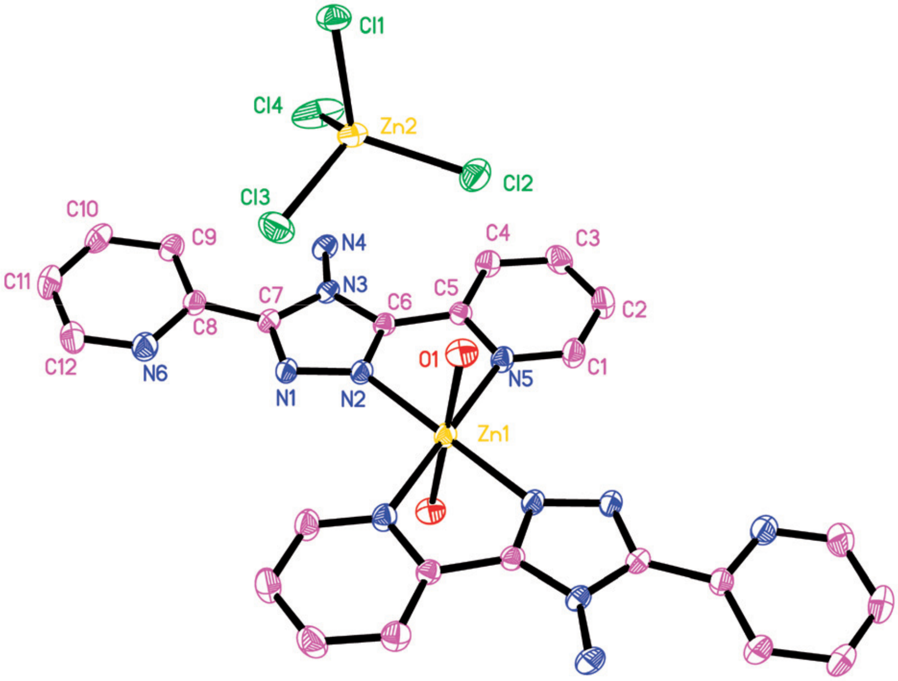


Figure 1. ORTEP drawing of **1** with atomic numbering scheme; thermal ellipsoids are drawn at the 30% level and hydrogens are omitted for clarity.

and two $[ZnCl_4]^{2-}$ anions. Zn^{2+}(1) coordinates to four nitrogens from two $\mathbf{L_1}$ ligands and two oxygens of water to form octahedral geometry, as shown in figure 1. $\mathbf{L_1}$ is a chelating ligand, using N(2) and N(5) coordinating to the same Zn^{2+} with bond lengths of Zn(1)–N(2) 2.074(3) Å, Zn(1)–N(5) 2.145(3) Å. Two oxygens of coordinated water are axial. Zn^{2+}(2) coordinates with four Cl^- ions. The most interesting part of the structure of **1** is the noncovalent anion–π interactions formed between Cl^- and the π-systems of $\mathbf{L_1}$ (figure 2a). Three Cl^- ions of $[ZnCl_4]^{2-}$ are involved in anion–π interactions, where Cl^-(2) interacts with the π-system of two pyridine rings and Cl^-(1) and Cl^-(3) interact with the π-system of triazole rings from two $\mathbf{L_1}$. The distance

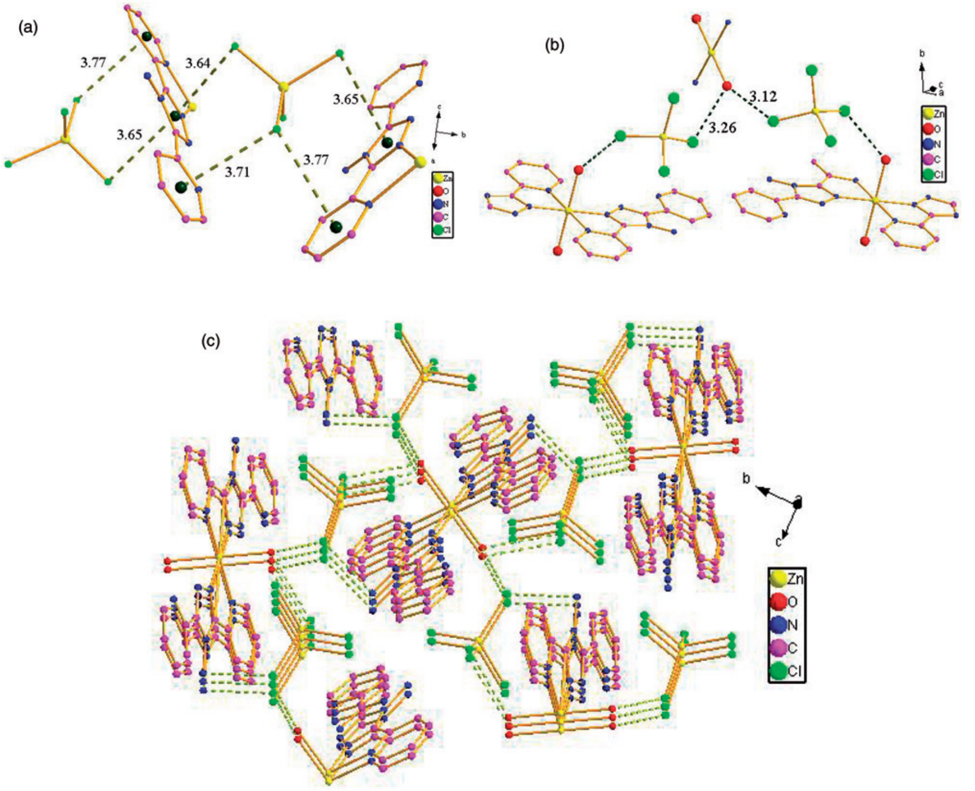


Figure 2. (a) View of the anion–π interactions in **1**. (b) View of the hydrogen bonds in **1**. (c) The crystal packing of **1**.

between $Cl^-(2)$ and the centroid of the two pyridine rings is 3.71 Å and the distances between $Cl^-(1)$, $Cl^-(3)$ and the centroid of triazole rings are 3.64, 3.65 Å. That these distances are shorter than 4 Å and the plane-centroid-anion angle ranges from 72° to 90°, which indicates the existence of anion–π interactions [17]. The anions of $[ZnCl_4]^{2-}$ and coordinated water take part in forming hydrogen bond. The coordinated water O(1) connects two adjacent $[ZnCl_4]^{2-}$ through $O(1)\cdots Cl(4^i)$ (3.132 Å) (symmetry code: (i) $-x+1$, $-0.5-y$, $-0.5+z$) and $O(1)\cdots Cl(2)$ (3.268 Å) (figure 2b). The formation of the π–π interactions between the parallel pyridine rings with distances below 3.6 Å are also observed. The range of supramolecular interactions including anion–π interactions, hydrogen bonds, and π–π stacking interactions link the units into a 3-D supramolecular network (figure 2c).

3.3. *The crystal structure of 2*

The X-ray structure determination reveals that **2** crystallizes in the triclinic system, space group *P*-1. Each unit of **2** consists of one central Cu^{2+}, two chlorides, and two $\mathbf{L_2}$ (figure 3). The Cu^{2+} coordinates to four triazole nitrogens coming from two $\mathbf{L_2}$ and two Cl^-. Complex **2** shows the preference of Cu(II) to form a tetragonally distorted

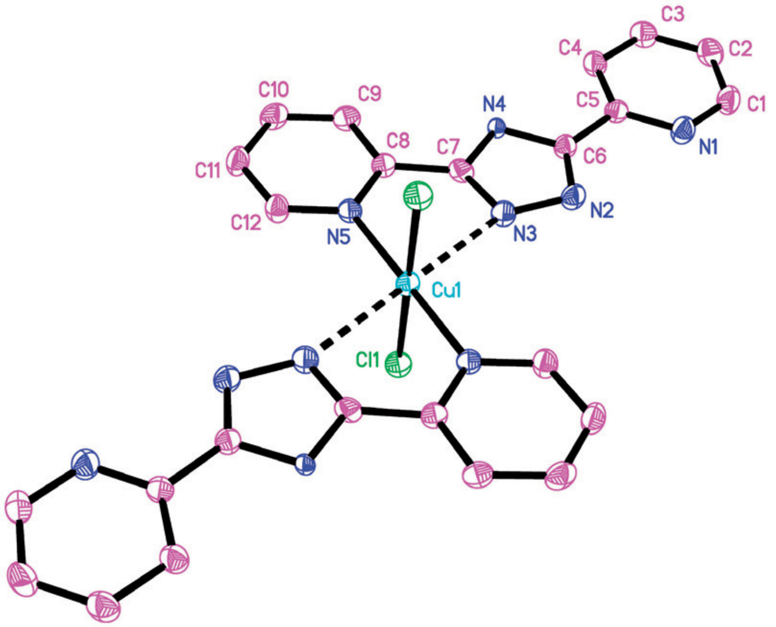


Figure 3. ORTEP drawing of **2** with atomic numbering scheme; thermal ellipsoids are drawn at the 30% level and hydrogens are omitted for clarity.

octahedron with four strong equatorial bonds and two longer axial bonds Cu(1)–N(3) (2.5224(25) Å). The shortest Cu $\cdots$ Cu^{i} (symmetry code: (i) x, y, z) is found to be 8.0 Å. In addition, the shortest centroid-to-centroid distance between aromatic rings of 1,2,4-triazole group and pyridine rings is 3.62 Å that indicates a π–π stacking interaction between aromatic rings (figure 4a). The crystal packing of **2** is shown in figure 4(b).

3.4. *The crystal structure of 3*

X-ray structural analysis reveals that **3** crystallize in the monoclinic system, space group $P2_1/c$. Each unit consists of a Zn^{2+}, one $\mathbf{L_3}$, and two Cl^- ions (figure 5). The Zn^{2+} coordinates to two nitrogens and an oxygen from tridentate $\mathbf{L_3}$ and two Cl^- ions to form a tetragonal pyramidal geometry. Hydrogen bonds of Cl $\cdots$ H–N are observed between molecules as shown in figure 6. Cl(1) connects two adjacent $\mathbf{L_3}$ through Cl(1) $\cdots$ N(3^{i}) (3.32 Å) (symmetry code: (i) $1-x$, $0.5+y$, $1.5-z$). The bond angle of Cl(1) $\cdots$ H–N(3^{i}) is 148.7°. Weak π–π stacking interactions are also observed between parallel pyridine rings of $\mathbf{L_3}$ with the centroid–centroid distance of 3.74 Å (figure 6).

3.5. *IR spectra*

The IR spectrum of **L** (figure S1) exhibits four absorption regions: the $\nu_{N\text{–}H}$ vibration at 3325 cm^{-1}, the sharpest band split into $\nu_{C=N}$ vibration band at 1625 cm^{-1}, $\nu_{N=N}$ vibration band at 1587 cm^{-1}, the bands of the C–N stretching vibration at 1040 cm^{-1},

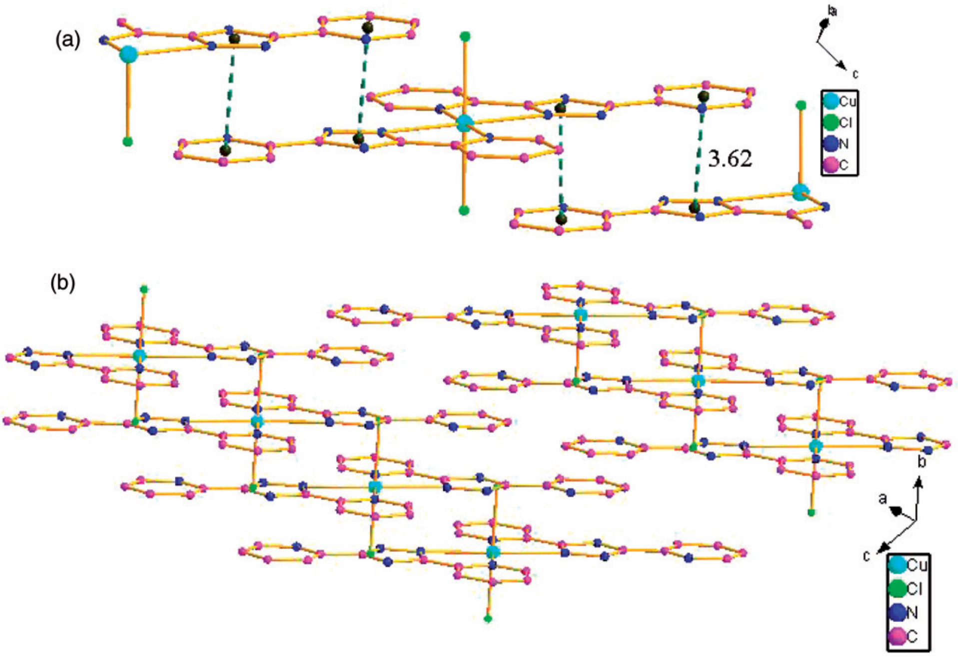


Figure 4. (a) View of π–π interactions in **2**. (b) The crystal packing of **2**.

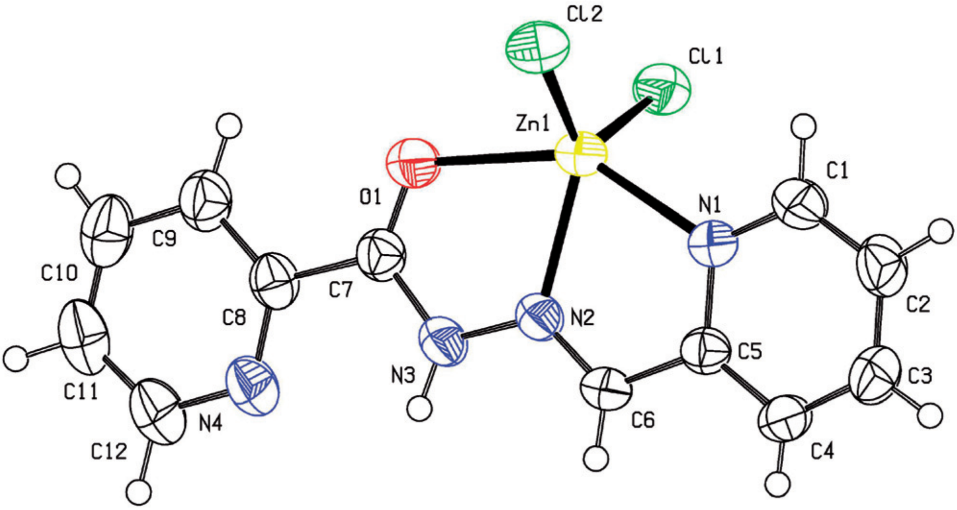


Figure 5. ORTEP drawing of **3** with atomic numbering scheme; thermal ellipsoids are drawn at the 30% level.

and the $\gamma_{\text{N–H}}$ vibration band at 744 cm^{-1}. In **1** and **2**, the $\nu_{\text{N–H}}$ vibration and $\gamma_{\text{N–H}}$ vibration shift to higher frequency (3618, 3446, 778 cm^{-1} for **1**, 3446, 776 cm^{-1} for **2**) due to the coordination of the imine nitrogen. In **3**, the $\nu_{\text{N–H}}$ vibration of the amide group at 3186 cm^{-1} and $\gamma_{\text{N–H}}$ vibration shift to higher frequency 783 cm^{-1}.

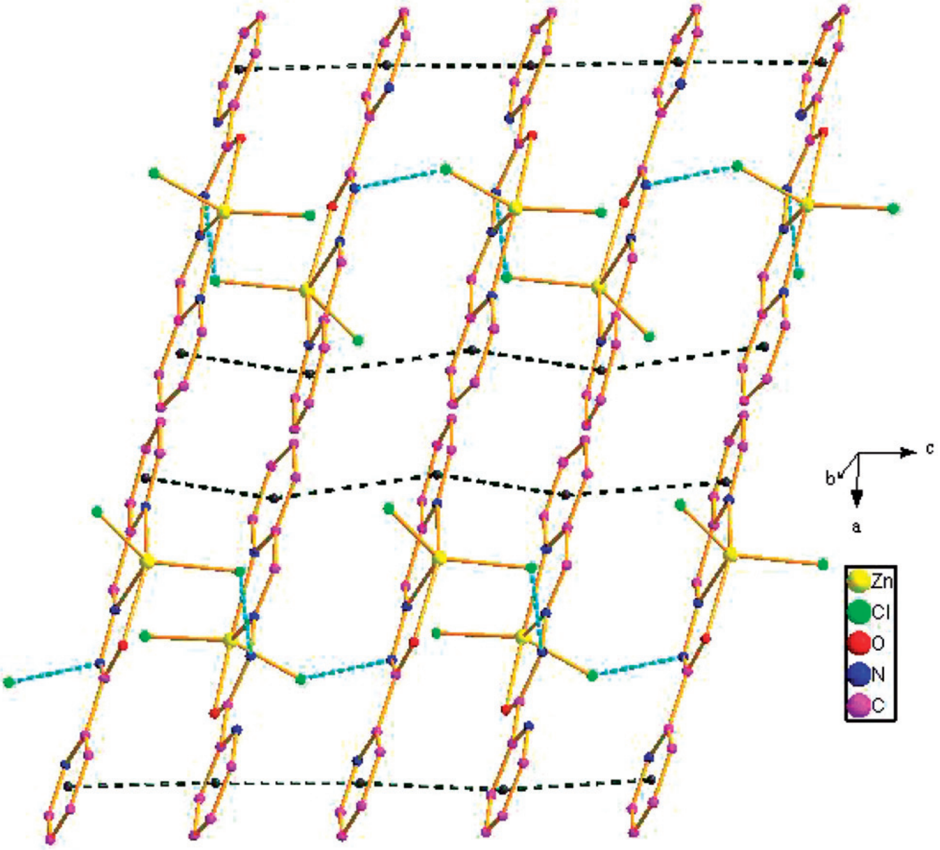


Figure 6. View of hydrogen bonds and π–π interactions in **3**.

3.6. *UV-Vis spectra of 1,2 and fluorescence spectra studies of 1,3*

The UV-Vis absorption spectra of **1** and **2** in water ($10^{-5}\,\mathrm{mol\,L^{-1}}$) are shown in "Supplementary material." Absorption spectra of **1** and **2** display similar peak positions around 305 nm ($\varepsilon_{\max} = 3.1 \times 10^4\,(\mathrm{mol\,L^{-1}})^{-1}\,\mathrm{cm^{-1}}$ for **1**, $\epsilon_{\max} = 1.62 \times 10^4\,(\mathrm{mol\,L^{-1}})^{-1}$ $\mathrm{cm^{-1}}$ for **2**), attributed to π–π^* transitions.

Photoluminescence spectra of solid samples of **1** and **3** at room temperature are shown in figure 7. The emission spectrum of **1** exhibits maximum at 451 nm with excitation wavelength at 305 nm (figure 7a). The excitation peak of solid **1** corresponds with the position of the UV-Vis absorption. The emission spectrum of **3** obtained with excitation wavelength at 395 nm exhibits maximum at 476 nm (figure 7b). The luminescence of **1** and **3** is attributed to π–π^* transitions of $\mathbf{L_1}$ and $\mathbf{L_3}$, respectively.

4. Conclusion

Three complexes have been synthesized and reported. Although many Zn(II) and Cu(II) complexes containing triazole or pyridinecarboxylate ligands have been found in recent

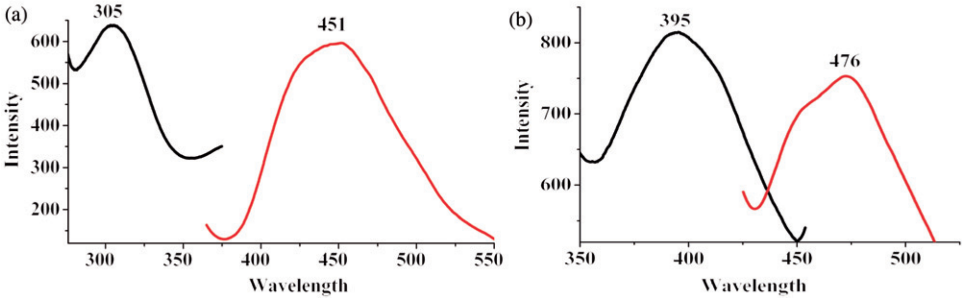


Figure 7. The emission (red line) and excitation (black line) spectra at room temperature for: (a) **1** and (b) **3**.

literature [21–25], the complexes **1**–**3** constructed from three different N-containing ligands $\mathbf{L}_1$–$\mathbf{L}_3$ derived *in situ* from 3,6-*bis*(2-pyridyl)-1,2-dihydro-1,2,4,5-tetrazine are first reported. The supramolecular interactions containing the anion–π, hydrogen bonding, and π–π stacking in **1**–**3** have been also found and discussed.

Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC No. 746304 for **1**, 746305 for **2**, and 746306 for **3**. Copies of this information can be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44 1223 336 033; E-mail: deposit@ccdc.cam.ac.uk).

Acknowledgments

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