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Zn Coordination polymers based on designed of tetrazole-yl acylamide: syntheses, structures and luminescence property

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Three Zn(II) coordination polymers, $[Zn(L^1)_2]_n$ (1), $[Zn(L^1)(5-ATZ)]_n$ (2) $(HL^1 = N-(1H-tetrazol-5-yl)benzamide)$, and $[Zn(L^2)_2(H_2O)]$ (3) $(L^2 = 4-methyl-N-(1H-tetrazol-5-yl)benzamide)$ have been synthesized under hydrothermal conditions and characterized by single-crystal X-ray diffraction. Single-crystal X-ray diffraction analyses showed that 1 and 2 both display two-dimensional architectures with diverse topologies. Compound 1 was a 4⁴ network based on Zn center, while compound 2 presents a 6³ topology. Compound 3 exhibits mononuclear structure, which was connected to three-dimensional architecture by hydrogen bonds. In addition, the fluorescence property and thermal stability of compound 1 have been studied.

Keywords: Zn(II) coordination polymers Topology Fluorescence property Thermal stability

In recent years, the design and synthesis of coordination polymers are gaining much more interest for their interesting structural topologies and wide potential applications as functional materials [1]. However, it is a long-standing challenge to design and synthesize new coordination polymers because of the central metals, the nature of the organic ligands as well as some external factors, such as solvent, temperature, pH value and anion, etc [2-8]. Therefore, in order to obtain novel structures with desirable functions, the selecting and designing reasonable organic ligands are particularly important [9]. Among diverse organic ligands, the polydentate N donor ligands have been employed extensively as chelating or bridging linkers [10]. 5-substitued 1*H*-tetrazoles have been extensively used in the synthesis of polymeric transition metal complexes due to their diversified coordination modes and structures [11]. However, examples of frameworks with the amide ligand of organic acid containing tetrazolate group have seldom been involved up to now [12]. Meanwhile, d¹⁰ metal ions present variable coordination numbers and geometries, and their compounds generally exhibit luminescent properties [13]. In this article, we use the amide ligand of benzoic acid containing tetrazolate group HL^1 and HL^2 successfully presenting their three novel Zn Coordination polymers, $[Zn(L^1)_2]_n$ (1), $[Zn(L^1)(5-ATZ)]_n$ (2) and $[Zn(L^2)_2(H_2O)]$ (3), and their structures were determined by single-crystal X-ray diffraction studies, also the fluorescence property and thermal stability of **1** have been studied.

1. Materials and method

All chemicals in the reactions were obtained from commercial sources without further purification excepting the ligands. The ligands N-(1H-tetrazol-5-yl) benzamide) (HL¹) and 4-methyl-N-(1*H*-tetrazol-5-yl)benzamide) (HL²) were synthesized according to the literature method [14,15], see part of syntheses.

Elemental analyses (C, H, and N) were performed on a Perkin-Elmer 240C analyzer, and IR spectra were measured on a Tensor 27 OPUS (Bruker) FT-IR spectrometer with KBr pellets. Emission spectra were taken on an F-4500 spectrofluorometer. Thermal stability (TG-DTA) studies were carried out on a Dupont thermal analyzer from room temperature to 800 °C at a rate of 10 °C·min-1. The X-ray powder

diffraction (XRPD) was recorded on a Rigaku D/Max-2500 diffractometer at 40 kV, 100 mA for a Cu-target tube and a graphite monochromator. Simulation of the XRPD spectra was carried out by the single-crystal data and diffraction-crystal module of the Mercury (Hg) program available free of charge via the Internet at <u>http://www.iucr.org</u>.

2. X-ray crystallography

Compounds 1-3 were collected on a computer-controlled carried out on a Scx-Mini diffractometer equipped with a graphite crystal monochromator situated in the incident beam. The determinations of unit cell parameters and data collections were performed with Mo-K α radiation ($\lambda = 0.71073$ Å) at 293(2) K and unit cell dimensions were obtained with least-squares refinements. The program SAINT [16a] was used for integration of the diffraction profiles. All the structures were solved by direct methods using the SHELXS program of the SHELXTL package and refined by full-matrix least-squares methods with SHELXL [16b]. Metal atoms in the compounds were located from the *E*-maps and other non-hydrogen atoms were located in successive difference Fourier syntheses and refined with anisotropic thermal parameters on F^2 . The hydrogen atoms of the ligand were generated geometrically. The detailed crystallographic data and selected bond dimensions for 1-3 are listed in Table 1 and 2. CCDC numbers: 899262, 899264, 899265.

Table 1

Crystallographic	data and	structure	refinement	details f	or compounds	1-3 .
2 2 1					1	

	1	2	3
Empirical formula	$C_{16}H_{12}ZnN_{10}O_2$	$C_9H_7ZnN_{10}O$	$C_{18}H_{18}ZnN_{10}O_3$
Formula weight	441.73	336.62	487.82
Crystal system	orthorhombic	monoclinic	monoclinic
Space group	Pbca	$P2_{1}/c$	Cc
T/K	293(2)	293(2)	293(2)
a/Å	8.4416(17)	10.270(2)	9.809(2)
b/Å	9.5149(19)	10.406(2)	11.830(2)
c/Å	20.268(4)	13.206(3)	17.643(3)
α/()	90	90	90
β⁄deg	90	97.08(3)	95.12(3)
∕/deg	90	90	90

$V/\text{\AA}^3$	1628.0(6)	1400.2(5)	2.39.1(6)
Z	4	4	4
$D_c/\mathrm{g~cm}^{-3}$	1.802	1.597	1.589
μ/mm^{-1}	1.551	1.770	1.250
F(000)	896	676	1000
RefInsmessured	15743	11799	10335
Obsd reflns	1859	2467	2331
$R_1^{a}/wR_2^{b}[I>2\sigma(I)]$	0.0388/0.0920	0.0597/0.0892	0.0682/0.1334
R_1 , wR_2 (all data)	0.0500/0.0969	0.0840/0.0952	0.1037/0.1479

 ${}^{a}\overline{R = \Sigma(||F_{o}| - |F_{c}||)/\Sigma|F_{o}|} {}^{b}wR = [\Sigma(|F_{o}|^{2} - |F_{c}|^{2})^{2}/\Sigma(F_{o}^{2})]^{1/2}$

Table 2

Selected bond distances (Å) and angles (deg) for $1\mathchar`-3$

1			
Zn(1)-N(2)	2.122(2)	Zn(1)-O(1)	2.0991(17)
Zn(1)-O(1B)	2.0991(17)	Zn(1)-N(2B)	2.122(2)
Zn(1)-N(5C)	2.265(2)	Zn(1)-N(5D)	2.265(2)
O(1)-Zn(1)-O(1B)	180.0	O(1)-Zn(1)-N(2)	85.58(7)
O(1B)-Zn(1)-N(2)	94.42(7)	O(1)-Zn(1)-N(2B)	94.42(7)
O(1B)-Zn(1)-N(2B)	85.58(7)	N(2)-Zn(1)-N(2B)	180.00(11)
O(1)-Zn(1)-N(5C)	92.47(7)	O(1B)-Zn(1)-N(5C)	87.53(7)
N(2)-Zn(1)-N(5C)	79.78(7)	N(2B)-Zn(1)-N(5C)	100.22(7)
O(1)-Zn(1)-N(5D)	87.53(7)	O(1B)-Zn(1)-N(5D)	92.47(7)
N(2)-Zn(1)-N(5D)	100.22(7)	N(2B)-Zn(1)-N(5D)	79.78(7)
N(5C)-Zn(1)-N(5D)	180.00(8)		
2			
Zn(1)-N(9A)	1.970(4)	Zn(1)-N(6)	1.977(4)
Zn(1)-N(1)	1.991(3)	Zn(1)-N(2B)	2.199(4)
Zn(1)-O(1)	2.212(3)		
Y.			
N(9A)-Zn(1)-N(6)	120.09(15)	N(9A)-Zn(1)-N(1)	118.77(14)
N(6)-Zn(1)-N(1)	119.56(15)	N(9A)-Zn(1)-N(2B)	91.34(15)
N(6)-Zn(1)-N(2B)	91.74(14)	N(1)-Zn(1)-N(2B)	99.48(13)
N(9A)-Zn(1)-O(1)	86.77(14)	N(6)-Zn(1)-O(1)	89.42(14)
N(1)-Zn(1)-O(1)	81.25(13)	N(2B)-Zn(1)-O(1)	178.09(14)

Zn(1)-O(1W)	1.990(5)	Zn(1)-N(5A)	2.010(3)
Zn(1)-N(5)	2.010(3)	Zn(1)-O(1A)	2.096(3)
Zn(1)-O(1)	2.096(3)		
O(1W)-Zn(1)-N(5A)	119.14(10)	O(1W)-Zn(1)-N(5)	119.14(10)
N(5A)-Zn(1)-N(5)	121.72(19)	O(1W)-Zn(1)-O(1A)	88.21(8)
N(5A)-Zn(1)-O(1A)	83.70(11)	N(5)-Zn(1)-O(1A)	98.07(11)
O(1W)-Zn(1)-O(1)	88.21(8)	N(5A)-Zn(1)-O(1)	98.07(11)
N(5)-Zn(1)-O(1)	83.70(11)	O(1A)-Zn(1)-O(1)	176.41(16)

Symmetry codes: For **1**: A: x+1/2,-y+1/2,-z+1; B: -x,-y+1,-z+1; C: x-1/2,-y+1/2,-z+1; D: -x+1/2,y+1/2,z.; For **2**: A: -x+1,y+1/2,-z+1/2; B: -x+1,-y+2,-z+1; C: -x+1,y-1/2,-z+1/2; For **3**: A:-x,y,-z+1/2.

3. Syntheses of part

3.1 N-(1*H*-tetrazol-5-yl)benzamide) (HL¹)

Anhydrous tetrahydrofuran (60 mL) containing 5-aminotetrazolate (0.1 mol) was in an ice bath for 30 min, benzoyl chloride (0.1 mol) was slowly added to the above solution, then the obtained solution was stirred overnight. The white solid was collected, washed with water, and dried to give 13.65g of the ligand, see Chart **1**. The yield was ~70% \circ m.p. 270 °C. Anal. Calcd for C₈H₇N₅O: C, 50.79; H, 3.73; N, 37.02. Found: C, 50.36; H, 3.81; N, 36.84. ¹H NMR(DMSO-*d*₆): 7.54~7.60 (m, 2H, ArH), 8.07~8.12 (m, 2H, PyH), 12.44 (s, 1H, N₄H) \circ IR (KBr pellet, cm⁻¹): 3212m, 3069m, 1669s, 1593s, 1547m, 1448w, 1228m, 1036s, 937w, 900s, 803m, 702s, 660m \circ

3.2 4-methyl-N-(1*H*-tetrazol-5-yl)benzamide) (HL²)

The preparation of HL^2 was synthesized in a similar procedure as HL^1 , by using 4-methyl benzoyl chloride in place of benzoyl chloride, and the yield was 60%. m.p. 155 °C. Anal. Calcd for C₉H₉N₅O: C, 53.20; H, 4.46; N, 34.47. Found: C, 52.93; H, 4.61; N, 34.22. ¹H NMR(DMSO-*d*₆): 2.35 (m, 3H, CH₃), 7.24 (m, 2H, ArH), 7.83(m, 2H, ArH), δ 8.0(s, H, Amide NH), 15.8(s, 1H, N₄H) $_{\circ}$ IR (KBr pellet, cm⁻¹): 3413(s), 1639(s), 1617(s), 1400(m), 1274(w), 1059(w), 995(w), 619(m).



Chart 1 Synthesis of HL^1 and HL^2

$3.3 [Zn(L^1)_2]_n (1)$

A mixture of ZnCl₂ (81.6 mg, 0.6 mmol), HL¹ (56.7 mg, 0.3 mmol) and water (12 mL) was sealed in a 25 mL Teflon-lined stainless steel vessel and heated at 140 °C for 48 h, then cooled to room temperature at a rate of 10 °C·h⁻¹. Colorless prism-shaped crystals of **1** were isolated and washed with water and ethanol and dried in air (ca. 36% yield based on HL¹). Anal. Calcd for $C_{16}H_{12}ZnN_{10}O_2$: C, 43.50; H, 2.74; N, 31.71. Found: C, 43.06; H, 2.91; N, 31.42. IR (KBr pellet, cm⁻¹): 3407(w), 3237(w), 1650(s), 1566(m), 1584(s), 1445(m), 1248(w), 1021(m), 929(w), 896(s), 808(m), 731(s), 657(m).

$3.4 [Zn(L^{1})(5-ATZ)]_{n} (2)$

A mixture of ZnCl₂ (68.0 mg, 0.5 mmol), HL^2 (56.7 mg, 0.3 mmol), NH_4VO_3 (70.2 mg, 0.6 mmol) and water (12 mL) was sealed in a 25 mL Teflon-lined stainless steel vessel and heated at 160 °C for 72 h, then cooled to room temperature at a rate of 5 °C·h⁻¹. Colorless bulk crystals of **2** were isolated and washed with water and ethanol and dried in air. IR (KBr pellet, cm⁻¹): 3430(w), 3239(w), 3026(w),1650(m), 1566(m), 1445(w), 1209(s), 1157(s), 914(m), 698(m), 657(w).

$3.5 [Zn(L^2)_2(H_2O)] (3)$

A mixture of ZnCl₂ (1.0 mmol), HL² (0.5 mmol), NaN₃ (1.0 mmol) and water (12 mL) was sealed in a 25 mL Teflon-lined stainless steel vessel and heated at 160 °C for 72 h, then cooled to room temperature at a rate of 5 °C·h⁻¹. Colorless prism-shaped crystals of **1** were isolated and washed with water and ethanol and dried in air. IR (KBr pellet, cm⁻¹): 3423(w), 3227(w), 1630(s), 1475(m), 1410(w), 1025(m), 920(m), 750(s), 685(m).

Caution! Although we have met no problems in handling tetrazoles and their compounds during this work, it should be treated with great caution owing to their potential explosive nature.

4. Molecular structure

4.1 $[Zn(L^1)_2]_n$ (1)

Single-crystal X-ray diffraction analysis shows that compound 1 crystallizes in Pcba space group, and the asymmetric unit contains half crystallographically independent Zn^{II} center and one L^1 ligand. Each Zn^{II} center shows a coordination number of six, surrounded by four N atoms and two O atoms from four independent L^1 . The Zn^{II} center is chelated by two L^1 through their O atoms and N atoms of tetrazolate rings, forming the equatorial plane that contains two six-membered rings, the N atoms of tetrazolate rings from other two L^1 occupying the axial positions (Fig. **1a**). The Zn–N distances are 2.122(2) and 2.265(2) Å and Zn–O bonds are 2.0991(17) Å, respectively, and coordination angles around Zn^{II} center vary from 79.78(7) to $180.00(7)^{\circ}$, which indicate the octahedral geometries around Zn^{II} center are slightly distorted. All the Zn-N and Zn-O are within the ranges expected for such complexes.¹⁷ The deprotonated L acts as tridentate bridging ligand linking two Zn^{II} centers with distance of 6.360 Å, the tetrazolate rings of L^1 in 1 adopt $\mu_{1,4}$ bridging coordination mode and the whole L^1 link Zn^{II} center into 2D structure in the *ab* plane. The connectivity of the network of 1 can be simplified to a (4^4) topology with the metal centers are simplied as 4-connecting nodes (Fig. 1b).



(a)



Fig. 1. (a) The local coordination geometry around the Zn^{II} centers in **1**; (b) The 2D (4⁴) framework structure.

$4.2 [Zn(L^1)(5-ATZ)]_n (2)$

When the ammonium metavanadate was added and the reaction temperature was up to 160°C, a different structure of **2** was obtained. Single-crystal X-ray diffraction analysis reveals that the asymmetric unit of **2** contains one Zn^{II} center, one L¹ and one 5-ATZ. The Zn^{II} center is five-coordinated by four nitrogen atoms and one oxygen atom from two 5-ATZ ligands and two L¹ ligands (Fig. **2a**). The Zn–N bond lengths vary from 1.970(4) to 2.199(4) Å and the Zn–O bond is 2.212(3) Å, the coordination angles around Zn^{II} center vary from 86.77(14) to 178.09(14)°. The 5-ATZ adopts a $\mu_{1,4}$ bridging coordination fashion, while L¹ takes tridentate coordination mode linking two Zn^{II} centers with the distance of 4.068 Å, in which the tetrazole ring adopt $\mu_{1,2}$ bridging coordination mode. In this way, the 5-ATZ and L¹ link the Zn^{II} centers to form a 2D layer (Fig. **2b**). Topologically, if the Zn^{II} ions are considered as 3-connected node, the 2D layer can be simplified as a 3-connected net with the (6³) topology (Fig. **2c**).



Fig. 2. (a) The local coordination geometry around the Zn^{II} centers in **2**; (b) The 2D layer in the *bc* plane; (c) The 2D (6³) framework structure.

 $4.3 [Zn(L^2)_2(H_2O)]$ (3)

The structure of **3** contains one independent crystallographically Zn^{II} center, two L^2 ligands in general position and one coordination water molecular. Each Zn^{II} center in the mononuclear motif is chelated by two L^2 ligand through their oxygen atoms and nitrogen atoms of tetrazolate rings as well as one oxygen atom of the coordinated water molecular to complete a distorted trigonal biyramidal geometry (Fig. **3a**). The Zn1–O distances are 1.990(5) and 2.096(3) Å and Zn1–N bonds are 2.010(3) Å, respectively, and the coordination angles around Zn^{II} center vary from 83.70(11) to 176.41(16)°. All the bond lengthes and bond angles around the Zn^{II} atom are in the normal ranges for such complexes[17]. The 3D structure of **3** are linked by C-H…N, C-H…O and O-H…N hydrogen bonds between the adjacent molecules (Table **S1** and Fig. **3b**).



Fig. 3. (a) The diagram of the coordination mode of Zn^{II} centers in **3**; (b) The 3D supramolecular structure of **3** connected by hydrogen bonds.

5. Summary of the Structures

Compound 1 was obtained through lignad HL^1 and $ZnCl_2$ at the temperature of 140 °C, while compound 2 was obtained when ammonium metavanadate was added and the reaction temperature was up to 160°C, and its structure is different from compound 1, also the 5-ATZ existing in compound 2, which may be from the decomposition of the L^1 under higher temperature. When the HL^2 and NaN_3 was used, compound 3 has been synthesized as mononuclear, where HL^2 have one more methyl than HL^1 in its structure. H_2L^1 and H_2L^2 can act as either bidentate or tridentate ligands when coordinating to Zn metal ions under different conditions, and three coordination modes for tetrazolate rings have been observed in these three compounds (Chart 2). The ligands are all deprotonated to form monovalent anionic derivatives.



Chart 2 (a) the coordination modes of HL^1 in complex 1; (b) the coordination modes of HL^1 in complex 2; (c) the coordination modes of HL^2 in complex 3.

Because of the poor productivity of compound 2 and 3, their photoluminescent property and thermal analyses have not been studied.

6. Photoluminescent property

It is well known that luminescence compounds have attracted much attention recently because of their potential applications in photochemistry, chemical sensors and structure electroluminescence (EL) display[18], especially for the luminescent properties of tetrazolate compounds with d¹⁰ metal ions[19]. Here we have been exploring the luminescence property of compound **1** in the solid state at room temperature (Fig.**4**). The free **HL**¹ ligand displays a very weak emission centered at ca. 370 nm in the solid state at ambient temperature. For compound **1**, its radiation emission maxima at ca. 375 nm, which shifts to longer wavelength only several nanometers, can likely be attributed to the π - π^* or π -n electronic transitions within ligand-centered aromatic systems, by comparison with other luminescent d¹⁰ metal coordination polymers[20].



Fig. 4. Solid-state fluorescence spectra of compound 1 at room temperature.

7. Thermal analyses

In order to determine the thermal stability of compound **1**, thermogravimetric analysis (TGA) for **1** was performed by heating the corresponding compound from 20 to 800 °C in air at a rate of 10 °C \cdot min⁻¹. The TGA result of **1** indicates that compound **1** decomposed upon to 340 °C, with further heating, a rapid weight loss occurs, which is due to the departure of organic ligand(Fig. S1). The thermal analysis indicates that complex **1** has high thermal stability.

8. XRPD Results

To confirm whether the analyzed crystal structures are truly representative of the bulk materials, XRPD experiments were carried out for compound **1**. The XRPD experimental and computer-simulated patterns of the corresponding compound is shown in Fig. S2. It is in good agreement with each other, which prove the phase purity of the as-synthesized product and the properties of compound **1** were believable.

9. Conclusion

In summary, three compounds have been obtained by a hydrothermal method using the tetrazole ligands HL^1 and HL^2 , as well as the corresponding Zn^{II} salts. The

difference in the frameworks of the target compounds are due to the influences of the nature of the reaction conditions and the different structures of the ligands. Also the fluorescence property and thermal stability of compound **1** have been studied. It is anticipated that more metal compounds containing our obtained ligands here with charming structures as well as physical properties will be synthesized in future.

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Zn Coordination polymers based on designed of tetrazole-yl acylamide: syntheses, structures and luminescence property

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- Compound 1 and 2 are both 2D structures with 4⁴ and 6³ networks, respectively.
- Compound 1 exhibits high thermal stability and intense fluorescence behavior.
- The structural differences are owing to different reaction conditions and ligands.