Construction of four d\textsuperscript{10} coordination polymers containing binuclear rings as building blocks from \textit{4′-(2H-tetrazol-5-yl)} biphienyl-4-carboxylic acid

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1. Introduction

Coordination polymers (CPs) have attracted attention because of their interesting topologies and properties, such as gas separation, luminescence, magnetism, and sensors [1–12]. Controllable synthesis of CPs is not clearly elucidated. Selecting a special CP for a particular application from a large number of CPs is still difficult. Therefore, investigating the syntheses and structures of CPs based on new ligands is necessary as ligands play an important role in the final structure. Both carboxylate and tetrazole groups are good donors, frequently used in CP syntheses. These groups display versatile coordination, suitable for the formation of secondary building blocks. A combination of these two functional groups may produce new ligands with interesting coordination behavior. However, CPs based on such ligands are
limited. Several ligands, such as 4-(1H-tetrazol-5-yl)benzoic acid [13–17], tetrazole-5-carboxylate [18–20] and 5-(1H-tetrazol-5-yl)isophthalic acid [21–24], have been applied to construct CPs with interesting structures and properties. In this study, 4′-(2H-tetrazol-5-yl)biphenyl-4-carboxylic acid (H₂TBPC), a ligand that possesses a long biphenyl spacer between the terminal tetrazole and carboxylate groups, was selected to prepare new functional CPs by following the same design strategy. Reactions of H₂TBPC with Zn(II) and Cd(II) result in four new d¹⁰ CPs. The structures and luminescence properties of these CPs were investigated. To the best of our knowledge, only a few H₂TBPC-based CPs have been reported [25, 26].

2. Experimental

2.1. Materials and instrumentation

H₂TBPC was bought from Jinan Camolai Trading Company. All other solvents and reagents were obtained from commercial vendors and used as purchased. Elemental analyses for C, H, and N were performed on a Perkin-Elmer 240C analytical instrument. IR spectra were recorded on a Nicolet FT-IR-170SX spectrophotometer in KBr pellets. X-ray powder diffraction measurements were measured using a Bruker D8 Advance diffractometer at 40 kV, 40 mA with a Cu-target tube and a graphite monochromator. Thermogravimetric analyses were performed on a Perkin-Elmer TGA7 analyzer with a heating rate of 10 °C min⁻¹ in flowing air. The luminescent spectra for the solid state were recorded at room temperature on Hitachi F-2500 and Edinburgh-FLS-920 instruments with a xenon arc lamp as the light source. In the measurements of emission and excitation spectra, the pass width is 5 nm.

2.2. Syntheses of 1–4

2.2.1. Synthesis of [Zn(μ₃-TBPC)(H₂O)]ₙ (1)

A mixture of H₂TBPC (0.20 mmol, 53.2 mg), Zn(CH₃COO)₂ (0.20 mmol, 36.7 mg), and H₂O (6 mL) was sealed into a 15 mL Teflon-lined stainless-steel autoclave, heated to 170 °C for 72 h under autogenous pressure, and then slowly cooled to room temperature at a rate of 5 °C/h. Pale-yellow block crystals of 1 were isolated, washed with distilled water, and dried in air (yield: 73%, based on Zn). Elemental Anal. Calcd (%) for C₁₄H₁₀N₄O₃Zn: C, 48.37; H, 2.90; N, 16.12. Found: C, 48.54; H, 3.02; N, 16.07%. IR (KBr, v/cm⁻¹): 3789w, 3405s, 2964w, 2919w, 1606m, 1589s, 1566w, 1514m, 1465w, 1420s, 1318m, 1257w, 1175w, 1155w, 1124w, 1050w, 873w, 847w, 783w, 764m, 703w, 498w.

2.2.2. Synthesis of [Zn(μ₃-TBPC)(Me₂NH)]ₙ (2)

A mixture of H₂TBPC (0.20 mmol, 53.2 mg), ZnSO₄·7H₂O (0.20 mmol, 57.5 mg), EtOH (3 mL), and DEF (3 mL) was sealed into a 15 mL Teflon-lined stainless-steel autoclave, heated to 170 °C for 72 h under autogenous pressure, and then slowly cooled to room temperature at a rate of 5 °C/h. Yellow block crystals of 2 were obtained by filtration and washed with EtOH several times, giving a yield of 67% (based on Zn). Elemental Anal. Calcd (%) for C₁₆H₁₅N₅O₂Zn: C, 51.28; H, 4.03; N, 18.69. Found: C, 51.56; H, 3.89; N, 18.76%. IR (KBr, v/cm⁻¹): 3788w, 3412m, 3280w, 2964w, 1611s, 1569w, 1514w, 1464w, 1398m, 1378w, 1261m, 1108s, 1046w, 1012w, 904w, 874w, 833w, 785w, 772w, 764m, 703w, 658w.

2.2.3. Synthesis of [Cd(μ₃-TBPC)(bpy)]ₙ (3)

A mixture of H₂TBPC (0.20 mmol, 53.2 mg), 2,2’-bipyridinel (0.20 mmol, 31.2 mg), Cd(NO₃)₂·4H₂O (0.20 mmol, 61.7 mg), CH₃CN (3 mL), and H₂O (3 mL) was sealed into a 15 mL Teflon-lined stainless-steel autoclave, heated to 170 °C for 72 h under autogenous pressure, and then slowly cooled to room temperature at a rate of 5 °C/h. Yellow block crystals of 3 were obtained with a yield of 30% (based on Cd) after washing with distilled water and drying in air. Elemental Anal. Calcd (%) for C₂₄H₁₆N₆O₂Cd: C, 54.10; H, 3.03; N, 15.77. Found: C, 53.95; H, 3.12; N, 15.68%. IR (KBr, v/cm⁻¹): 3795w, 3408w, 2973w, 1588s, 1567w, 1531s, 1477w, 1437w, 1428w, 1398s, 1307w, 1245w, 1170w, 1156w, 1104w, 1041w, 845w, 787w, 762s, 736w, 688w, 648w.
2.2.4. Synthesis of $[\text{Cd}(\mu_4-\text{TBPC})(\text{H}_2\text{O})]_n$ (4)

A mixture of H$_2$TBPC (0.20 mmol, 53.2 mg), CdBr$_2$ (0.20 mmol, 54.5 mg), CH$_3$CN (3 mL), and H$_2$O (3 mL) was heated to 170 °C for 72 h in a 15 mL Teflon-lined stainless-steel reactor and then slowly cooled to room temperature at a rate of 5 °C/h. Colorless block crystals of 4 were collected by filtration and washed with distilled water several times. yield: 65% (based on Cd). elemental Anal. Calcd (%) for C$_{14}$H$_{10}$N$_4$O$_3$Cd: C, 42.61; H, 2.55; N, 14.20. Found: C, 42.86; H, 2.63; N, 14.33%. IR (KBr, ν/cm$^{-1}$): 3789w, 3372s, 2975w, 1680w, 1606w, 1588m, 1566w, 1549m, 1459m, 1411s, 1357w, 1252w, 1168w, 1152w, 1048w, 1005w, 863w, 832w, 787w, 777w, 728w, 689w, 565w, 461w, 417w.

2.3. X-ray data collection and structure refinement

Data collections were performed at 298 K on a Bruker Smart Apex II diffractometer with graphite-monochromated Mo K$_\alpha$ radiation ($\lambda = 0.71073$ Å) for 1–4. Multi-scan absorption corrections were applied using SADABS [27]. Structural solutions and refinements based on $F^2$ for all complexes were performed with SHELXL [28]. All non-H atoms were refined with anisotropic thermal parameters. All hydrogens were placed in idealized positions. Hydrogens on hydroxyl for 2 were not added but included in the formula. The crystal parameters, data collections, and refinements for 1–4 are listed in table (1). CCDC reference numbers are 1408241-1408244 for 1–4.

3. Results and discussion

3.1. Crystal structure of 1–4

Single crystal X-ray diffraction shows that the compounds possess different structures, but contain an M$_2$L$_2$ ring as the building block. The coordination angle between carboxylate and N on the 2-position of tetrazole is approximately 70°, which may be suitable for constructing a small ring. In 1–4, two H$_2$TBPC ligands and two metal ions are bridged via carboxylate and N on the 2-position of tetrazole to form a binuclear grid (scheme 1).

The formation of a binuclear ring cannot use all the coordination sites of the metal ions, and some coordination atoms remain uncoordinated on TBPC ligands. Hence, such rings may act as building units to assemble further into a higher dimensional network. Each Zn(II) in 1 is five-coordinate with two nitrogens and two carboxylate oxygens from three individual TBPC$^{2-}$ ligands and one oxygen from...
coordinated water, forming a distorted square-pyramidal geometry (figure 1(a)). Each binuclear ring is connected to four adjacent binuclear rings by Zn–N bonds between Zn(II) ions and three nitrogens on TBPC$^{2-}$ dianions, resulting in a 2-D network structure in the bc plane (figure 1(b)). Each $\mu_3$-TBPC$^{2-}$ connects to three Zn(II) ions and can be considered as a 3-connecting node, while each Zn(II) ion is coordinated by three $\mu_3$-TBPC$^{2-}$ ligands and also can be regarded as a 3-connecting node. Therefore, the resulting structure of 1 can be described as a binodal (3,3)-connected FeS-type 2-D network with the Schlafli symbol of (4·8$^2$) (figure 1(c)). In addition, the O–H⋯N hydrogen bond between coordinated water molecule and the tetrazolate group of the TBPC$^{2-}$ ligand [O1W–H1WA⋯N4, H1WA⋯N4 = 2.04 Å, O1W⋯N4 = 2.857(5) Å, $\angle$ O1W–H1WA⋯N4 = 172°, symmetry code: $-1 + x, y, z$], as well as the O–H⋯O hydrogen bond between coordinated water and the carboxylate of TBPC$^{2-}$ [O1W–H1WB⋯O2, H1WB⋯O2 = 1.97 Å, O1W⋯O2 = 2.757(6) Å, $\angle$ O1W–H1WB⋯O2 = 162°, symmetry code: $1 - x, -y, 1 - z$] together assemble the neighboring 2-D layers into a 3-D supramolecular network (figure 1(d)).

Compound 2 can be obtained when using DEF/ethanol as the solvents. Each Zn(II) displays a slightly distorted [ZnN$_3$O] coordination configuration. This configuration is defined by one carboxylate oxygen [Zn−O, 1.920(3) Å] and two tetrazole nitrogens [Zn−N, 2.040(4)−2.071(4) Å] from three individual TBPC$^{2-}$ ligands, and one nitrogen from one coordinated Me$_2$NH [Zn−N, 2.051(4) Å] (figure 2(a)). A binuclear unit similar to that in 1 is also observed (figure 2(b)). The TBPC$^{2-}$ is a tridentate ligand that connects three Zn(II) ions. However, apart from the coordination donors that are used in the binuclear ring, TBPC$^{2-}$ using the N4 rather than the N3 (observed in 1) coordinate to the third Zn(II) ion. Therefore, the interconnection of binuclear rings via Zn–N bonds between Zn(II) and N4 forms a 2-D layer, in which the binuclear rings are almost perpendicular to the plane with an angle of 87.4° (figure 2(c)). These 2-D layers are stacked along the a direction in an –AAAA– sequence, and no noticeable interaction exists between the neighboring layers. The topological method was also performed to further analyze the 2-D framework of 2. As mentioned above, each $\mu_3$-TBPC$^{2-}$ bonds to three Zn(II) ions and each Zn(II) ion also links to three $\mu_4$-TBPC$^{2-}$ ligands, thus both of them can be viewed as 3-connected nodes. Therefore, the whole structure of 2 can be represented as (3,3)-connected 2-D FeS-type topology (figure 2(d)), which is the same as compared with 1.

Compound 3 crystallizes in the monoclinic space group P2(1)/c. Its asymmetric unit comprises one Cd(II), one TBPC$^{2-}$, and one bpy neutral ligand. As illustrated in figure 3(a), each Cd(II) is six-coordinate in a distorted octahedral [CdN$_4$O$_2$] geometry. This configuration is surrounded by two tetrazole nitrogens and two carboxylate oxygens from three different TBPC$^{2-}$ ligands, and two pyridine nitrogens

Scheme 1. The formation of binuclear ring in 1–4.
from one bpy chelating ligand. The coordination mode of the tetrazole group on TBPC$^{2-}$ is the same as that in 2, hence, a similar 2-D layer based on the binuclear ring and Cd1–N4 bonds is formed (figure 3(b)). A (4-8$^2$) network topology is also formed when both the metal ion and TBPC$^{2-}$ anion serve as 3-connected nodes (figure 3(c)). The coordinated bpy molecules are located on the sides of the 2-D layer. As a result, a 3-D supramolecular framework is formed by linking these 2-D layers through weak n···n stacking interactions between bpy rings from neighboring layers, with a centroid–centroid distance of 3.867 Å (figure 3(d)).
Single crystal X-ray diffraction shows that 4 crystallizes in the monoclinic system with P2(1)/c space group and the asymmetric unit contains only one crystallographically independent Cd(II), one TBPC$_2^-$, and one coordinated water. The local coordination geometry around the Cd(II) ion can be described as a [CdN$_3$O$_3$] slightly distorted octahedron [29, 30], with the axial positions occupied by one nitrogen from one TBPC$_2^-$ and one oxygen from one coordinated water (figure 4(a)). The equatorial plane consists of two nitrogens and two carboxylate oxygens from the other three different TBPC$_2^-$ ligands.

In 4, each TBPC$_2^-$ has a unique μ$_4$-O,O$'$:kN,kN',kN" quadridentate bridging mode. Thus, the binuclear units are connected by Zn–N bonds between Zn and N2 and N4, giving a 3-D pillar-layered framework (figure 4(b)). The 2-D layer constructed by Zn(II) and tetrazole groups is shown in figure 4(c). Two tetrazole groups simultaneously bridge two neighboring Cd(II) ions to form a binuclear [CdN$_2$]$_2$ unit. These units are further connected to a 2-D network extending along the bc plane (figure 4(c)). The topological analysis approach was employed for 4. Each μ$_4$-TBPC$_2^-$ bonds to four Cd(II) ions and each Cd(II) also links to four μ$_4$-TBPC$_2^-$ ligands, thus both can be viewed as 4-connected nodes (figure 4(d)). On the basis of this simplification, the resulting structure of 4 can be described as a binodal (4,4)-connected PtS-type 3-D framework [31–33] with the Schläfli symbol of (4$^2$·8$^4$) analyzed by TOPOS (figure 4(e)).

Thus, a binuclear ring is often formed in 1–4, regardless the of metal ion, anion, and coligand used. This indicates that H$_2$TBPC possesses the ability to form a binuclear unit easily during the assembly process, which may help predict the structures of new CPs from H$_2$TBPC ligand.

Figure 3. (a) Coordination environment of Cd(II) in 3 (hydrogens are omitted for clarity). Symmetry codes: (i) −x + 2, −y + 2, −z + 2; (ii) x + 1, −y + 3/2, z − 1/2. (b) The 2-D network structure of 3 in the bc plane. (c) A schematic representation of binodal (3,3)-connected PeS-type 2-D framework of 3. (d) The 3-D supramolecular framework of 3 constructed by the weak π⋯π stacking interactions between neighboring 2-D layers.
3.2. PXRD and thermogravimetric analyses of 1–4

PXRD experiments show that the phase purity of 1–4 is high (figures S1–S4). Thermogravimetric analyses (TG) of 1–4 were carried out under air with a heating rate of 10 °C min⁻¹. As described in figure (5), the TG curve for 1 indicates a gradual weight loss of 5.6% from room temperature to 270 °C, which can be ascribed to the removal of water (Calcd 5.2%) in the asymmetric unit. Then a major weight loss above 330 °C is attributable to the decomposition of the whole coordination framework. Seen from the TG diagram of 2, a gradual weight loss of 10.4% from room temperature to 300 °C may be due to the release of N(CH₃)₂ (Calcd 12.6%), and the framework begins to decompose above that temperature. For 3, there is no noticeable weight loss before 370 °C, and further heating leads to decomposition of the framework. For 4, the coordination water is lost before 260 °C (observed 5.0%, Calcd 4.7%), and the framework is stable to about 360 °C.

3.3. Luminescent properties of 1–4

Previous studies have shown that CPs containing d¹⁰ ions exhibit photoluminescent properties [34–39]. Thus, photoluminescent properties of 1–4 and the ligand in the solid state have been investigated at room temperature, as shown in figure (6). The intense broad emissions at 412 nm (λₑₓ = 358 nm) for 1, 395 nm (λₑₓ = 327 nm) for 2, 403 nm (λₑₓ = 331 nm) for 3, and 383 nm (λₑₓ = 323 nm) for 4 can be observed. Compared with the emission spectra of free H₂TBPC (the emission at 400 nm with λₑₓ = 340 nm), only slight blue shifts of 12 and 9 nm for 2 and 4, and red shifts of 12 and 3 nm for 1 and 3 occur, which may be attributed to ligand-centered emission [40, 41].

Figure 4. (a) Local coordination environment of Cd(II) in 4 (hydrogens are omitted for clarity). Symmetry codes: (i) −x + 1, y + 1/2, −z + 3/2; (ii) −x + 1, −y + 1, −z + 1; (iii) x + 1, −y + 1/2, z + 1/2. (b) The 2-D network built by binuclear [CdN₂]₂ units in 4. (c) Polyhedral view of the 3-D pillar-layered coordination framework of 4. (d) 4-Connected μ₂-TBPC²⁻ node and 4-connected Cd(II) node in 1. (e) A schematic representation of binodal (4,4)-connected Pt⁵-type 3-D framework of 4.
Conclusion

Four new d^{10} CPs were constructed based on a new tetrazole-based carboxylate ligand with biphenyl spacer. All compounds contain a binuclear ring as building blocks and display different structures and topologies. Compounds 1–3 are 2-D networks with 4·8^2 topology, whereas 4 is a 3-D framework with PtS-type topology. This study confirms that H₂TBPC is suitable for constructing CPs with interesting structures.

Disclosure statement

No potential conflict of interest was reported by the authors.

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Figure 5. TG curves for 1–4.

Figure 6. Solid-state emission spectrum for H₂TPBC and 1–4 in the solid state at room temperature.
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