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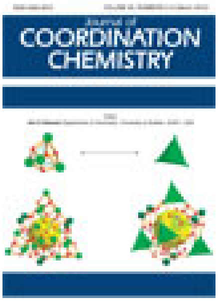

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Syntheses, structures, and magnetism of two coordination compounds with 3,3′,4,4′-biphenyltetracarboxylic acid and 1,10-phenanthroline

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Syntheses, structures, and magnetism of two coordination compounds with 3,3′,4,4′-biphenyltetracarboxylic acid and 1,10-phenanthroline

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Two coordination polymers, $\{[Mn(1,10\text{-phen})_2(3,4\text{-}H_2bptc)]\cdot(H_2O)\}_{2n}$ (**1**) and $\{[Co_2(1,10\text{-phen})(3,4\text{-bptc})(H_2O)_2]\cdot H_2O\}_n$ (**2**) ($3,4\text{-}H_4bptc$ = 3,3′,4,4′-biphenyltetracarboxylic acid, 1,10-phen = 1,10-phenanthroline), have been prepared and structurally characterized. In both compounds, $3,4\text{-}H_2bptc^{2-}$ and $3,4\text{-bptc}^{4-}$ exhibit different coordination modes and lead to various architectures. Compound **1** displays 1-D zigzag chains constructed with hydrogen bonds. Compound **2** exhibits a 2-D layer constructed from $bpta^{4-}$ between the dinuclear Co(II) units. The thermal stabilities and magnetic properties are also reported. In addition, **1** was explored as a luminescent material. The carboxylic oxygen atoms act as H-bond acceptors and donors forming a 1-D ladder-like arrangement.

Keywords: Coordination polymer; 3,3′,4,4′-Biphenyltetracarboxylic acid; Magnetic property

1. Introduction

Design of metal–organic coordination polymers has achieved considerable progress in supramolecular chemistry and crystal engineering. Interest is justified for intriguing topologies, structural diversity, such as helixes and diamondoid nets, and potential applications as functional materials, such as heterogeneous catalysis, molecular recognition, magnetism, gas storage, ion exchange, nonlinear optics, and electrical conductivity [1–12]. Polycarboxylates are often good candidates as bridging ligands to construct coordination polymers owing to their versatile coordination modes and high structural stability; 1,2-benzenedicarboxylate, 1,4-benzenedicarboxylate, 1,3,5-benzenetricarboxylate, and 1,2,4,5-benzenetetracarboxylate have been widely used to form metal–organic polymers with various structures and properties [13–29]. Considerable efforts have been devoted to developing flexible or V-shaped multidentate bridging ligands [30–40]. However, the nature of the rigid aromatic multidentate ligands to form the overall configuration has been scarcely investigated so far [41], particularly for the

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magnetic study of the transition metal coordination polymers bridged by carboxylate ligand [42–55].

In this regard, 3,3′,4,4′-biphenyltetracarboxylic acid (3,4-H_4bptc) was examined to take advantage of unique features of hydroxypolycarboxylic acids: (1) versatile coordination behavior from completely or partially deprotonated acid; (2) polycarboxylics are nonchelating, chelating, bridging, and hydrogen-bond acceptors and donors to form extended structures; (3) two phenyl rings can be rotated around the C–C single bond [42, 56].

Introduction of auxiliary ligands into carboxylate systems usually leads to polymeric frameworks, modulating structure geometry, and providing products more easily [57, 58]. Herein, phen is used in combination with H_4bptc to form the compounds.

To get insight into the coordination chemistry of H_4bptc and study the magnetic interactions between transition metal centers bridged by this carboxylate ligand, we synthesized Mn(II) and Co(II) coordination polymers with 3,4-H_4bptc and phen and investigated their magnetic properties.

2. Experimental

2.1. *Materials and instruments*

All reagents were purchased commercially and used without purification. Elemental analyses were carried out with an Elementar Vario EL III analyzer. Infrared (IR) spectra were recorded with a Tensor 27 spectrometer (Bruker Optics, Ettlingen, Germany). Fluorescence spectra were measured at room temperature with an Edinburgh FL-FS90 TCSPC system. Thermogravimetric measurements were performed with a Netzsch STA449C apparatus under nitrogen with a heating rate of $10^{\circ}C\,min^{-1}$ from 30°C to 900°C. X-ray powder diffraction (XRPD) patterns were measured on a Rigaku D/max-IIIA X-ray diffractometer with graphite monochromated Cu-Kα ($\lambda = 0.154056$ nm) radiation. Variable-temperature (2–300 K) magnetic susceptibility measurements were carried out on a Maglab System 2000 susceptometer (made in Oxford, UK). The magnetometer was operated at 1 T magnetic field. The diamagnetic corrections were evaluated from Pascal's constants.

2.2. *Synthesis*

2.2.1. Synthesis of $\{[Mn(1,10\text{-}phen)_2(3,4\text{-}H_2bptc)]\cdot(H_2O)\}_{2n}$ (1).

A mixture containing $MnCl_2\cdot 4H_2O$ (0.1 mmol, 0.0197 g), 3,4-H_4bptc (0.05 mmol, 0.0147 g), 1,10-phen (0.1 mmol, 0.0198 g), and water (6 mL) with pH = 6 was sealed in a 10 mL Teflon-lined stainless steel vessel and heated at 170°C for 1 day and then cooled to room temperature at $5^{\circ}C\,h^{-1}$. Yellow prism crystals of **1** were collected in a yield of 35% (based on Mn). Anal. Calcd for **1** ($C_{80}H_{52}N_8O_{18}Mn_2$) (%): C, 63.03; H, 3.41; N, 7.35. Found (%): C, 63.01; H, 3.39; N, 7.34. IR (KBr)/cm^{-1}: 3473(s), 3416(s), 1588(m), 1513(w), 1382(m), 1299(w), 1142(m), 1100(m), 992(m), 846(m), 805(w), 780(m), 765(m), 726(m), 570(m), 482(w).

2.2.2. Synthesis of {[Co_2(1,10-phen)(3,4-bptc)(H_2O)$_2$] · H_2O}$_n$ (2). A mixture of $CoCl_2 \cdot 6H_2O$ (0.10 mmol, 0.0238 g), 3,4-H_4bptc (0.1 mmol, 0.0294 g), 1,10-phen (0.35 mmol, 0.0594 g), CH_3OH (1 mL), and H_2O (5 mL) with pH of 7 adjusted by 0.5 mol L^{-1} NaOH solution was sealed in a 10 mL Teflon-lined stainless steel vessel and heated at 170°C for 3 days. Then the reaction mixture was cooled to room temperature at 10°C h^{-1}. Purple block crystals of **2** suitable for X-ray diffraction were isolated in 38% yield (based on Co). Anal. Calcd for **2** ($C_{28}H_{20}N_2O_{11}Co_2$) (%): C, 49.53; H, 2.95; N, 4.13. Found (%): C, 50.01; H, 2.82; N, 4.32. IR (KBr)/cm^{-1}: 3447(s), 2929(w), 1697(m), 1625(s), 1382(s), 1299(w), 1103(m), 846(m), 785(w), 780(w), 765(w), 726(w), 669(w), 485(w).

2.3. *X-ray crystal structure determinations*

All diffraction data were collected on a Bruker/Siemens Smart Apex IICCD diffractometer with graphite monochromated Mo-Kα radiation ($k = 0.71073$ Å) at 293(2) K. Cell parameters were retrieved using SMART [59] software and refined using SAINT-PLUS [60, 61] for all observed reflections. Data reduction and correction for Lp and decay were performed using SAINT-PLUS software. Absorption corrections were applied using SADABS [62]. The structures were solved by direct methods using SHELXS of the SHELXTL-97 package and refined with SHELXL. Crystallographic details are summarized in table S1. Selected bond lengths and angles of the compounds are shown in table S2. These observed hydrogen-bonding interactions, with the donor–acceptor distances, are shown in table S3.

3. Results and discussion

3.1. *General characterization*

Compounds **1** and **2** were obtained under hydrothermal conditions; both are stable in air. X-ray structural analysis and the electric neutrality principle shows 3,4-H_2bptc^{2-} in **1** and 3,4-bptc^{4-} in **2**. Bidentate bridging of the carboxylates exists in these two compounds, similar to previous reports for modes of carboxylic groups [58, 63, 64].

3.2. *Structural description*

3.2.1. {[Mn(1,10-phen)$_2$(3,4-H_2bptc)] · H_2O}$_{2n}$. The asymmetric unit of **1** contains two Mn(II), two 3,4-H_2bptc^{2-}, and four 1,10-phen ligands. Two –COOH groups of 3,4-H_2bptc^2 do not lose protons and exhibit C–O and C = O bond lengths of 1.273 and 1.209 Å, respectively, while the deprotonated –COOH has C–O bond lengths of 1.227 Å. As shown in figure 1(A), the central Mn(II) is six-coordinate with a slightly distorted octahedral configuration. The equatorial square is composed of one oxygen (Mn1–O8 2.149(5) Å) from a carboxylic group and three nitrogen atoms (Mn1–N1 2.302(7) Å, Mn1–N2 2.275(6) Å, Mn1–N4 2.271(6) Å) from two phen ligands; the axial position is occupied by one oxygen (Mn1–O7 2.091(4) Å) from another carboxylic group and one nitrogen (Mn1–N3 2.253(5) Å) from one phen. Adjacent metal ions are connected by

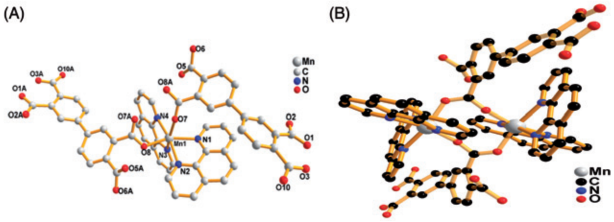

Figure 1. (A) The coordination environment of Mn1 in **1**. (B) The binuclear unit in **1**. All hydrogen atoms are omitted for clarity.

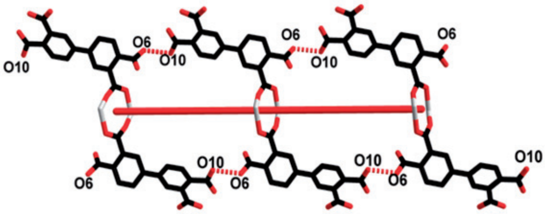

Figure 2. The carboxylic oxygen atoms are H-bond acceptors and donors forming 1-D ladder-like arrangement. Hydrogen bonds are shown as dashed lines. All hydrogen atoms, H_2O molecules, and phen ligands are omitted for clarity.

carboxylic groups with bidentate bridging modes in a *syn-anti* fashion to form a binuclear unit (figure 1B). These units are linked by hydrogen-bonding interactions (O(6)–H(9 A) ··· O(10)) to generate 1-D ribbon-like chains which contain 38-membered rings, as shown in figure 2.

3.2.2. $\{[Co_2(1,10\text{-bpen})(3,4\text{-bptc})(H_2O)_2] \cdot H_2O\}_n$.

Single-crystal X-ray structural analysis shows **2** is a double-layer complex containing two crystallographically independent Co ions (figure 3). Each octahedral Co1 is attached to one chelated phen (Co1–N1 2.114(4) Å, Co1–N2 2.148(4) Å), three monodentate bptc^{4-} (Co1–O2 2.017(3) Å, Co1–O4 2.156(3) Å, Co1–O9 2.115(3) Å), and one water molecule (Co1–O1 2.158(3) Å); octahedral Co2 is coordinated to six oxygen atoms with O4, O5, O6, O7, O8, O9 (Co1–O4 2.156(3) Å, Co1–O5 2.089(3) Å, Co1–O6 2.187(3) Å, Co1–O7 2.148(3) Å, Co1–O8 2.030(3) Å, Co1–O9 2.214(3) Å) from four bptc^{4-} and one coordinated water molecule. Two adjacent Co ions are bridged by bptc^{4-} into a dinuclear core with a short Co1–Co2 contact of 3.207 Å. The carboxylates of four bpta^{4-} exhibit the same coordination mode and connect with two Co ions into 1-D chains. Then bptc^{4-} link the chains to generate a 2-D coordination layer by monodentate/bis(monodentate) modes (figure 4).

Additionally, there are intramolecular and intermolecular hydrogen bonds in **2**. Hydrogen bonds form between coordinated water and oxygen from carboxyl (O(7)–H(7 A) ··· O(11)), and interaction of uncoordinated water and oxygen from carboxyl (O(11)–H(11 A) ··· O(10), O(11)–H(11B) ··· O(10)). Thus the free water molecules act as H-bond acceptors and donors forming a 3-D supramolecular structure with channels along the *c*-axis, as shown in figure 5.

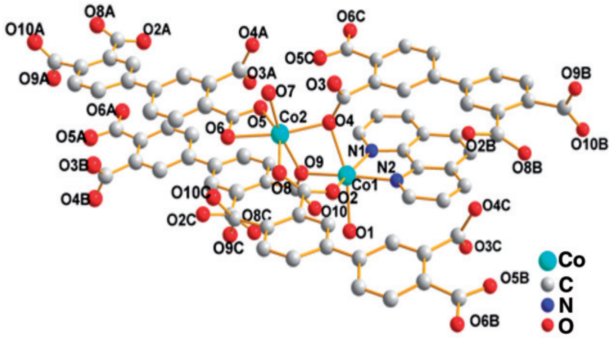

Figure 3. The coordination environments of Co1 and Co2.

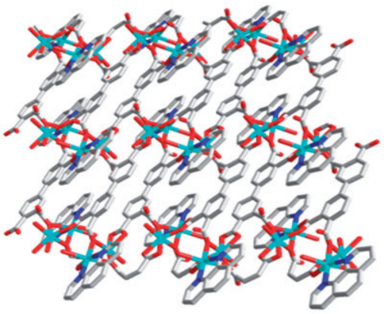

Figure 4. 2-D layered coordination framework of **2**. All hydrogen atoms are omitted for clarity.

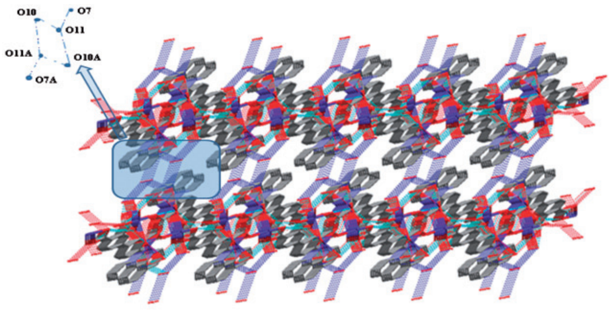

Figure 5. The free water molecules as H-bond acceptors and donors forming a 3-D supramolecular structure with channels along the *c*-axis. Hydrogen bonds are shown as dashed lines in black. All hydrogen atoms are omitted for clarity.

3.3. *Spectroscopic characterization*

IR spectra of **1** and **2** were performed as KBr pellets from 4000 to 400 cm^{-1}; ν(O–H) of H_2O in both are observed between 3500 and 3300 cm^{-1}. Compound **1** shows characteristic bands of carboxylates at 1588(m) for asymmetric stretching and at 1382(m) for symmetric stretching; $\Delta[\nu_{as}(OCO)–\nu_s(COO)] > 200\ cm^{-1}$ for **1** reveals that the carboxylates are bidentate bridging. The strong and broad peak at 3416 cm^{-1}

suggests the presence of hydrogen bonds. For **2**, the bands of carboxylates occur at 1697(m) and 1625(s) for asymmetric stretching and at 1382(s) and 1299(w) for symmetric stretching, consistent with different coordination of carboxylates, chelating, bis-monodentate, and bridging bidentate modes, in accord with the results of single-crystal X-ray analysis.

The solid-state photoluminescence spectrum of **1** is depicted in "Supplementary material." Upon excitation at *ca* 330 nm for **1**, the emission peak is located at 466 nm, a red shift of *ca* 78 nm relative to that of free biphenyl-3,3′,4,4′-tetracarboxylic dianhydride [42, 65]. We tentatively assign it to ligand-to-metal charge transfer (LMCT) [66, 67]. The weak peak at 397 nm is assigned to intraligand fluorescence. Free phen shows fluorescence emission in the solid state at 365 nm and 388 nm ($\lambda_{ex} = 310$ nm) [68]. Compared with phen, a red shift occurs in **1**, which might also be attributed to LMCT [69–71]. The strong fluorescence emissions of **1** make it potentially useful as photoactive materials.

3.4. *Thermal gravimetric analysis and XRPD measurement*

To examine thermal stability of these frameworks, thermogravimetric analyses (TG-DTG) for the compounds were performed with a NETZSCH STA 449C thermogravimetric instrument from 30°C to 900°C at a heating rate of 10°C $\min^{-1}$ under static air. The simultaneous TG profiles of **1** and **2** are shown in "Supplementary material." Compound **1** displays three weight loss stages. The first with 2.49% (Calcd 2.36%) (50−231.8°C) corresponds to release of two guest water molecules per formula unit. The second step shows a weight loss of 34.09% from 231.8°C to 353.8°C, which closely matches the calculated weight loss of 32.43% for removing one phen and one H_2bptc^{2-} from the framework of **1**. The third step occurring between 353.8°C and 472.4°C corresponds to oxidation of other phen and other H_2bptc^{2-} ligands. Compound **2** also displays three main steps of weight loss, beginning to collapse at 350.4°C with the total weight loss of 78.60% (Calcd 78.12%). A weight loss of 10.01% (Calcd 10.23%) at 50–170.8°C corresponds to release of lattice water and coordinated water; then decomposition of other components started beyond 350°C.

To confirm the phase-purity for **1** and **2**, samples were characterized by XRPD at room temperature (Supplementary material). The patterns simulated from the single-crystal X-ray data of **1** and **2** are in agreement with the experimental ones, proving each sample is a single phase.

3.5. *Magnetism*

The temperature dependence of χ_M and inverse magnetic susceptibility ($1/\chi_M$) for **1** and **2** at 1000 Oe from 2 to 300 K are shown in figures 6 and 7. Both $1/\chi_M$ *versus* T plots are almost linear down to very low temperatures. Fitting the curves to the Curie–Weiss law [$1/\chi_M = (T-\theta)/C$] gives a Curie–Weiss constant of $C = 8.277\ \mathrm{cm^3\,K\,mol^{-1}}$ and a Weiss temperature, $\theta = 1.41$ K for **1** and $\theta = -0.456$ K for **2**. The θ value for **2** is indicative of strong antiferromagnetic interactions [72, 73], while **1** displays weak ferromagnetic interactions. The antiferromagnetic interactions are obviously due to a long-range superexchange pathway between adjacent magnetic centers [74, 75]; the bridging carboxylic groups may lead to antiferromagnetic behavior, as shown in $\{[Co_3(nbtb)_2$

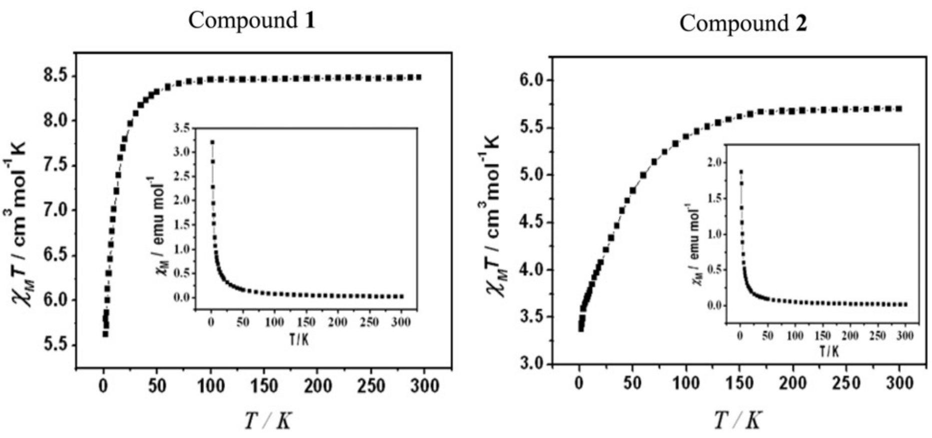

Figure 6. χ_M and $\chi_M T$ *vs.* T for **1** and **2**.

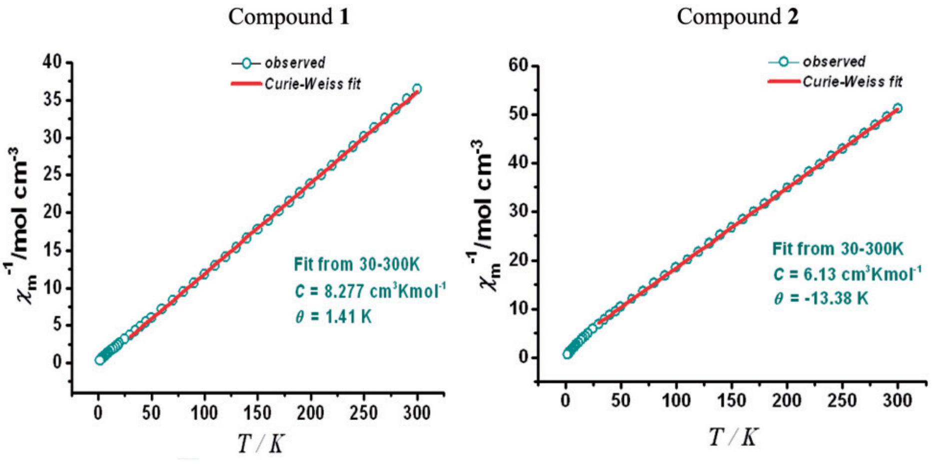

Figure 7. The reverse susceptibility $1/\chi_M$ of the complex magnetism over temperature.

$(bpe)_3(H_2O)_2] \cdot 2H_2O\}_n$, $[Mn(H_2bptc)(2,2'\text{-bpy})_2] \cdot 2H_2O$, and $[Mn_3(Hbptc)_2(2,2'\text{-bpy})_3(H_2O)_8] \cdot 2H_2O$ [63, 64]. The analysis of magnetic data for **1** is complicated by spin–orbit coupling, distortion from regular stereochemistry, electron delocalization, and crystal field mixing of excited states into the ground state, affecting the magnetic properties [76]. In spite of these difficulties, a comparative investigation of structurally analogous **1** and **2** helps to learn about the contribution of different coordination modes of bridging ligands. The difference between **1** and **2** may result from the fact that the metal–metal separation bridged by bpdc^{4-} in **2** is shorter than that in **1**.

4. Conclusion

Two coordination compounds based on 3,3′,4,4′-biphenyltetracarboxylic acid (3,4-H_4bptc) have been synthesized. Hydrogen-bonding interactions in these systems further enhance the stability and extend to form high-dimensional frameworks. Compound **1** in the solid state exhibits strong fluorescent emission bands with red shifts, which presumably arises from LMCT. The two transition metal coordination polymers bridged by carboxylates are different, implying difference in mediating magnetic coupling interactions for different coordination modes of the ligand.

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