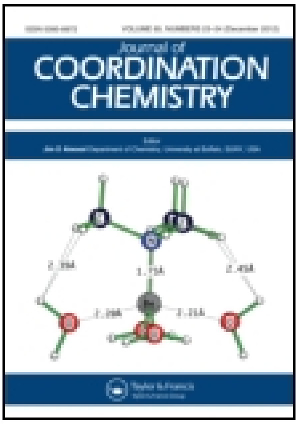

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# Exploratory syntheses, crystal structures, and properties of two new 3-D coordination polymers of Mn(II) and Fe(II) with $(5^4.6^2)$ $(5^{10}.6^3.7.8)$ topological spaces

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# Exploratory syntheses, crystal structures, and properties of two new 3-D coordination polymers of Mn(II) and Fe(II) with $(5^4.6^2)(5^{10}.6^3.7.8)$ topological spaces

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Solvothermal reactions of terphenyl-2,5,2′,5′-tetracarboxylic acid ($H_4$qptc) and $M^{II}$ (M = Mn, Fe) in the presence of 1,2-bis(diphenylphosphino)ethane (dppe) afford two new coordination polymers, $[M(qptc)(dppe\text{-}O)]_n$ (M = Mn (**1**), Fe (**2**)) (1,2-bis(diphenylphosphoryl)ethane = dppe-O). The structures have been determined by single-crystal X-ray diffraction analyses and further characterized by elemental analyses, IR, TGA, and magnetism. The $M^{II}$ in **1** and **2** are linked by $qptc^{4-}$ to form 2-D layers, which are further connected *via* the oxidized dppe (dppe-O) exhibiting a (4,6)-connected network with the $(5^4.6^2)(5^{10}.6^3.7.8)$ topology.

*Keywords*: terphenyl-2,5,2′,5′-tetracarboxylic acid; 1,2-bis(diphenylphosphoryl)ethane; manganese(II); iron(II)

## 1. Introduction

Synthesis of coordination polymers (CPs) is interesting because of potential applications in gas storage, microelectronics, molecular magnets, chemical separations, nonlinear optics and heterogeneous catalysis and interesting topologies [1–5]. Diverse organic spacers with suitable metal salts have been used to construct various molecular structures with aromatic polycarboxylates as excellent candidates for building the desired CPs due to their bent backbones and versatile bridging [6–11]. Structures of such materials depend on the metal ions, pH, solvents, synthetic methods, etc. [12–15].

In assembly of MOFs, selection of organic polycarboxylates with effective coordination and suitable conjugated systems is important [16]. Metal-organic structural motifs, including honeycomb, brick wall, bilayer, ladder, herringbone, diamondoid, and rectangular grid, have been observed by employing bridging polycarboxylates [17–19]. Aromatic polycarboxylates serve as excellent candidates for building highly connected, self-penetrating, or helical coordination frameworks [20, 21]. In this contribution,

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terphenyl-2,5,2′,5′-tetracarboxylic acid was selected for the following reasons: (i) it is a tetracarboxylate with variable coordination; (ii) it forms multidimensional CPs due to rigid, symmetrical, and long character. Secondary ligands such as phen also play an important role in the preparation of CPs, providing recognition sites for aromatic stacking interactions [22–25].

Our recent study on coordination assemblies using terphenyl-2,5,2′,5′-tetracarboxylic acid and phen outlines a strategy to obtain new coordination nets [26]. A minor change may be applied to realize good structural control of the resulting CPs. Thus, these considerations inspired us to explore coordination frameworks with P-donor 1,2-bis(diphenylphosphoryl)ethane. Complexes based on oxidized P-donors are less common in construction of CPs [27–31]. Compared with phen, dppe is more flexible and longer, possessing interesting character: (i) it has two terminal phosphorus donors and can bridge and (ii) phosphorus can be oxidized in the reaction providing new coordination. These attributes may lead to cavities, interpenetration, helical structures, and other motifs [32–35]. Recently, we began to assemble $H_4$qptc and different transitional metal ions into polymeric complexes with dppe as the bridge under the solvothermal conditions. In this article, we report syntheses and characterization of two CPs, $[M(qptc)(dppe\text{-}O)]_n$ ($M = $ Mn (**1**), Fe (**2**)), which are isostructural and display a (4,6)-connected network with the $(5^4.6^2)(5^{10}.6^3.7.8)$ topology.

## 2. Experimental

### 2.1. *Materials and methods*

Terphenyl-2,5,2′,5′-tetracarboxylic acid and 1,2-bis(diphenylphosphino)ethane were purchased from Jinan Henghua Sci. & Tec. Co. Ltd. and used without purification. C, N, and H analyses were performed on an EA1110 CHNS-0 CE elemental analyzer. IR (KBr pellet) spectra were recorded on a Nicolet Magna 750FT-IR spectrometer. Thermogravimetric measurements were carried out in a nitrogen stream using a Netzsch STA449C apparatus with a heating rate of 10°C min$^{-1}$. Variable-temperature magnetic susceptibilities were measured on polycrystalline samples with a Quantum Design MPMS SQUID susceptometer operating at a magnetic field of 1000 Oe from 1.8 to 300 K.

### 2.2. *Synthesis*

**2.2.1. Synthesis of [Mn(qptc)(dppe-O)]$_n$ (1).** The synthesis is performed in 25 mL Teflon-lined stainless steel vessels. A mixture of $H_4$qptc (0.15 mmol, 0.050 g), manganese(II) sulfate monohydrate (0.20 mmol, 0.048 g), dppe-O (0.32 mmol, 0.050 g), NaOH (0.3 mmol, 0.012 g), 6 mL $H_2O$, and 6 mL EtOH was heated to 120°C for 3 days, followed by slow cooling (descent rate of 10°C h$^{-1}$) to room temperature, giving block crystals. Yield 36% (based on Mn). Anal. Calcd (%) for $C_{48}H_{34}MnO_{10}P_2$ (887.66): C, 64.95; H, 3.86; P, 6.98. Found (%): C, 64.85; H, 3.81; P, 6.86. IR (KBr pellet, cm$^{-1}$): 3445(vs), 3069(v), 2934(v), 1708(vs), 1589(vs), 1453(vs), 1231(s), 1098(s), 850(m), 779(m), 523(w).

**2.2.2. Synthesis of [Fe(qptc)(dppe-O)]$_n$ (2).** The synthesis is performed in 25 mL Teflon-lined stainless steel vessels. A mixture of $H_4$qptc (0.15 mmol, 0.050 g), iron(II) chloride (0.20 mmol, 0.048 g), dppe-O (0.32 mmol, 0.050 g), NaOH (0.3 mmol, 0.012 g), 6 mL $H_2O$, and 6 mL EtOH was heated to 120°C for 3 days, followed by slow cooling (descent rate of 10°C $h^{-1}$) to room temperature, giving block crystals. Yield 36% (based on Fe). Anal. Calcd (%) for $C_{48}H34FeO_{10}P_2$ (888.57): C, 64.88; H, 3.86; P, 6.97. Found (%): C, 64.83; H, 3.75; P, 6.79. IR (KBr pellet, $cm^{-1}$): 3436(vs), 3073(v), 2921(v), 1810(vs), 1551(vs), 1417(vs), 1251(s), 1105(s), 852(m), 773(m), 517(w).

**2.2.3. IR spectra discussion.** IR spectra of **1** and **2** show vibration of carboxylic acids. Strong peaks (779, 850, 3069, 3445 $cm^{-1}$ in **1**; 773, 852, 3073, 3436 $cm^{-1}$ in **2**) are attributed to the stretch of benzene rings. The peaks (1098, 1589, 1708, 2934 $cm^{-1}$ in **1**; 1115, 1551, 1810, 2921 $cm^{-1}$ in **2**) could be assigned to coordinated carboxylate. Strong peaks of 1453 and 1231 $cm^{-1}$ in **1** and 1417 and 1251 $cm^{-1}$ in **2** are mainly due to P=O, P-benzene interaction. IR spectra of **1** and **2** are in agreement with the crystal structure.

### 2.3. *X-ray crystallography*

Intensity data collection was carried out on a Siemens SMART diffractometer equipped with a CCD detector using Mo-K$\alpha$ monochromated radiation ($\lambda = 0.71073$ Å) at 293(2) K. The absorption correction was based on multiple and symmetry-equivalent reflections in the data set using SADABS based on the method of Blessing. The structures were solved by direct methods and refined by full-matrix least-squares using the SHELXTL package [36, 37]. Crystallographic data for **1** and **2** are given in table 1. Selected bond lengths and angles are listed in table 2.

## 3. Results and discussion

### 3.1. *Structure descriptions of [M(qptc)(dppe-O)]$_n$ (M = Mn (1), Fe (2))*

The single-crystal X-ray diffraction analysis reveals that *1* and *2* are isostructural and crystallize in the monoclinic system with different space groups: $P2(1)/n$ for **1** and $P2(1)/c$ for **2**. Here only the structure of **1** will be discussed as a representative. The asymmetric unit of **1** consists of half of a $Mn^{II}$, half of a dppe-O, and half of a qptc$^{4-}$. Each $Mn^{II}$ has a distorted octahedral geometry, coordinated by four qptc$^{4-}$ [Mn(1)–O(2) = 2.124(2) Å, Mn(1)–O(3) = 2.2594(18) Å] and two dppe-O bridging ligands [Mn(1)–O(1) = 2.107(2) Å] showing a distorted octahedral coordination geometry (figure 1).

The $H_4$qptc in **1** and **2** is completely deprotonated under the hydrothermal condition and the four carboxylates of qptc$^{4-}$ exhibit $\mu_1$-$\eta^1$:$\eta^0$ coordination to link four $M^{II}$ ions, forming one 2-D layer in the *ac* plane (figure 2a). Bridging dppe-O ligands preserve the original anti conformation of donors with dihedral angles between O=P–C···C–P=O planes from 155.4° to 180° for the antiparallel (anti) and from 9.2° to 29.8° for parallel (syn) alignments of the MnO groups. The 2-D layers above are further linked *via* dppe-O ligands to form the (4,6)-connected network with the $(5^4.6^2)(5^{10}.6^3.7.8)$

Table 1. Crystallographic data and details of diffraction experiments for **1** and **2**.

| | **1** | **2** |
|---|---|---|
| Empirical formula | $C_{48}H_{34}MnO_{10}P_2$ | $C_{48}H_{34}FeO_{10}P_2$ |
| Formula weight | 887.63 | 888.54 |
| Crystal system | Monoclinic | Monoclinic |
| Space group | $P2(1)/n$ | $P2(1)/c$ |
| Unit cell dimensions (Å, °) | | |
| $a$ | 13.233(3) | 13.338(3) |
| $b$ | 9.506(2) | 9.704(2) |
| $c$ | 16.443(4) | 18.866(3) |
| $\alpha$ | 90.00 | 90.00 |
| $\beta$ | 102.171(2) | 121.223(12) |
| $\gamma$ | 90.00 | 90.00 |
| Volume (Å$^3$) | 2022.0(7) | 2088.2(7) |
| $Z$ | 2 | 2 |
| Calculated density (g cm$^{-3}$) | 1.458 | 1.413 |
| Absorption coefficient (mm$^{-1}$) | 0.469 | 0.499 |
| Temperature (K) | 298(2) | 298(2) |
| $R_{\text{int}}$ | 0.0393 | 0.0538 |
| $R\ [I > 2\sigma(I)]^{a,b}$ | $R_1 = 0.0445$, $wR_2 = 0.1091$ | $R_1 = 0.0505$, $wR_2 = 0.0713$ |
| $R$ (all data)$^{a,b}$ | $R_1 = 0.0650$, $wR_2 = 0.1198$ | $R_1 = 0.0664$, $wR_2 = 0.0936$ |
| Goodness-of-fit$^c$ | 1.001 | 1.004 |
| $(\Delta\rho)_{\max}$ (e Å$^{-3}$) | 0.583 | 0.682 |
| $(\Delta\rho)_{\min}$ (e Å$^{-3}$) | −0.394 | −0.452 |

[a] $R_1 = \Sigma\|F_{\text{obsd.}}| - |F_{\text{calcd}}\|/\Sigma|F_{\text{obsd.}}|$.
[b] $wR_2 = \{\Sigma[w(F^2_{\text{obsd.}} - F^2_{\text{calcd}})^2]/\Sigma[w(F^2_{\text{obsd.}})^2]\}^{1/2}$. $w = 1/[\sigma^2(F_o^2) + xP + (yP)^2]$; with $P = (F_o^2 + 2F_c^2)/3$.
[c] Goodness-of-fit $= [\Sigma w(F^2_{\text{obsd.}} - F^2_{\text{calcd}})^2/(n-p)]^{1/2}$, where $n$ = number of reflections, $p$ = parameter used.

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

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Complex **1** | | | | | | | |
| Mn1–O1 | 2.107(2) | Mn1–O2 | 2.124(2) | Mn1–O3 | 2.2594(18) | | |
| O1#1–Mn1–O1 | 180.0 | O1#1–Mn1–O2#1 | 88.76(12) | O1–Mn1–O2#1 | 91.24(12) | O1#1–Mn1–O2 | 91.24(12) |
| O1–Mn1–O2 | 88.76(12) | O2#1–Mn1–O2 | 180.0 | O1#1–Mn1–O3 | 82.73(8) | O1–Mn1–O3 | 97.27(8) |
| O2#1–Mn1–O3 | 97.33(8) | O2–Mn1–O3 | 82.67(8) | O1#1–Mn1–O3#1 | 97.27(8) | O1–Mn1–O3#1 | 82.73(8) |
| O2#1–Mn1–O3#1 | 82.67(8) | O2–Mn1–O3#1 | 97.33(8) | O3–Mn1–O3#1 | 180.0 | | |
| Complex **2** | | | | | | | |
| Fe1–O3#1 | 2.228(5) | Fe1–O3 | 2.228(5) | Fe1–O1#1 | 2.237(5) | Fe1–O1 | 2.237(5) |
| Fe1–O4 | 2.322(4) | Fe1–O4#1 | 2.322(4) | | | | |
| O3#1–Fe1–O3 | 180.000(1) | O3#1–Fe1–O1#1 | 89.3(3) | O3–Fe1–O1#1 | 90.7(3) | O3#1–Fe1–O1 | 90.7(3) |
| O3–Fe1–O1 | 89.3(3) | O1#1–Fe1–O1 | 180.000(1) | O3#1–Fe1–O4 | 92.71(19) | O3–Fe1–O4 | 87.29(19) |
| O1#1–Fe1–O4 | 81.3(2) | O1–Fe1–O4 | 98.7(2) | O3#1–Fe1–O4#1 | 87.29(19) | O3–Fe1–O4#1 | 92.71(19) |
| O1#1–Fe1–O4#1 | 98.7(2) | O1–Fe1–O4#1 | 81.3(2) | O4–Fe1–O4#1 | 180.000(1) | | |

Symmetry codes: For complex **1**: #1 $-x = 1$, $-y = 2$, $-z$; for complex **2**: #1 $-x = 1$, $-y = 1$, $-z = 1$.

topology (figure 3). The 3-D polymeric structures of **1** and **2** consist of alternately arranged left- and right-handed helical chains along the *b*-axis (figure 2b).

### 3.2. *Magnetic studies*

The temperature dependence of the magnetic susceptibility of a powder sample was measured for **1** and **2** by a Quantum Design SQUID magnetometer on the MPMS-7 system; their magnetic behavior is shown in figure 4. The overall magnetic behavior of **1**

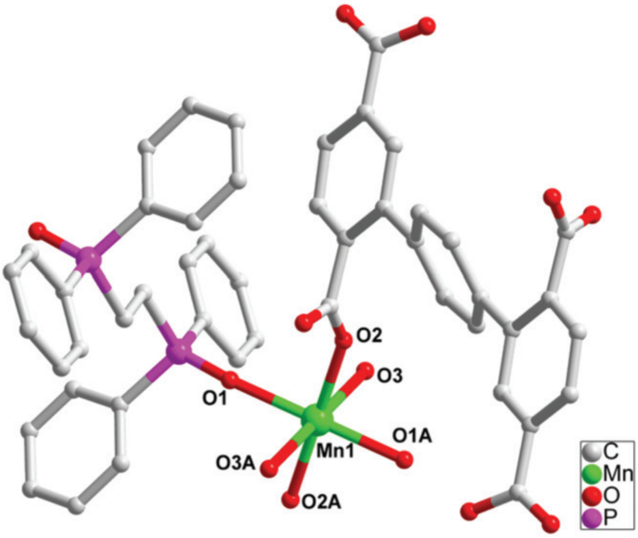

Figure 1. Coordination environment of $Mn^{II}$ in **1** with thermal ellipsoids at 30% probability level (symmetry codes: (A): $-x,-y+1,-z+1$).

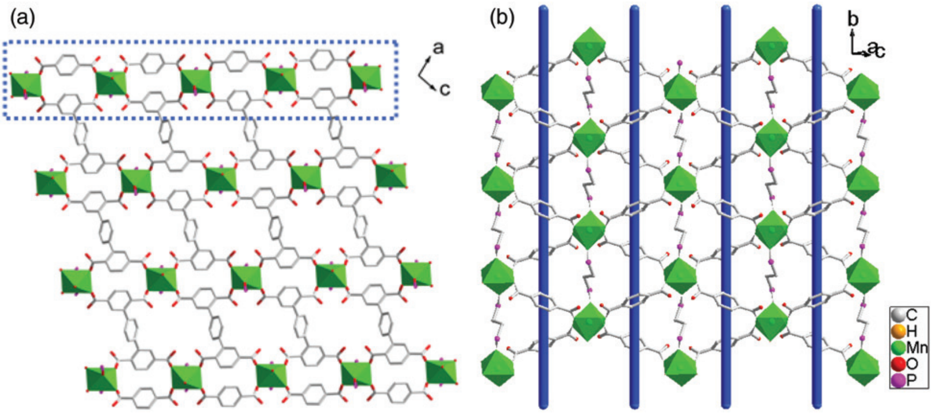

Figure 2. (a) View of the 2-D layer of **1** and **2**; (b) View of the 2-D layer of **1** and **2** containing right-handed helices.

and **2** corresponds to paramagnets with magnetic moment values of 4.62 $\mu_B$ for **1** and 3.58 $\mu_B$ for **2** similar to the magnetic moment of single $Mn^{2+}$ cation (4.38 $\mu_B$) and $Fe^{2+}$ cation (3.00 $\mu_B$).

## 3.3. *TGA analysis*

To reveal the thermal stabilities of **1** and **2**, TGA measurements have been carried out on a Perkin-Elmer Diamond TG/DTA instrument with a flow of dry air and a heating rate of 5°C $min^{-1}$ from room temperature to 1000°C. As shown in Supplementary

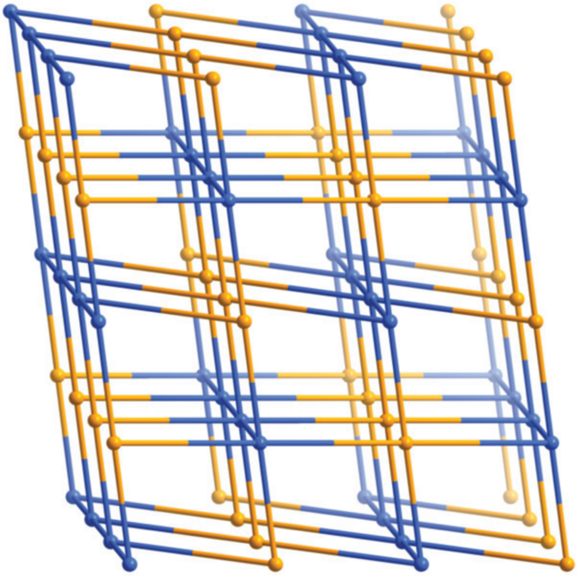

Figure 3. A perspective view of the 3-D supramolecular structure of **1** and **2**.

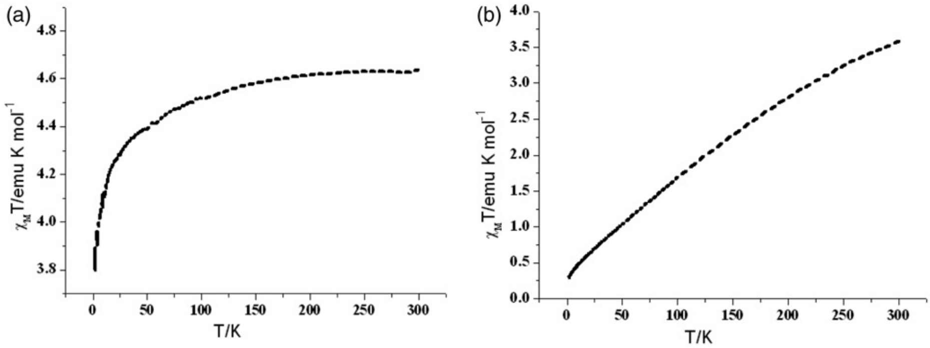

Figure 4. (a) Plot of the effective magnetic moment as a function of temperature for **1** in constant magnetic field of 1000 Oe. (b) Plot of the effective magnetic moment as a function of temperature for **2** in constant magnetic field of 1000 Oe.

Material, TGA curve indicates that **1** and **2** are unstable with increasing temperature. Over 600°C, qptc$^{4-}$ and dppe-O were completely decomposed. The residual weight of **1** is *ca* 8.1% (Calcd for MnO: 8.0%). The residual weight of **2** is *ca* 9.6% (Calcd for $Fe_2O_3$: 9.0%).

## 4. Conclusion

We have developed a synthetic strategy for CPs by employing terphenyl-2,5,2′,5′-tetracarboxylic acid ($H_4$qptc). Two CPs, $[M(qptc)(dppe\text{-}O)]_n$ ($M = $ Mn (**1**), Fe (**2**)), based on $H_4$qptc and dppe have been obtained under solvothermal conditions. Structural comparisons of **1** and **2** indicate that the backbone of qptc$^{4-}$ plays an important role in structures of these metal-organic coordination architectures. Further systematic studies for the design and synthesis of such crystalline materials with $H_4$qptc and other N/P-donor building blocks are underway in our laboratory.

## Supplementary material

For **1** and **2**, further details on the crystal structure investigations may be obtained from the Cambridge Crystallographic Data Centre, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [Telephone: +44-(0)1223-762-910; Fax: +44-(0)1223-336-033; Email: deposit@ccdc.cam.ac.uk; http://www.ccdc.cam.ac.uk/deposit], on quoting the depository number CCDC-856757 for **1** and 856756 for **2**.

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