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# Carboxylato-bridged 3D polymeric networks of Pr(III): Synthesis, crystal structure, magnetic property and thermal behavior

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# ABSTRACT

A novel polymeric carboxylato-bridged praseodymium(III) complex of fumarate,  $[Pr_2(fum)_3(H_2O)_4]$ -5H<sub>2</sub>O (fum, fumarate dianion) has been synthesized and characterized by single-crystal X-ray diffraction studies, magnetic measurements and thermal studies. Single crystal X-ray structural determination reveals that it is a new porous three dimensional pillared layer framework with 1D channel for the accommodation of tetrameric water clusters. A preliminary treatment of the variable-temperature magnetic susceptibility using an expression including the ligand field effects and molecular field approximation leads to  $zJ' = -1.60 \text{ cm}^{-1}$ ,  $\Delta = 0.5 \text{ cm}^{-1}$  and g = 0.84, suggesting the existence of a weak antiferromagnetic coupling between the praseodymium ions.

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# 1. Introduction

The exponential growth of the field of metal-organic materials, particularly metal-organic frameworks (MOFs), has been seen over the past decades due to their fascinating network topologies and their many potential applications as functional materials [1]. This structural versatility is reflected in applications to areas as diverse as catalysis, proton conductivity, ion exchange, intercalation chemistry, photochemistry and magnetic materials [2-9]. Even more remarkably, these applications can be combined and integrated into individual frameworks to form multifunctional MOFs [10]. Lanthanide (Ln)-containing MOFs have attracted much interest due to their ability to incorporate both photoluminescent centers and magnetic properties, making them ideal for developing new multifunctional materials [11]. Furthermore, the high affinity of lanthanides for oxygen donor atoms makes carboxylates excellent candidates as bridging ligands, because they can adopt a variety of coordination modes and result in diverse multidimensional architectures [12]. Among the dicarboxylates being employed in the construction of such rare earth MOFs, fumarate (hereinafter fum) [13] is unique because of its relatively small central moiety (CH=CH), compared with others such as the most common benzene-dicarboxylate ligands [14], which allows for more versatile coordination modes and possible porosity in the constructed frameworks. Several structural studies have been published on lanthanide fumarates [13,15]. For example, Gao et al. reported a new three dimensional (3D) Sm–fum framework,  $[Sm_2(fum)_3(H_2O)_4]$ .  $3H_2O$ , which possesses a three dimensional structure but has 0D cavities, and each cavity supports a hexameric water cluster. Interestingly, the material exhibits a reversible dehydration–rehydration procedure and framework dynamics [13a].

The present contribution reports the hydrothermal synthesis, crystal structure, magnetic study and thermal behavior of the new praseodymium (III) frameworks with fum, namely  $[Pr_2(fum)_3(H_2O)_4]\cdot5H_2O$  (complex **1**). The compound has the same Ln/fum ratio of 2:3 as  $[Sm_2(fum)_3(H_2O)_4]\cdot3H_2O$  [13a], but with different structure. Complex **1**, containing a rigid anion, is 3D polymeric network of Pr(III) with 10 coordination geometry and has a small void in the crystal filled with water molecules. Low-temperature magnetic susceptibility measurements show the existence of a weak antiferromagnetic coupling among the praseodymium ions.

# 2. Experimental

#### 2.1. Materials and apparatus

Pr(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O was prepared by dissolving the oxide in dilute nitric acid and the filtrate was subsequently dried under steam bath to obtain the nitrate salt. All other chemicals were purchased and used as received without further purification. Elemental analysis was carried out on a Perkin–Elmer 2400 elemental analyzer. TGA 7 thermogravimetric analyzer was used to carry out thermogravimetric analysis in the air with a heating rate of 10 °C min<sup>-1</sup>.





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Variable-temperature magnetic susceptibilitie was measured using a MPMSXL-7 SQUID magnetometer. The magnetic data were corrected for the sample holder and the diamagnetic contributions.

# 2.2. Preparation of $[Pr_2(fum)_3(H_2O)_4] \cdot 5H_2O(1)$

 $Pr(NO_3)_3$ ·6H<sub>2</sub>O (0.5 mmol, 0.218 g) was dissolved in 10 mL deionized water, to which H<sub>2</sub>fum (1 mmol, 0.116 g) and glutamic acid (1 mmol, 0.147 g) were added while stirring. Finally, NaOH aqueous solution was added to adjust the pH value of the mixture to about 3.5–4. A homogeneous reaction gel was formed after stirring for 2 h, and was transferred to a Teflon-lined stainless steel autoclave (15 mL). The vessel was sealed and heated at 150 °C for 5 d under autogenous pressure and then cooled to room temperature. After filtered, the product was washed with ethanol and then dried under ambient conditions. Quadrate crystals of the complex 1 were collected. Elemental analysis calcd (%) for 1 in powder form: C 18.32, H 3.05, O 42.74; found: C 18.21, H 3.12, O 42.51.

# 2.3. Single-crystal X-ray diffraction

Suitable single crystal with dimensions of  $0.15 \times 0.12 \times$ 0.11 mm<sup>3</sup> for compound **1** was selected for single-crystal X-ray diffraction analysis. Crystallographic data were collected at a temperature of -86 ± 2 °C on a Bruker ApexII CCD diffractometer with graphite monochromated Mo K $\alpha$  radiation ( $\lambda = 0.71073$  Å). Data processing was accomplished with the SAINT processing program. The structure was solved by the direct methods and refined on  $F^2$ by full-matrix least-squares using SHELXTL97 [16] The locations of Pr atom was easily determined, and O, C, and H atoms were subsequently determined from the difference Fourier maps. All non-hydrogen atoms were refined with anisotropic thermal parameters. Crystal data:  $[Pr_2(fum)_3(H_2O)_4] \cdot 5H_2O$ : Mr = 786.13, monoclinic system, space group P2(1)/c (No. 14), a = 9.1999(5) Å, b = 15.8570(8) Å, c = 8.6559(5) Å,  $\alpha = 90^{\circ}$ ,  $\beta = 114.4380(10)^{\circ}$ ,  $\gamma$  = 90, V = 1149.62(11) Å<sup>3</sup>, and Z = 2,  $\mu$  = 4.29 mm<sup>-1</sup>,  $\rho_{calcd}$  = 2.271 g cm<sup>-3</sup>. The final  $wR_2$  (all data) was 0.0603 and  $R_1$  was 0.0333. Experimental details for the structural determination are described in Table 1. The selected bond distances and bond angles are given in Tables S1.

#### Table 1

Crystal data and structure refinement for 1.

Identification code	1
Empirical formula	$C_{12}H_{24} \cdot O_{21}Pr_2$
Formula weight	786.13
Temperature	187 K
Wavelength	0.71073 A
Crystal system, space group	P2(1)/c, monoclinic
Unit cell dimensions	$a = 9.1999(5)$ Å, $\alpha = 90^{\circ}$
	$b = 15.857(8)$ Å, $\beta = 114.438(10)^{\circ}$
	$c = 8.6559(5)$ Å, $\gamma = 90^{\circ}$
Volume	1149.62 (11)
Z, calculated density	4, 2.271 Mg/m <sup>3</sup>
Absorption coefficient	$4.286 \text{ mm}^{-1}$
F(000)	764
Crystal size	$0.15\times0.12\times0.11\ mm^3$
Theta range for data collection	2.43-26.02°
Limiting indices	$11 \leqslant h \leqslant 8$ , $-19 \leqslant k \leqslant 18$ , $-10 \leqslant l \leqslant 10$
Reflections collected/unique	6301/2263 [ <i>R</i> (int) = 0.0290]
Completeness to theta = 26.02	99.9%
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data/restraints/parameters	2263/12/209
Goodness-of-fit on F <sup>2</sup>	1.050
Final R indices [I > 2sigma(I)]	$R_1 = 0.0264, wR_2 = 0.0579$
R indices (all data)	$R_1 = 0.0333, wR_2 = 0.0603$
Largest diff. peak and hole	0.846 and –0.832 <i>e</i> Å <sup>–3</sup>

# 3. Results and discussion

# 3.1. Crystal structure description of 1

As shown in Fig. 1, there is only one type of Pr(III) atom environment in the asymmetric unit of 1. Each praseodymium atom is coordinated by 10 oxygen atoms, two of which belong to the two coordinated water molecules, while the remaining eight belong to six fumarate ligands. There are one and a half crystallographically independent fumarate molecules in the asymmetrical unit of **1**. Two types of coordination modes of fumarate ligands exist in complex 1: (i) two chelating/bridging tridentate [Scheme 1a], the fumarate anions are centrosymmetric and bonded in this mode to Pr(III) atoms along the a-axis; (ii) two bridging bidentate [Scheme 1b], the fumarate anions are bonded in this mode to Pr(III) cations along the *b*-axis. All the interatomic distances are usual: The Pr-O distances are in the range of 2.428-2.737 Å and Pr-Pr distances is 4.370 Å, as well as C-O (1.249-1.271 Å) and C-C distances (1.314–1.493) Å seemingly typical for praseodymium(III) carboxylates [15].

Complex 1 possesses three dimensional structure, which is built up from complex chains of Pr(III) polyhedra, linked in the two other directions by fumarate. The chains of compound **1** are built up from the edge-sharing linkage of 10-coordinated Pr(III) polyhedra along the *a* axis, as shown in Fig. 2a, which are further linked by fumarate in chelating and bridging mode to give rise to a 2D sheets parallel to the *ac* plane (Fig. 2b). The other type fumarate in syn-syn bridging mode is approximately perpendicular to the ac plane, pillaring the sheets and a global 3D network is formed (Fig. 2c). As a consequence, large rhombus 1D channels are formed in the direction of *a* and *c* axis with the cavities of ca. 11.525  $\times$  8.696 Å (Pr  $\cdots$  Pr distance) and ca. 10.046  $\times$  6.567 (C2...C2 distance) Å, respectively. The free water molecules, located in the tunnels, give numerous hydrogen bonds with the terminal H<sub>2</sub>O molecules of the skeleton, which point toward the center of the tunnels. As a comparison, the 3D structure of [Sm<sub>2</sub>(fum)<sub>3</sub>(H<sub>2</sub>O)<sub>4</sub>]·3H<sub>2</sub>O [13a], containing a different number of lattice water molecules, originates from a supramolecular isomer. It comprises of dinuclear entities connected by dicarboxylate anions, rather than containing chains of praseodymium atoms at a distance of ca. 4.37 Å in complex **1**. The structure possesses three kinds of fum ligands: each has its two COO groups adopting different coordination modes. While there are two kinds of fum ligands and two COO groups of each fum ligand adopting the same coordination modes in complex 1. Such diversity coordination



Fig. 1. The coordination environments of Pr(III) in complex 1.



Scheme 1. Coordination modes of fumarate groups in complex 1.



**Fig. 2.** (a) Polyhedral view of the inorganic chains of complex **1**; (b) 2-D network of **1** parallel to the *ac* plane; (c) view of the structure along *c*-axis. The pillaring ligand links the layers between them. The coordinated water molecules point into the channel. Lattice water molecules are in the channel.

modes of organic ligands induce the synthesis of many novel structures.

There are five crystallographically independent water molecules in **1**. Two of them, O(4w) and O(5w), are from the coordinated water molecules, and the other three, O(1w), O(2w) and O(3w) come from the uncoordinated water molecules. They are self-assembled through hydrogen bonds. The hydrogen bonding geometry and the water network are shown in Table 2 and Fig. 3a, respectively. O(1w), O(3w), O(4w), O(5w) forms a tetrameric water cluster regarding the short O...O distances of 2.645–2.861 Å along the ring (Fig. 3b). O(1w) acts as both hydrogen bond donor and acceptor, connecting with adjacent O(4w)  $(O(1w) \cdots O(4w) \ 2.809 \ \text{\AA})$  and  $O4 \ (O(1w) \cdots O4 \ 2.745 \ \text{\AA})$ . O(2w) is lied on 21 screw axis and the hydrogen atoms of which forms two kind of C-O···H hydrogen bonds with C-O of fumarate, the distances of C-O4 $\cdot$  H-O(2w) and C-O2 $\cdot$  H-O(2w) are 3.062 and 3.276 Å, respectively. O(3w) forms two hydrogen bonds with the two coordinated water molecules and the distances of  $O(3w) \cdots O(4w)$  and  $O(3w) \cdots O(5w)$  are 2.839 and 2.861 Å, respectively.

# 3.2. Thermal analysis

Thermogravimetric analysis of compound 1 was carried out in air atmosphere in the range of 35–900 °C. As seen in Fig. 4, the compound displays mainly two thermal processes. The first weight loss occurred at ca. 35–240 °C, with a weight loss of 14.5%. This is the release of the lattice and a part of coordinated water molecules. The second weight loss process occurred at ca. 400–580 °C, which due to decomposition of the material. The final residue of 47.8% is a little high than the calculated 36.9% base on  $Pr_2O_3$ . It can be attributed that the residue is the admixture of  $Pr_2O_3$  and  $PrO_2$ .

# 3.3. Magnetic measurements

Magnetic susceptibility studies of **1** have been carried out in an applied magnetic field of 1000 Oe in the temperature range 300–2 K. The plot of  $\mu_{\text{eff}}$  vs. *T* is shown in Fig. 5. At 300 K, the  $\mu_{\text{eff}}$  value of 3.58 B.M., which is in fair agreement with the expected value of

Table 2						
Hydrogen	bonds	for	1	[Å	and	deg].

D–H···A	d(D-H)	$d(H{\cdot}{\cdot}{\cdot}A)$	$d(D{\cdots}A)$	<(DHA)
OW4−H(6) · · · OW3#6	0.88(2)	2.00(3)	2.861(7)	165(7)
OW4−H(7) · · · OW1#1	0.90(2)	1.85(2)	2.745(5)	173(7)
OW1−H(2) · · · O(4)#7	0.88(2)	2.00(3)	2.809(5)	152(6)
OW5−H(8) · · · OW3	0.90(2)	1.95(3)	2.839(6)	169(9)
OW3−H(4) · · · O(2)#8	0.89(2)	2.57(5)	3.250(7)	134(6)
OW5−H(9) · · · OW2	0.89(2)	2.01(3)	2.885(3)	164(7)
OW3−H(5) · · · OW1	0.90(2)	2.54(4)	3.402(10)	161(8)
OW1−H(1) · · · OW3	0.87(2)	2.57(3)	3.402(10)	160(6)
OW2−H(3) · · · O(2)	0.88(2)	2.48(5)	3.276(3)	151(8)
OW2−H(3) ···O(4)	0.88(2)	2.48(8)	3.062(3)	124(7)

Symmetry transformations used to generate equivalent atoms: (c) #1 x, -y + 1/2, z - 1/2; #2 x, -y + 1/2, z + 1/2; #3 x - 1, y, z - 1; #4 x + 1, y, z + 1; #5 -x + 1, -y, -z + 1; #6 -x, y - 1/2, -z + 1/2; #7 x - 1, y, z; #8 -x, -y + 1, -z.



**Fig. 3.** (a) The hydrogen bonding network of complex **1**. (b) Tetrameric water clusters formed by the coordinated and uncoordinated water molecules in **1** viewed along the *b*-axis.





**Fig. 5.** The  $\mu_{\text{eff}}$  vs. *T* plot of complex **1**.

3.57 B.M. for one uncoupled Pr(III) ions (S = 1, L = 5,  ${}^{3}H_{4}$ , g = 4/5).  $\mu_{eff}$  gradually decreases until 50 K and then further decreases to reach a minimum of 0.83 B.M. at 2 K. Indeed, the effects of the thermal depopulation of Pr(III) excited states that result from spin–orbit coupling might also be partially responsible for the decrease of the  $\mu_{eff}$  when the temperature is lowered [17]. A fit of the experimental data to a Curie–Weiss law above 2 K leads to the Curie and Weiss constants of 1.77 cm<sup>3</sup> K mol<sup>-1</sup> and –18.12 K, respectively. The negative Weiss ( $\theta$ ) constant combined with roughly linearly shaped isothermal *M* vs. *H* plot (see Fig. S1 in the Supporting Information), in which *M* reaches only 0.60  $\mu_{\rm B}$  at 7 T and 2 K, is clearly indicative of the presence of some antiferromagnetic interactions between spin carriers in this complex.

To gain some information concerning the interaction between Pr(III) ions, as a preliminary treatment, we assumed that owing to the ligand field effects, Pr(III) ion may exhibit a splitting of  $m_j$  energy levels ( $\hat{H} = \Delta \hat{J}_z^2$ ), resulting in a magnetic susceptibility Eq. (1) previously derived by McPherson and co-workers [18].

$$\chi_{\rm Pr} = \frac{Ng^2\beta^2}{kT} \frac{\left[2e^{(-\Delta/kT)} + 8e^{(-4\Delta/kT)} + 18e^{(-9\Delta/kT)} + 32e^{(-16\Delta/kT)}\right]}{\left[1 + 2e^{(-\Delta/kT)} + 2e^{(-4\Delta/kT)} + 2e^{(-9\Delta/kT)} + 2e^{(-16\Delta/kT)}\right]}$$
(1)

In this expression,  $\Delta$  is the zero field splitting parameter and N, g,  $\beta$  and k have their usual meanings. Using the above equation and considering the molecular field theory with zJ' as the total exchange parameter between Pr(III) ions, we can fit our experimental data with the following equation.

$$\chi = \frac{\chi_{\rm Pr}}{1 - (2zJ'/Ng^2\beta^2)\chi_{\rm Pr}}$$
(2)

The best fitting of the susceptibility data in the temperature range 2–300 K gives  $zJ' = -1.60 \text{ cm}^{-1}$ ,  $\Delta = 0.50 \text{ cm}^{-1}$ , g = 0.84, and  $R = \sum (\chi_{obsd} - \chi_{cald})^2 / \sum \chi_{obsd}^2 = 6.8 \times 10^{-3}$ . This molecular field approximation [19–21] with zJ' characterizing the total exchange parameter between the Pr(III) ions, indicates that an overall antiferromagnetic interaction is operative between the lanthanide ions (since zJ' < 0), which is probably mediated through carboxylate groups.

## 4. Conclusion

In this paper we have presented the synthesis, crystal structure, and magnetic study of the novel polymeric architectures of praseodymium(III) with dicarboxylate anions. It possesses fumarate pillared lanthanide–fumarate layers with 1D channels for accommodation of guest water. The magnetic investigation reveals weak antiferromagnetic interactions between metal ions in this complex.

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# Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molstruc.2010.06.019.

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