

两个基于 $[\text{Fe}(\text{bpca})(\text{CN})_3]^-$ 的一维 3d-4f 异金属配合物的合成和晶体结构

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摘要: 以 $[(\text{bpca})\text{Fe}(\text{CN})_3]^-$ (bpca=二(2-吡啶羰基)酰胺阴离子)为构筑基元, 设计合成了 2 个新颖的 3d-4f 异金属配合物, $[(\text{bpca})\text{Fe}(\text{CN})_3\text{Pr}(\text{H}_2\text{O})_5]\text{Cl}_2$ (**1**) 和 $[(\text{bpca})_2\text{Fe}_2(\text{CN})_6\text{Pr}(\text{H}_2\text{O})_6]\text{Cl} \cdot 4\text{H}_2\text{O}$ (**2**), 并测定了它们的晶体结构。化合物 **1** 的晶体属正交晶系, $Pnma$ 空间群; 而化合物 **2** 属三斜晶系, $P\bar{1}$ 空间群。在这 2 个化合物中, $[(\text{bpca})\text{Fe}(\text{CN})_3]^-$ 和 $[\text{Pr}(\text{H}_2\text{O})_x]^{3+}$ (**1**, $x=5$; **2**, $x=6$) 交替排列形成一维链状结构, 并通过 π - π 堆积作用、氢键作用及分子间短距离相互作用形成三维网络结构。

关键词: 3d-4f 异金属配合物; 氰基桥联; 链状配合物; 晶体结构

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Syntheses and Crystal Structures of Two One-dimensional 3d-4f Heterobimetallic Complexes Based on $[\text{Fe}(\text{bpca})(\text{CN})_3]^-$

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Abstract: Two 3d-4f bimetallic complexes, $[(\text{bpca})\text{Fe}(\text{CN})_3\text{Pr}(\text{H}_2\text{O})_5]\text{Cl}_2$ (**1**) and $[(\text{bpca})_2\text{Fe}_2(\text{CN})_6\text{Pr}(\text{H}_2\text{O})_6]\text{Cl} \cdot 4\text{H}_2\text{O}$ (**2**) (bpca=bis(2-pyridylcarbonyl)amidate anion), have been synthesized and their crystal structures were determined. Complex **1** crystallizes in the orthorhombic space group $Pnma$ while complex **2** is in the triclinic space group $P\bar{1}$. Both complexes possess novel one-dimensional (1D) chain structures with alternating arrays of $[(\text{bpca})\text{Fe}(\text{CN})_3]^-$ and $[\text{Pr}(\text{H}_2\text{O})_x]^{3+}$ ($x=5$ for **1** and 6 for **2**) fragments. They exhibit 3D supramolecular architectures through π - π stacking interactions, hydrogen bonds and short-distance intermolecular interactions. CCDC: 624698, **1**; 624699, **2**.

Key words: 3d-4f bimetallic complexes; cyanide bridged; chain complexes; crystal structures

0 Introduction

For more than two decades, increasing attention has been paid to the construction of 3d-4f heterometallic complexes with interesting architectures and novel topologies, and some of them may have potential applications as functional materials^[1]. A successful

strategy leading to the 3d-4f heteropolymetallic system is based on the use of building-blocks containing a transition-metal ion as ligands towards lanthanide ions^[1a,b]. The hexacyanometalate anions $[\text{M}(\text{CN})_6]^{n-}$ (M is a transition-metal atom such as Fe, Co, Cr or Mn) have been used widely as building blocks to construct cyano-bridged 3d-4f metal complexes with different dimensio-

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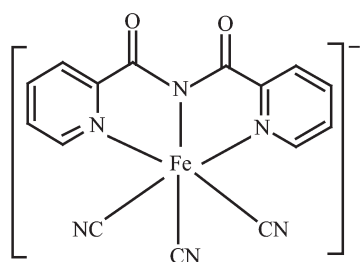
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nalities^[1d,e,2-9], as cyanide is an efficient bridging ligand to construct the framework due to the singularities that it can strongly bridge two different metal atoms in an end-to-end fashion^[10]. However, there are rare similar reports involving the building blocks of modified cyanometalates $[(L)_mM(CN)_x]^{(x-m)-}$ (M is a transition-metal ion; L is an organic polydentate ligand)^[11-14].

In an effort to extend this interesting chemistry, we choose the tailored cyanometalate precursor, $[(bpca)Fe(CN)_3]^-$ (Scheme 1), which has been used to prepare several clusters and polymers with 3d transition metal ions^[15]. This precursor consists of three CN^- groups and the ligand of bis(2-pyridylcarbonyl)amidate anion (bpca), affording N-donor and O-donor, both of which are able to coordinate to transition and lanthanide metal ions. Thus, it may direct the formation of new 3d-4f complexes with interesting structures. Marilena et al. have prepared a binuclear single molecule magnet $[Fe(bpca)(\mu-bpca)Dy(NO_3)_4]$ with the use of the ligand of bpca^[16]. Herein, we report the synthesis and crystal structures of two one-dimensional (1D) cyano- and bpca bridged 3d-4f polymers, $\{[(bpca)Fe(CN)_3Pr(H_2O)_5]Cl_2\}_n$ (**1**) and $\{[(bpca)_2Fe_2(CN)_6Pr(H_2O)_6]Cl \cdot 4H_2O\}_n$ (**2**).



Scheme 1

1 Experimental

1.1 Materials and measurements

All chemicals were reagent grade and used as received. $(Bu_4N)[(bpca)Fe(CN)_3] \cdot H_2O$ was prepared according to the literature method^[15b]. Although no problems were encountered in this work, cyanides are toxic and should be handled with great care!

The IR spectra were taken on a Vector22 Bruker spectrophotometer with KBr pellets in the range 4000~400 cm^{-1} . Elemental analysis for C, H, N were performed on a Perkin-Elmer 240C analyzer.

1.2 Preparation of complex 1

A solution of $PrCl_3 \cdot 6H_2O$ (0.05 mmol, 17.8 mg) in 2 mL of water was added to a solution of $(Bu_4N)[(bpca)Fe(CN)_3] \cdot H_2O$ (31.1 mg, 0.05 mmol) in 25 mL of methanol. Slow evaporation of the resulting solution in air, resulted to the formation of yellow plate-like crystals after two weeks. Yield: 35%. Anal. Calcd. for $C_{15}H_{18}FePrN_6O_7Cl_2$ (%): C, 27.21; H, 2.74; N, 12.69. Found (%): C, 27.26; H, 2.69; N, 12.72.

1.3 Preparation of complex 2

$PrCl_3 \cdot 6H_2O$ (8.9 mg, 0.025 mmol) was added to a 20 mL acetonitrile solution of $(Bu_4N)[(bpca)Fe(CN)_3] \cdot H_2O$ (31.1 mg, 0.05 mmol) and the mixture precipitated immediately. Water was added dropwise to the mixture until the precipitate disappeared. Yellow needlelike crystals were obtained after two weeks upon the evaporation of the solvent. Yield: 42%. Anal. Calcd. for $C_{30}H_{36}Fe_2PrN_{12}O_{14}Cl$ (%): C, 33.46; H, 3.37; N, 15.61. Found (%): C, 33.38; H, 3.34; N, 15.70.

1.4 Crystal structure determination

The crystal structures of complexes **1** and **2** were determined on a Bruker SMART Apex CCD diffractometer using monochromatized Mo $K\alpha$ radiation ($\lambda=0.071\ 073$ nm) at room temperature. Cell parameters were retrieved using SMART software. Data were collected using a narrow-frame method with scan widths of 0.30° in ω and an exposure time of 10 s/frame. The highly redundant data sets were reduced using SAINT^[17] and corrected for Lorentz and polarization effects. Absorption corrections were applied using SADABS^[18] supplied by Bruker. Structures were solved by direct methods using the program SHELXS-97^[19]. The positions of the metal atoms and their first coordination spheres were located from direct-method E maps; other non-hydrogen atoms were found using alternating difference Fourier syntheses and least-squared refinement cycles and, during the final cycles, were refined anisotropically. Hydrogen atoms were placed in calculated position and refined with a riding mode with a uniform value of U_{iso} . Information concerning crystallographic data collection and structure refinement is summarized in Table 1.

CCDC: 624698, **1**; 624699, **2**.

Table 1 Crystal and refinement data for complexes **1** and **2**

	1	2
Formula	C ₁₅ H ₁₈ FePrN ₆ O ₇ Cl ₂	C ₃₀ H ₃₆ Fe ₂ PrN ₁₂ O ₁₄ Cl
Formular weight	662.01	1 076.77
Crystal system	Orthorhombic	Triclinic
Space group	<i>Pnma</i>	<i>P</i> $\bar{1}$
<i>a</i> / nm	2.101 8(4)	0.870 0(3)
<i>b</i> / nm	1.505 1(3)	1.126 3(1)
<i>c</i> / nm	0.772 4(1)	2.224 2(1)
α / (°)		77.611(2)
β / (°)		80.489(2)
γ / (°)		82.805(2)
<i>Z</i>	4	2
<i>V</i> / nm ³	2.443 4(8)	2.090 4(1)
<i>D</i> _{calc} / (g·cm ⁻³)	1.800	1.711
<i>T</i> / K	293(2)	293(2)
λ / nm	0.071 073	0.071 073
μ / mm ⁻¹	2.828	1.972
<i>F</i> (000)	1 300	1 080
θ range / (°)	3.12~26.00	1.86~30.62
<i>hkl</i> range	-25 ≤ <i>h</i> ≤ 25, -18 ≤ <i>k</i> ≤ 18, -9 ≤ <i>l</i> ≤ 9	-12 ≤ <i>h</i> ≤ 12, -15 ≤ <i>k</i> ≤ 16, -31 ≤ <i>l</i> ≤ 31
Collected	17 684	47 619
Unique	2 478	12 575
Parameters	148	541
Goodness of fit	1.055	1.130
<i>R</i> ₁ (<i>I</i> > 2σ(<i>I</i>))	0.039 4	0.049 0
<i>wR</i> ₂ (<i>I</i> > 2σ(<i>I</i>))	0.083 3	0.122 1
Largest diff. peak and hole / (e·nm ⁻³)	650, -1 407	1 460, -2 354

2 Results and discussion

2.1 Synthesis and identification

The anionic precursor [(bpca)Fe(CN)₃]⁻ is chosen as the building block to prepare cyanide and bpca bridged 3d-4f heterobimetallic coordination polymers. The tetrabutylammonium salt of this precursor is soluble in common organic solvent such as acetonitrile, methanol and acetone. Only one C≡N stretching vibration (2 122 cm⁻¹) has been observed in the IR spectrum and the stretching frequencies of the C=O bonds are located at 1 715 cm⁻¹. The reaction of (Bu₄N)[(bpca)Fe(CN)₃] with PrCl₃·6H₂O yielded different crystalline products when different solvents and stoichiometries were used. The reaction in 1:1 molar ratios in methanol and water afforded yellow plate-like crystals of complex

1. With the same starting materials, complex **2** was obtained as yellow needlelike crystals in 1:2 (Pr/Fe) molar ratios in acetonitrile and water. In the IR spectra, the stretching frequencies of the C≡N bonds are located at 2 159 and 2 132 cm⁻¹ for **1**, and 2 161 and 2 126 cm⁻¹ for **2**, respectively, which are consistent with the presence of bridging and terminal cyanide ligands. The strong absorptions at 1 703 cm⁻¹ for **1** and 1 713 cm⁻¹ for **2** can be assigned to the C=O stretching vibration, which shift to lower frequencies compared to the starting material (1 715 cm⁻¹).

2.2 Crystal structures

Selected bond lengths and angles for complexes **1** and **2** are listed in Tables 2~3.

The crystallographic analyses reveal that complex **1** crystallizes in the orthorhombic space group *Pnma*

Table 2 Selected bond lengths (nm) and angles (°) for complex **1**

Fe(1)-N(1)	0.185 0(1)	Fe(1)-N(2)	0.190 7(5)	Fe(1)-C(9)	0.190 6(7)
Fe(1)-C(8)	0.193 8(7)	Fe(1)-C(7)	0.196 0(6)	Pr(1)-O(2)	0.252 4(5)
Pr(1)-O(3)	0.252 1(4)	Pr(1)-O(4)	0.260 9(3)	Pr(1)-O(1C)	0.254 9(3)
Pr(1)-N(5)	0.254 0(6)	C(8)-N(4)	0.115 4(9)	C(9)-N(5)	0.115 2(9)
C(7)-N(3)	0.112 6(9)				
N(1)-Fe(1)-N(2)	82.10(8)	N(1)-Fe(1)-C(9)	97.91(8)	C(9)-Fe(1)-N(2)	177.4(3)
C(9)-Fe(1)-C(8)	85.6(3)	C(9)-Fe(1)-C(7)	84.8(3)	C(8)-Fe(1)-C(7)	170.4(3)
N(5)-C(9)-Fe(1)	178.4(6)	N(3)-C(7)-Fe(1)	177.4(6)	N(4)-C(8)-Fe(1)	177.3(7)
O(3)-Pr(1)-O(1B)	127.03(12)	O(1C)-Pr(1)-O(1B)	66.67(14)	O(3)-Pr(1)-O(1C)	67.80(13)
C(9)-N(5)-Pr(1)	161.7(6)	C(6)-O(1)-Pr(1A)	134.0(3)		

Symmetry codes: (A) $x+1/2, y, -z+3/2$; (B) $x-1/2, y, -z+3/2$; (C) $x-1/2, -y+3/2, -z+3/2$.

Table 3 Selected bond (nm) and angles (°) for complex **2**

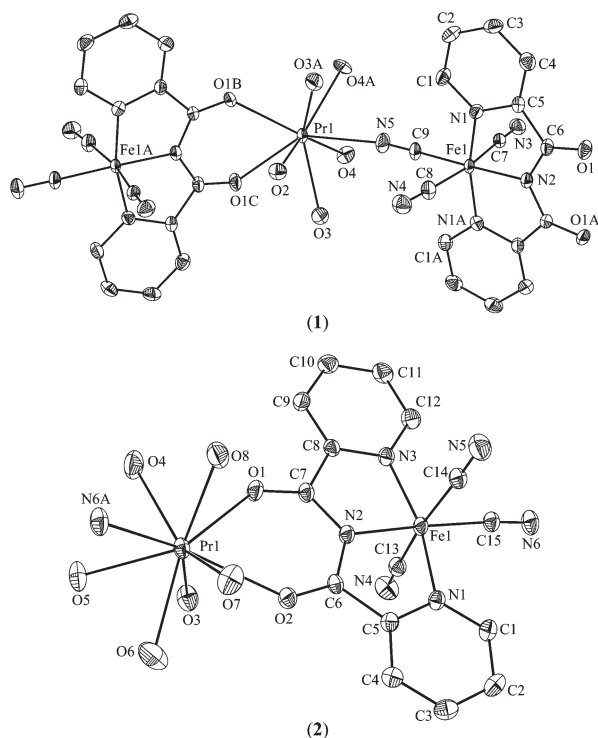
Fe(1)-C(13)	0.196 5(4)	Fe(1)-C(14)	0.195 1(4)	Fe(1)-C(15)	0.192 7(4)
Pr(1)-O(1)	0.256 1(3)	Pr(1)-O(2)	0.260 5(3)	Pr(1)-O(3)	0.247 8(3)
Pr(1)-O(4)	0.247 6(3)	Pr(1)-O(5)	0.258 4(3)	Pr(1)-O(6)	0.248 9(3)
Pr(1)-O(7)	0.2525(3)	Pr(1)-O(8)	0.246 4(3)	Pr(1)-N(6A)	0.254 1(3)
C(13)-N(4)	0.113 9(5)	C(14)-N(5)	0.114 2(5)	C(15)-N(6)	0.114 8(5)
C(14)-Fe(1)-C(13)	169.03(15)	C(15)-Fe(1)-C(13)	82.30(16)	C(15)-Fe(1)-C(14)	87.16(16)
N(4)-C(13)-Fe(1)	176.9(4)	N(5)-C(14)-Fe(1)	177.6(4)	N(6)-C(15)-Fe(1)	173.7(4)
O(1)-Pr(1)-O(2)	65.56(8)	C(15)-N(6)-Pr(1B)	158.6(3)	C(7)-O(1)-Pr(1)	138.5(2)
C(6)-O(2)-Pr(1)	137.1(2)				

Symmetry codes: (A) $x, y-1, z$; (B) $x, y+1, z$.

while complex **2** is in the triclinic space group $P\bar{1}$. The ORTEP drawings for **1** and **2** with the atom labeling schemes are represented in Fig.1. Both complexes possess novel 1D chain structures made up of alternating arrays of $[(\text{bpca})\text{Fe}(\text{CN})_3]^-$ and $[\text{Pr}(\text{H}_2\text{O})_x]^{3+}$ ($x=5$ for **1** and 6 for **2**) fragments along the a axis for **1** and b axis for **2** (Fig.2). In the chains of both complexes, each $[(\text{bpca})\text{Fe}(\text{CN})_3]^-$ unit uses two carbonyl groups and one of its cyanide ligands located in the bpca plane to connect two praseodymium(III) ions. The Fe(1)-C(cyano) bond lengths (0.190 6(7)~0.196 0(6) nm for **1** and 0.192 7(4)~0.196 5(4) nm for **2**) and the Fe-C \equiv N angles (177.3(7)°~178.4(6)° for **1** and 173.7(4)°~177.6(4)° for **2**) are in good agreement with those observed in other complexes containing $[(\text{bpca})\text{Fe}(\text{CN})_3]^-$ unit^[15]. Each Pr(III) ion is linked to two $[(\text{bpca})\text{Fe}(\text{CN})_3]^-$ units in a *trans* fashion. In **1**, the praseodymium(III) ion is eight-coordinated with a distorted square antiprism geometry, completed by five oxygen atoms from five

water molecules, two oxygen atoms from the bridging bpca ligand and one nitrogen atom from the bridging cyanide group. However, the Pr(III) ion is nine-coordinated with one more water oxygen atom in complex **2**. The Pr-O distances vary from 0.252 1(4) to 0.260 9 (3) nm for **1** and from 0.246 4 (3) to 0.260 5(3) nm for **2**. The Pr-N bonds length is 0.254 0(6) nm for **1** and 0.254 1(3) nm for **2**, respectively. All of the cyanide bridges in **2** are a little bentier than that in **1**, as reflected in the Fe-C \equiv N (178.4(6)° for **1** and 173.7(4)° for **2**) and Pr-N \equiv C (161.7(6)° for **1** and 158.6(3)° for **2**) angles.

In complex **2**, the Pr(III) ion is almost coplanar with the coordinated bpca ligand, but in complex **1**, the dihedral angle between the bpca ligand and the plane formed by Pr and carbonyl oxygen atoms is 16.38°. In **1**, the adjacent intrachain Fe \cdots Pr separations are 0.551 8 nm for Fe(1) \cdots Pr(1) and 0.580 6 nm for Fe(1) \cdots Pr(1A) (symmetric code: (A) $x+1/2, y, -z+3/2$), and the



Free $[(\text{bpca})\text{Fe}(\text{CN})_3]^-$ anions (in **2**), chloride and hydrogen atoms have been omitted for clarity

Fig.1 ORTEP views of complexes **1** (upper) and **2** (lower) with atom labeling schemes (thermal ellipsoids drawn at the 30% probability level)

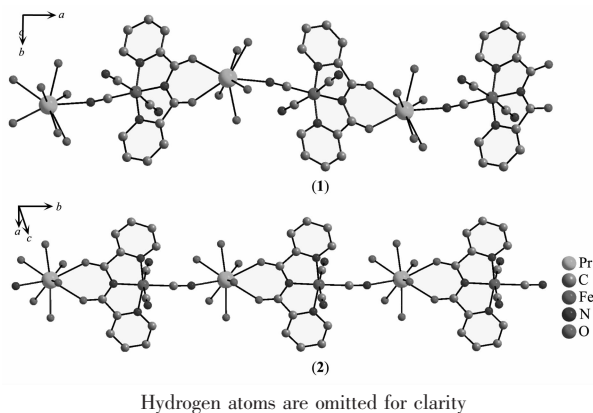


Fig.2 1D chain structures of complexes **1** (upper) and **2** (lower) closest intrachain Fe...Fe, Pr...Pr separations are 1.051 0 and 1.132 4 nm. The shortest interchain Fe...Pr, Fe...Fe, Pr...Pr distances are 0.765 4, 0.772 4 and 0.772 4 nm, respectively. In **2**, The adjacent intrachain Fe...Pr separations are 0.591 1 nm for Fe (1) ...Pr(1) and 0.547 6 nm for Fe(1)...Pr(1B) (symmetry codes: (B) $x, y+1, z$), and the closest intrachain Fe...Fe, Pr...Pr separations are 1.126 3 and 1.126 3 nm, respectively. The shortest interchain Fe...Pr, Fe...Fe, Pr...Pr

distances are 0.746 5, 0.740 9 and 0.870 0 nm, respectively.

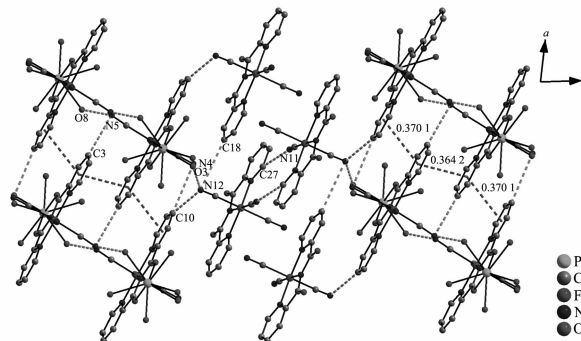
A 3D supramolecular structure is created by the coexistence of π - π stacking interactions, hydrogen bonds and shorter distance intermolecular interactions between neighboring $\{[(\text{bpca})\text{Fe}(\text{CN})_3\text{Pr}(\text{H}_2\text{O})_5]\}_n^{2n+}$ chains in **1** (Fig.3). There are hydrogen bonds occurring at O4-H4C...N4 (O4...N4=0.299 1 nm) along the c axis and a shorter C3-H3...N3 (C3...N3=0.339 6 nm) contact along the b axis. The π - π stacking interaction (0.377 3 nm) from pyridyl rings (N1, C1, C2, C3, C4, C5) provides additional stabilization of the crystal structure.



Chlorate and hydrogen atoms are omitted for clarity

Fig.3 Packing diagram of complex **1** with green dashed lines representing hydrogen bonds and π - π stacking interaction

In **2**, as shown in Fig.4, 1D chains are connected through the C10-H10...N4 short-distance intermolecular interactions (C10...N4=0.349 6 nm) and π - π stacking interactions (0.370 1 nm) through pyridyl rings to form the 2D sheets along the a direction. Two adjacent sheets are connected by π - π stacking



Water molecules, chlorate and hydrogen atoms are omitted for clarity

Fig.4 3D supramolecular structure of complex **2** formed by hydrogen bonds or short-distance intermolecular interactions (green dashed lines) and π - π stacking interactions (pink dashed lines)

interactions with the separation of 0.364 2 nm, H-bonds of O8–H8C \cdots N5 (O8 \cdots N5=0.277 4 nm) and short-distance intermolecular interactions of C3–H3 \cdots N5 (C3 \cdots N5=0.346 6 nm). Each two sheets is separated by the free [(bpca)Fe(CN) $_3$] $^-$ anions. The O3–H3B \cdots N12 and O3–H3C \cdots N12 (O3 \cdots N12=0.283 3 nm) hydrogen bonds and several shorter intermolecular contacts (C10 \cdots N12, 0.337 1 nm; C18 \cdots N12, 0.355 7 nm; C27 \cdots N11, 0.321 4 nm) further connect the free anions and sheets to lead to the 3D network.

In summary, with the use of [Fe(bpca)(CN) $_3$] $^-$ as the building block, two one-dimensional 3d-4f polymers have been synthesized and structurally characterized. They are among the rare examples of 3d-4f complexes involving the building blocks of modified cyanometalates.

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