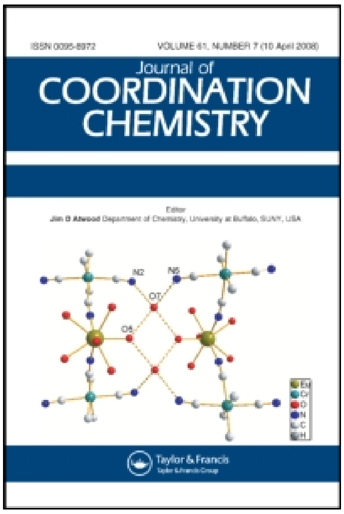

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Syntheses, crystal structures and fluorescent properties of four one-dimensional lanthanide coordination polymers with 3-cyanobenzoato

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Syntheses, crystal structures and fluorescent properties of four one-dimensional lanthanide coordination polymers with 3-cyanobenzoato

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The hydrothermal reactions of $Nd(ClO_4)_3 \cdot 6H_2O$, $Gd(ClO_4)_3 \cdot 6H_2O$, $Dy(ClO_4)_3 \cdot 6H_2O$, $Er(ClO_4)_3 \cdot 6H_2O$ with 1,3-dicyanobenzene, give rise to four one-dimensional rare earth-based coordination polymers: $[M(3\text{-}CNC_6H_4COO)_3(H_2O)_2]_n$ (where M = Nd (**1**), Gd (**2**), Dy (**3**), Er (**4**), $3\text{-}CNC_6H_4COO$ = 3-cyanobenzoato), respectively. Their solid-state structures have been characterized by X-ray single-crystal diffraction studies. The results show that 1,3-dicyanobenzene hydrolyzed to give 3-cyanobenzoato under hydrothermal condition, and the four complexes are isomorphous. Crystal data for **1**: triclinic, space group *P*-1, $a = 9.4063(19)$, $b = 11.485(2)$, $c = 12.616(3)$ Å, $\alpha = 66.38(3)$, $\beta = 74.01(3)$, $\gamma = 86.96(3)^\circ$, $V = 1197.9(4)$ Å^3, $Z = 1$, $D_c = 1.704\ \mathrm{Mg\,m^{-3}}$; for **2**: triclinic, space group *P*-1, $a = 9.3712(19)$, $b = 11.446(2)$, $c = 12.627(3)$ Å, $\alpha = 65.86(3)$, $\beta = 73.89(3)$, $\gamma = 86.84(3)^\circ$, $V = 1184.8(4)$ Å^3, $Z = 1$, $D_c = 1.759\ \mathrm{Mg\,m^{-3}}$; for **3**: triclinic, space group *P*-1, $a = 9.3425(19)$, $b = 11.432(2)$, $c = 12.703(3)$ Å, $\alpha = 65.28(3)$, $\beta = 73.80(3)$, $\gamma = 86.86(3)^\circ$, $V = 1180.6(4)$ Å^3, $Z = 1$, $D_c = 1.780\ \mathrm{Mg\,m^{-3}}$; for **4**: triclinic, space group *P*-1, $a = 9.3425(19)$, $b = 11.432(2)$, $c = 12.703(3)$ Å, $\alpha = 65.28(3)$, $\beta = 73.80(3)$, $\gamma = 86.86(3)^\circ$, $V = 1180.6(4)$ Å^3, $Z = 1$, $D_c = 1.7794\ \mathrm{Mg\,m^{-3}}$. The fluorescence emission spectra of compounds **1** to **4** are also reported.

Keywords: Lanthanide complexes; 3-Cyanobenzoato; Crystal structure; Fluorescence

1. Introduction

Lanthanide complexation chemistry has been studied intensively over the past two decades and much progress has been made with well-defined, kinetically robust systems tailored to applications as bioactive probes for magnetic resonance and luminescence [1–7]. Of the numerous lanthanide complexes with carboxylato ligands widely studied, most possess a variety of dimeric or infinite chain structures in which the carboxylate groups act as bridges between metal atoms, and the number of bridging

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carboxylato groups varies in different complexes [8]. Aromatic polycarboxylic acids such as 2,6-naphthalenedicarboxylic acid [9], 1,4-benzenedicarboxylaic acid [10] and 1,3-benzenedicarboxylic acid [11, 12] are used extensively in the synthesis of coordination polymers, whereas utilization of monobenzoato and derivatives to construct polymeric Ln(III) complexes are much less well known, although several examples have been reported recently [13].

In this paper, four one-dimensional lanthanide coordination polymers: $[M(3\text{-}CNC_6H_4COO)_3(H_2O)_2]_n$ (where M = Nd (**1**), Gd (**2**), Dy (**3**), Er (**4**), $3\text{-}CNC_6H_4COO$ = 3-cyanobenzoato), have been synthesized through hydrothermal reactions using 1,3-dicyanobenzene in the presence of lanthanide perchlorate hexadhydrate, and their crystal structures and photoluminescence properties have been investigated.

2. Experimental

2.1. *Materials and apparatus*

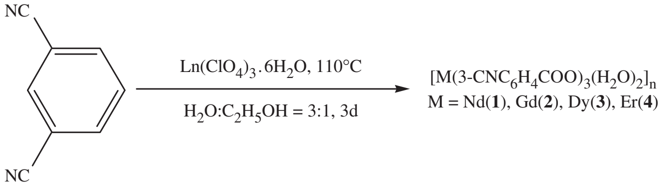
1,3-Dicyanobenzene was purchased from Acro Chemical Co. and all other chemicals were of reagent grade and used as purchased without further purification.

C, H and N data were obtained using a Perkin-Elmer 2400II CHNS/O elemental analyzer. IR spectra were obtained as KBr pellets on a Perkin-Elmer Spectrum One FT-IR spectrometer in the range of 4000–400 cm^{-1}. Photoluminescence spectra were measured on a fluorimeter (Edinburgh Instruments F900).

Caution! Perchlorate salts of metal complexes with organic ligands are potentially exposive and should be handled with care.

2.2. *Preparation of the complexes*

The compounds **1**, **2**, **3** and **4** were synthesized by similar procedures (scheme 1). In a typical reaction, a mixture of 1,3-dicyanobenzene (0.192 g, 1.5 mmol) and $Ln(ClO_4)_3 \cdot 6H_2O$ (Nd 0.275 g; Gd 0.282 g; Dy 0.284 g; Er 0.287 g; 0.5 mmol), H_2O (15 mL) and ethanol (5 mL) was placed in a Teflon-lined stainless steel vessel (25 mL) and the vessel was sealed and heated to 110°C. Colorless single crystals of $[M(3\text{-}CNC_6H_4COO)_3(H_2O)_2]_n$ were obtained after three days, yielding 61, 57, 53 and 50% for Nd (**1**), Gd (**2**), Dy (**3**), and Er (**4**), respectively. The products are stable in air and not soluble in water or most common organic solvents. Elemental analysis for compound **1**: Calcd C, 46.60; H, 2.61; N, 6.79. Found: C, 46.71; H, 2.68; N, 6.83%; compound **2**: Calcd C, 45.64; H, 2.55; N, 6.65. Found: C, 45.73; H, 2.61; N, 6.72%; compound **3**: Calcd C, 45.26; H, 2.53; N, 6.60. Found: C, 45.34; H, 2.58; N, 6.69%; compound **4**: Calcd C, 44.93; H, 2.51; N, 6.55. Found: C, 45.11; H, 2.63; N, 6.62%.



Scheme 1. Preparation of complexes **1** to **4**.

Table 1. Summary of crystallographic data for compounds **1**, **2**, **3** and **4**.

	1	**2**	**3**	**4**
Formula	$C_{48}H_{32}N_6Nd_2O_{16}$	$C_{48}H_{32}N_6Gd_2O_{16}$	$C_{48}H_{32}Dy_2N_6O_{16}$	$C_{48}H_{32}$ $Er_2N_6O_{16}$
Formula weight	1237.27	1263.29	1273.79	1283.31
Crystal system	Triclinic	Triclinic	Triclinic	Triclinic
Space group	*P*-1	*P*-1	*P*-1	*P*-1
a (Å)	9.4063(19)	9.3712(19)	9.3425(19)	9.3425(19)
b (Å)	11.485(2)	11.446(2)	11.432(2)	11.432(2)
c (Å)	12.616(3)	12.627(3)	12.703(3)	12.703(3)
α (°)	66.38(3)	65.86(3)	65.28(3)	65.28(3)
β (°)	74.01(3)	73.89(3)	73.80(3)	73.80(3)
γ (°)	86.96(3)	86.84(3)	86.86(3)	86.86(3)
V (Å^3)	1197.9(4)	1184.8(4)	1180.6(4)	1180.6(4)
Z	1	1	1	1
$F(000)$	602	610	614	618
ρ_{Calcd} (Mg m^{-3})	1.704	1.759	1.780	1.794
T (K)	293(2)	293(2)	293(2)	293(2)
$R_1[I>2\sigma(I)]$	0.0375	0.0336	0.0303	0.0265
$wR_2[I>2\sigma(I)]$	0.0870	0.0617	0.0702	0.0681

The IR spectrum of these compounds show characteristic peaks for COO^- and –CN groups. A strong peak at 1607 cm^{-1}, indicates that the carboxylic acid is deprotonated (cf 1700 cm^{-1} in free 3-cyanobenzoic acid), implying that 1,3-dicyanobenzene is hydrolysized. The band at 2800–3100 cm^{-1}(b, s) corresponding to ν(O–H) in the aqua ligand is shifted to higher frequency (3400–3500 cm^{-1}). The medium peak at 2200 cm^{-1}, related to ν(C–N) is not changed, and it is illustrated that one of the two cyano groups of 1,3-dicyanobenzene is neither hydrolysized nor coordinated [14].

2.3. *Single-crystal X-ray diffraction studies of complexes 1–4*

The X-ray single crystal data collections for complexes **1–4** were performed on a Rigaku R-axis RAPID IP diffractometer with graphite monochromated Mo-kα radiation ($\lambda = 0.71073$ Å) using the ω scan technique at 293(2) K. The structure was solved by direct methods and refined by full-matrix least-squares on F^2 using SHELXS-97 [15]. All non-hydrogen atoms were treated anisotropically. H atoms were generated geometrically and allowed to ride on parent atoms. SHELXL-97 refinement programs are used [15]. Details of the crystal structure solutions and refinements are listed in table 1. Selected bond lengths and angles for the complexes **1** to **4** are given in table 2.

3. Results and discussion

3.1. *Syntheses*

A hydrothermal reaction of an aqueous mixture of 1,3-dicyanobenzene and lanthanide perchlorate in a molar ratio of 3 : 1 afforded $[M(3\text{-}CNC_6H_4COO)_3(H_2O)_2]_n$ ($M = Nd$, Gd, Dy, Er), in which 1,3-dicyanobenzene hydrolyzed partially into 3-cyanobenzoato,

Table 2. Selected bond distances (Å) and angles (°) for compounds **1**, **2**, **3** and **4**.

Compound **1**			
Nd(1)–O(1)	2.564(3)	Nd(1)–O(2)	2.517(3)
Nd(1)–O(3)	2.445(3)	Nd(1)–O(4)$^{\#1}$	2.416(3)
Nd(1)–O(5)$^{\#2}$	2.432(3)	Nd(1)–O(6)	2.342(3)
Nd(1)–O(7)	2.488(3)	Nd(1)–O(8)	2.548(3)
O(1)–Nd(1)–O(2)	51.17(9)	O(1)–Nd(1)–O(3)	79.66(10)
O(2)–Nd(1)–O(3)	73.89(10)	O(2)–Nd(1)–O(7)	135.27(9)
O(3)–Nd(1)–O(7)	74.75(10)	O(6)–Nd(1)–O(7)	82.71(11)
O(6)–Nd(1)–O(8)	73.99(11)	O(2)–Nd(1)–O(4)$^{\#1}$	86.70(11)
O(4)$^{\#1}$–Nd(1)–O(5)$^{\#2}$	74.13(11)	O(5)$^{\#2}$–Nd(1)–O(8)	73.12(11)
Compound **2**			
Gd(1)–O(1)	2.512(3)	Gd(1)–O(2)	2.468(3)
Gd(1)–O(3)	2.346(3)	Gd(1)–O(4)$^{\#1}$	2.341(3)
Gd(1)–O(5)$^{\#2}$	2.376(3)	Gd(1)–O(6)	2.282(3)
Gd(1)–O(7)	2.442(3)	Gd(1)–O(8)	2.493(3)
O(1)–Gd(1)–O(2)	52.27(8)	O(1)-Gd(1)–O(3)	78.98(9)
O(2)–Gd(1)–O(3)	73.81(10)	O(2)–Gd(1)–O(7)	132.63(9)
O(3)–Gd(1)–O(7)	73.56(10)	O(6)–Gd(1)–O(7)	83.04(10)
O(6)–Gd(1)–O(8)	74.51(10)	O(2)–Gd(1)–O(4)$^{\#1}$	83.96(10)
O(4)$^{\#1}$–Gd(1)–O(5)$^{\#2}$	75.65(10)	O(5)$^{\#2}$–Gd(1)–O(8)	73.06(10)
Compound **3**			
Dy(1)–O(1)	2.476(3)	Dy(1)–O(2)	2.443(3)
Dy(1)–O(3)	2.316(3)	Dy(1)–O(4)$^{\#1}$	2.324(3)
Dy(1)–O(5)$^{\#2}$	2.345(3)	Dy(1)–O(6)	2.262(3)
Dy(1)–O(7)	2.406(3)	Dy(1)–O(8)	2.459(3)
O(1)–Dy(1)–O(2)	52.93(9)	O(1)–Dy(1)–O(3)	78.84(10)
O(2)–Dy(1)–O(3)	74.02(10)	O(2)–Dy(1)–O(7)	131.67(9)
O(3)–Dy(1)–O(7)	73.13(11)	O(6)–Dy(1)–O(7)	82.93(10)
O(6)–Dy(1)–O(8)	74.80(11)	O(2)–Dy(1)–O(4)$^{\#1}$	82.57(10)
O(4)$^{\#1}$–Dy(1)–O(5)$^{\#2}$	76.10(10)	O(5)$^{\#2}$–Dy(1)–O(8)	72.86(11)
Compound **4**			
Er(1)–O(1)	2.465(3)	Er(1)–O(2)	2.425(3)
Er(1)–O(3)	2.286(3)	Er(1)–O(4)$^{\#1}$	2.291(3)
Er(1)–O(5)$^{\#2}$	2.322(3)	Er(1)–O(6)	2.235(3)
Er(1)–O(7)	2.389(3)	Er(1)–O(8)	2.443(3)
O(1)–Er(1)–O(2)	53.27(8)	O(1)–Er(1)–O(3)	78.71(9)
O(2)–Er(1)–O(3)	74.08(9)	O(2)–Er(1)–O(7)	131.14(9)
O(3)–Er(1)–O(7)	73.21(10)	O(6)–Er(1)–O(7)	83.28(10)
O(6)–Er(1)–O(8)	74.64(10)	O(2)–Er(1)–O(4)$^{\#1}$	81.82(10)
O(4)$^{\#1}$–Er(1)–O(5)$^{\#2}$	76.88(10)	O(5)$^{\#2}$–Er(1)–O(8)	72.23(10)

Symmetry transformations used to generate equivalent atoms for compounds **1**, **2**, **3** and **4**: #1 $-x, -y, -z+1$; #2 $-x+1, -y, -z+1$.

different from its triple helical calcium-based coordination polymer [16]. In the latter case, the 1,3-dicyanobenzene hydrolyzed into 1,3-benzenecarboxylate.

3.2. *Crystal structures of 1–4*

Single-crystal X-ray diffraction studies reveal that $[M(3\text{-}CNC_6H_4COO)_3(H_2O)_2]_n$ (where M = Nd (**1**), Gd (**2**), Dy (**3**), Er (**4**), $3\text{-}CNC_6H_4COO$ = 3-cyanobenzoate) are isomorphous, thus only the structural characteristics of **1** are discussed in detail. The asymmetric unit of **1** contains an eight-coordinate Nd(III) (figure 1) which is surrounded by an O_8 environment composed of two O atoms from one bidentate

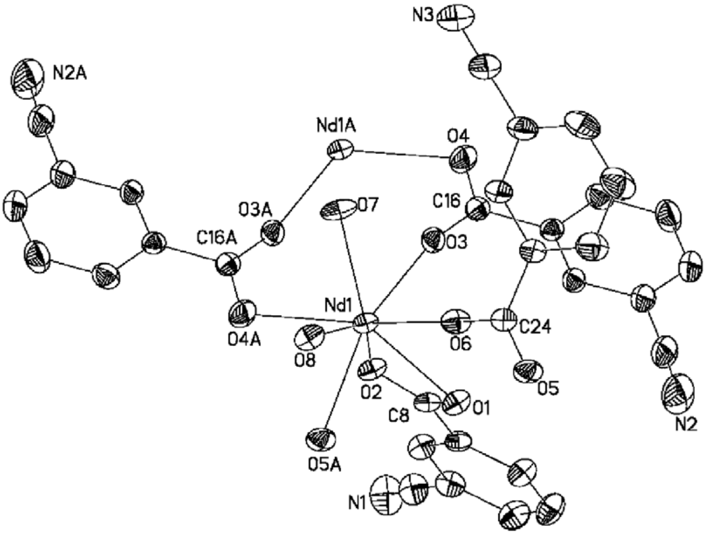


Figure 1. The coordination environment of the Nd(III) in **1** (30% probability ellipses). All hydrogen atoms are omitted for clarity.

chelating 3-cyanobenzoate and four O atoms from four bidentate bridging 3-cyanobenzoates and finally two O atoms from the water molecules, resulting in a distorted square antiprism. The two bidentate bridging 3-cyanobenzoates link two Nd centers in *syn-anti* mode to give rise to an eight-membered ring [Nd(1)O(3)C(16)O(4)Nd(1A)O(3A)C(16A)O(4A)]. The carboxylato groups are involved in two different coordination modes: bidentate chelating with a small bite angle [O(1)–Nd(1)–O(2) 51.17(9)°] and a common bidentate bridging mode in the *syn-anti* configuration [C(16)–O(3)–Nd(1), 166.7(3)° and C(16)–O(4)–Nd(1A), 111.3(3)°] [8]. The nearest Nd···Nd separation is 4.537 Å, indicating the lack of direct metal–metal interaction. The Nd–O bond distances range from 2.342 (3) to 2.564(3) Å. The average distance is 2.469(3) Å, similar to other related Nd–O distances [12]. Other bond distances and angles of 3-cyanobenzoate are comparable to those for $[Ca(OCOC_6H_4CN)_2(H_2O)_2]_n$ [14]. Unlike other copper(I) complexes with 1,3-dicyanobenzene [17], none of cyano groups coordinate to lanthanide ions in complexes **1** to **4**. There is a weak intramolecular hydrogen bond in compound **1**, e.g. C(23)–H(23A)···O(7) (3.422(6) Å. The most distinctive structural feature of complex **1** in the solid state is that it forms an infinite chain along the *a*-axis (figure 2); viewing down the *a*-axis, the cyanide sheath is around the coordination polymer (figure 3).

The M–M separations of four complexes follow the order: Nd–Nd (4.997 Å) > Gd–Gd(4.898 Å) > Dy–Dy (4.851 Å) > Er–Er (4.832 Å). The average M–O bond distances among the four complexes are in the same order; Nd–O (2.451 Å) > Gd–O(2.397 Å) > Dy–O (2.379 Å) > Er–O (2.350 Å), demonstrating that the lanthanide contraction displays an important role in crystal structures of **1** to **4** [18].

3.3. *Fluorescence emission spectra of 1 to 4*

Fluorescence analyses of compounds **1** to **4** (measured in solid state at room temperature) reveal well known lanthanide emissions. Upon excitation at 330 nm,

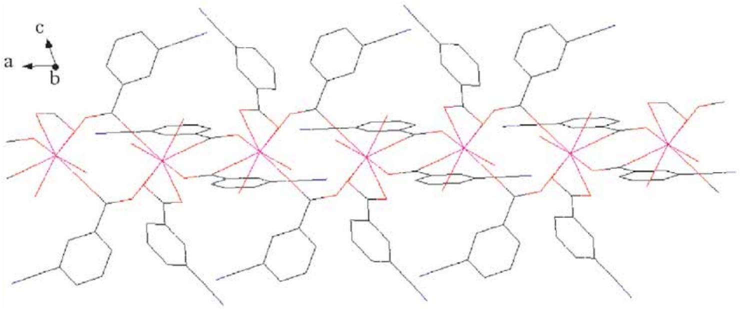


Figure 2. A one-dimensional chain perspective view showing the *syn-anti* bridging benzoate of the polymeric structure, viewed down the *b*-axis.

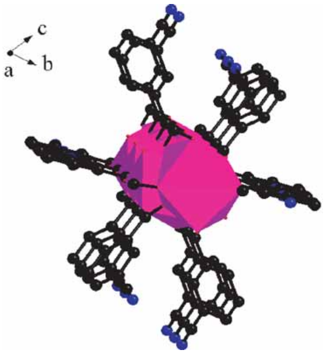


Figure 3. A one-dimensional chain representation viewed down the *a*-axis, highlighting the Nd square antiprism and the cyanide that is around the coordination polymer.

complex **1** displays emission at 676 nm corresponding to the $^4F_{9/2} \rightarrow {}^4I_{9/2}$ transition of Nd(III) [12, 19], whereas in compound **2**, a broad band centered at 555 nm was attributed to the 3-cyanobenzoato ligand emission [20]. The emission spectrum of **3** has a broad band around 673 nm corresponding to the $^4F_{9/2} \rightarrow {}^4H_{11/2}$ transition of Dy(III) [21, 22], and those of **4** involves two groups of narrow peaks, attributed to the $^4F_{7/2} \rightarrow {}^4I_{15/2}$ (510 nm) and $^4F_{9/2} \rightarrow {}^4I_{15/2}$ (624 nm) transitions of Er(III) [13, 18], respectively.

4. Conclusions

In summary, we have synthesized four one-dimensional lanthanide metal coordination polymers (**1**–**4**) via hydrothermal reactions. These compounds crystallize in a new structural type with the separations of M···M and the average M–O distances of four lanthanide complexes steadily decreasing from Nd to Er as a result of lanthanide contraction.

Supplementary material

Details of the crystal structures of **1** to **4** have been deposited with the Cambridge Crystallographic Data Center under CCDC No. 284587–284590. Copies of these data can be obtained, free of charge, on application to The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336-033; E-mail: deposit@ccdc.cam.ac.uk or www:http//www.ccdc.cam.ac.uk).

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