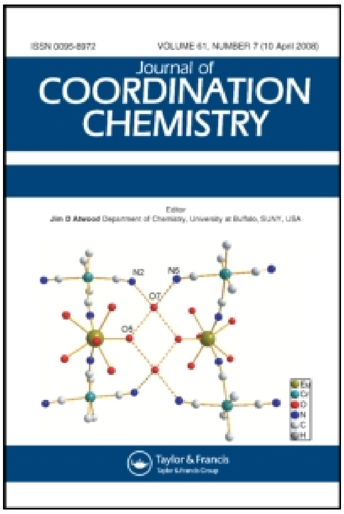

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Synthesis and crystal structure of two lanthanide coordination compounds of 4-(quinolin-8-yloxy)butanoic acid

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Synthesis and crystal structure of two lanthanide coordination compounds of 4-(quinolin-8-yloxy)butanoic acid

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Two lanthanide coordination compounds, $[La(QYBA)_3H_2O]_2 \cdot 2H_2O$ (**1**) and $[Eu(QYBA)_3(phen)]_2 \cdot 2H_2O$ (**2**), have been synthesized and characterized structurally by X-ray diffraction, where QYBA = 4-(quinolin-8-yloxy)butanoic acid and phen = 1,10-phenanthroline. Their solid-state structures have been characterized by elemental analysis and IR spectroscopy. Both form a dimeric molecular structure through bridging oxygen atoms of carboxylate groups and both are nine-coordinate. Adjacent dimers in **1** are interlinked by QYBA into an infinite chain structure, while the dimers in **2** are interlinked by hydrogen bonds and π–π stacking interactions into a two-dimensional network.

Keywords: Lanthanide coordination compounds; Dimeric complex; Hydrogen bonds; π–π stacking; 4-(Quinolin-8-yloxy)butanoic acid

1. Introduction

Lanthanide complexes have interesting photophysical properties with potential application as luminescence probes for chemical or biological macromolecules and the active center for luminescent materials [1–4]. Magnetic [5, 6] and luminescent properties [7, 8] of lanthanide complexes have aroused attention for decades, and studies have been focused on the design and assembly of lanthanide complexes with organic ligands such as aromatic carboxylic acids, β-diketone, cryptands, calixarenes, heterocyclic ligands, etc. Lanthanide complexes with aromatic carboxylic acids show higher thermal or luminescent stabilities for practical application than other lanthanide systems because they readily form dimer or infinite chain polymeric structures [9–12].

8-Quinolinyloxyacetic acid and its derivatives possess a broad spectrum of biological activities [13]. Compared to other carboxylic acids, the chemistry of 8-quinolinyloxyacetic acid has been investigated in a limited manner so far [14, 15]. We reported the synthesis and crystal structure of 4-(quinolin-8-yloxy)butanoic acid [16]. In this article, we describe the synthesis and structure of two lanthanide dimer complexes,

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Table 1. Crystal data and structure refinement for $[La(QYBA)_3H_2O]_2 \cdot 2H_2O$ (**1**) and $[Eu(QYBA)_3(phen)]_2 \cdot 2H_2O$ (**2**).

Complex	**1**	**2**
Empirical formula	$C_{39}H_{42}LaN_3O_{12}$	$C_{102}H_{96}Eu_2N_{10}O_{22}$
Formula weight	883.67	2117.81
Wavelength (Å)	0.71073	0.71073
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
a (Å)	7.931(2)	11.561(2)
b (Å)	16.232(4)	14.226(3)
c (Å)	16.739(4)	14.763(3)
α (°)	66.131(9)	83.23(3)
β (°)	87.677(10)	86.19(3)
γ (°)	80.073(10)	75.01(3)
Z	2	1
D_{Calcd} ($\mathrm{Mg\,m^{-3}}$)	1.513	1.511
$F(000)$	900	1080
μ ($\mathrm{mm^{-1}}$)	1.167	1.415
Crystal size ($\mathrm{mm^3}$)	$0.28 \times 0.22 \times 0.16$	$0.28 \times 0.26 \times 0.19$
θ range (°)	1.39–25.00	1.39–25.00
Index ranges	$-9 \le h \le 9$ $-19 \le k \le 18$ $-19 \le l \le 19$	$-13 \le h \le 13$ $-16 \le k \le 14$ $-17 \le l \le 17$
Reflections collected	22373	26777
Unique reflections	6811 ($R_{\mathrm{int}} = 0.0765$)	8164 ($R_{\mathrm{int}} = 0.0374$)
Absorption correction	Semi-empirical from equivalents	Semi-empirical from equivalents
Refinement method	Full-matrix least-equivalents on F^2	Full-matrix least-equivalents on F^2
Data/restraints/parameters	6811/6/496	8164/13/613
Goodness of fit on F^2	1.030	1.073
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0466$, $wR_2 = 0.0993$	$R_1 = 0.0321$, $wR_2 = 0.0834$
R indices (all data)	$R_1 = 0.0660$, $wR_2 = 0.1087$	$R_1 = 0.0397$, $wR_2 = 0.0925$

$[La(QYBA)_3H_2O]_2 \cdot 2H_2O$ (**1**) and $[Eu(QYBA)_3(phen)]_2 \cdot 2H_2O$ (**2**) (QYBA = 4-(quinolin-8-yloxy)butanoic acid, phen = 1,10-phenanthroline).

2. Experimental

2.1. *Synthesis of* $[La(QYBA)_3H_2O]_2 \cdot 2H_2O$

4-(Quinolin-8-yloxy)butanoic acid was synthesized according to the literature [16]. $La(NO_3)_3 \cdot 6H_2O$ and $Eu(NO_3)_3 \cdot 6H_2O$ were prepared from the corresponding lanthanide oxides with concentrated nitric acid. All other reagents were commercially available and used as received.

Stoichiometric amounts of $La(NO_3)_3 \cdot 6H_2O$ (87 mg, 0.2 mmol) and 4-(quinolin-8-yloxy)butanoic acid (139 mg, 0.6 mmol) were separately dissolved in CH_3OH. The pH of solution was adjusted to 6–7 with 0.2 M aqueous NaOH, then the solution of QYBA was added dropwise to the solution of $La(NO_3)_3$; the mixed solution was stirred for 6 h at room temperature. The solid which precipitated was filtered off, dissolved in dimethylformamide, filtered, and the filtrate allowed to stand at room temperature. After three weeks, well-shaped, colorless, single crystals suitable for analysis were obtained. Anal Calcd for $C_{39}H_{42}LaN_3O_{12}$: C, 53.01; H, 4.79; N, 4.75. Found: C, 52.98; H, 4.81; N, 4.77. The IR spectrum exhibits a complicated pattern of bands in the range 4000–400 cm^{-1}: 3419 cm^{-1} ($\nu_{sO–H}$), 1603 and 1558 cm^{-1} ($\nu_{sC=O}$).

2.2. *Synthesis of [Eu(QYBA)$_3$(phen)]$_2$•2H$_2$O*

$Eu(NO_3)_3 \cdot 6H_2O$ (90 mg, 0.2 mmol), QYBA (139 mg, 0.6 mmol) and phen (40 mg, 0.2 mmol) were mixed in 10 mL CH_3OH. The pH of solution was adjusted to 6–7 with 0.2 M aqueous NaOH, the mixed solution was stirred for 6 h at room temperature, filtered, and the filtrate allowed to stand at room temperature. After five days, well-shaped, colorless, single crystals suitable for analysis were obtained. Anal Calcd for $C_{102}H_{96}Eu_2N_{10}O_{22}$: C, 57.84; H, 4.57; N, 6.61. Found: C, 57.80; H, 4.58; N, 6.63. The IR spectrum exhibits a complicated pattern of bands in the range 4000–400 cm^{-1}: 3427 cm^{-1} ($\nu_{sO–H}$), 1602 and 1582 cm^{-1} ($\nu_{sC=O}$).

2.3. *X-ray crystallography*

Diffraction data for crystals with dimensions $0.28 \times 0.22 \times 0.16\,mm^3$ and $0.28 \times 0.26 \times 0.19\,mm^3$ were performed with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å) on a Bruker APEX-II area detector. A semiempirical absorption correction was applied to the data. The structure was solved by direct methods using SHELXS-97 and refined against F^2 by full matrix least-squares using SHELXL-97. Hydrogen atoms were placed in calculated positions. Crystal data and experimental details of the structure determinations are listed in table 1.

2.4. *Physical measurements*

Elemental analyses (C, H, N) were determined on an Elementar Cario EL elemental analyzer. Infrared spectroscopy on KBr pellets was performed on a Nexus 912 AO446 FT-IR spectrophotometer in the 4000–400 cm^{-1} range.

3. Results and discussion

3.1. *Description of the crystal structure for* [*La*(*QYBA*)$_3$*H*$_2$*O*]$_2$•2*H*$_2$*O*

$[La(QYBA)_3H_2O]_2 \cdot 2H_2O$ crystallizes in the triclinic system with space group $P\bar{1}$. Figure 1 shows the coordination geometry and atom labeling in the crystal structure. Structure analysis reveals a dimeric formulation $[La(QYBA)_3H_2O]_2 \cdot 2H_2O$ with two equivalent structural units corresponding to one half of the dimer related by a crystallographic inversion center. The two La ions are in the same coordination environment. The two La(III) atoms are bridged by four QYBA molecules with a La(III)–La(III) distance of 4.091 Å. Each La(III) is nine-coordinate, with eight oxygen atoms from six bridged QYBA groups (O1, O1A, O2, O4, O5B, O7, O9 and O9A) and one oxygen atom from water (O10), to form a distorted monocapped square

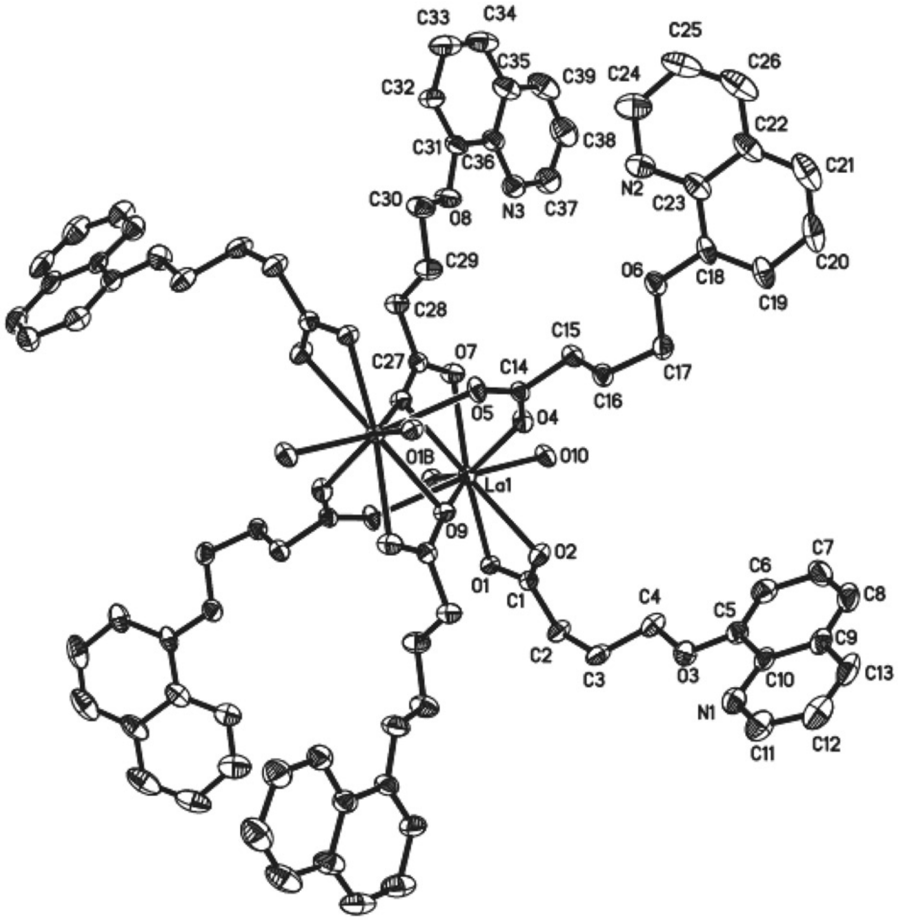
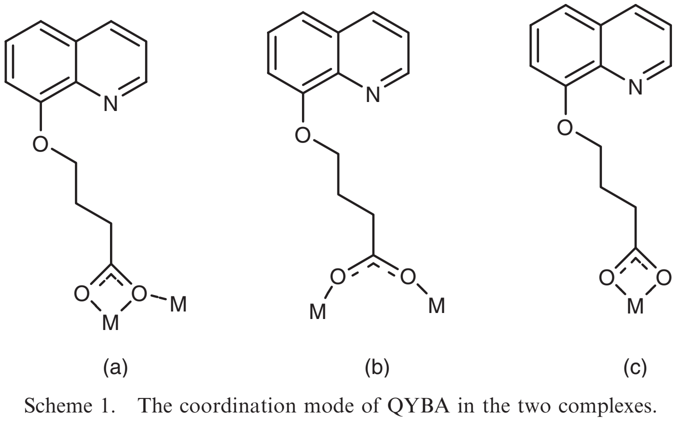


Figure 1. ORTEP drawing (50% thermal) of molecular structure of **1**.



(a) (b) (c)

Scheme 1. The coordination mode of QYBA in the two complexes.

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

La(1)–O(4)	2.453(3)	La(1)–O(7)	2.594(3)
La(1)–O(10)	2.489(3)	La(1)–O(1)	2.626(3)
La(1)–O(9)	2.491(3)	La(1)–O(9)#2	2.637(3)
La(1)–O(1)#1	2.526(3)	La(1)–O(2)	2.637(3)
La(1)–O(5)#2	2.552(3)		
O(4)–La(1)–O(10)	82.08(11)	O(1)#1–La(1)–O(1)	66.16(10)
O(4)–La(1)–O(9)	72.08(11)	O(5)#2–La(1)–O(1)	81.44(10)
O(10)–La(1)–O(9)	140.58(11)	O(7)–La(1)–O(1)	137.14(10)
O(4)–La(1)–O(1)#1	145.14(10)	O(4)–La(1)–O(9)#2	72.09(10)
O(10)–La(1)–O(1)#1	74.71(11)	O(10)–La(1)–O(9)#2	125.59(11)
O(9)–La(1)–O(1)#1	140.29(10)	O(9)–La(1)–O(9)#2	74.18(10)
O(4)–La(1)–O(5)#2	135.92(10)	O(1)#1–La(1)–O(9)#2	100.83(10)
O(10)–La(1)–O(5)#2	141.63(11)	O(5)#2–La(1)–O(9)#2	74.41(10)
O(9)–La(1)–O(5)#2	71.82(10)	O(7)–La(1)–O(9)#2	49.41(9)
O(1)#1–La(1)–O(5)#2	68.99(10)	O(1)–La(1)–O(9)#2	155.54(10)
O(4)–La(1)–O(7)	73.34(11)	O(4)–La(1)–O(2)	83.82(11)
O(10)–La(1)–O(7)	77.82(11)	O(10)–La(1)–O(2)	74.92(11)
O(9)–La(1)–O(7)	120.30(10)	O(9)–La(1)–O(2)	73.17(10)
O(1)#1–La(1)–O(7)	76.55(10)	O(1)#1–La(1)–O(2)	113.88(10)
O(5)#2–La(1)–O(7)	104.75(11)	O(5)#2–La(1)–O(2)	108.73(11)
O(4)–La(1)–O(1)	130.60(10)	O(7)–La(1)–O(2)	146.46(11)
O(10)–La(1)–O(1)	72.76(11)	O(1)–La(1)–O(2)	49.17(10)
O(9)–La(1)–O(1)	102.09(9)	O(9)#2–La(1)–O(2)	144.03(10)

Symmetry transformations used to generate equivalent atoms: #1: $-x+2$, $-y+1$, $-z+1$; #2: $-x+1$, $-y+1$, $-z+1$.

antiprismatic coordination geometry. One of the quadrangular faces is defined by four carboxylate oxygen atoms (O1A, O5B, O7, O9A) with a mean deviation of 0.0437 Å from the least-squares plane; the other one, which is capped by one oxygen atom (O2) from one carboxylate oxygen atom, is defined by three oxygen atoms from QYBA ligands and one coordinated water oxygen atom (O1, O4, O9, O10) with a mean deviation of 0.1136 Å from the least-squares plane. The dihedral angle between the two square faces is ca 4.0°.

There are two coordination modes of QYBA anions existing in $[La(QYBA)_3H_2O]_2 \cdot 2H_2O$, bidentate bridging (scheme 1b) and tridentate bridging (scheme 1a). Six tridentate bridging oxygens have La–O distances of 2.491(3) ~ 2.637(3) Å. Two bidentate bridging oxygens have La–O distances of 2.453(3) and 2.552(3) Å. In addition, the coordinated water molecule has a bond distance of 2.489(3) Å. The average La–O distance is 2.556(3) Å (table 2), comparable to those found in the lanthanum complex with *o*-aminobenzoate [17]. Through the tridentate bridging coordination, the dimeric molecules in this complex were interlinked by QYBA groups along the *a* direction into an infinite chain structure (figure 2), similar to lanthanum complexes with benzoate [18]. There are also two lattice water molecules per dimer, which connected with QYBA groups and coordinated waters through multiple strong hydrogen bonds (table 4).

3.2. *Description of the crystal structure for* $[Eu(QYBA)_3(phen)]_2 \cdot 2H_2O$

Figure 3 shows the molecular structure of **2**, comprising a center-related dinuclear $[Eu_2(CO_2)_6]$ unit. Four carboxylate groups link a pair of Eu(III) atoms in the O,O-bridging mode to generate a centrosymmetric dimer with Eu(III)–Eu(III) distances

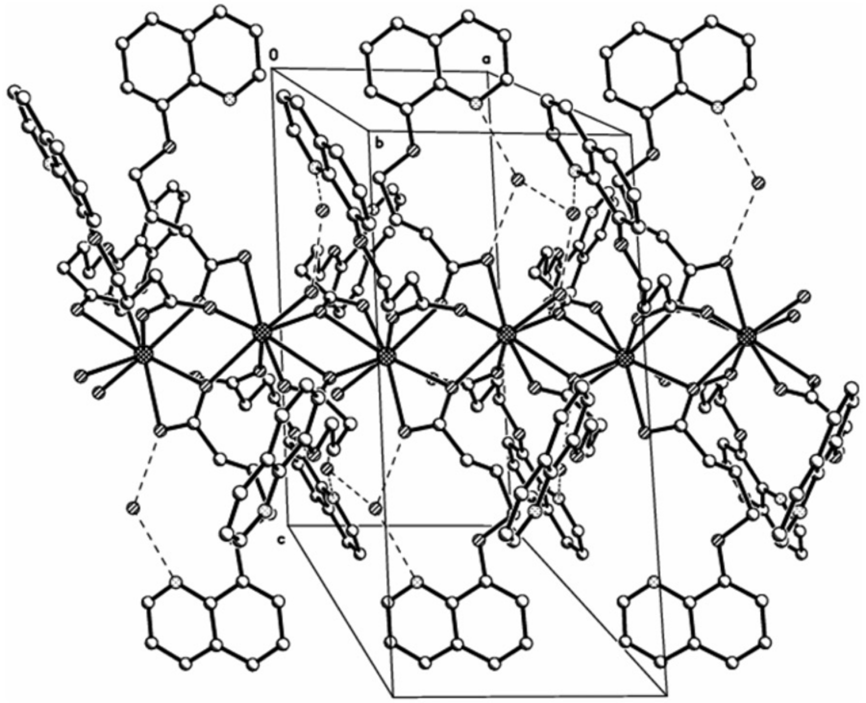


Figure 2. View of the 1D polymeric chain of **1**.

Table 3. Selected bond distances (Å) and angles (°) for **2**.

Eu(1)–O(2)#1	2.352(2)	Eu(1)–O(5)	2.541(2)
Eu(1)–O(7)	2.374(2)	Eu(1)–O(2)	2.568(2)
Eu(1)–O(9)	2.389(2)	Eu(1)–N(5)	2.590(3)
Eu(1)–O(4)	2.432(2)	Eu(1)–N(4)	2.635(3)
Eu(1)–O(1)	2.522(2)		
O(2)#1–Eu(1)–(7)	75.50(8)	O(4)–Eu(1)–O(2)	146.01(8)
O(2)#1–Eu(1)–O(9)	76.63(8)	O(1)–Eu(1)–O(2)	50.66(7)
O(7)–Eu(1)–O(9)	136.92(8)	O(5)–Eu(1)–O(2)	141.34(8)
O(2)#1–Eu(1)–O(4)	83.86(8)	O(2)#1–Eu(1)–N(5)	144.09(8)
O(7)–Eu(1)–O(4)	78.53(9)	O(7)–Eu(1)–N(5)	138.78(8)
O(9)–Eu(1)–O(4)	129.87(8)	O(9)–Eu(1)–N(5)	78.71(8)
O(2)#1–Eu(1)–O(1)	124.15(7)	O(4)–Eu(1)–N(5)	92.43(9)
O(7)–Eu(1)–O(1)	89.72(8)	O(1)–Eu(1)–N(5)	75.60(8)
O(9)–Eu(1)–O(1)	79.56(8)	O(5)–Eu(1)–N(5)	71.86(9)
O(4)–Eu(1)–O(1)	146.04(8)	O(2)–Eu(1)–N(5)	120.66(8)
O(2)#1–Eu(1)–O(5)	77.98(8)	O(2)#1–Eu(1)–N(4)	146.25(8)
O(7)–Eu(1)–O(5)	125.69(9)	O(7)–Eu(1)–N(4)	76.15(9)
O(9)–Eu(1)–O(5)	78.67(9)	O(9)–Eu(1)–N(4)	137.12(8)
O(4)–Eu(1)–O(5)	52.06(9)	O(4)–Eu(1)–N(4)	72.86(9)
O(1)–Eu(1)–O(5)	143.67(8)	O(1)–Eu(1)–N(4)	73.41(8)
O(2)#1–Eu(1)–O(2)	73.72(8)	O(5)–Eu(1)–N(4)	104.67(9)
O(7)–Eu(1)–O(2)	71.41(8)	O(2)–Eu(1)–N(4)	113.53(8)
O(9)–Eu(1)–O(2)	69.62(8)	N(5)–Eu(1)–N(4)	62.82(9)

Symmetry transformations used to generate equivalent atoms: #1: $-x$, $-y+2$, $-z+1$.

Table 4. The hydrogen bonds in **1**.

D–H···A	d(D–H)	d(H···A)	d(D···A)	∠(DHA)
O10–H40···O5#1	0.850	1.902	2.749	174.07
O10–H41···O11	0.850	1.813	2.649	167.45
O11–H42···O12#2	0.850	2.085	2.774	137.73
O11–H43···N2#1	0.851	2.064	2.864	156.39
O12–H45···O7#2	0.853	2.205	2.841	131.24

Symmetry transformations used to generate equivalent atoms: #1: $x+1$, y, z; #2: $-x+1$, $-y+1$, $-z+1$.

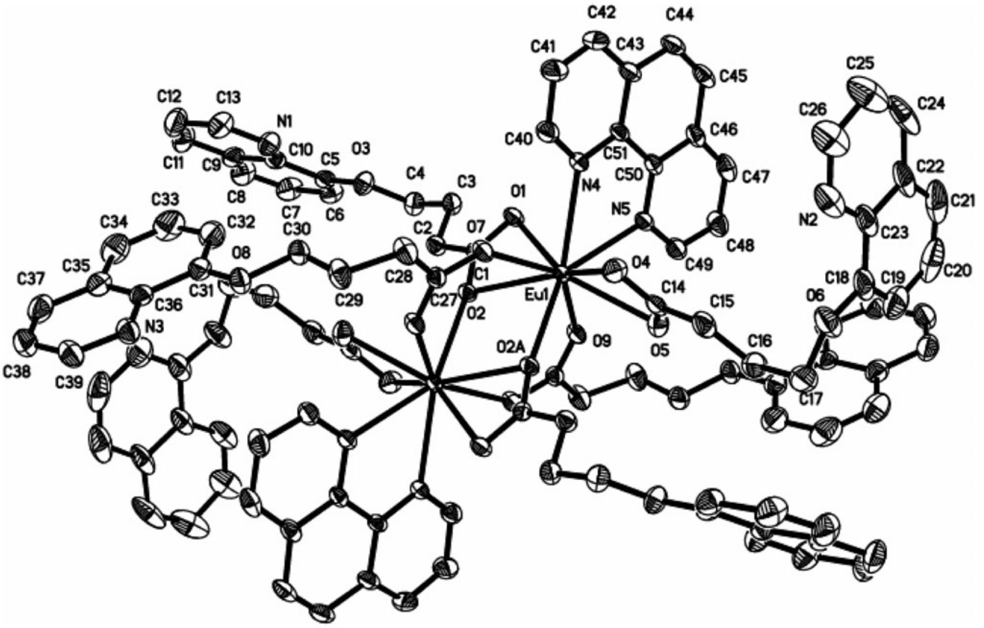


Figure 3. ORTEP drawing (50% thermal) of molecular structure of **2**.

of 3.939 Å. The Eu(III) atom is coordinated by five O atoms from four bridging QYBA ligands, two O atoms from a chelating QYBA and two *N* atoms from one chelating phen molecule. The nine coordination atoms form a distorted monocapped square antiprismatic geometry. One of the quadrangular faces is defined by two carboxylate oxygen atoms (O4, O5) and two nitrogen atoms (N4, N5) from phen with a mean deviation of 0.0750 Å from the least-squares plane; the other one, which is capped by one oxygen atom (O2) from one carboxylate oxygen atom, is defined by four oxygen atoms from QYBA ligands (O1, O2A, O7, O9) with a mean deviation of 0.1388 Å from the least-squares plane. The dihedral angle between the two square faces is Ca 3.7°. The bond distances of Eu–N (Eu–N4 = 2.635(3) Å, Eu–N5 = 2.590(3) Å) are comparable to those of a previously reported phen-containing Eu(III) complex [19]. The Eu–O bond distances fall in the range 2.352(2)–2.568(2) Å (table 3), and the average Eu–O distance is 2.454(2) Å, similar to those of Eu(III) complexes with phenoxyacetate [20]. The carboxylate groups are

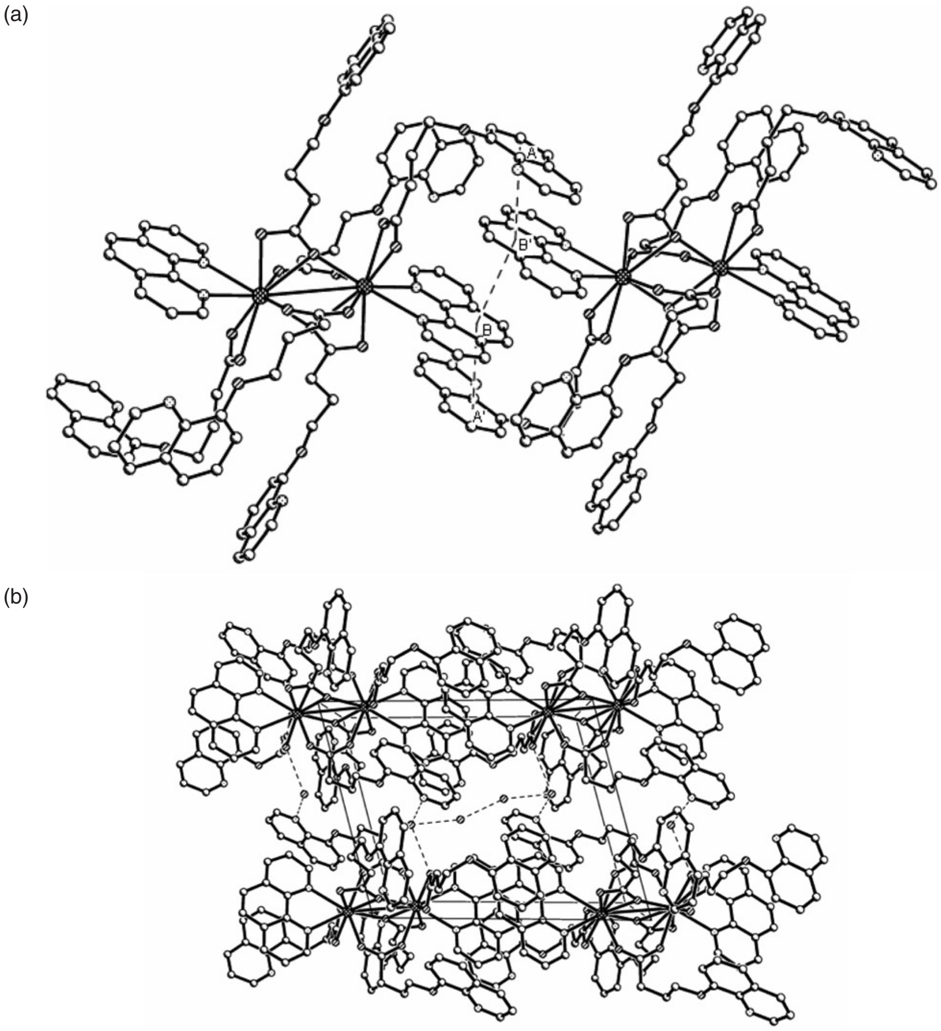


Figure 4. View of the packing structure of **2** formed by hydrogen bonds; (b) and π–π stacking (a) interactions.

Table 5. The hydrogen bonds in **2**.

D–H···A	d(D–H)	d(H···A)	d(D···A)	∠(DHA)
O10–H50···O11#1	0.851	2.110	2.847	144.70
O10–H50···O4	0.851	2.463	2.921	114.54
O10–H51···N3#2	0.850	2.233	3.042	159.02
O11–H53···O11#1	0.850	2.389	2.740	105.33

Symmetry transformations used to generate equivalent atoms: #1: $-x+1$, $-y+1$, $-z+1$; #2: $-x+1$, $-y$, $-z+1$.

bound to Eu(III) in three different coordination fashions: a chelating mode (scheme 1c), a bidentate bridging mode (scheme 1b) and a chelating-bridging mode (scheme 1a), which are similar to those of the Eu(III) complexes with phenoxyacetate [20] and benzoic acid [21], but different to those found in substituted benzoic acid [6, 22]. In these Eu(III) complexes, the coordination number of Eu(III) is eight. Supramolecular interactions also play an important role in the crystal packing and stabilization of **2** with hydrogen bonds and π–π stacking interactions. The face-to-face π–π stacking interactions between the QYBA rings (A and A′) and phen molecules (B′ and B) with a dihedral angle of 4.0 have a centroid-to-centroid distance of 3.634 Å. Face-to-face π–π stacking interactions between rings B and B′ from neighboring phen molecules (the centroid-to-centroid distance is 3.576 Å) generate a 1D chain network along the *b* direction (figure 4a). Adjacent chains are further connected to form a two-dimensional (2D) network parallel to the *ab* plane through hydrogen bonds resulting from lattice water and QYBA (table 5 and figure 4b).

In summary, the syntheses and crystal structures of two homodinuclear lanthanide complexes containing the 4-(quinolin-8-yloxy)butanoic acid ligand have been reported. The dimers in **1** are interlinked by tridentate bridging oxygens generating a zigzag chain along *a*, while the dimers in **2** are interlinked by hydrogen bonds and π–π stacking interactions into a two-dimensional network.

Supplementary material

Crystallographic data (excluding structure factors) for the structure reported in this article have been deposited with the Cambridge Crystallographic Data Centre as Supplementary Publication No. CCDC–648801 and 648802. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (Fax: (+44) 1223-336-033; Email: deposit@ccdc.cam.ac.uk).

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