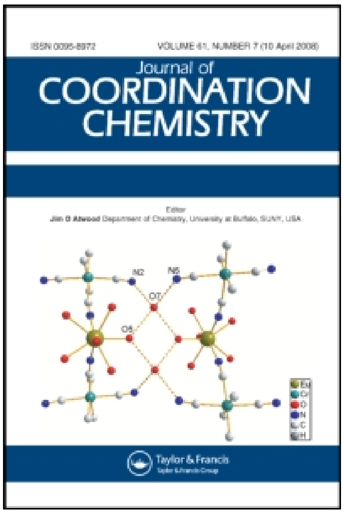

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Syntheses, crystal structures, and properties of 2-(1H-tetrazol-1-yl) acetic acid complexes

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Syntheses, crystal structures, and properties of 2-(1H-tetrazol-1-yl) acetic acid complexes

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Three new complexes have been prepared based on 2-(1H-tetrazol-1-yl) acetic acid (Htza) and the Ln(III) salts $La(NO)_3 \cdot 6H_2O$, $PrCl_3 \cdot 6H_2O$, and $NdCl_3 \cdot 6H_2O$. Their crystal structures have been characterized by single crystal X-ray crystallography. In each instance, the tetrazole ligand is both bridging and chelating-bridging connecting Ln(III) ions *via* the oxygens from carboxylate moieties. Thus infinite 1-D coordination polymers are formed. In addition, solid state UV spectra of the complexes are investigated.

Keywords: Ln(III) complexes; Crystal structure; UV properties

1. Introduction

Supramolecular chemistry has yielded many beautiful structures of different shapes and sizes through self-assembly of smaller complementary subunits [1]. Attention has been devoted to the design and synthesis of lanthanide complexes due to their potential applications as magnetic materials [2, 3], luminescent materials, catalysts, radiopharmaceuticals, and selective gas absorption reagents [4–7]. Lanthanides possess rich coordination requirements and highly desirable luminescent properties which can be probed to elucidate the nature of the supramolecular structure [8, 9]. Ligand design has been based mainly on structures that encapsulate the lanthanide [10–12], imposing bulkiness around the metal. Literature abounds with reports of heterocyclic acid ligands [13–26], but those formed from tetrazole-1-acetic acid with a lanthanide ion are rare. We chose tetrazole-1-acetic acid (Htza) as organic ligand based on versatile coordination modes of tetrazole-1-acetic acid giving lanthanide carboxylate chains or layers obtained through chelating and bridging with lanthanide ions [27]. Herein, we report syntheses and characterizations of three lanthanide coordination polymers

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$[La(tza)_3(CH_3OH)_2]_n$ (**1**), $[Pr(tza)_3(CH_3OH)_2]_n$ (**2**), and $[Nd(tza)_3(CH_3OH)_2]_n$ (**3**), in which supramolecular architectures are constructed by intermolecular hydrogen bonds.

2. Experimental

2.1. *General details*

All chemicals were of reagent grade obtained from commercial sources and used without purification. IR data were recorded on a Bruker Tensor 27 spectrophotometer with KBr pellets from 400–4000 cm^{-1}. UV-Vis absorption spectra in the solid state were taken using a Cary 5000 UV-Vis-NIR spectrophotometer.

2.2. *Syntheses of compounds 1–3*

Single crystals suitable for X-ray analyses were obtained by similar methods. All reactions were carried out in the existence of triethylamine with 1 : 2 molar ratio for Ln(III) : ligand species.

2.2.1. $[La(tza)_3(CH_3OH)_2]_n$ (1). $La(NO)_3 \cdot 6H_2O$ (16.8 mg, 0.05 mmol) and Htza (12.8 mg, 0.1 mmol) were dispersed in 5 mL of methanol. Triethylamine methanol solution (2 mL, 0.05 mol L^{-1}) was added. The solution was homogenized at room temperature for several minutes and the filtrate was allowed to stand at room temperature. Colorless block single crystals of **1** were obtained by slow evaporation of the solvent in 55% yield (based on the Htza). IR data (KBr, cm^{-1}): 3422(m), 3143(m), 1651(s), 1592(s), 1483(w), 1442(m), 1401(m), 1318(m), 1260(w), 1178(w), 1108(w), 801(w), 704(w), 580(w).

2.2.2. $[Pr(tza)_3(CH_3OH)_2]_n$ (2). Yield: 50%. IR data (KBr, cm^{-1}): 3436(m), 3156(m), 1659(s), 1590(s), 1484(w), 1449(m), 1399(m), 1322(m), 1174(w), 1108(w), 978(w), 805(w), 712(w), 581(w).

2.2.3. $[Nd(tza)_3(CH_3OH)_2]_n$ (3). Yield: 51%. IR data (KBr, cm^{-1}): 3357(m), 3141(m), 2964(w), 2855(w), 1656(s), 1590(s), 1485(w), 1447(s), 1402(s), 1320(m), 1262(w), 1178(m), 1106(m), 1021(w), 976(w), 881(w), 803(m), 711(m), 580(w).

2.3. *X-ray crystallography*

A summary of selected crystallographic data for **1**–**3** is given in table 1. The intensities were collected on a Bruker SMART-CCD diffractometer with graphite-monochromated Mo-Kα ($\lambda = 0.71073$ Å) using SMART and SAINT programs [28]. Forty-five frames of data were collected at 298 K with an oscillation range of 1° per frame and an exposure time of 10 s per frame. Indexing and unit cell refinement were based on all observed reflections from those 45 frames. The structure was solved by direct methods and refined on F^2 by full-matrix least-squares methods with *SHELXTL* version 5.1 [29].

Table 1. Crystallographic data and structure refinement parameters for **1**–**3**.

	1	**2**	**3**
Formula	$C_{11}H_{17}LaN_{12}O_8$	$C_{11}H_{17}PrN_{12}O_8$	$C_{11}H_{17}NdN_{12}O_8$
Moleculer weight	584.28	586.28	589.61
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	P_21/n	P_21/n	P_21/n
Unit cell dimeneions (Å, 0)			
a	10.422(2)	10.3572(15)	10.347(2)
b	8.9901(17)	8.9686(13)	8.9420(18)
c	22.596(4)	22.593(3)	22.583(5)
β	94.734(2)	94.588(2)	94.503(2)
V(Å^3)	2110.0(7)	2091.9(5)	2083.0(7)
Z	4	4	4
D_{Calcd} (g cm^{-3})	1.839	1.862	1.880
R_1 ($I > 2\sigma(I)$)	0.0255	0.0191	0.0314
wR_2	0.0577	0.0443	0.1115

$R_1 = \sum \|F_o| - |F_c\| / \sum |F_o|$; $wR_2 = [\sum w(F_o^2 - F_c^2)^2 / w(F_o^2)^2]^{1/2}$.

All non-hydrogen atoms were refined anisotropically. Hydrogens of the organic ligands were added geometrically and refined using the riding model. Selected bond lengths and bond angles are listed in table 2.

3. Results and discussion

Single-crystal X-ray diffraction reveals that compound **1** crystallizes in monoclinic space group P_21/n and exhibits a 1-D wave-like structure. The asymmetric unit consists of one La(III), three tza$^-$ ligands, and two coordinated methanols. The perspective view together with atomic numbering scheme is illustrated in figure 1. Each La(III) is nine-coordinate by seven oxygens belonging to six different carboxylate groups of six tza$^-$ ligands (O1, O2A, O3, O4, O5, O4B, and O6A) and two methanol molecules (O7 and O8) with a distorted mono-capped square-antiprismatic geometry. The La–O (methanol) distances are 2.624 and 2.697 Å and the La–O (carboxylate) bond lengths are in the range of 2.443–2.708 Å, comparable to those reported for lanthanum complexes [30]. All tza$^-$ ligands are deprotonated, and there are two types of the tza$^-$, bridged or chelated-bridged. One is a $\mu_{1,3}$ bridging ligand connecting two crystallographically equivalent La(III) ions, and the other uses two oxygens of the carboxylate to chelate a La1 and uses one oxygen to bridge two crystallographically equivalent La(III) ions. The bent configuration of carboxylate has thus formed 1-D polymeric chains running along the b-axis and containing both doubly and quadruply carboxylate-bridged units (figure S2). Through quadruple carboxylate-bridging, La1 and La1A form two closed eight-number rings (La1–O1–C3–O2–La1A–O1A–C3A–O2A and La1–O5–C9–O6–La1A–O5A–C9A–O6A), which are almost coplanar and perpendicular to each other. The La1–La1A distance is 4.58 Å. La1, La1B, and two bridging (O4, O4B) carboxylate oxygens form a rhombic plane with La1–La1B distance being 4.54 Å. The eight-number rings and the rhombic plane in **1** arrange alternately to afford a 1–D infinite chain lying along the b-axis. The tetrazole nitrogens of the ligands do not

Table 2. Selected bond Lengths (Å) and angles (°) for **1**–**3**.[a]

Complex **1**			
La(1)–O(2)#1	2.442(2)	La(1)–O(8)	2.623(2)
La(1)–O(4)	2.7072(19)	La(1)–O(6)#1	2.4825(19)
La(1)–O(3)	2.633(2)	La(1)–O(7)	2.699(2)
La(1)–O(1)	2.530(2)	La(1)–O(4)#2	2.5786(18)
La(1)–O(5)	2.4953(19)		
O(2)#1–La(1)–O(6)#1	71.43(7)	O(2)#1–La(1)–O(5)	74.84(7)
O(6)#1–La(1)–O(5)	123.04(6)	O(2)#1–La(1)–O(1)	117.88(7)
O(6)#1–La(1)–O(1)	78.08(7)	O(5)–La(1)–O(1)	79.02(7)
O(2)#1–La(1)–O(4)#2	144.03(7)	O(6)#1–La(1)–O(4)#2	142.58(6)
O(5)–La(1)–O(4)#2	74.10(6)	O(1)–La(1)–O(4)#2	73.01(6)
O(2)#1–La(1)–O(8)	138.08(7)	O(6)#1–La(1)–O(8)	70.47(7)
O(5)–La(1)–O(8)	142.80(7)	O(1)–La(1)–O(8)	69.94(7)
O(4)#2–La(1)–O(8)	77.54(6)	O(2)#1–La(1)–O(3)	83.35(7)
O(6)#1–La(1)–O(3)	77.90(7)	O(5)–La(1)–O(3)	140.84(7)
O(1)–La(1)–O(3)	140.12(7)	O(8)–La(1)–O(3)	72.22(7)
O(4)#2–La(1)–O(3)	110.52(6)	O(2)#1–La(1)–O(7)	73.44(7)
O(6)#1–La(1)–O(7)	137.24(7)	O(5)–La(1)–O(7)	68.27(7)
O(1)–La(1)–O(7)	141.35(7)	O(4)#2–La(1)–O(7)	78.54(6)
O(8)–La(1)–O(7)	128.38(6)	O(3)–La(1)–O(7)	74.56(6)
O(2)#1–La(1)–O(4)	122.51(7)	O(6)#1–La(1)–O(4)	116.64(6)
O(5)–La(1)–O(4)	120.09(6)	O(1)–La(1)–O(4)	119.43(6)
O(4)#2–La(1)–O(4)	61.58(7)	O(8)–La(1)–O(4)	63.14(6)
O(3)–La(1)–O(4)	48.95(6)	O(7)–La(1)–O(4)	65.24(6)
Complex **2**			
Pr(1)–O(2)#1	2.4015(14)	Pr(1)–O(6)#1	2.4455(14)
Pr(1)–O(5)	2.4562(14)	Pr(1)–O(1)	2.4857(14)
Pr(1)–O(4)#2	2.5381(13)	Pr(1)–O(8)	2.5704(15)
Pr(1)–O(3)	2.5898(14)	Pr(1)–O(7)	2.6630(14)
Pr(1)–O(4)	2.6928(13)		
O(2)#1–Pr(1)–O(6)#1	71.71(5)	O(2)#1–Pr(1)–O(5)	75.11(5)
O(6)#1–Pr(1)–O(5)	121.47(5)	O(2)#1–Pr(1)–O(1)	117.66(5)
O(6)#1–Pr(1)–O(1)	76.96(5)	O(5)–Pr(1)–O(1)	77.79(5)
O(2)#1–Pr(1)–O(4)#2	143.72(5)	O(6)#1–Pr(1)–O(4)#2	142.74(5)
O(5)–Pr(1)–O(4)#2	74.52(4)	O(1)–Pr(1)–O(4)#2	74.23(5)
O(2)#1–Pr(1)–O(8)	138.17(5)	O(6)#1–Pr(1)–O(8)	71.02(5)
O(5)–Pr(1)–O(8)	142.51(5)	O(1)–Pr(1)–O(8)	70.89(5)
O(4)#2–Pr(1)–O(8)	77.51(5)	O(2)#1–Pr(1)–O(3)	82.08(5)
O(6)#1–Pr(1)–O(3)	78.46(5)	O(5)–Pr(1)–O(3)	141.37(5)
O(1)–Pr(1)–O(3)	140.84(5)	O(4)#2–Pr(1)–O(3)	110.75(4)
O(8)–Pr(1)–O(3)	72.51(5)	O(2)#1–Pr(1)–O(7)	72.48(5)
O(6)#1–Pr(1)–O(7)	137.65(5)	O(5)–Pr(1)–O(7)	68.70(5)
O(1)–Pr(1)–O(7)	141.17(5)	O(4)#2–Pr(1)–O(7)	78.32(4)
O(8)–Pr(1)–O(7)	128.66(4)	O(3)–Pr(1)–O(7)	74.93(5)
O(2)#1–Pr(1)–O(4)	121.23(5)	O(6)#1–Pr(1)–O(4)	117.76(4)
O(5)–Pr(1)–O(4)	120.58(4)	O(1)–Pr(1)–O(4)	120.96(4)
O(4)#2–Pr(1)–O(4)	61.34(5)	O(8)–Pr(1)–O(4)	63.45(4)
O(3)–Pr(1)–O(4)	49.41(4)	O(7)–Pr(1)–O(4)	65.21(4)
Complex **3**			
Nd(1)–O(2)#1	2.385(3)	Nd(1)–O(6)#1	2.433(3)
Nd(1)–O(5)	2.443(3)	Nd(1)–O(1)	2.468(3)
Nd(1)–O(4)#2	2.522(2)	Nd(1)–O(8)	2.553(3)
Nd(1)–O(3)	2.569(3)	Nd(1)–O(7)	2.645(3)
Nd(1)–O(4)	2.682(3)		
O(2)#1–Nd(1)–O(6)#1	71.82(10)	O(2)#1–Nd(1)–O(5)	75.02(10)
O(6)#1–Nd(1)–O(5)	121.18(9)	O(2)#1–Nd(1)–O(1)	117.41(10)
O(6)#1–Nd(1)–O(1)	76.64(10)	O(5)–Nd(1)–O(1)	77.58(10)
O(2)#1–Nd(1)–O(4)#2	143.66(9)	O(6)#1–Nd(1)–O(4)#2	142.67(9)

(*continued*)

Table 2. Continued.

O(5)–Nd(1)–O(4)#2	74.64(8)	O(1)–Nd(1)–O(4)#2	74.49(9)
O(2)#1–Nd(1)–O(8)	138.07(10)	O(6)#1–Nd(1)–O(8)	70.92(9)
O(5)–Nd(1)–O(8)	142.65(10)	O(1)–Nd(1)–O(8)	71.18(10)
O(4)#2–Nd(1)–O(8)	77.66(9)	O(2)#1–Nd(1)–O(3)	81.81(10)
O(6)#1–Nd(1)–O(3)	78.32(9)	O(5)–Nd(1)–O(3)	141.49(9)
O(1)–Nd(1)–O(3)	140.90(10)	O(4)#2–Nd(1)–O(3)	111.09(8)
O(8)–Nd(1)–O(3)	72.49(10)	O(2)#1–Nd(1)–O(7)	72.32(9)
O(6)#1–Nd(1)–O(7)	137.80(9)	O(5)–Nd(1)–O(7)	68.65(9)
O(1)–Nd(1)–O(7)	141.08(9)	O(4)#2–Nd(1)–O(7)	78.32(8)
O(8)–Nd(1)–O(7)	128.85(8)	O(3)–Nd(1)–O(7)	75.25(9)
O(2)#1–Nd(1)–O(4)	121.07(9)	O(6)#1–Nd(1)–O(4)	117.95(8)
O(5)–Nd(1)–O(4)	120.68(8)	O(1)–Nd(1)–O(4)	121.38(8)
O(4)#2–Nd(1)–O(4)	61.37(9)	O(8)–Nd(1)–O(4)	63.60(8)
O(3)–Nd(1)–O(4)	49.73(7)	O(7)–Nd(1)–O(4)	65.25(8)

[a] Symmetry codes for **1**, **2**, and **3**: (#1) $-x, -y+1, -z+2$; (#2) $-x, -y+2, -z+2$.

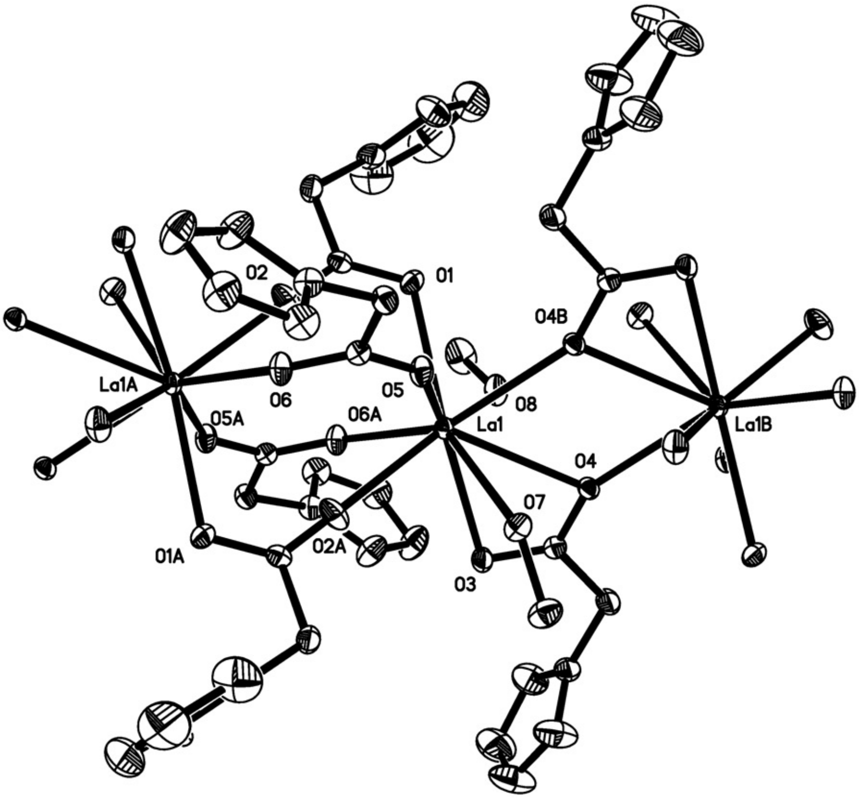

Figure 1. Coordination environment of La^{3+} at 30% probability displacement ellipsoids in **1**. H atoms are omitted.

coordinate and dangle along the 1–D chain. As would be expected, the remaining nitrogen N7 engages in hydrogen bonding with the methanol H10 of an adjacent chain; the N7C–O7 separation is 2.77 Å. The interaction results in generation of the 2–D network.

Complexes **2** and **3** are isostructural with **1**, with only very small differences among them. Because the radii of Pr(III) and Nd(III) are slightly smaller than La(III), most of the Ln(III)–O bonds in **2** and **3** are slightly shorter than the corresponding ones in **1** (table 2).

The solid state UV spectra of **2** and **3** (figure S3) show peak position and spectral shape of **2** and **3** at 307 nm accord with those of Htza, attributed to the intraligand π–π^* transition of tetraazole. The shift of peaks occurring in the complexes is probably due to different configuration of ligand and coordination environment around metal ions.

4. Conclusion

We prepared crystalline products from reaction of Htza with $La(NO)_3 \cdot 6H_2O$, $PrCl_3 \cdot 6H_2O$ and $NdCl_3 \cdot 6H_2O$ and characterized their crystal structures. In each instance, the tetrazole derived ligand serves as both bridging and chelating-bridging, connecting Ln(III) ions *via* coordination to the oxygen atoms from carboxylate moieties. Hydrogen bonds link the infinite 1–D chains into 2–D networks.

Supplementary material

CCDC 699186–699188 contains the supplementary crystallographic data for this article. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif

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