Supramolecular structures constructed from three novel rare earth metal complexes

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Abstract. Three rare earth metal supramolecular complexes, $\{[\text{Tb}(\text{L}_2)_4](\text{ClO}_4)_3 \cdot 2\text{H}_2\text{O}(\mathbf{1})$, $[\text{Eu}(\text{L}_2)_2(\text{H}_2\text{O})_5](\text{ClO}_4)_3(\mathbf{2})$ and $[\text{Gd}(\text{NO}_3)_3(\text{L}_2)_2] \cdot 2\text{CH}_3\text{CH}_2\text{OH}(\mathbf{3})$ ($\text{L}_2 = 3\text{-Dimethylamino-1-pyridin-2-yl-propenone}$), have been synthesized and characterized by elemental analysis, IR and single crystal X-ray diffraction. The crystal structure analysis reveals that the coordination numbers of three complexes ($\mathbf{1-3}$) are 8, 9 and 10, respectively. Three complexes assembled into 3D frameworks based on $\text{C-H} \cdots \text{O}$, $\text{O-H} \cdots \text{O}$ hydrogen bond linkages.

Keywords. Rare earth metal complex; crystal structure; hydrogen bonds; 3D supramolecular structure.

1. Introduction

The self-assembly of supramolecules via non-covalent bonds is currently an interesting topic of research. Their diverse structures and stability have attracted the attention of chemists and biologists.^{1–6} In the self-assembling hydrogen bonding structures, $\text{CH} \cdots \pi$ and $\pi \cdots \pi$ stacking play important roles in higher dimensional supramolecular framework construction. They are indispensable for architecture formation and can reinforce the structural stability with a wide range of potential applications in gas storage, sensor, catalysis, semiconductor, superconductor, optical switch and solar cell. Compared with other forces of interactions, the hydrogen bond has been considered as the most important interaction in supramolecular chemistry due to its relative strength, directionality and ability to provide a directing force in determining the molecular conformation and arrangement in solids.^{7–10} The design of a number of supramolecular frameworks with significantly different motifs, such as layers, ribbons, tubes, sheets and spheres, can be achieved through strong $\text{N-H} \cdots \text{O}$ and $\text{O-H} \cdots \text{O}$ hydrogen bonds.^{11,12} In addition, it is worthy to note that the weak $\text{C-H} \cdots \text{O}$ hydrogen bonds also play an important role in further stabilizing the 3-D network structures.

In our previous work, we have reported several complexes derived from two ligand entities and transition metal salts^{13–17} (scheme 1). Herein, we describe three supramolecules of coordination compounds $\{[\text{Tb}(\text{L}_2)_4](\text{ClO}_4)_3(\mathbf{1})$, $[\text{Eu}(\text{L}_2)_2(\text{H}_2\text{O})_5](\text{ClO}_4)_3(\mathbf{2})$, $[\text{Gd}(\text{NO}_3)_3(\text{L}_2)_2](\mathbf{3})\}$ from L_2 ($\text{L}_2 = 3\text{-Dimethylamino-1-pyridin-2-yl-propenone}$), constructed by strong $\text{O-H} \cdots \text{O}$ hydrogen bonds and weak $\text{C-H} \cdots \text{O}$ hydrogen bonds. These studies have advanced our understanding of hydrogen-bonding and interactions for the construction of structures with varied architecture.

2. Experimental

All reagents were of AR grade and used without purification. 3-(Dimethylamino) - 1-(pyridine-2-yl) prop-2-en-1-one was prepared by similar procedure reported in the literature.¹⁷ Elemental analysis was measured on a Perkin-Elmer 1400C analyzer. Infrared spectra ($4000\text{--}400\text{ cm}^{-1}$) were measured with a Bruker Vector 22 FT-IR spectrophotometer on KBr disks.

2.1 Synthesis

The same method reported in literature¹⁴ was used in the synthesis of $\mathbf{1-3}$ except the mole ratio between ligand L_2 and three rare earth metal salts $[\text{Tb}(\text{ClO}_4)_3]$, $[\text{Eu}(\text{ClO}_4)_3]$, $[\text{Gd}(\text{NO}_3)_3]$. A solution of the ligand (0.1 mmol) and rare earth metal salt (0.1 mmol) in 40 mL of

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Scheme 1. The molecular structures of two ligands.

ethanol was refluxed for 2 h, and then cooled to room temperature and filtered. Single crystals suitable for X-ray analysis were grown from the mother solution by slow evaporation at room temperature in air.

- 1: Anal. calcd for $C_{40}H_{52}Cl_3N_8O_{18}Tb$: C, 40.10; H, 4.37; N, 9.35%. Found: C, 40.02; H, 4.46; N, 9.19%. IR (KBr, cm^{-1}): 3469(m), 1657(m), 1606(m), 1518(m), 1467(m), 1386(s), 1221(w), 1086(s), 1024(w), 833(w).
- 2: Anal. calcd for $C_{20}H_{34}Cl_3EuN_4O_{19}$: C, 26.91; H, 3.84; N, 6.28%. Found: C, 26.88; H, 3.87; N, 6.26%. IR (KBr, cm^{-1}): 3461(m), 1664(m), 1613(m), 1526(m), 1467(m), 1380(s), 1232(w), 1102(s), 1026(w), 840(w).
- 3: Anal. calcd for $C_{24}H_{36}GdN_7O_{13}$: C, 36.59; H, 4.61; N, 12.45%. Found: C, 36.62; H, 4.71; N, 12.18%. IR (KBr, cm^{-1}): 3470(m), 1652(m), 1596(m), 1533(m), 1508(vs), 1467(m), 1393(s),

1294(s), 1228(w), 1021(w), 830(w), 819(w), 742(w).

2.2 X-ray crystallography

X-ray data for complexes **1–3** were collected on a Bruker SMART 1K CCD diffractometer at 293 K employing graphite-monochromatized $MoK\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$). The data were collected using SMART and reduced by the program SAINT.¹⁸ The structure was solved by the direct method and refined by full-matrix least squares fitting on F^2_{obs} by SHELXTL-PC.¹⁹ All non-hydrogen atoms were refined with anisotropic thermal parameters. The C–H hydrogen atoms were positioned geometrically and refined using a riding model with $d(CH_{aro}) = 0.95 \text{ \AA}$, $U_{iso} = 1.2U_{eq}(C)$ for aromatic and $0.98 \text{ \AA}$, $U_{iso} = 1.2U_{eq}(C)$ for CH_3 . The O–H hydrogen atoms were localized by difference Fourier maps and refined by fixing the bond lengths to $0.840(1) \text{ \AA}$. The crystallographic data for complexes **1–3** are listed in table 1.

3. Results and Discussion

3.1 Synthesis and characterization

All the complexes were prepared in 1:1 metal/ligand molar ratio. Infrared spectra of **1–3** display bands

Table 1. Crystallographic data and refinement parameters of complexes **1–3**.

complex	1	2	3
Empirical formula	$C_{40}H_{52}Cl_3N_8O_{18}Tb$	$C_{20}H_{34}Cl_3EuN_4O_{19}$	$C_{24}H_{36}GdN_7O_{13}$
Formula weight	1198.18	892.82	787.85
Crystal size (mm)	0.23 x 0.25 x 0.30	0.29 x 0.32 x 0.34	0.29 x 0.30 x 0.32
Crystal system	monoclinic	monoclinic	monoclinic
Space group	$P 2_1/c$	$C2/c$	$C2/c$
a (Å)	16.1966(10)	17.949(4)	21.322(2)
b (Å)	21.4944(13)	12.194(3)	10.9876(11)
c (Å)	22.4537(10)	17.427(4)	16.3844(16)
α [°]	95.00	90.00	90
β [°]	128.901(3)	116.301(4)	121.020(2)
γ [°]	90.00	90.00	90
V , Å ³	6083.4(6)	3419.4(14)	3289.6(6)
Z	4	4	4
D_{calcd} , (Mg/m ³)	1.308	1.734	1.591
μ , mm ⁻¹	1.359	2.150	2.086
$F(000)$	2432	1792	1588
Ref. collected/unique parameters	32323/11873 ($R_{int} = 0.065$)	8128/2957 ($R_{int} = 0.054$)	8669/3225 ($R_{int} = 0.054$)
$R1^a$	0.0728	0.0627	0.0549
$wR2^b$	0.1750	0.1657	0.1128
GOF	1.01	1.11	1.04
Min. / max. ρ (e·Å ⁻³)	−0.69/1.29	−2.06/1.71	−0.49/1.32

^a $R1 = \sum ||F_o| - |F_c|| / \sum |F_o|$, ^b $wR2 = [\sum (w(F_o^2 - F_c^2)^2) / \sum w(F_o^2)^2]^{1/2}$

Table 2. Selected bond lengths (Å) and angles (°) of three complexes^a.

1					
Tb1-O1	2.284(6)	Tb1-O2	2.313(8)	Tb1-O3	2.319(5)
Tb1-O4	2.292(7)	Tb1-N1	2.521(5)	Tb1-N3	2.539(8)
Tb1-N5	2.521(8)	Tb1-N7	2.538(9)	O1-Tb1-O2	130.7(2)
O1-Tb1-O3	78.1(2)	O1-Tb1-O4	124.3(3)	O1-Tb1-N1	64.8(2)
O1-Tb1-N3	80.9(3)	O1-Tb1-N5	142.8(2)	O1-Tb1-N7	81.4(3)
O2-Tb1-O3 O9	124.5(2)	O2-Tb1-N7	140.1(3)	O3-Tb1-O4	130.6(2)
O3-Tb1-N1	141.9(2)	O4-Tb1-N3	141.0(3)	N1-Tb1-N5	152.2(3)
N1-Tb1-N7	102.2(3)	N3-Tb1-N7	154.8(3)	N5-Tb1-N7	88.9(3)
N3-Tb1-N5	94.6(3)	N1-Tb1-N3	86.2(2)	O4-Tb1-N5	81.5(3)
2					
Eu1-O1	2.315(5)	Eu1-O2	2.448(8)	Eu1-O3	2.388(10)
Eu1-O4	2.541(12)	Eu1-N1	2.536(8)	Eu1-O1 ^I	2.315(5)
Eu1-O2 ^I	2.448(8)	Eu1-O3 ^I	2.388(10)	Eu1-N1 ^I	2.536(8)
O1-Eu1-O2	132.3(2)	O1-Eu1-O3	76.1(3)	O1-Eu1-O4	115.90(18)
O1-Eu1-O1 ^I	128.2(3)	O1-Eu1-O3 ^I	139.7(3)	O2-Eu1-O2 ^I	138.5(3)
O2-Eu1-N1 ^I	133.4(3)	O1 ^I -Eu1-O3	139.7(3)	O3-Eu1-N1 ^I	153.4(3)
O4-Eu1-N1	135.6(2)	O1 ^I -Eu1-O4	115.90(18)	O4-Eu1-N1 ^I	135.6(2)
O2 ^I -Eu1-N1	133.4(3)	O3 ^I -Eu1-N1	153.4(3)	O1 ^I -Eu1-O2 ^I	132.3(2)
3					
Gd1-O1	2.349(5)	Gd1-O2	2.548(6)	Gd1-O3	2.544(7)
Gd1-O5	2.499(5)	Gd1-N1	2.552(4)	Gd1-O1 ^{II}	2.349(5)
Gd1-O2 ^{II}	2.548(6)	Gd1-O3 ^{II}	2.544(7)	Gd1-O5 ^{II}	2.499(5)
Gd1-N1 ^{II}	2.552(4)	O1-Gd1-O2	74.35(15)	O1-Gd1-O3	69.57(15)
O1-Gd1-O5	137.50(19)	O1-Gd1-N1	64.35(17)	O1-Gd1-O1 ^{II}	71.29(16)
O1-Gd1-O2 ^{II}	145.61(15)	O1-Gd1-O3 ^{II}	116.65(15)	O1-Gd1-O5 ^{II}	137.03(15)
O1 ^{II} -Gd1-O2	145.61(15)	O2-Gd1-O2 ^{II}	140.03(15)	O2-Gd1-O3 ^{II}	127.10(16)
O2-Gd1-N1 ^{II}	117.90(19)	O3-Gd1-N1	113.71(18)	O1 ^{II} -Gd1-O3	116.65(15)
O2 ^{II} -Gd1-O3	127.10(16)	O3-Gd1-O3 ^{II}	172.99(14)	O5-Gd1-N1	128.15(16)
O1 ^{II} -Gd1-O5	137.03(15)	O2 ^{II} -Gd1-N1	117.90(19)	N1-Gd1-N1 ^{II}	149.74(17)
O1 ^{II} -Gd1-O5 ^{II}	137.5(2)	O3 ^{II} -Gd1-N1 ^{II}	113.71(18)	O5 ^{II} -Gd1-N1 ^{II}	128.15(16)

^a Symmetry transformations used to generate equivalent atoms: (I) 1-x,y,1/2-z, (II) 1-x,y,3/2-z.**Table 3.** Selected hydrogen bond lengths (Å) and angles (°) of complexes **1–3**.

D-H...A	D-H	H...A	D...A	∠DHA	A
1					
C12-H12...O12	0.930	2.570	3.49(3)	173.0	1+x,y,z
C20-H20A...O12	0.960	2.510	3.400(19)	155.00	
C10- H10C...O9	0.960	2.420	3.34(3)	162.00	
C35-H35...O16	0.930	2.550	3.38(2)	150.0	
C24- H24...O14	0.930	2.300	3.133(16)	149.00	
C19- H19C...O8	0.960	2.590	3.139(19)	116.00	-x,-2-y,1-z
C21- H21...O5	0.930	2.570	3.311(18)	137.00	
2					
O2- H2C...O5'	0.8500	2.090	2.79(3)	140.00	x,y,-1+z
O2-H2D...O10	0.8500	2.540	3.11(2)	125.00	
O2-H2D...O11	0.8500	2.050 63.00	2.879(17)	163.00	1/2-x,1/2-y,1-z
O3- H3C...O5'	0.8500	2.580	3.31(3)	144.0	1-x,y,3/2-z
O3- H3D...O9	0.8500	2.160	2.90(2)	145.0	
O3-H3D...O10	0.8500	2.580	3.38(3)	157.0	1-x,y,3/2-z
O3-H3D...O12	0.8500	1.930	2.75(3)	162.0	1/2+x,1/2-y,-1/2+z
O4- H4C...O8'	0.8500	1.990	2.83(5)	169.0	
C1- H1...O7'	0.9300	2.590	3.47(4)	158.0	1/2-x,1/2+y,1/2-z
C8- H8...O11	0.9300	2.500	3.292(19)	142.0	1/2+x,1/2-y,-1/2+z
C9- H9A...O12	0.9600	2.540	3.15(3)	121.0	1-x,y,3/2-z
C9- H9B...O8'	0.9600	2.570	3.42(4)	148.0	1/2+x,1/2-y,-1/2+z
3					
O7- H7...O3	0.8200	2.2400	3.046(14)	169.00	3/2-x,-1/2+y,3/2-z
C8- H8...O1	0.9300	2.4100	2.767(8)	103.00	
C12- H12C...O4	0.9600	2.5100	3.455(16)	170.00	

characteristic of the O-H stretching vibration in the 3470–3450 cm^{-1} region. The absorption bands in the 1400–1670 cm^{-1} region are in accordance with the skeletal vibrations of ligand for these complexes. The strong bands of ClO_4^- appear near 1100 cm^{-1} in the IR spectra of **1** and **2**. The bands of NO_3^- appear near 1508, 1294, 1021 cm^{-1} in the IR spectra of **3** (tables 2 and 3).

3.2 Structural descriptions of complexes **1**–**3**

The complex **1**, $[\text{Tb}(\text{L}_2)_4](\text{ClO}_4)_3 \cdot 2\text{H}_2\text{O}$, crystallizes in the monoclinic system, with space group $P2_1/c$. The least unit of the complex consists of one central metal

Tb(III), four ligands, three free perchlorate counter-anions and two lattice water molecules, as shown in figure 1a. The Tb centre is eight-coordinated by four oxygen atoms and four nitrogen atoms from four ligands with inverse square prism geometry. The bond distances between the terbium ion and oxygen atoms are from 2.284(6) Å to 2.319(5) Å, while the Tb–N bonds range from 2.521(5) Å to 2.539(8) Å.

For complex **1**, independent mononuclear units are linked together via heteromeric hydrogen bonds, C12–H12...O12, C20–H20A...O12 and C10–H10C...O9 to form a tiled chain. As shown in figure 1b, these hydrogen bonds are involved with oxygen atoms of one

Figure 1. (a) Left: Perspective view of **1** with the atom-numbering scheme; Right: Geometry around the Tb(III) atom of **1**. (b) One-dimensional chain structure in **1**. (c) Two-dimensional planar structure in **1**.

perchlorate anion and the hydrogen of organic ligands. On the one hand, the resulting chains are spread through hydrogen bonds, C35-H35...O16 and C24-H24...O14, parallel to *bc* plane to produce fluctuant 2D hydrogen-bonded layers (figure 1c), which are formed by the oxygen atoms of second perchlorate and the hydrogen of organic ligands. The third perchlorate anion is connected to mononuclear units by the hydrogen-bonding.

The complex **2** crystallizes in monoclinic group *C2/c* and displays a discrete mononuclear structure. The basic unit of the complex **2** consists of one central metal Eu(III), two ligands, three free perchlorate counter-anions and five coordinated water molecules. Figure 2a shows a perspective view of the molecular structure with atoms labelling.

As depicted in figure 2a, the Eu(III) centre in **2** is nine-coordinated by two N and seven O atoms from two ligands and five water molecules with a distorted mono-capped square antiprism. The bond distances between the Europium ion and oxygen vary from 2.315(7) Å to 2.448(8) Å, which are slightly longer than the Tb-O bond lengths, on the other hand the Eu-N bonds are 2.536(8) Å and similar to Tb-N bonds.

As shown in figure 2b, independent mononuclear units are linked together via heteromeric hydrogen

bonds, C1-H1...O7', and O4-H4C...O8' to form a zigzag chain along *c* axis. On the other hand, the independent units are linked together via classic hydrogen bonds between the coordination water molecules and perchlorate counter-anions, O2-H2D...O11, O3-H3D...O9, O3-H3D...O10 and O3-H3D...O12 to form a chain along *b* axis (figure 2c). The above two kinds of hydrogen bonds are interwoven with each other to produce fluctuant 2D hydrogen-bonded layers parallel to *bc* plane (figure 2d). Meanwhile the above planes are connected through hydrogen bonds, C8-H8...O11, C9-H9A...O12 and C9-H9B...O8', to form 3D hydrogen-bonding network.

Treatment of L_1 with $Gd(NO_3)_3$ generates complex **3**. Similarly with complex **2**, complex **3** crystallizes in the monoclinic space group *C2/c*, consisting of one Gadolinium(III) ion, two ligands, three coordinated nitrates and two free ethanol molecules (figure 3a). The coordination geometry around the Gd (III) ion is a distorted double-capped dodecahedron composed of two N atoms and two O atoms from two ligands and six O atoms from three nitrates. The average Gd-O bond lengths are 2.485 Å, which are slightly longer than the Tb-O bond lengths or Eu-O bond lengths.

Figure 2. (a) Left: Perspective view of **2** with the atom-numbering scheme; Right: Geometry around the Eu(III) atom of **2**. (b) Zig-zag chain structure in **2**. (c) One-dimensional chain in **2**. (d) Two-dimensional planar structure in **2**.

Figure 3. (a) Left: Perspective view of **3** with the atom-numbering scheme; Right: Geometry around the Gd(III) atom of **3**. (b) Two-dimensional planar structure in **3**.

It's interesting to note that the free ethanol molecules play an important role in the crystal structure, acting as hydrogen-bonding donors ($\text{C12-H12C} \cdots \text{O4}$ and $\text{O7-H7} \cdots \text{O3}$ (figure 3b)), to connect the surrounding monocular units and form an undulating two-dimensional plane.

4. Conclusion

Three rare earth metal supramolecular complexes, $\{[\text{Tb}(\text{L}_2)_4](\text{ClO}_4)_3 \cdot 2\text{H}_2\text{O}(\mathbf{1})$, $[\text{Eu}(\text{L}_2)_2(\text{H}_2\text{O})_5](\text{ClO}_4)_3$ (**2**) and $[\text{Gd}(\text{NO}_3)_3(\text{L}_2)_2] \cdot 2\text{CH}_3\text{CH}_2\text{OH}(\mathbf{3})$, have been synthesized and characterized. The crystal structure analysis reveals that the ligand acts as N⁴O chelation and the coordination numbers of three complexes (**1–3**) are 8, 9 and 10, respectively. The most common coordination numbers of rare earth complexes are 8 or 9. While some rare earth complexes with a coordination numbers of 11 or 12 often contain NO_3^- as ligand, because NO_3^- anion is small in size and low

charge (small repulsion), it can also provide two coordination numbers in the formation of complexes.^{20,21} Because of the lanthanide contraction, ionic radius of Eu (94.7pm), Gd (93.8pm), Tb (92.3pm) decreases; hence, coordination numbers of complexes **1** and **2** are 8 or 9, while the coordination number of complex **3**, containing NO_3^- with O⁴O chelation, is 10. In addition, the free perchlorates in complexes **1** and **2** take part in hydrogen-bonding to connect near units while the nitrates in complex **3** not only adopts the O⁴O chelation but also act as hydrogen bond acceptor. These complexes assembled into 3D hydrogen-bonding frameworks based on $\text{C-H} \cdots \text{O}$ and $\text{O-H} \cdots \text{O}$ hydrogen bond linkages.

Supplementary Information

Crystallographic data for the structural analysis have been deposited at the Cambridge Crystallographic Data Center with CCDC deposition numbers 1016118-

1016120 for the complexes. Copies of this information may be obtained free of charge via www.ccdc.cam.ac.uk/datarequest.cif.

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