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Supporting Information for

Coordination polymers of lanthanide complexes with benzene dicarboxylato ligands

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Synthesis of complexes 2−9, 11−13 and 14.

$Nd(L^1)_{1.5}(H_2O)_2\cdot H_2O$ (2): The procedure is similar to the synthesis of **1** except $Pr(NO_3)_3\cdot 6H_2O$ was replaced by $Nd(NO_3)_3\cdot 6H_2O$ (43.8 mg, 0.1 mmol). Light-purple block crystals of **2** were obtained in a yield of 65% based on Nd. Elemental Anal. Calcd for $C_{33}H_{24}NdO_{12}$ (756.76): C, 52.37; H, 3.19%. Found: C, 52.45; H, 3.13%. IR bands (KBr pellet, cm^{-1}): 3420 (m), 3058 (w), 1668 (s), 1640 (m), 1596 (m), 1479 (w), 1444 (w), 1398 (s), 1339 (m), 1255 (m), 1177 (w), 1020 (w), 950 (w), 923 (m), 891 (m), 803 (m), 770 (m), 692 (m), 620 (m), 523 (m), 489(m).

$Sm(L^1)_{1.5}(H_2O)_2\cdot H_2O$ (3): The procedure is similar to the synthesis of **1** except $Pr(NO_3)_3\cdot 6H_2O$ was replaced by $Sm(NO_3)_3\cdot 6H_2O$ (44.4 mg, 0.1 mmol). Light-yellow block crystals of **3** were obtained in a yield of 60% based on Sm. Elemental Anal. Calcd for $C_{33}H_{24}SmO_{12}$ (762.87): C, 51.95; H, 3.17%. Found: C, 51.87; H, 3.23%. IR bands (KBr pellet, cm^{-1}): 3444 (m), 3058 (w), 1670 (s), 1596 (s), 1481 (m), 1445 (m), 1400 (s), 1338 (m), 1255 (s), 1177 (m), 1147 (m), 1043 (w), 951 (w), 922 (m), 892 (w), 804 (m), 772 (w), 716 (m), 694 (m), 621 (w), 522(m), 490(m).

$Eu(L^1)_{1.5}(H_2O)_2\cdot H_2O$ (4): The procedure is similar to the synthesis of **1** except $Pr(NO_3)_3\cdot 6H_2O$ was replaced by $Eu(NO_3)_3\cdot 6H_2O$ (44.6 mg, 0.1 mmol). Colorless block crystals of **4** were obtained in a yield of 65% based on Eu. Elemental Anal. Calcd for $C_{33}H_{24}EuO_{12}$ (764.48): C, 51.84; H, 3.16%. Found: C, 51.77; H, 3.20%. IR bands (KBr pellet, cm^{-1}): 3445 (m), 3058 (w), 1659 (s), 1597 (s), 1482 (m), 1446 (m), 1402 (s), 1340 (m), 1255 (m), 1178 (w), 1147 (w), 1043 (w), 951 (w), 922 (m), 840 (w), 805 (m), 773 (w), 716 (w), 696 (m), 621 (w), 523(m), 492(m).

$Gd(L^1)_{1.5}(H_2O)_2\cdot H_2O$ (5): The procedure is similar to the synthesis of **1** except $Pr(NO_3)_3\cdot 6H_2O$ was replaced by $Gd(NO_3)_3\cdot 6H_2O$ (45.1 mg, 0.1 mmol). Colorless block crystals of **5** were obtained in a yield of 55% based on Gd. Elemental Anal. Calcd for $C_{33}H_{24}GdO_{12}$ (769.77): C, 51.49; H, 3.14%. Found: C, 51.55; H, 3.19%. IR bands (KBr pellet, cm^{-1}): 3445 (m), 3059 (w), 1660 (s), 1640 (m), 1598 (s), 1482 (m), 1447 (m), 1402 (s), 1341 (m), 1255 (m), 1178 (w), 1022

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(w), 950 (w), 923 (m), 891 (m), 805 (m), 772 (w), 694 (w), 628 (w), 523 (m), 492(m).

$Tb(L^1)_{1.5}(H_2O)_2 \cdot H_2O$ (6): The procedure is similar to the synthesis of **1** except $Pr(NO_3)_3 \cdot 6H_2O$ was replaced by $Tb(NO_3)_3 \cdot 6H_2O$ (45.3 mg, 0.1 mmol). Colorless block crystals of **6** were obtained in a yield of 45% based on Tb. Elemental Anal. Calcd for $C_{33}H_{24}TbO_{12}$ (771.46): C, 51.38; H, 3.14%. Found: C, 51.45; H, 3.20%.

$Dy(L^1)_{1.5}(H_2O)_2 \cdot H_2O$ (7): The procedure is similar to the synthesis of **1** except $Pr(NO_3)_3 \cdot 6H_2O$ was replaced by $Dy(NO_3)_3 \cdot 6H_2O$ (45.6 mg, 0.1 mmol). Colorless block crystals of **7** were obtained in a yield of 40% based on Dy. Elemental Anal. Calcd for $C_{33}H_{24}DyO_{12}$ (775.04): C, 51.14; H, 3.12%. Found: C, 51.25; H, 3.10%.

$Sm(L^2)(phen)(OH)$ (8): The procedure is similar to the synthesis of **10** except $Tb(NO_3)_3 \cdot 6H_2O$ was replaced by $Sm(NO_3)_3 \cdot 6H_2O$ (44.4 mg, 0.1 mmol). Light-yellow prismatic crystals of **8** were obtained in a yield of 43% based on Sm. Elemental Anal. Calcd for $C_{34}H_{21}SmN_2O_7$ (719.90): C, 56.73; H, 2.94; N, 3.89%. Found: C, 56.60; H, 2.90; N, 3.83%.

$Eu(L^2)(phen)(OH)$ (9): The procedure is similar to the synthesis of **10** except $Tb(NO_3)_3 \cdot 6H_2O$ was replaced by $Eu(NO_3)_3 \cdot 6H_2O$ (44.6 mg, 0.1 mmol). Colorless prismatic crystals of **9** were obtained in a yield of 48% based on Eu. Elemental Anal. Calcd for $C_{34}H_{21}EuN_2O_7$ (721.50): C, 56.60; H, 2.93; N, 3.88%. Found: C, 56.78; H, 2.90; N, 3.83%.

$Dy(L^2)(phen)(OH)$ (11): The procedure is similar to the synthesis of **10** except $Tb(NO_3)_3 \cdot 6H_2O$ was replaced by $Dy(NO_3)_3 \cdot 6H_2O$ (45.6 mg, 0.1 mmol). Colorless prismatic crystals of **11** were obtained in a yield of 58% based on Dy. Elemental Anal. Calcd for $C_{34}H_{21}DyN_2O_7$ (732.03): C, 55.78; H, 2.89; N, 3.83%. Found: C, 55.83; H, 2.91; N, 3.80%. IR bands (KBr pellet, cm^{-1}): 3446 (s), 3059 (w), 1666 (s), 1594 (s), 1481 (m), 1447 (m), 1401 (s), 1340 (m), 1312 (w), 1256 (s), 1178 (s), 1140 (w), 1102 (w), 1020 (w), 951 (w), 926 (w), 892 (w), 806 (w), 767 (w), 723 (w), 625 (w), 572 (w), 493(w).

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Er(L^2)(phen)(OH) (12): The procedure is similar to the synthesis of **10** except that $Tb(NO_3)_3·6H_2O$ was replaced by $Er(NO_3)_3·6H_2O$ (46.1 mg, 0.1 mmol). Pink prismatic crystals of **12** were obtained in a yield of 43% based on Er. Elemental Anal. Calcd for $C_{34}H_{21}ErN_2O_7$ (736.79): C, 55.42; H, 2.87; N, 3.80%. Found: C, 55.49; H, 2.83; N, 3.84%. IR bands (KBr pellet, cm^{-1}): 3430 (s), 3073 (w), 1669 (s), 1589 (s), 1555 (m), 1518 (w), 1484 (m), 1394 (s), 1313 (w), 1254 (m), 1177 (w), 1139 (w), 1101 (w), 1043 (w), 920 (w), 892 (w), 844 (m), 776 (m), 725(m), 609(w), 498(w), 431(w).

Yb(L^2)(phen)(OH) (13): The procedure is similar to the synthesis of **10** except $Tb(NO_3)_3·6H_2O$ was replaced by $Yb(NO_3)_3·5H_2O$ (44.9 mg, 0.1 mmol). Colorless prismatic crystals of **13** were obtained in a yield of 50% based on Yb. Elemental Anal. Calcd for $C_{34}H_{21}YbN_2O_7$ (742.57): C, 54.99; H, 2.85; N, 3.77%. Found: C, 55.03; H, 2.82; N, 3.81%. IR bands (KBr pellet, cm^{-1}): 3445 (s), 3060 (w), 1664 (s), 1596 (m), 1532 (w), 1481 (w), 1447 (m), 1405 (s), 1340 (m), 1257 (m), 1179 (w), 1073 (w), 1044 (w), 1021 (w), 1001(w), 927 (w), 894 (w), 837 (w), 807(m), 767 (m), 691(m), 524(w), 495(w).

Er(L^1)$_{0.5}$(L^2)(H_2O)$_2$ (14): The procedure is similar to the synthesis of **15** except $Ho(NO_3)_3·5H_2O$ was replaced by $Er(NO_3)_3·6H_2O$ (46.1 mg, 0.1 mmol). Pink block crystals of **14** were obtained in a yield of 62% based on Er. Elemental Anal. Calcd for $C_{33}H_{22}ErO_{11}$ (761.77): C, 52.03; H, 2.91%. Found: C, 52.08; H, 2.88%. IR bands (KBr pellet, cm^{-1}): 3435 (m), 3059 (w), 1664 (s), 1595 (s), 1481 (m), 1447 (m), 1404 (s), 1340 (m), 1257 (s), 1179 (w), 1154 (w), 1043 (w), 952 (w), 927 (w), 894 (w), 807 (m), 767 (w), 738 (w), 641 (w), 626 (w), 525(w), 493(w).

Due to bad quality of crystals, we can not collect the single-crystal XRD data of **7** (Dy(III), **8** (Sm(III)) and **9** (Eu(III)). The structure of compounds **7**−**9** was determined by powder XRD measurement. According to the powder XRD measurements, compounds **1**−**7** are isostructural and compounds **8**−**13** are isostructural.

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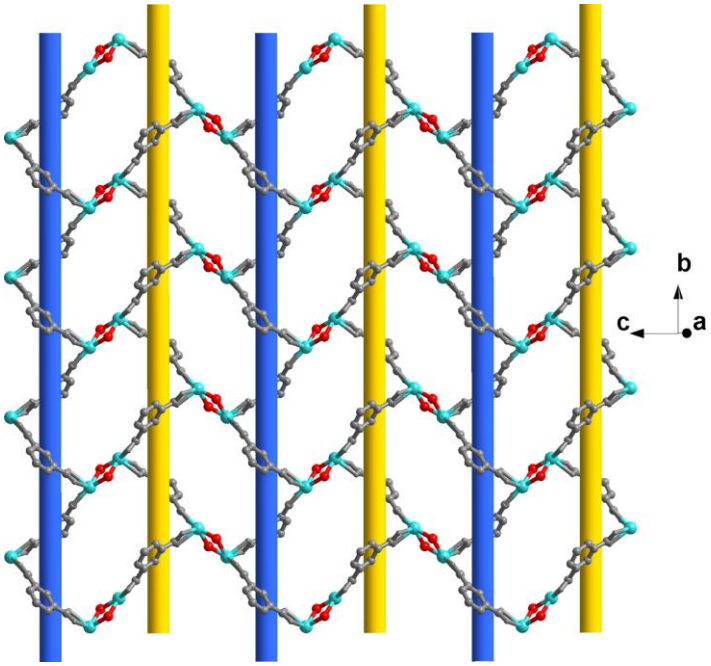

Figure S1 The alternately arranged right- and left-handed chains are bridged by μ_2–OH groups leading to an achiral 2D monolayer in **10**.

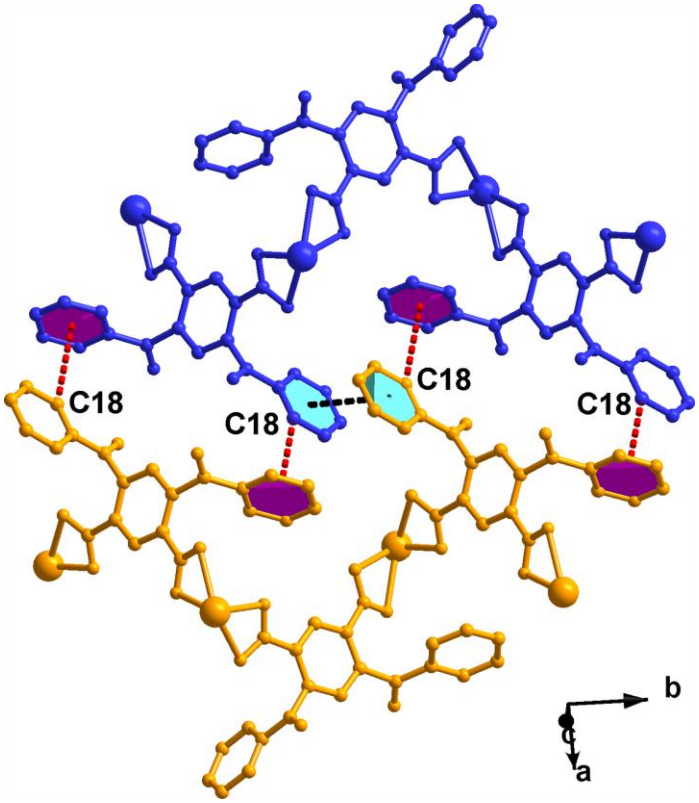

Figure S2 The red and black dotted lines represent C18–H18$\cdots\pi$ and $\pi\cdots\pi$ interactions in **10**, respectively. Different layers are high-lighted with different colors.

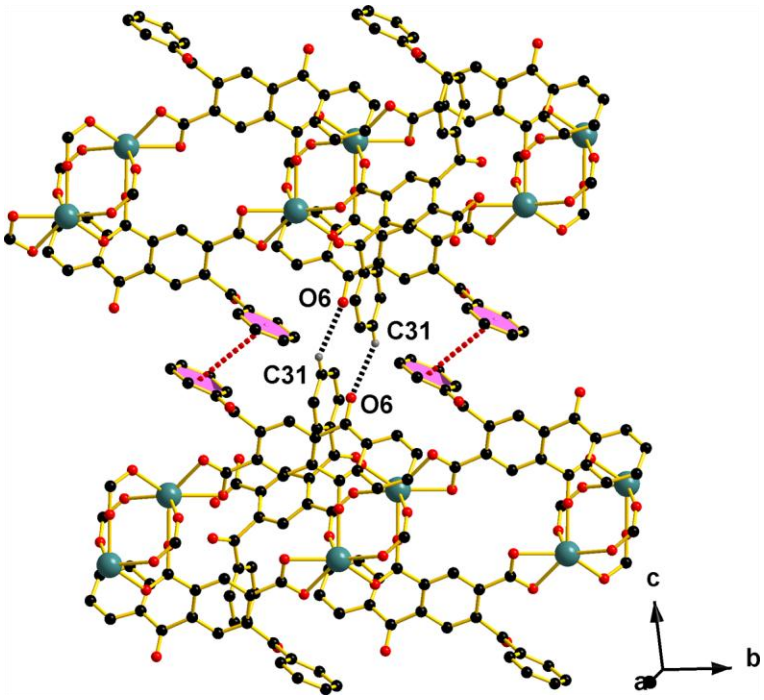

Figure S3 The black and red dotted lines represent C31–H31···O6 and $\pi\cdots\pi$ interactions in **15**, respectively.

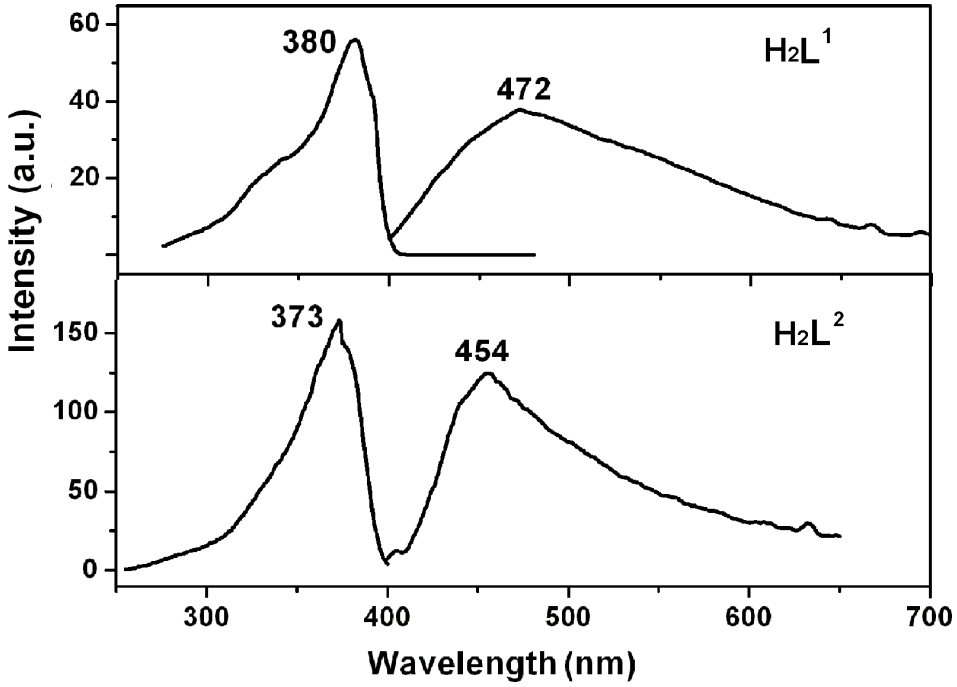

Figure S4 Solid-state excitation and emission spectra of free ligands H_2L^1 and H_2L^2 at room temperature.

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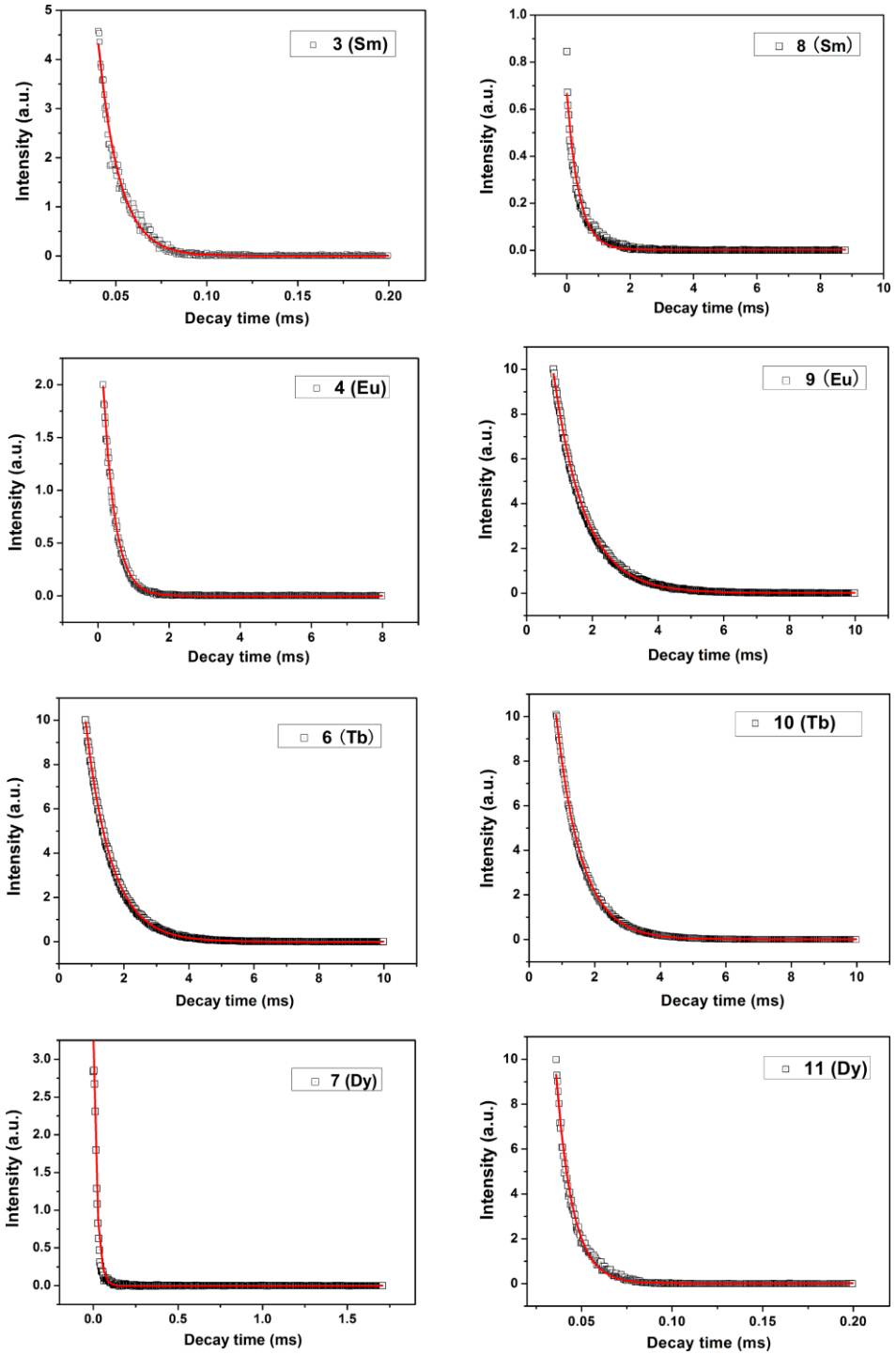

Figure S5 Typical fluorescence decay curves (black) observed for **3**–**4** and **6**–**11** in the solid state at room temperature, and red lines are the corresponding monoexponential fits.

Magnetic Property

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The variable-temperature magnetic susceptibility of compound **5** was measured with a Quantum Design MPMS-XL superconducting quantum interference device (SQUID) magnetometer in the temperature range of 2−300 K at a field of 1 kOe, and diamagnetic corrections were made using Pascal's constants. The result is depicted in Figure 6 as $\chi_M T$ versus T curve (χ_M being the molar paramagnetic susceptibility). At 300 K, $\chi_M T$ for **5** is 16.45 cm^3 K mol^{-1} close to the expected value of 15.75 cm^3 K mol^{-1} for two uncoupled free Gd(III) (S = 7/2, L = 0, $^8S_{7/2}$, g = 2.0, C = 7.875 cm^3 K mol^{-1}) ions. $\chi_M T$ remains almost constant at a value of ~16.11 cm^3 K mol^{-1} as the temperature decreases to ~11 K, where the value starts to steadily drop to a value of 15.58 cm^3 K mol^{-1} at 2.0 K. This behavior is indicative of weak antiferromagnetic exchange interaction between the Gd(III) ions in the molecule. A fit of the Curie−Weiss law $\chi = C / (T - \theta)$ (Figure S6) between 2−300 K for **5** in a 1000 Oe applied field yields C = 16.37 cm^3 K mol^{-1} and θ = −0.16 K, suggesting an overall antiferromagnetic interaction among the Gd(III) ions. An attempt was made to fit the magnetic data to an isotropic exchange expression for a binuclear S = 7/2 model. The equation (1) deduced from spin Hamiltonian, $H = -JS_{Gd1}S_{Gd2}$, is employed to simulation the magnetic susceptibility of the binuclear system.[1]

$$\chi_M T = \frac{2Ng^2\beta^2}{k} \frac{[e^{J/kT} + 5e^{3J/kT} + 14e^{6J/kT} + 30e^{10J/kT} + 55e^{15J/kT} + 91e^{21J/kT} + 140e^{28J/kT}]}{[1 + 3e^{J/kT} + 5e^{3J/kT} + 7e^{6J/kT} + 9e^{10J/kT} + 11e^{15J/kT} + 13e^{21J/kT} + 15e^{28J/kT}]} \quad (1)$$

In this expression, J denotes the exchange parameter between Gd(III) ions and N, g, β and k have their usual meanings. The best fitting of the susceptibility data in the temperature range 2–300 K gives J = −0.014 m^{-1}, g = 2.04. The small negative value of J further indicate that the presence of antiferromagnetic interactions among Gd(III) ions.

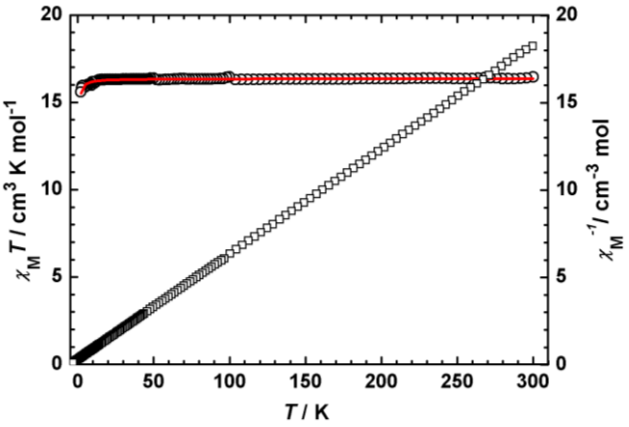

Figure S6 Temperature dependence of $\chi_M T$ (open circles) at 1000 Oe and χ_M^{-1} (squares) for **5**. Solid red line is the best fit obtained with the model as described in the text.

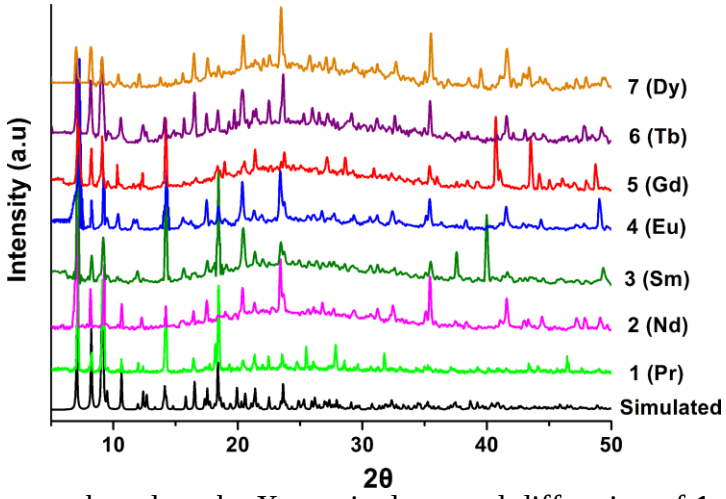

Figure S7 PXRD patterns based on the X-ray single crystal diffraction of **1** and the experimental samples of **1**−**7**, respectively.

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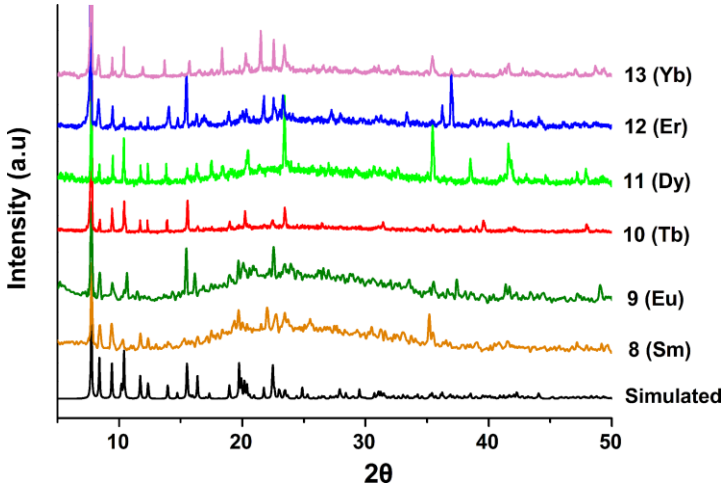

Figure S8 PXRD patterns based on the X-ray single crystal diffraction of **10** and the experimental samples of **8**−**13**, respectively.

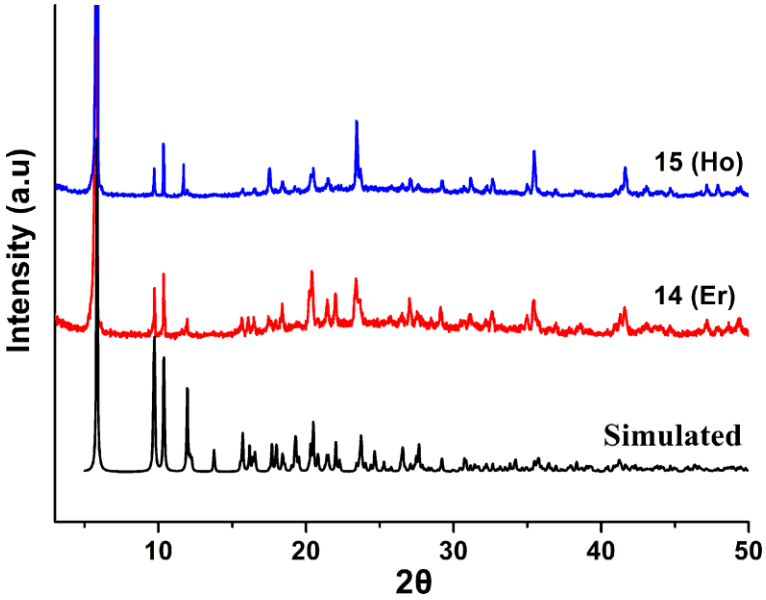

Figure S9 PXRD patterns based on the X-ray single crystal diffraction of **14** and the experimental samples of **14**−**15**, respectively.

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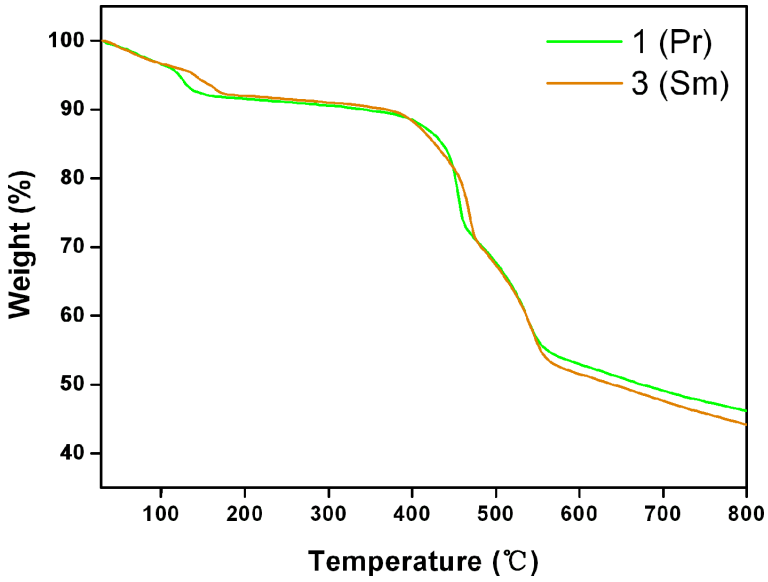

Figure S10 TGA curves of **1** and **3** under N_2 atmosphere (5 °C min^{-1}).

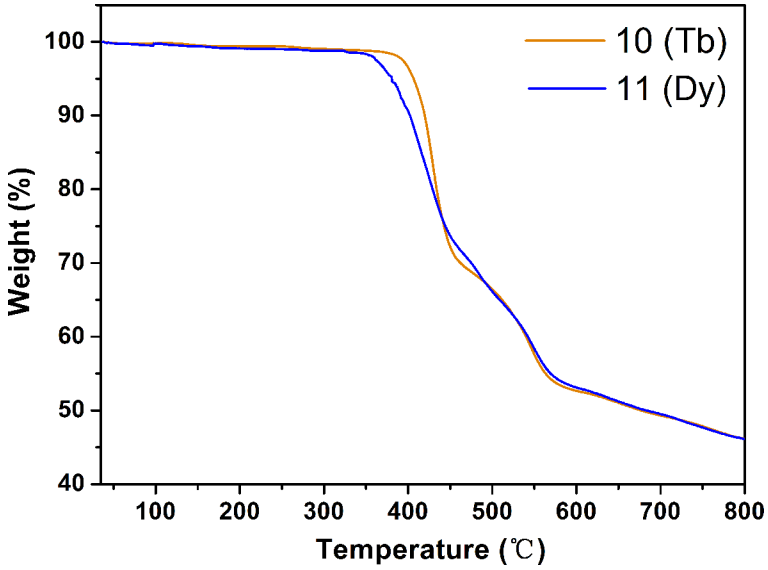

Figure S11 TGA curves of **10** and **11** under N_2 atmosphere (5 °C min^{-1}).

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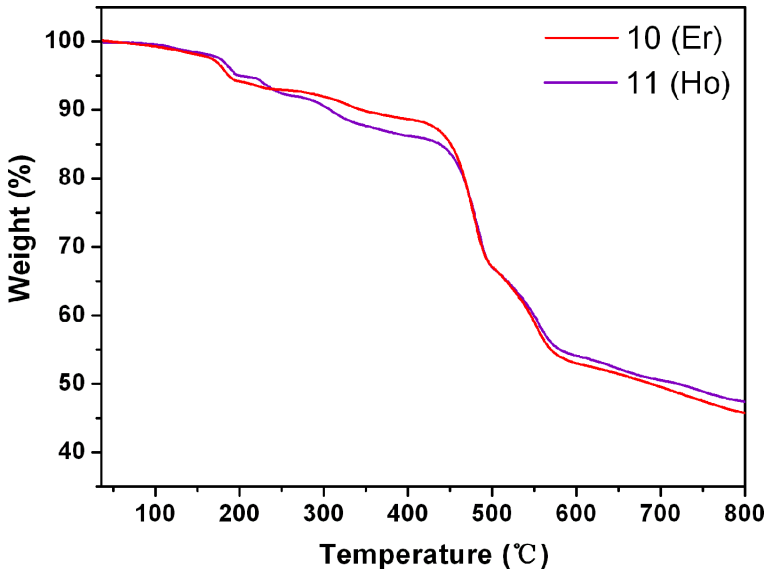

Figure S12 TGA curves of **14** and **15** under N_2 atmosphere (5 °C min^{-1}).

Table S1. Crystal data and structure refinement parameters for **2**−**5, 7**−**9** and **10**.

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Complexes	**2-Nd**	**3-Sm**	**4-Eu**	**5-Gd**	**6-Tb**
Formula	$C_{33}H_{24}NdO_{12}$	$C_{33}H_{24}SmO_{12}$	$C_{33}H_{24}EuO_{12}$	$C_{33}H_{24}GdO_{12}$	$C_{33}H_{24}O_{12}Tb$
Formula weight	756.76	762.87	764.48	769.77	771.44
Space group	*P*$\bar{1}$	*P*$\bar{1}$	*P*$\bar{1}$	*P*$\bar{1}$	*P*$\bar{1}$
a/Å	10.4475(11)	10.4130(11)	10.3979(6)	10.4002(6)	10.3248(19)
b/Å	11.5142(6)	11.4804(18)	11.4388(6)	11.4239(6)	11.3302(18)
c/Å	14.0367(16)	14.1232(19)	14.1007(8)	14.1035(8)	14.0870(3)
α/°	102.313(7)	102.540(6)	102.635(5)	102.780(1)	102.938(14)
β/°	110.938(10)	111.188(1)	111.321(5)	111.354(1)	111.170(17)
γ/°	100.587(6)	100.429(3)	100.381(5)	100.275(1)	100.561(14)
V/Å^3	1476.9(2)	1472.9(3)	1460.95(14)	1458.80(14)	1433.0(5)
Z	2	2	2	2	2
μ/mm^{-1}	1.826	2.062	2.216	2.342	2.538
F(000)	756	760	762	764	766
R_{int}	0.0516	0.0301	0.0349	0.0180	0.0358
GOF	1.033	1.082	1.030	1.041	0.990
$R_1{}^a$, w$R_2{}^b$ [$I>2\sigma(I)$]	0.0414, 0.0878	0.0329, 0.0873	0.0360, 0.0658	0.0280, 0.0669	0.0331, 0.0693
$R_1{}^a$, w$R_2{}^b$ (all data)	0.0511, 0.0931	0.0393, 0.1026	0.0467, 0.0702	0.0308, 0.0687	0.0387, 0.0752

Complexes	**7-Dy**	**8-Er**	**9-Yb**	**10-Er**
Formula	$C_{34}H_{20}DyN_2O_7$	$C_{34}H_{21}ErN_2O_7$	$C_{34}H_{21}YbN_2O_7$	$C_{33}H_{22}ErO_{11}$
Formula weight	732.02	736.79	742.57	761.77
Space group	$P2_1/c$	$P2_1/c$	$P2_1/c$	*P*$\bar{1}$
a/Å	11.9684(6)	12.0185(4)	12.0745(18)	10.1324(8)
b/Å	12.6499(4)	12.6199(5)	12.584(2)	10.1905(9)
c/Å	19.6347(17)	19.5860(8)	19.537(2)	15.5442(10)
α/°	90	90	90	96.333(2)

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β/°	108.000(6)	108.006(3)	108.000(7)	97.155(3)
γ/°	90	90	90	114.009(3)
V/Å^3	2827.2(3)	2825.16(19)	2823.3(7)	1431.59(19)
Z	4	4	4	2
μ/mm^{-1}	2.698	3.026	3.368	2.997
F(000)	1440	1452	1460	752
R_{int}	0.0340	0.0522	0.0693	0.0515
GOF	1.038	0.900	1.076	1.109
$R_1{}^a$, w$R_2{}^b$ [$I>2\sigma(I)$]	0.0349, 0.0706	0.0327, 0.0476	0.0422, 0.0748	0.0602, 0.1755
$R_1{}^a$, w$R_2{}^b$ (all data)	0.0498, 0.0755	0.0494, 0.0519	0.0618, 0.0827	0.0662, 0.1956

$^aR_1 = \sum||F_0| - |Fc||/\sum|F_0|$. ^{b}w$R_2 = \sum[w(F_0^2 - Fc^2)^2]/\sum[w(F_0^2)^2]^{1/2}$

Table S2. The Ln···Ln distances (Å) and the average bonds length (Å) Ln–O and Ln–N of complexes **1**−**6** and **10**−**15**.

	1−6					
Complexes	**1-Pr**	**2-Nd**	**3-Sm**	**4-Eu**	**5-Gd**	**6-Tb**
Ln···Ln	4.0611(8)	4.0326(7)	4.0136(6)	3.9903(4)	3.9804(3)	3.9597(12)
Ln–O	2.5262	2.5067	2.4840	2.4687	2.4593	2.4324
	10−13				**14−15**	
Complexes	**10-Tb**	**11-Dy**	**12-Er**	**13-Yb**	**14-Er**	**15-Ho**
Ln···Ln	3.7009(3)	3.6758(3)	3.6435(5)	3.6073(6)	4.1971(5)	4.1902(4)
Ln–O	2.3858	2.3692	2.3545	2.3368	2.3636	2.3647
Ln–N	2.567	2.544	2.527	2.500		

Table S3. Hydrogen-bonding geometry parameters (Å, °) for Complexes **1**−**6** and **10**−**15**.

D−H···A	d(D−H)(Å)	d(H···A)(Å)	d(D···A)(Å)	∠(DHA)(°)	Symmetry code

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Complex **1**					
O1W-H1WA···O3W	0.85	2.05	2.865(7)	160	
O2W-H2WA···O9	0.85	2.20	2.890(5)	138	2-*x*, 1-*y*, 1-*z*
O3W-H3WA···O2	0.85	2.07	2.842(7)	151	
O3W-H3WB···O7	0.85	2.34	3.071(6)	144	
C14-H14···O3	0.93	2.48	3.278(5)	143	
C25-H25···O2W	0.93	2.49	3.334(5)	151	
C30-H30···O3W	0.93	2.58	3.356(8)	142	*x*, *y*, -1+*z*
Complex **2**					
O1W-H1WB···O6	0.85	2.14	2.904(6)	149	2-*x*, 1-*y*, 1-*z*
O2W-H2WA···O3W	0.85	2.06	2.881(8)	163	1-*x*, 1-*y*, 1-*z*
O3W-H3WA···O2	0.85	2.06	2.839(7)	152	1-*x*, 1-*y*, 1-*z*
O3W-H3WB···O4	0.86	2.40	3.075(6)	136	
C14-H14···O1W	0.93	2.46	3.310(6)	152	
C21-H21···O3W	0.93	2.56	3.336(9)	142	1-*x*, 1-*y*, -*z*
C25-H25···O3	0.93	2.48	3.280(7)	144	
Complex **3**					
O1W-H1WB···O6	0.86	2.23	2.928(7)	138	2-*x*, 1-*y*, 1-*z*
O2W-H2WB···O3W	0.86	2.09	2.877(8)	152	
O3W-H3WA···O2	0.85	2.14	2.830(9)	137	
O3W-H3WB···O5	0.85	2.34	3.113(7)	150	1-*x*, 1-*y*, 1-*z*
C14-H14A···O1W	0.93	2.44	3.297(7)	154	
C21-H21A···O3W	0.93	2.53	3.320(10)	143	*x*, *y*, -1+*z*
C25-H25A···O3	0.93	2.49	3.282(7)	143	
Complex **4**					
O1W-H1WB···O3W	0.85	2.03	2.860(7)	167	1-*x*, 2-*y*, 1-*z*
O2W-H2WA···O6	0.85	2.12	2.944(6)	164	2-*x*, 1-*y*, 1-*z*
O3W-H3WA···O1	0.85	2.06	2.824(7)	149	1-*x*, 2-*y*, 1-*z*
O3W-H3WB···O5	0.85	2.51	3.097(6)	126	*x*, 1+*y*, *z*
C14-H14···O2W	0.93	2.41	3.278(6)	155	
C21-H21···O3W	0.93	2.52	3.314(8)	143	1-*x*, 2-*y*, -*z*
C25-H25···O3	0.93	2.48	3.266(7)	143	
Complex **5**					
O1W-H1WA···O9	0.86	2.12	2.957(5)	163	2-*x*, 1-*y*, 1-*z*
O2W-H2WA···O3W	0.85	2.17	2.856(7)	138	
O3W-H3WC···O1	0.86	2.06	2.824(7)	149	
O3W-H3WD···O7	0.85	2.34	3.109(6)	150	1-*x*, 1-*y*, 1-*z*
C14-H14···O3	0.93	2.47	3.264(5)	143	
C25-H25···O1W	0.93	2.40	3.275(5)	156	
C32-H32···O3W	0.93	2.52	3.308(8)	143	*x*, *y*, -1+*z*
Complex **6**					
O1W-H1WA···O6	0.85	2.12	2.961(3)	146	1-x,-y,1-z
O2W-H2WB···O3W	0.85	2.17	2.854(6)	132	1-x,1-y,1-z
O3W-H3WC···O5	0.85	2.06	3.130(7)	149	1-x,1-y,1-z
O3W-H3WD···O8	0.85	2.34	2.804(8)	148	1-*x*, 1-*y*, 1-*z*

C14-H14···O9	0.93	2.47	3.244(5)	144	
C25-H25···O1W	0.93	2.40	3.231(4)	157	
C32-H32···O3W	0.93	2.52	3.284(6)	143	1-x,1-y,-z
Complex **10**					
C21-H21···O3	0.93	2.47	3.322(9)	153	2-*x*,-1/2+*y*,3/2-*z*
Complex **11**					
C19-H19···O5	0.93	2.47	3.324(8)	153	-1+*x*, *y*, *z*
Complex **12**					
C12-H12···O5	0.93	2.44	3.295(7)	153	2-*x*,1/2+*y*, 3/2-*z*
Complex **13**					
C20-H20···O6	0.93	2.43	3.287(11)	153	1+*x*, *y*, *z*
Complex **14**					
C31-H31···O5	0.93	2.61	3.3778(11)	140	*x*, *y*, -1+*z*
Complex **15**					
C31-H31···O6	0.93	2.63	3.3762(86)	138	1-*x*, 1-*y*, -*z*

Table S4. X–H···π Interactions in Complexes **1**−**6** and **10**−**15**[c].

X–H···π	H···C(g) (Å)	X–H···C(g) (°)	X···C(g) (Å)	Symmetry code
Complex **1**				
C31-H31>Cg(2)	2.67	129	3.332(6)	2-*x*, 2- *y*, 1- *z*
Complex **2**				
C20-H20>Cg(2)	2.67	129	3.329(9)	2- *x*, 2- *y*, 1- *z*
Complex **3**				
C20-H20A>Cg(2)	2.66	128	3.316(9)	2- *x*, 2- *y*, 1- *z*
Complex **4**				
C20-H20>Cg(2)	2.66	129	3.319(8)	2- *x*, 2- *y*, 1- *z*
Complex **5**				
C31-H31>Cg(2)	2.65	128	3.305(7)	2- *x*, 2- *y*, 1- *z*
Complex **6**				
C18-H18>Cg(4)	2.91	136	3.637(6)	2- *x*, 1- *y*, 2- *z*
Complex **7**				
C22-H22> Cg(5)	2.91	136	3.642(6)	-1+*x*, 1/2-*y*, -1/2+*z*
Complex **8**				
C15-H15> Cg(5)	2.92	137	3.658(6)	2- *x*, 1- *y*, 2- *z*
Complex **9**				
C17-H17> Cg(5)	2.96	135	3.678(8)	1+ *x*, 3/2- *y*, 1/2+ *z*
Complex **10**				
O2W-H2WA>Cg(4)	2.51	161	3.330(8)	*x*, *y*, *z*
C12-H12>Cg(6)	2.76	150	3.595(10)	2-*x*, 2- *y*, 1- *z*
C21-H21>Cg(3)	2.80	135	3.522(13)	-1+ *x*, *y*, *z*
Complex **11**				

O2W-H2WB>Cg(3)	2.54	156	3.350(5)	*x*, *y*, *z*
C12-H12>Cg(4)	2.80	136	3.526(8)	- *x*, 1- *y*, 1- *z*
C19-H19>Cg(6)	2.79	150	3.619(6)	-1+ *x*, -1+ *y*, *z*

[c]For **1**: Cg(2): C6>C7>C8>C9>C10>C11
For **2**: Cg(2): C6>C7>C8>C9>C10>C11
For **3**: Cg(2): C6>C7>C8>C9>C10>C11
For **4**: Cg(2): C6>C7>C8>C9>C10>C11
For **5**: Cg(2): C6>C7>C8>C9>C10>C11
For **10**: Cg(4): C6>C7>C8>C9>C10>C11
For **11**: Cg(5): C10>C11>C12>C13>C14>C15
For **12**: Cg(5): C17>C18>C19>C20>C21>C22
For **13**: Cg(5): C9>C10>C11>C12>C13>C14
For **14**: Cg(4): C17>C18>C19>C20>C21>C22; Cg(6): C28>C29>C30>C31>C32>C33; Cg(3): C10>C11>C12>C13>C14>C15
For **15**: Cg(3): C10>C11>C12>C13>C14>C15; Cg(4): C17>C18>C19>C20>C21>C22; Cg(6): C28>C29>C30>C31>C32>C33

Table S5. $\pi\cdots\pi$ Interactions in Complexes **10**–**15**[d].

Two rings	Symmetry code	Centroid-to-centroid distance (Å)	Dihedral angel (°)	Perpendicular distance of Cg(I) on ring J (Å)	Perpendicular distance of Cg(J) on ring I (Å)
Complex **10**					
Cg(5)>Cg(5)	2- *x*, - *y*, 2- *z*	3.935(4)	0	-3.302(3)	-3.302(3)
Cg(6)>Cg(6)	1- *x*, 2- *y*, 1- *z*	3.646(4)	0	-3.490(3)	-3.490(3)
Complex **11**					
Cg(6)>Cg(6)	- *x*, 1- *y*, 1- *z*	3.919(3)	0	3.309(2)	3.310(2)
Cg(7)>Cg(7)	1- *x*, - *y*, 1- *z*	3.610(3)	0	-3.457(3)	-3.457(3)
Complex **12**					
Cg(4)>Cg(4)	2- *x*, 2- *y*, 2- *z*	3.881(3)	0	-3.297(2)	-3.296(2)
Cg(6)>Cg(6)	1- *x*, - *y*, 1- *z*	3.6211(3)	0	-3.447(2)	-3.447(2)
Complex **13**					
Cg(6)>Cg(6)	2- *x*, 1- *y*, 1- *z*	3.840(5)	0	-3.272(3)	-3.272(3)
Cg(7)>Cg(7)	1- *x*, 2- *y*, 1- *z*	3.613(7)	0	3.442(3)	3.442(3)
Complex **14**					
Cg(4)>Cg(4)	1- *x*, 2- *y*, 2- *z*	4.088(6)	0	3.740(4)	3.739(4)
Complex **15**					
Cg(3)>Cg(3)	1- *x*, 2-*y*, 2- *z*	4.082(4)	0	3.731(3)	3.731(3)

[d]For **10**: Cg(5): C17>C18>C19>C20>C21>C22; Cg(6): C26>C27>C28>C29>C30>C31
For **11**: Cg(6): C17>C18>C19>C20>C21>C22; Cg(7): C26>C27>C28>C29>C30>C31

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For **12**: Cg(4): C10>C11>C12>C13>C14>C15; Cg(6): C26>C27>C28>C29>C33>C34
For **13**: Cg(6): C16>C17>C18>C19>C20>C21; Cg(7): C26>C27>C28>C29>C33>C34
For **14**: Cg(4): C17>C18>C19>C20>C21>C22
For **15**: Cg(3): C10>C11>C12>C13>C14>C15

Table S6. Maximum emission wavelengths, λ_{ex} (nm), τ (μs) and $\Phi_{overall}$ (%)of **3**, **4** and **6**–**11**.

Complexes	λ_{ex} (nm)	τ (μs)	$\Phi_{overall}$ (%)	Maximum emission wavelengths
3(Sm)	340	11.5	0.3	562 ($^4G_{5/2} \rightarrow {}^6H_{5/2}$), 596 ($^4G_{5/2} \rightarrow {}^6H_{7/2}$), 642 ($^4G_{5/2} \rightarrow {}^6H_{9/2}$), 703 ($^4G_{5/2} \rightarrow {}^6H_{11/2}$)
4(Eu)	350	332	17.2	578 ($^5D_0 \rightarrow {}^7F_0$), 590 ($^5D_0 \rightarrow {}^7F_1$), 613 ($^5D_0 \rightarrow {}^7F_2$), 651 ($^5D_0 \rightarrow {}^7F_3$), 696 ($^5D_0 \rightarrow {}^7F_4$)
6(Tb)	290	675	26.3	488 ($^5D_4 \rightarrow {}^7F_6$), 545 ($^5D_4 \rightarrow {}^7F_5$), 586 ($^5D_4 \rightarrow {}^7F_4$), 622 ($^5D_4 \rightarrow {}^7F_3$), 652 ($^5D_4 \rightarrow {}^7F_2$), 668 ($^5D_4 \rightarrow {}^7F_1$), 680 ($^5D_4 \rightarrow {}^7F_0$)
7(Dy)	290	1.27	0.9	479 ($^4F_{9/2} \rightarrow {}^6H_{15/2}$), 574 ($^4F_{9/2} \rightarrow {}^6H_{13/2}$), 662 ($^4F_{9/2} \rightarrow {}^6H_{11/2}$), 751 ($^4F_{9/2} \rightarrow {}^6H_{9/2}$)
8(Sm)	290	49.2	5.3	563 ($^4G_{5/2} \rightarrow {}^6H_{5/2}$), 598 ($^4G_{5/2} \rightarrow {}^6H_{7/2}$), 645 ($^4G_{5/2} \rightarrow {}^6H_{9/2}$), 704 ($^4G_{5/2} \rightarrow {}^6H_{11/2}$)
9(Eu)	290	981	55.4	579 ($^5D_0 \rightarrow {}^7F_0$), 592 ($^5D_0 \rightarrow {}^7F_1$), 615 ($^5D_0 \rightarrow {}^7F_2$), 651 ($^5D_0 \rightarrow {}^7F_3$), 698 ($^5D_0 \rightarrow {}^7F_4$)
10(Tb)	365	762	40.2	489 ($^5D_4 \rightarrow {}^7F_6$), 548 ($^5D_4 \rightarrow {}^7F_5$), 585 ($^5D_4 \rightarrow {}^7F_4$), 621 ($^5D_4 \rightarrow {}^7F_3$), 651 ($^5D_4 \rightarrow {}^7F_2$), 669 ($^5D_4 \rightarrow {}^7F_1$), 680 ($^5D_4 \rightarrow {}^7F_0$)
11(Dy)	365	10.4	2.0	479 ($^4F_{9/2} \rightarrow {}^6H_{15/2}$), 574 ($^4F_{9/2} \rightarrow {}^6H_{13/2}$), 662 ($^4F_{9/2} \rightarrow {}^6H_{11/2}$), 750 ($^4F_{9/2} \rightarrow {}^6H_{9/2}$)

Table S7. Selected Bond distances (Å) and Angles (°) of **1**–**6** and **10**–**15**[e].

1					
Pr(1)-O(8)	2.440(2)	Pr(1)-O(2)	2.500(2)	Pr(1)-O(2W)	2.548(3)
Pr(1)-O(4)	2.457(2)	Pr(1)-O(7)	2.511(2)	Pr(1)-O(1W)	2.581(3)
Pr(1)-O(5)	2.472(2)	Pr(1)-O(1)	2.517(2)	Pr(1)-O(8)#1	2.710(2)
O(8)-Pr(1)-O(4)	74.96(8)	O(5)-Pr(1)-O(1)	135.49(9)	O(2)-Pr(1)-O(1W)	72.93(10)

O(8)-Pr(1)-O(5)	74.49(8)	O(2)-Pr(1)-O(1)	52.04(8)	O(7)-Pr(1)-O(1W)	69.45(9)
O(4)-Pr(1)-O(5)	136.31(8)	O(7)-Pr(1)-O(1)	125.82(8)	O(1)-Pr(1)-O(1W)	109.84(10)
O(8)-Pr(1)-O(2)	139.20(8)	O(8)-Pr(1)-O(2W)	95.50(9)	O(2W)-Pr(1)-O(1W)	65.91(10)
O(4)-Pr(1)-O(2)	81.02(9)	O(4)-Pr(1)-O(2W)	139.51(9)	O(8)-Pr(1)-O(8)#1	76.03(8)
O(5)-Pr(1)-O(2)	140.87(9)	O(5)-Pr(1)-O(2W)	73.99(9)	O(4)-Pr(1)-O(8)#1	68.76(8)
O(8)-Pr(1)-O(7)	124.95(8)	O(2)-Pr(1)-O(2W)	81.86(10)	O(5)-Pr(1)-O(8)#1	74.06(8)
O(4)-Pr(1)-O(7)	76.65(9)	O(7)-Pr(1)-O(2W)	134.86(9)	O(2)-Pr(1)-O(8)#1	124.60(8)
O(5)-Pr(1)-O(7)	96.45(9)	O(1)-Pr(1)-O(2W)	66.46(9)	O(7)-Pr(1)-O(8)#1	49.84(8)
O(2)-Pr(1)-O(7)	79.20(8)	O(8)-Pr(1)-O(1W)	142.53(9)	O(1)-Pr(1)-O(8)#1	142.42(8)
O(8)-Pr(1)-O(1)	89.43(8)	O(4)-Pr(1)-O(1W)	140.25(9)	O(2W)-Pr(1)-O(8)#1	148.06(8)
O(4)-Pr(1)-O(1)	74.07(9)	O(5)-Pr(1)-O(1W)	69.26(10)	O(1W)-Pr(1)-O(8)#1	102.24(9)
2					
Nd(1)-O(5)	2.414(3)	Nd(1)-O(2)	2.476(3)	Nd(1)-O(1W)	2.529(3)
Nd(1)-O(8)	2.437(3)	Nd(1)-O(4)#1	2.491(3)	Nd(1)-O(2W)	2.557(3)
Nd(1)-O(7)	2.453(3)	Nd(1)-O(1)	2.502(3)	Nd(1)-O(5)#1	2.701(3)
O(5)-Nd(1)-O(8)	75.44(10)	O(7)-Nd(1)-O(1)	135.34(11)	O(2)-Nd(1)-O(2W)	73.14(11)
O(5)-Nd(1)-O(7)	74.32(10)	O(2)-Nd(1)-O(1)	52.32(10)	O(4)#1-Nd(1)-O(2W)	69.75(11)
O(8)-Nd(1)-O(7)	136.34(10)	O(4)#1-Nd(1)-O(1)	126.20(10)	O(1)-Nd(1)-O(2W)	109.89(12)
O(5)-Nd(1)-O(2)	139.43(11)	O(5)-Nd(1)-O(1W)	94.93(10)	O(1W)-Nd(1)-O(2W)	65.91(12)
O(8)-Nd(1)-O(2)	80.96(11)	O(8)-Nd(1)-O(1W)	139.51(12)	O(5)-Nd(1)-O(5)#1	76.07(10)
O(7)-Nd(1)-O(2)	140.79(11)	O(7)-Nd(1)-O(1W)	74.01(11)	O(8)-Nd(1)-O(5)#1	68.42(10)
O(5)-Nd(1)-O(4)#1	125.06(10)	O(2)-Nd(1)-O(1W)	82.08(11)	O(7)-Nd(1)-O(5)#1	74.22(10)
O(8)-Nd(1)-O(4)#1	76.51(11)	O(4)#1-Nd(1)-O(1W)	135.17(11)	O(2)-Nd(1)-O(5)#1	124.46(10)
O(7)-Nd(1)-O(4)#1	96.21(11)	O(1)-Nd(1)-O(1W)	66.20(12)	O(4)#1-Nd(1)-O(5)#1	49.78(9)
O(2)-Nd(1)-O(4)#1	79.25(11)	O(5)-Nd(1)-O(2W)	141.87(11)	O(1)-Nd(1)-O(5)#1	142.29(11)
O(5)-Nd(1)-O(1)	89.33(10)	O(8)-Nd(1)-O(2W)	140.45(11)	O(1W)-Nd(1)-O(5)#1	148.23(10)
O(8)-Nd(1)-O(1)	74.33(11)	O(7)-Nd(1)-O(2W)	68.88(11)	O(2W)-Nd(1)-O(5)#1	102.57(11)
3					
Sm(1)-O(4)	2.385(3)	Sm(1)-O(5)#1	2.451(3)	Sm(1)-O(1W)	2.500(3)
Sm(1)-O(8)	2.420(3)	Sm(1)-O(2)	2.455(4)	Sm(1)-O(2W)	2.537(4)
Sm(1)-O(7)	2.420(3)	Sm(1)-O(1)	2.488(3)	Sm(1)-O(4)#1	2.700(3)

O(4)-Sm(1)-O(8)	75.39(11)	O(7)-Sm(1)-O(1)	135.47(12)	O(5)#1-Sm(1)-O(2W)	69.38(12)
O(4)-Sm(1)-O(7)	74.94(11)	O(5)#1-Sm(1)-O(1)	126.83(12)	O(2)-Sm(1)-O(2W)	72.80(13)
O(8)-Sm(1)-O(7)	136.64(11)	O(2)-Sm(1)-O(1)	52.90(11)	O(1)-Sm(1)-O(2W)	110.18(12)
O(4)-Sm(1)-O(5)#1	125.45(11)	O(4)-Sm(1)-O(1W)	94.46(12)	O(1W)-Sm(1)-O(2W)	66.18(12)
O(8)-Sm(1)-O(5)#1	77.09(11)	O(8)-Sm(1)-O(1W)	139.12(12)	O(4)-Sm(1)-O(4)#1	75.94(11)
O(7)-Sm(1)-O(5)#1	95.50(12)	O(7)-Sm(1)-O(1W)	74.10(12)	O(8)-Sm(1)-O(4)#1	69.02(10)
O(4)-Sm(1)-O(2)	139.17(11)	O(5)#1-Sm(1)-O(1W)	135.15(12)	O(7)-Sm(1)-O(4)#1	73.59(11)
O(8)-Sm(1)-O(2)	80.86(12)	O(2)-Sm(1)-O(1W)	82.27(13)	O(5)#1-Sm(1)-O(4)#1	50.33(11)
O(7)-Sm(1)-O(2)	140.48(12)	O(1)-Sm(1)-O(1W)	66.26(12)	O(2)-Sm(1)-O(4)#1	125.05(11)
O(5)#1-Sm(1)-O(2)	79.34(12)	O(4)-Sm(1)-O(2W)	142.28(12)	O(1)-Sm(1)-O(4)#1	142.23(11)
O(4)-Sm(1)-O(1)	88.42(11)	O(8)-Sm(1)-O(2W)	140.33(12)	O(1W)-Sm(1)-O(4)#1	147.67(11)
O(8)-Sm(1)-O(1)	73.86(11)	O(7)-Sm(1)-O(2W)	68.82(13)	O(2W)-Sm(1)-O(4)#1	102.78(12)
4					
Eu(1)-O(4)	2.367(2)	Eu(1)-O(5)#1	2.435(3)	Eu(1)-O(2W)	2.481(3)
Eu(1)-O(7)	2.403(3)	Eu(1)-O(1)	2.435(3)	Eu(1)-O(1W)	2.519(3)
Eu(1)-O(8)#1	2.403(3)	Eu(1)-O(2)	2.480(3)	Eu(1)-O(4)#1	2.695(3)
O(4)-Eu(1)-O(7)	75.65(9)	O(8)#1-Eu(1)-O(2)	135.58(10)	O(5)#1-Eu(1)-O(1W)	69.67(10)
O(4)-Eu(1)-O(8)#1	75.16(9)	O(5)#1-Eu(1)-O(2)	127.03(10)	O(1)-Eu(1)-O(1W)	72.75(10)
O(7)-Eu(1)-O(8)#1	136.68(10)	O(1)-Eu(1)-O(2)	53.08(9)	O(2)-Eu(1)-O(1W)	109.90(11)
O(4)-Eu(1)-O(5)#1	125.72(10)	O(4)-Eu(1)-O(2W)	94.02(10)	O(2W)-Eu(1)-O(1W)	66.10(10)
O(7)-Eu(1)-O(5)#1	77.01(10)	O(7)-Eu(1)-O(2W)	138.96(11)	O(4)-Eu(1)-O(4)#1	76.15(10)
O(8)#1-Eu(1)-O(5)#1	95.18(10)	O(8)#1-Eu(1)-O(2W)	74.38(11)	O(7)-Eu(1)-O(4)#1	68.97(9)
O(4)-Eu(1)-O(1)	139.00(10)	O(5)#1-Eu(1)-O(2W)	135.38(10)	O(8)#1-Eu(1)-O(4)#1	73.44(9)
O(7)-Eu(1)-O(1)	80.61(10)	O(1)-Eu(1)-O(2W)	82.45(10)	O(5)#1-Eu(1)-O(4)#1	50.37(9)
O(8)#1-Eu(1)-O(1)	140.50(10)	O(2)-Eu(1)-O(2W)	65.97(11)	O(1)-Eu(1)-O(4)#1	124.95(9)
O(5)#1-Eu(1)-O(1)	79.27(10)	O(4)-Eu(1)-O(1W)	142.20(10)	O(2)-Eu(1)-O(4)#1	142.23(9)
O(4)-Eu(1)-O(2)	88.10(10)	O(7)-Eu(1)-O(1W)	140.35(9)	O(2W)-Eu(1)-O(4)#1	147.78(10)
O(7)-Eu(1)-O(2)	73.96(10)	O(8)#1-Eu(1)-O(1W)	68.75(10)	O(1W)-Eu(1)-O(4)#1	103.20(10)
5					
Gd(1)-O(8)	2.354(2)	Gd(1)-O(7)#1	2.424(2)	Gd(1)-O(1W)	2.475(3)
Gd(1)-O(5)	2.388(2)	Gd(1)-O(1)	2.425(2)	Gd(1)-O(2W)	2.509(3)

Gd(1)-O(4)#1	2.395(2)	Gd(1)-O(2)	2.465(3)	Gd(1)-O(8)#1	2.699(3)
O(8)-Gd(1)-O(5)	75.94(8)	O(4)#1-Gd(1)-O(2)	135.32(9)	O(7)#1-Gd(1)-O(2W)	69.49(9)
O(8)-Gd(1)-O(4)#1	75.15(9)	O(7)#1-Gd(1)-O(2)	127.53(9)	O(1)-Gd(1)-O(2W)	72.69(10)
O(5)-Gd(1)-O(4)#1	136.81(8)	O(1)-Gd(1)-O(2)	53.29(9)	O(2)-Gd(1)-O(2W)	109.89(10)
O(8)-Gd(1)-O(7)#1	125.96(9)	O(8)-Gd(1)-O(1W)	93.67(9)	O(1W)-Gd(1)-O(2W)	66.09(10)
O(5)-Gd(1)-O(7)#1	77.16(9)	O(5)-Gd(1)-O(1W)	139.11(10)	O(8)-Gd(1)-O(8)#1	76.25(9)
O(4)#1-Gd(1)-O(7)#1	94.92(9)	O(4)#1-Gd(1)-O(1W)	74.16(9)	O(5)-Gd(1)-O(8)#1	68.97(8)
O(8)-Gd(1)-O(1)	138.91(9)	O(7)#1-Gd(1)-O(1W)	135.26(9)	O(4)#1-Gd(1)-O(8)#1	73.44(8)
O(5)-Gd(1)-O(1)	80.48(9)	O(1)-Gd(1)-O(1W)	82.66(10)	O(7)#1-Gd(1)-O(8)#1	50.44(8)
O(4)#1-Gd(1)-O(1)	140.50(9)	O(2)-Gd(1)-O(1W)	65.97(10)	O(1)-Gd(1)-O(8)#1	125.05(8)
O(7)#1-Gd(1)-O(1)	79.44(9)	O(8)-Gd(1)-O(2W)	142.17(9)	O(2)-Gd(1)-O(8)#1	142.26(9)
O(8)-Gd(1)-O(2)	87.77(9)	O(5)-Gd(1)-O(2W)	140.16(9)	O(1W)-Gd(1)-O(8)#1	147.54(9)
O(5)-Gd(1)-O(2)	74.09(9)	O(4)#1-Gd(1)-O(2W)	68.81(9)	O(2W)-Gd(1)-O(8)#1	103.35(9)
6					
Tb(1)-O(4)	2.319(3)	Tb(1)-O(5)#1	2.379(3)	Tb(1)-O(1W)	2.461(3)
Tb(1)-O(1)	2.363(3)	Tb(1)-O(8)	2.398(3)	Tb(1)-O(2W)	2.488(3)
Tb(1)-O(2)#1	2.366(3)	Tb(1)-O(7)	2.422(3)	Tb(1)-O(4)#1	2.696(3)
O(4)-Tb(1)-O(1)	76.22(10)	O(5)#1-Tb(1)-O(7)	128.34(11)	O(2)#1-Tb(1)-O(7)	134.80(11)
O(4)-Tb(1)-O(2)#1	75.03(10)	O(8)-Tb(1)-O(7)	53.50(10)	O(8)-Tb(1)-O(2W)	72.17(12)
O(1)-Tb(1)-O(2)#1	136.67(10)	O(4)-Tb(1)-O(1W)	92.85(11)	O(7)-Tb(1)-O(2W)	108.38(12)
O(4)-Tb(1)-O(5)#1	126.04(10)	O(1)-Tb(1)-O(1W)	138.96(11)	O(1W)-Tb(1)-O(2W)	65.44(11)
O(1)-Tb(1)-O(5)#1	77.23(10)	O(2)#1-Tb(1)-O(1W)	74.21(11)	O(4)-Tb(1)-O(4)#1	75.94(10)
O(2)#1-Tb(1)-O(5)#1	94.52(11)	O(5)#1-Tb(1)-O(1W)	135.78(11)	O(1)-Tb(1)-O(4)#1	68.47(9)
O(4)-Tb(1)-O(8)	138.91(10)	O(8)-Tb(1)-O(1W)	83.05(12)	O(2)#1-Tb(1)-O(4)#1	73.55(9)
O(1)-Tb(1)-O(8)	80.46(10)	O(7)-Tb(1)-O(1W)	65.17(12)	O(5)#1-Tb(1)-O(4)#1	50.70(9)
O(2)#1-Tb(1)-O(8)	140.58(10)	O(4)-Tb(1)-O(2W)	141.89(11)	O(8)-Tb(1)-O(4)#1	125.20(10)
O(5)#1-Tb(1)-O(8)	79.70(11)	O(1)-Tb(1)-O(2W)	140.63(11)	O(7)-Tb(1)-O(4)#1	142.34(10)
O(4)-Tb(1)-O(7)	87.66(11)	O(2)#1-Tb(1)-O(2W)	69.19(12)	O(1W)-Tb(1)-O(4)#1	147.62(11)
O(1)-Tb(1)-O(7)	74.83(11)	O(5)#1-Tb(1)-O(2W)	70.55(11)	O(2W)-Tb(1)-O(4)#1	105.24(10)
10					
Tb(1)-O(7)#1	2.252(3)	Tb(1)-O(1)	2.411(4)	Tb(1)-N(2)	2.560(4)

Tb(1)-O(7)	2.270(3)	Tb(1)-O(2)	2.465(4)	Tb(1)-N(1)	2.574(4)
Tb(1)-O(5)#2	2.400(4)	Tb(1)-O(4)#2	2.517(4)		
O(7)#1-Tb(1)-O(7)	70.13(14)	O(7)#1-Tb(1)-O(4)#2	82.91(12)	O(2)-Tb(1)-N(2)	80.37(13)
O(7)#1-Tb(1)-O(5)#2	135.28(13)	O(7)-Tb(1)-O(4)#2	147.70(12)	O(4)#2-Tb(1)-N(2)	113.05(14)
O(7)-Tb(1)-O(5)#2	153.76(13)	O(5)#2-Tb(1)-O(4)#2	53.15(13)	O(7)#1-Tb(1)-N(1)	85.69(15)
O(7)#1-Tb(1)-O(1)	85.26(14)	O(1)-Tb(1)-O(4)#2	86.42(14)	O(7)-Tb(1)-N(1)	84.99(15)
O(7)-Tb(1)-O(1)	108.03(15)	O(2)-Tb(1)-O(4)#2	125.24(13)	O(5)#2-Tb(1)-N(1)	90.17(16)
O(5)#2-Tb(1)-O(1)	84.17(17)	O(7)#1-Tb(1)-N(2)	138.76(13)	O(1)-Tb(1)-N(1)	160.47(15)
O(7)#1-Tb(1)-O(2)	122.51(13)	O(7)-Tb(1)-N(2)	79.36(13)	O(2)-Tb(1)-N(1)	144.36(14)
O(7)-Tb(1)-O(2)	85.26(13)	O(5)#2-Tb(1)-N(2)	75.35(15)	O(4)#2-Tb(1)-N(1)	75.31(15)
O(5)#2-Tb(1)-O(2)	83.78(14)	O(1)-Tb(1)-N(2)	131.42(14)	N(2)-Tb(1)-N(1)	64.15(15)
O(1)-Tb(1)-O(2)	53.60(12)				
11					
Dy(1)-O(7)	2.238(3)	Dy(1)-O(2)	2.395(3)	Dy(1)-N(2)	2.539(4)
Dy(1)-O(7)#1	2.254(3)	Dy(1)-O(1)	2.450(3)	Dy(1)-N(1)	2.549(4)
Dy(1)-O(3)	2.379(4)	Dy(1)-O(4)	2.499(4)		
O(7)-Dy(1)-O(7)#1	70.15(13)	O(7)-Dy(1)-O(4)	82.64(11)	O(1)-Dy(1)-N(2)	80.02(12)
O(7)-Dy(1)-O(3)	135.45(12)	O(7)#1-Dy(1)-O(4)	147.74(11)	O(4)-Dy(1)-N(2)	113.37(12)
O(7)#1-Dy(1)-O(3)	153.54(12)	O(3)-Dy(1)-O(4)	53.51(11)	O(7)-Dy(1)-N(1)	85.68(13)
O(7)-Dy(1)-O(2)	85.30(12)	O(2)-Dy(1)-O(4)	85.97(13)	O(7)#1-Dy(1)-N(1)	85.66(14)
O(7)#1-Dy(1)-O(2)	108.02(13)	O(1)-Dy(1)-O(4)	125.33(12)	O(3)-Dy(1)-N(1)	89.57(14)
O(3)-Dy(1)-O(2)	84.32(14)	O(7)-Dy(1)-N(2)	139.07(14)	O(2)-Dy(1)-N(1)	159.86(14)
O(7)-Dy(1)-O(1)	122.54(11)	O(7)#1-Dy(1)-N(2)	79.63(13)	O(1)-Dy(1)-N(1)	144.53(12)
O(7)#1-Dy(1)-O(1)	84.98(12)	O(3)-Dy(1)-N(2)	74.83(14)	O(4)-Dy(1)-N(1)	75.04(14)
O(3)-Dy(1)-O(1)	83.93(12)	O(2)-Dy(1)-N(2)	131.17(12)	N(2)-Dy(1)-N(1)	64.64(13)
O(2)-Dy(1)-O(1)	53.85(12)				
12					
Er(1)-O(7)#1	2.218(3)	Er(1)-O(2)	2.377(3)	Er(1)-N(1)	2.524(4)
Er(1)-O(7)	2.250(3)	Er(1)-O(1)	2.433(3)	Er(1)-N(2)	2.529(4)
Er(1)-O(4)	2.365(3)	Er(1)-O(3)	2.484(3)		
O(7)#1-Er(1)-O(7)	70.70(12)	O(7)#1-Er(1)-O(3)	81.60(11)	O(1)-Er(1)-N(1)	79.63(12)

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O(7)#1-Er(1)-O(4)	135.25(12)	O(7)-Er(1)-O(3)	147.78(10)	O(3)-Er(1)-N(1)	114.62(12)
O(7)-Er(1)-O(4)	153.24(12)	O(4)-Er(1)-O(3)	54.23(11)	O(7)#1-Er(1)-N(2)	85.34(12)
O(7)#1-Er(1)-O(2)	85.12(11)	O(2)-Er(1)-O(3)	84.51(12)	O(7)-Er(1)-N(2)	85.87(12)
O(7)-Er(1)-O(2)	108.48(12)	O(1)-Er(1)-O(3)	124.86(11)	O(4)-Er(1)-N(2)	89.71(13)
O(4)-Er(1)-O(2)	83.97(13)	O(7)#1-Er(1)-N(1)	139.54(12)	O(2)-Er(1)-N(2)	158.95(13)
O(7)#1-Er(1)-O(1)	122.85(11)	O(7)-Er(1)-N(1)	79.49(12)	O(1)-Er(1)-N(2)	144.91(12)
O(7)-Er(1)-O(1)	85.01(11)	O(4)-Er(1)-N(1)	74.69(13)	O(3)-Er(1)-N(2)	75.56(13)
O(4)-Er(1)-O(1)	83.57(11)	O(2)-Er(1)-N(1)	131.10(12)	N(1)-Er(1)-N(2)	65.41(13)
O(2)-Er(1)-O(1)	54.27(11)				

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Yb(1)-O(7)#1	2.196(4)	Yb(1)-O(2)	2.353(4)	Yb(1)-N(1)	2.496(6)
Yb(1)-O(7)	2.229(4)	Yb(1)-O(1)	2.422(5)	Yb(1)-N(2)	2.504(5)
Yb(1)-O(3)	2.347(4)	Yb(1)-O(4)	2.474(5)		
O(7)#1-Yb(1)-O(7)	70.78(17)	O(7)#1-Yb(1)-O(4)	80.81(15)	O(1)-Yb(1)-N(1)	145.18(17)
O(7)#1-Yb(1)-O(3)	135.36(16)	O(7)-Yb(1)-O(4)	147.50(15)	O(4)-Yb(1)-N(1)	75.82(17)
O(7)-Yb(1)-O(3)	153.03(16)	O(3)-Yb(1)-O(4)	55.03(15)	O(7)#1-Yb(1)-N(2)	139.86(17)
O(7)#1-Yb(1)-O(2)	84.73(17)	O(2)-Yb(1)-O(4)	83.44(17)	O(7)-Yb(1)-N(2)	79.62(16)
O(7)-Yb(1)-O(2)	108.64(17)	O(1)-Yb(1)-O(4)	125.15(15)	O(3)-Yb(1)-N(2)	74.33(17)
O(3)-Yb(1)-O(2)	84.14(18)	O(7)#1-Yb(1)-N(1)	85.20(18)	O(2)-Yb(1)-N(2)	131.27(18)
O(7)#1-Yb(1)-O(1)	122.59(17)	O(7)-Yb(1)-N(1)	86.15(18)	O(1)-Yb(1)-N(2)	79.38(17)
O(7)-Yb(1)-O(1)	84.53(16)	O(3)-Yb(1)-N(1)	89.51(18)	O(4)-Yb(1)-N(2)	115.61(17)
O(3)-Yb(1)-O(1)	83.97(17)	O(2)-Yb(1)-N(1)	158.14(19)	N(1)-Yb(1)-N(2)	65.95(19)
O(2)-Yb(1)-O(1)	54.90(16)				

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Er(1)-O(3)	2.241(5)	Er(1)-O(1)	2.417(6)	Er(1)-O(1W)	2.452(6)
Er(1)-O(7)	2.292(6)	Er(1)-O(2W)	2.427(6)	Er(1)-O(2)	2.392(6)
Er(1)-O(4)	2.322(5)	Er(1)-O(8)#1	2.366(5)		
O(3)-Er(1)-O(7)	73.6(2)	O(2)-Er(1)-O(1)	54.30(19)	O(2)-Er(1)-O(2W)	91.9(2)
O(3)-Er(1)-O(4)	123.8(2)	O(3)-Er(1)-O(2W)	146.2(2)	O(1)-Er(1)-O(2W)	69.6(2)
O(7)-Er(1)-O(4)	80.6(2)	O(7)-Er(1)-O(2W)	140.1(2)	O(3)-Er(1)-O(1W)	76.1(3)
O(3)-Er(1)-O(8)#1	81.1(2)	O(4)-Er(1)-O(2W)	75.2(2)	O(7)-Er(1)-O(1W)	141.7(2)

O(7)-Er(1)-O(8)#1	127.3(2)	O(8)#1-Er(1)-O(2W)	76.8(2)	O(4)-Er(1)-O(1W)	136.6(2)
O(4)-Er(1)-O(8)#1	76.42(19)	O(3)-Er(1)-O(1)	134.2(2)	O(8)#1-Er(1)-O(1W)	68.9(2)
O(3)-Er(1)-O(2)	88.9(2)	O(7)-Er(1)-O(1)	76.8(2)	O(2)-Er(1)-O(1W)	71.3(2)
O(7)-Er(1)-O(2)	85.3(2)	O(4)-Er(1)-O(1)	83.81(18)	O(1)-Er(1)-O(1W)	110.1(2)
O(4)-Er(1)-O(2)	137.9(2)	O(8)#1-Er(1)-O(1)	144.35(19)	O(2W)-Er(1)-O(1W)	72.3(2)
O(8)#1-Er(1)-O(2)	140.2(2)				
15					
Ho(1)-O(2)	2.250(4)	Ho(1)-O(2W)	2.399(4)	Ho(1)-O(1W)	2.454(4)
Ho(1)-O(8)	2.283(3)	Ho(1)-O(4)	2.427(4)	Ho(1)-O(3)	2.386(3)
Ho(1)-O(1)#1	2.339(3)	Ho(1)-O(7)#1	2.380(3)		
O(2)-Ho(1)-O(8)	73.63(14)	O(3)-Ho(1)-O(2W)	91.69(14)	O(3)-Ho(1)-O(4)	54.39(12)
O(2)-Ho(1)-O(1)#1	123.79(14)	O(2)-Ho(1)-O(4)	134.84(12)	O(2W)-Ho(1)-O(4)	69.55(13)
O(8)-Ho(1)-O(1)#1	81.02(12)	O(8)-Ho(1)-O(4)	76.57(14)	O(2)-Ho(1)-O(1W)	75.60(15)
O(2)-Ho(1)-O(7)#1	80.66(13)	O(1)#1-Ho(1)-O(4)	83.16(12)	O(8)-Ho(1)-O(1W)	140.75(13)
O(8)-Ho(1)-O(7)#1	128.26(12)	O(7)#1-Ho(1)-O(4)	144.21(13)	O(1)#1-Ho(1)-O(1W)	137.25(12)
O(1)#1-Ho(1)-O(7)#1	77.24(12)	O(2)-Ho(1)-O(2W)	145.64(13)	O(7)#1-Ho(1)-O(1W)	68.51(12)
O(2)-Ho(1)-O(3)	89.31(14)	O(8)-Ho(1)-O(2W)	140.62(14)	O(3)-Ho(1)-O(1W)	71.50(13)
O(8)-Ho(1)-O(3)	84.30(13)	O(1)#1-Ho(1)-O(2W)	75.68(13)	O(2W)-Ho(1)-O(1W)	72.22(14)
O(1)#1-Ho(1)-O(3)	137.29(13)	O(7)#1-Ho(1)-O(2W)	76.68(13)	O(4)-Ho(1)-O(1W)	110.42(13)
O(7)#1-Ho(1)-O(3)	140.01(12)				

[e]Symmetry transformations used to generate equivalent atoms: For **1:** #1, −*x* + 1, −*y* + 1, −*z* + 1. For **2:** #1, −*x* + 1, −*y* + 1, −*z* + 1. For **3:** #1, −*x* + 2, −*y* + 2, −*z* + 1. For **4:** #1, −*x* + 1, −*y* + 1, −z. For **5:** #1, −*x* + 1, −*y*, −*z* + 1. For **6:** #1, −*x*, −*y*, −*z* + 1. For **10:** #1, −*x* + 1, −*y* + 1, −*z* + 1; #2, −*x* + 1, −*y* + 1/2, −*z* +3/2. For **11:** #1, −*x* + 1, −*y* + 1, −*z* + 1. For **12:** #1, −*x* + 1, −*y* + 1, −*z* + 1. For **13:** #1, −*x* + 1, −*y* + 1, −*z* + 1. For **14:** #1, −*x* + 1, −*y* + 1, −*z* + 1. For **15:** #1, −*x* + 1, −*y* + 1, −*z* + 1.

Reference

1. A. Panagiotopoulos, T. F. Zafiropoulos, S. P. Perlepes, E. Bakalbassis, I. Masson-Ramade, O. Kahn, A. Terzis and C. P. Raptopoulou, *Inorg. Chem.*, 1995, **34**, 4918-4920.