

Supporting information

**P-C Bond Cleavage-Assisted Lanthanide Phosphate
Coordination Polymers**

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Experimental Section

Reagents and general procedures

Solvents and other general reagents used in this work were purified according to standard procedures.⁶² Trimethyl phosphate (S.D. Fine India), 2,6-Bis-(hydroxymethyl)-*p*-cresol, Eu(NO₃)₃·5H₂O, Dy(NO₃)₃·5H₂O and Gd(NO₃)₃·6H₂O salt (Aldrich U.S.A.) were used as received. The phosphonic acid 4-methyl-2,6-bis-(phosphonomethyl) phenol (H₅mbpp⁶³) was prepared following a reported procedure.⁶³

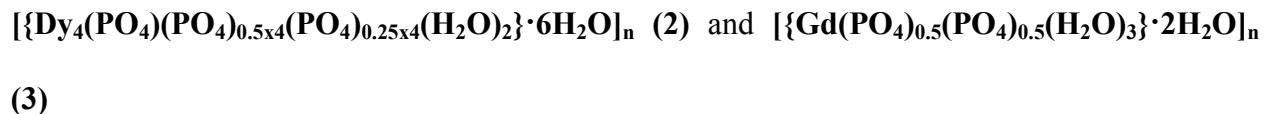
Instrumentation

¹H and ³¹P NMR spectra were recorded in D₂O solutions using a JEOL-JNM LAMBDA 400 model NMR spectrometer operating at 400.0 and 161.7 MHz respectively. Chemical shifts are referenced to tetramethylsilane. IR spectra were recorded as KBr pellets on a Bruker Vector 22 FT-IR spectrophotometer operating from 400-4000 cm⁻¹. ESI-MS spectra were recorded on a MICROMASS QUATTRO II triple quadrupole mass spectrometer. Elemental analyses of the compounds were obtained using a Thermoquest CE instrument CHNS-O, EA/110 model. Thermogravimetric analysis (heating rate of 10 °C/min) was carried out on a Perkin-Elmer Pyris 6 machine under argon atmosphere. All samples were subjected to variable temperature (2–300 K) magnetic susceptibility and variable field (0-7 T) magnetisation measurements. For compounds **2** and **3**, ac susceptibility measurements were also carried out using variable temperature and frequency. All measurements were performed using a Quantum Design MPMS-XL SQUID magnetometer at the University of Manchester. Diamagnetic corrections were calculated using Pascal's constants, and an experimental correction for the sample holder was applied.

Synthesis



An aqueous solution (5 mL) of 4-methyl-2,6-bis-(phosphonomethyl) phenol (H_5mbpp) (0.069 g, 0.233 mmol) was added to an aqueous solution (5 mL) of $\text{Eu}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ (1.000 g, 0.233 mmol) with constant stirring for 15 min at room temperature. The resultant reaction mixture was transferred into a teflon-lined stainless steel autoclave and was heated under autogenous pressure at 160 °C for two days. Colorless block-shaped crystals suitable for X-ray analysis were obtained overnight by cooling the vessel to room temperature (~30 °C). The crystalline compound of **1** was washed with water followed by diethyl ether and air-dried. Yield: 0.056 g, 60% (based on europium). $\text{Eu}_4\text{O}_{44}\text{P}_9\text{H}_{16}$ (1606.74). IR (KBr, cm^{-1}): 3265(br), 2853(w), 1640(m), 1434(s), 1118(s), 991(s), 750(m), 652(s), 616(s).



2 and **3** were prepared by using the same procedure as above using similar molar quantities. The lanthanide reagents used were $\text{Dy}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ and $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$. The isolated yields of **2** and **3** were 0.055 g, 57% (based on dysprosium) and 0.06 g, 59% (based on gadolinium) respectively.

$\text{Dy}_4\text{O}_{44}\text{P}_9\text{H}_{16}$ (1648.86). IR (KBr, cm^{-1}): 3444(br), 3276(s), 2098(w), 1629(s), 1426(s), 1177(s), 1088(s), 999(m), 969(w), 663(s), 631(s).

$\text{GdO}_{13}\text{P}_2\text{H}_{10}$ (437.27). IR (KBr, cm^{-1}): 3273(br), 2858(w), 1638(m), 1434(s), 1117(s), 1068(s), 992(m), 654(s), 618(s).

X-ray Crystallography

Single-crystal X-ray structural studies of **1-3** were performed using a CCD Bruker SMART APEX diffractometer equipped with an Oxford Instruments low-temperature attachment. Data were collected using graphite-monochromated MoK α radiation (λ_{α} = 0.71073 Å). The crystals did not degrade/decompose during data collection. Data collection, structure solution and refinement were performed using *SMART*, *SAINT* and *SHELXTL* programs respectively.⁶⁴⁻⁶⁹ All calculations for data reduction were done using the Bruker SADABS program. All the non-hydrogen atoms were refined anisotropically using full-matrix least-square procedures. All the hydrogen atoms were included in idealized positions and a riding model was used. All the molecular drawings were obtained from DIAMOND (version 3.1).CCDC Numbers for the Crystal data of **1-3**:1033450-1033452

Table S1. Crystal data and structure refinement parameters of **1**, **2** and **3**

| Compound | 1 | 2 | 3 |
|--------------------------------|---|---|--|
| Formula | $\text{Eu}_4\text{H}_{16}\text{O}_{44}\text{P}_9$ | $\text{Dy}_4\text{H}_{16}\text{O}_{44}\text{P}_9$ | $\text{GdH}_{10}\text{O}_{13}\text{P}_2$ |
| Formula weight | 1606.74 | 1648.86 | 437.27 |
| Temp. (K) | 100(2) | 100(2) | 100(2) |
| Crystal system | Monoclinic | Monoclinic | Monoclinic |
| Space group | $C2/c$ | $C2/c$ | $P2_1/c$ |
| Unit cell dimensions | a (Å) | 9.040(5) | 8.965(5) |
| | b (Å) | 18.306(5) | 18.187(5) |
| | c (Å) | 21.435(5) | 21.316(5) |
| | α (°) | 90 | 90 |
| | β (°) | 95.240(5) | 95.672(5) |
| | γ (°) | 90 | 90 |
| | Volume (Å ³); Z | 3532(2); 4 | 3458(2); 4 |
| Density(Mgm ⁻³) | 3.022 | 3.166 | 2.752 |
| Abs. coef. (mm ⁻¹) | 7.557 | 9.104 | 6.650 |
| $F(000)$ | 3020 | 3068 | 832 |
| Crystal size (mm) | 0.12 x 0.11 x 0.09 | 0.12 x 0.11 x 0.09 | 0.16 x 0.14 x 0.12 |
| θ range (°) | 1.91 to 25.50 | 2.24 to 25.50 | 2.60 to 25.50 |
| Limiting indices | $-10 \leq h \leq 10$ | $-18 \leq h \leq 10$ | $-7 \leq h \leq 7$ |
| | $-22 \leq k \leq 21$ | $-22 \leq k \leq 22$ | $-22 \leq k \leq 22$ |
| | $-25 \leq l \leq 16$ | $-25 \leq l \leq 25$ | $-7 \leq l \leq 10$ |

| | | | |
|--|--|--|--|
| Reflections collected | 9043 | 9086 | 7119 |
| Unique reflections [R _{int}] | 3182 [0.0323] | 3157 [0.0449] | 1964[0.0313] |
| Completeness to θ | 96.6 % (25.50°) | 98.0 % (25.50°) | 99.9 % (25.50°) |
| Data/restraints/parameters | 3182/1/274 | 3157/6/258 | 1964/30/130 |
| GOOF on F ² | 1.094 | 1.049 | 1.100 |
| Final R indices [I>2σ (I)] | R ₁ = 0.0257, wR ₂ = 0.0588 | R ₁ = 0.0327, wR ₂ = 0.0731 | R ₁ = 0.0546, wR ₂ = 0.1268 |
| R indices (all data) | R ₁ = 0.0281, wR ₂ = 0.0601 | R ₁ = 0.0424, wR ₂ = 0.0775 | R ₁ = 0.0569, wR ₂ = 0.1287 |
| Largest diff peak/hole (eÅ ⁻³) | 1.431 and -1.893 | 1.656 and -1.225 | 4.800 and -4.800 |

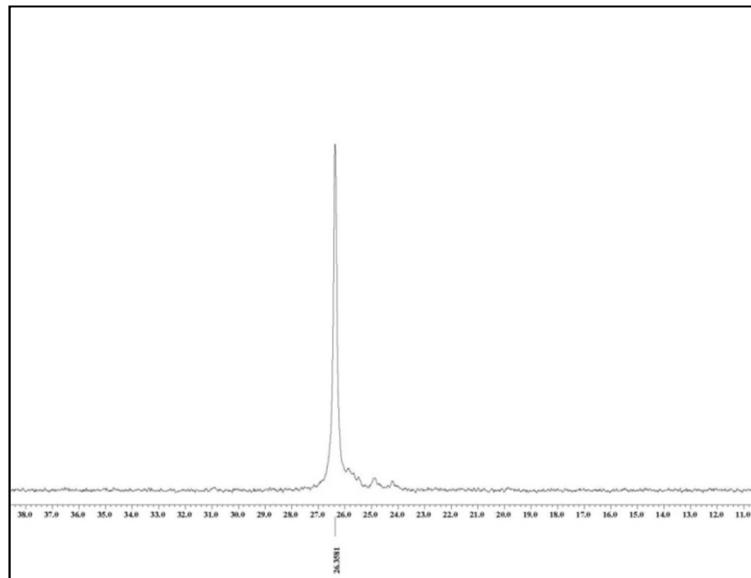
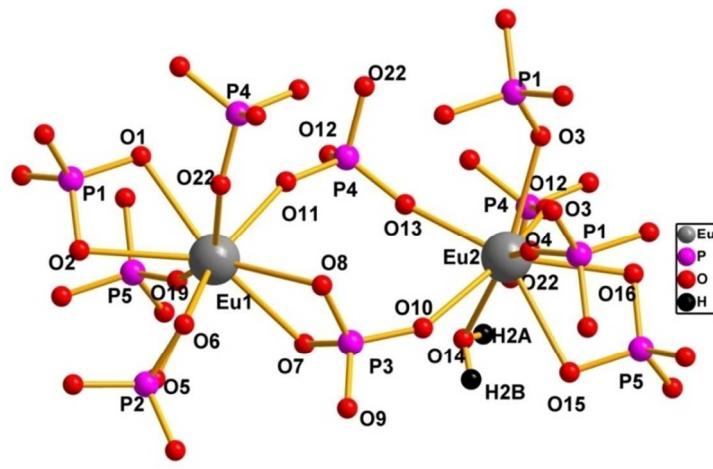


Figure S1. ^{31}P NMR (D_2O) spectrum of a blank reaction involving H_5mbpp under hydrothermal reaction conditions, indicating that the integrity of the ligand is preserved (in absence of the lanthanide metal salt)



(a)

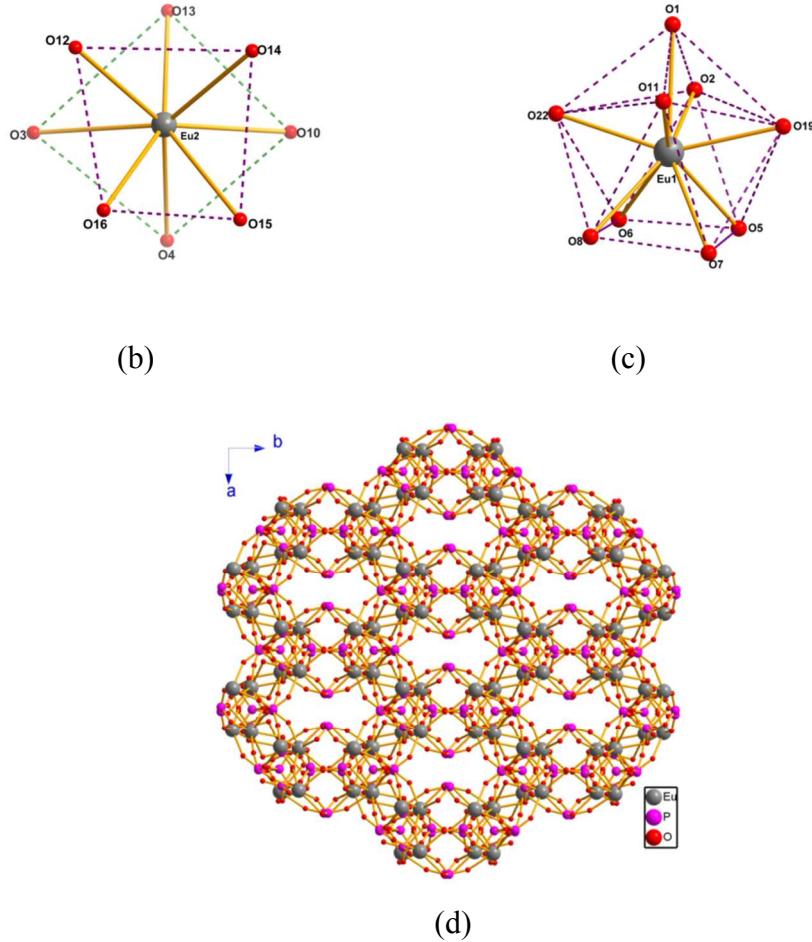


Figure S2. (a) Molecular structure of **1**. All the hydrogen atoms have been omitted for clarity. Important bond lengths (\AA) and bond angles (deg): Eu(1)-O(19), 2.301(7); Eu(1)-O(22), 2.345(6); Eu(1)-O(11), 2.365(7); Eu(1)-O(6), 2.436(7); Eu(1)-O(8), 2.456(6); Eu(1)-O(1), 2.492(7); Eu(1)-O(7), 2.521(7); Eu(1)-O(5), 2.568(6); Eu(1)-O(2), 2.638(7); Eu(2)-O(13), 2.302(6); Eu(2)-O(14), 2.321(7); Eu(2)-O(4)*, 2.349(6); Eu(2)-O(10), 2.375(7); Eu(2)-O(12)*, 2.404(7); Eu(2)-O(3)*, 2.453(6); Eu(2)-O(16), 2.470(7); Eu(2)-O(15), 2.569(7); O(19)-Eu(1)-O(22), 149.3(2); O(19)-Eu(1)-O(11), 80.0(2); O(22)-Eu(1)-O(11), 88.4(2); O(19)-Eu(1)-O(6), 127.4(2); O(22)-Eu(1)-O(6), 71.2(2); O(11)-Eu(1)-O(6), 151.5(2); O(19)-Eu(1)-O(8), 133.1(2); O(22)-Eu(1)-O(8), 72.3(2); O(11)-Eu(1)-O(8), 82.9(2); O(6)-Eu(1)-O(8), 72.2(2); O(19)-Eu(1)-O(1), 77.4(2); O(22)-Eu(1)-O(1), 72.0(2); O(11)-Eu(1)-O(1), 74.1(2); O(6)-Eu(1)-O(1), 115.8(2); O(8)-Eu(1)-O(1), 137.8(2); O(19)-Eu(1)-O(7), 77.9(2); O(22)-Eu(1)-O(7), 128.4(2); O(11)-Eu(1)-O(7), 80.5(2); O(6)-Eu(1)-O(7), 96.4(2); O(8)-Eu(1)-O(7), 56.3(2); O(1)-Eu(1)-O(7), 147.2(2); O(19)-Eu(1)-O(5), 72.7(2); O(22)-Eu(1)-O(5), 125.3(2); O(11)-Eu(1)-O(5), 146.0(2); O(6)-Eu(1)-O(5), 55.8(2); O(8)-Eu(1)-O(5), 101.1(2); O(1)-Eu(1)-O(5), 117.8(2); O(7)-Eu(1)-O(5), 74.3(2); O(19)-Eu(1)-O(2), 81.2(2); O(22)-Eu(1)-O(2), 83.9(2); O(11)-Eu(1)-O(2),

127.9(2); O(6)-Eu(1)-O(2), 70.9(2); O(8)-Eu(1)-O(2), 140.9(2); O(1)-Eu(1)-O(2), 54.5(2); O(7)-Eu(1)-O(2), 140.6(2); O(5)-Eu(1)-O(2), 67.7(2). (b) & (c) immediate coordination geometry around the metal center.(d) Interconnected three-dimensional polymeric structure of **1**.

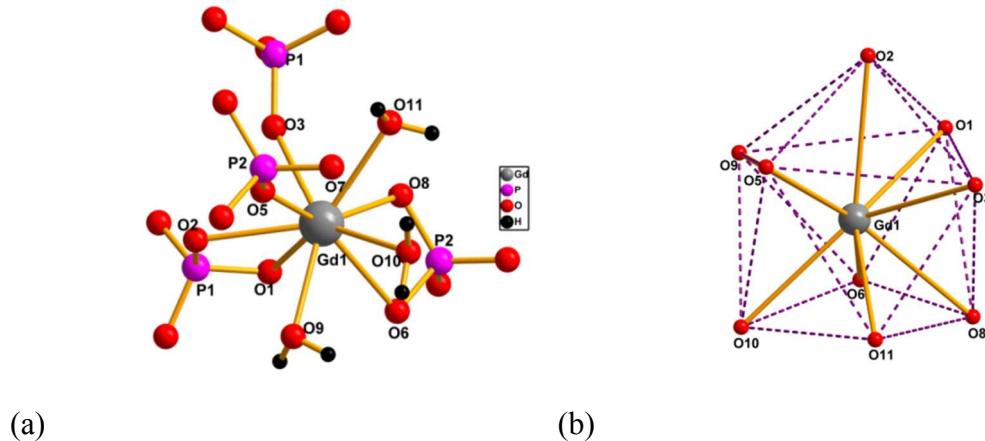


Figure S3. Molecular structure of **3**. All the hydrogen atoms have been omitted for clarity. Important bond lengths (\AA) and bond angles (deg): Gd(1)-O(3)*, 2.343(8); Gd(1)-O(5), 2.354(8); Gd(1)-O(10), 2.388(8); Gd(1)-O(9), 2.419(8); Gd(1)-O(8)*, 2.461(8); Gd(1)-O(1), 2.462(8); Gd(1)-O(11), 2.482(7); Gd(1)-O(2), 2.510(7); Gd(1)-O(6)*, 2.516(10); O(3)*-Gd(1)-O(5), 86.4(3); O(3)*-Gd(1)-O(10), 139.2(3); O(5)-Gd(1)-O(10), 82.4(3); O(3)*-Gd(1)-O(9), 142.5(3); O(5)-Gd(1)-O(9), 81.5(3); O(10)-Gd(1)-O(9), 74.1(3); O(3)*-Gd(1)-O(8)*, 75.5(3); O(5)-Gd(1)-O(8)*, 149.4(3); O(10)-Gd(1)-O(8)*, 95.2(3); O(9)-Gd(1)-O(8)*, 127.4(3); O(3)*-Gd(1)-O(1), 77.8(3); O(5)-Gd(1)-O(1), 125.7(3); O(10)-Gd(1)-O(1), 138.9(3); O(9)-Gd(1)-O(1), 80.8(3); O(8)*-Gd(1)-O(1), 74.8(3); O(3)*-Gd(1)-O(11), 71.4(3); O(5)-Gd(1)-O(11), 80.1(3); O(10)-Gd(1)-O(11), 68.1(3); O(9)-Gd(1)-O(11), 139.6(3); O(8)*-Gd(1)-O(11), 70.9(3); O(1)-Gd(1)-O(11), 138.3(3); O(3)*-Gd(1)-O(2), 70.2(3); O(5)-Gd(1)-O(2), 69.1(2); O(10)-Gd(1)-O(2), 138.4(3); O(9)-Gd(1)-O(2), 72.3(3); O(8)*-Gd(1)-O(2), 124.7(2); O(1)-Gd(1)-O(2), 56.7(2); O(11)-Gd(1)-O(2), 131.4(2); O(3)*-Gd(1)-O(6)*, 126.6(3); O(5)-Gd(1)-O(6)*, 146.9(3); O(10)-Gd(1)-O(6)*, 73.1(3); O(9)-Gd(1)-O(6)*, 70.7(3); O(8)*-Gd(1)-O(6)*, 57.1(3); O(1)-Gd(1)-O(6)*, 68.0(3); O(11)-Gd(1)-O(6)*, 109.8(3); O(2)-Gd(1)-O(6)*, 116.6(3). (b) Coordination geometry of the gadolinium ion in **3**.

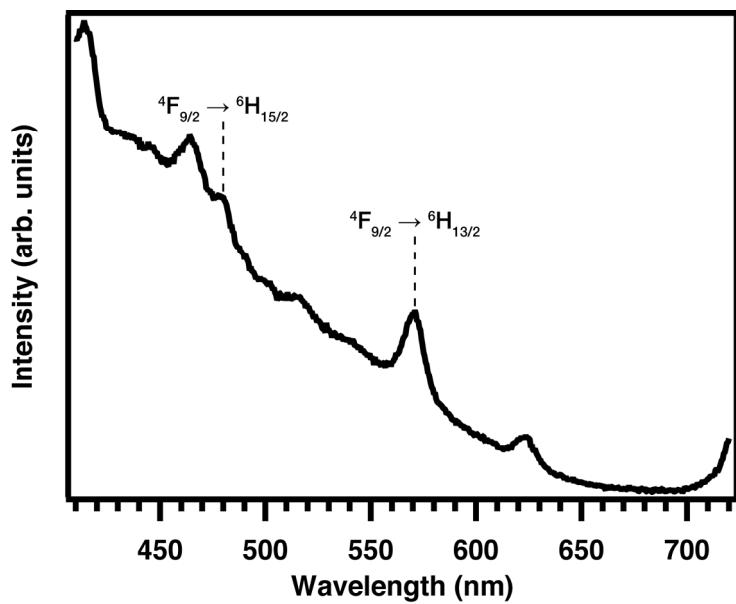


Figure S4. Room-temperature solid-state luminescence spectra of **2** (excited at 380 nm)

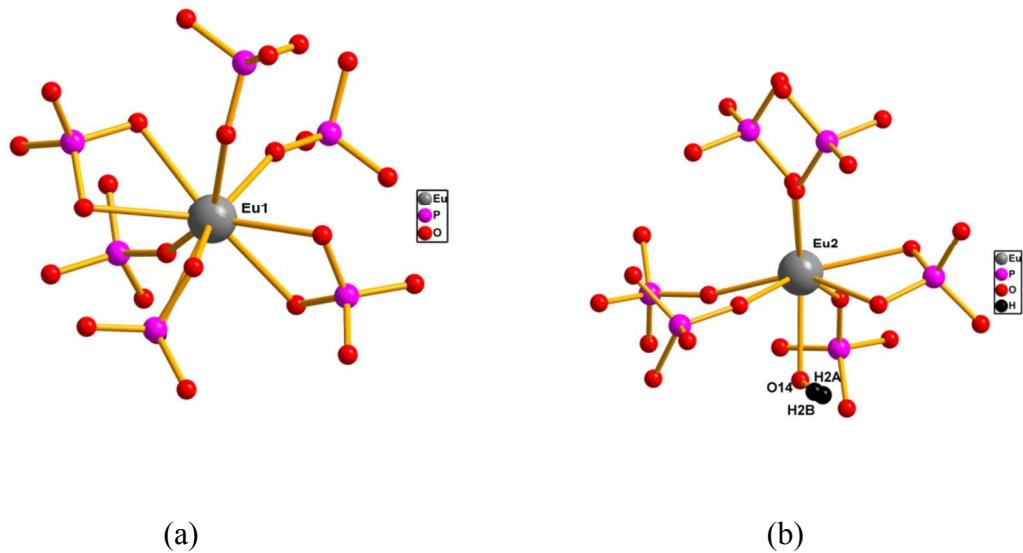


Figure S5. The coordination environment around lanthanide centers of compound **1**

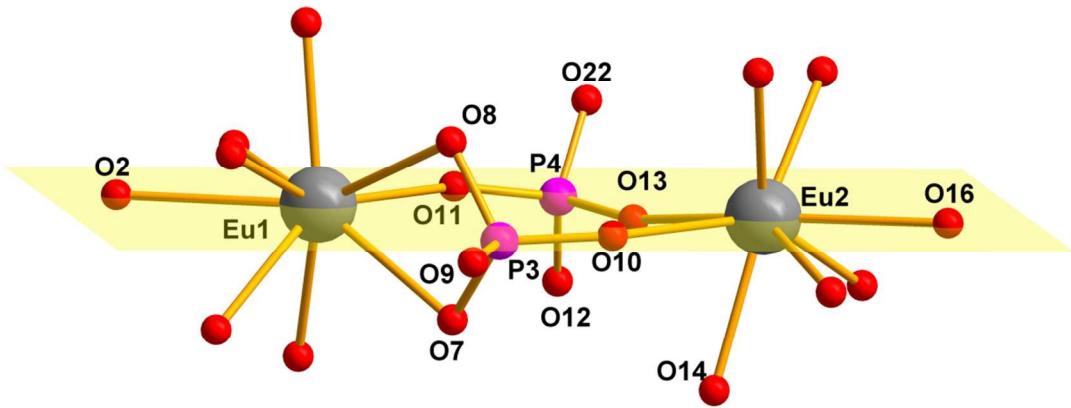


Figure S6. Mean plane of the dimeric motif in **1**

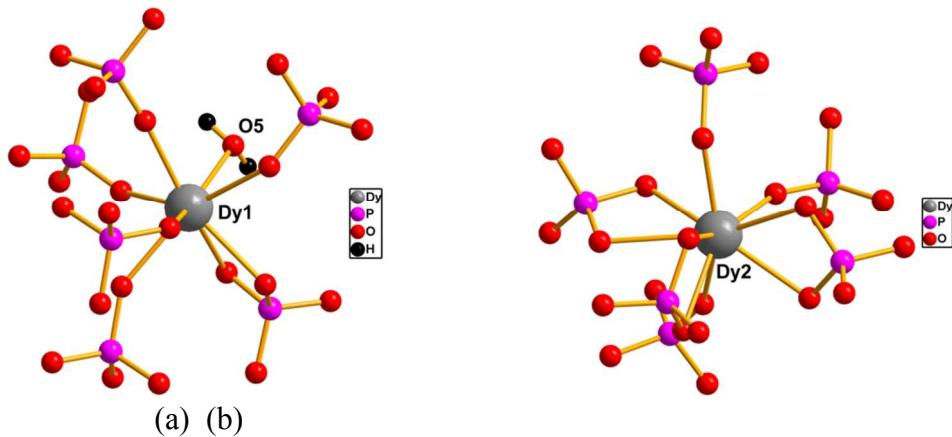


Figure S7. The coordination environment around the distinct lanthanide centers of compound **2**.

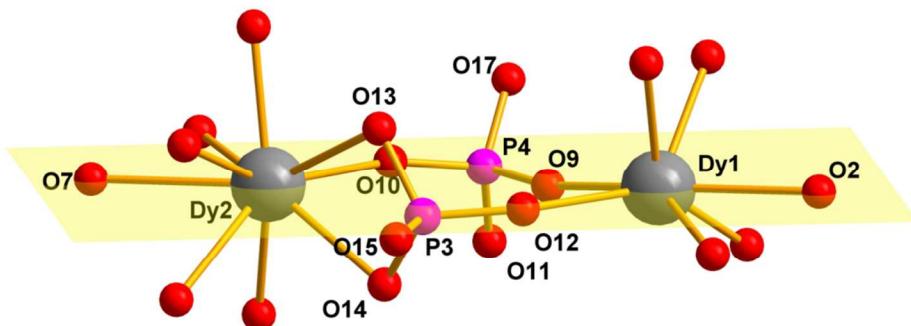


Figure S8. Mean plane of the dimeric motif **2**

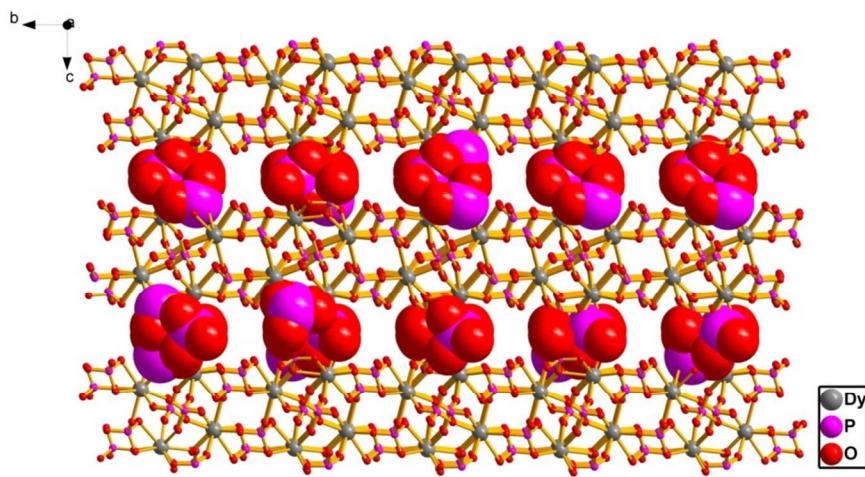


Figure S9. Layered architecture of **2** with each pair of layers being interconnected by bridging phosphate ligands

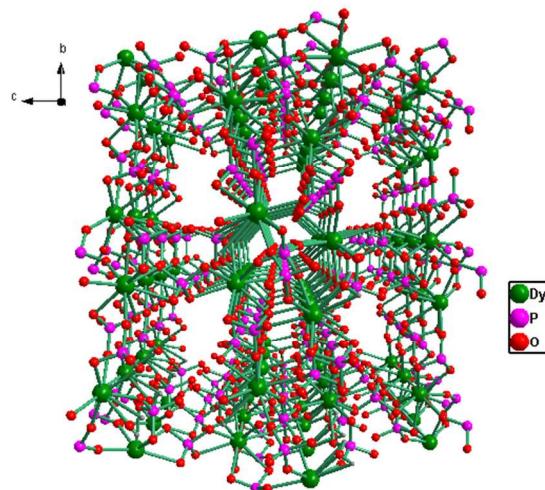


Figure S10. View down the axis of **2**

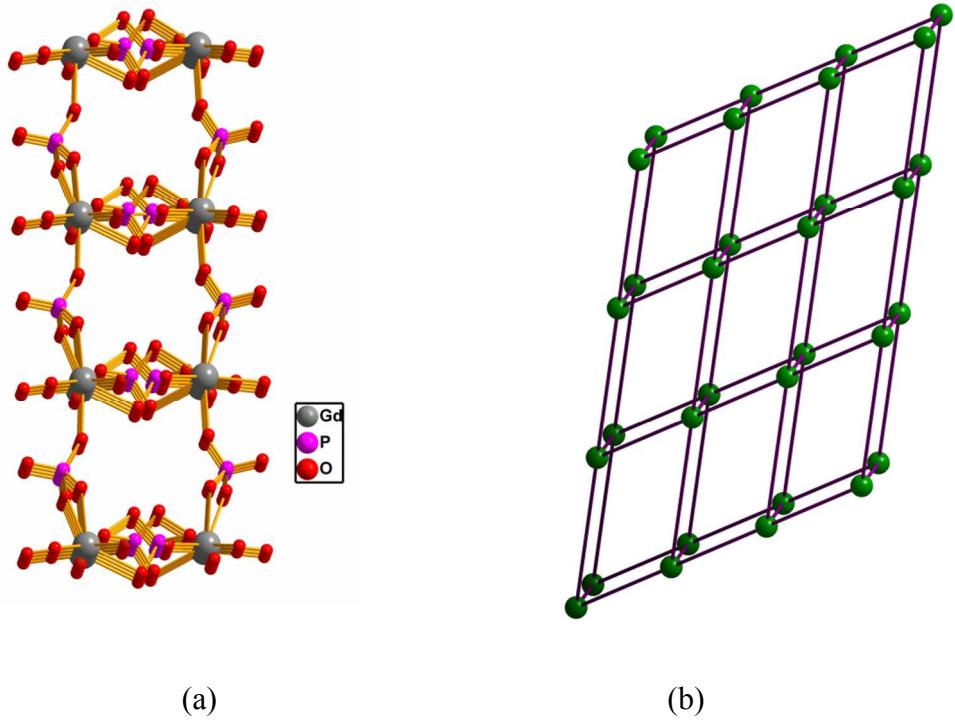


Figure S11.(a) the interconnected dinuclear motifs in **3** and (b) a view showing only the metal ions

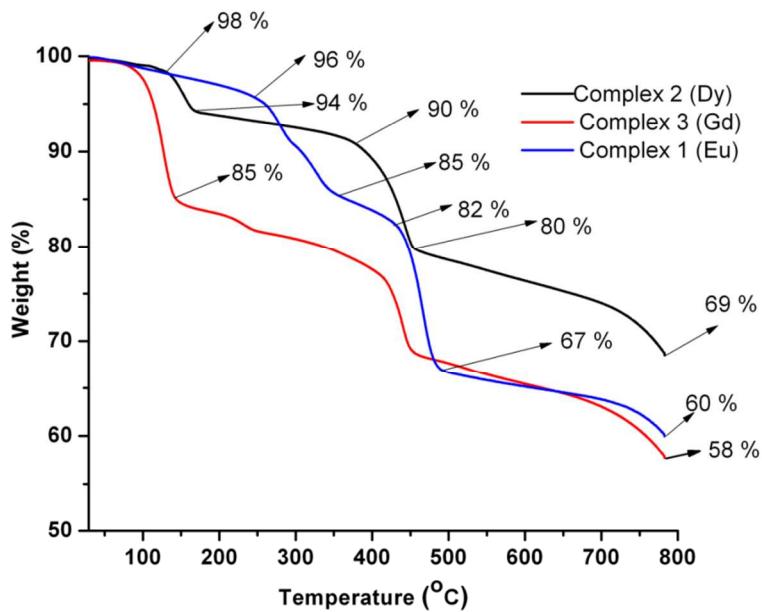


Figure S12. TGA curve for **1-3** (Heating rate: 10° per min)

TGA analysis

The TGA study of complex **1** shows mainly a three-step gradual weight loss process (Figure S12). The first event between 30-246 °C, corresponds to a small weight loss, ~4%, which is probably due to the loss of four non coordinated water molecule. The second step (246-356) °C corresponds to a further weight loss, ~11% , (loss of two phosphate ligands). The third step (431-490) °C corresponds to a further weight loss, ~15% , (loss of three phosphate ligands).The final char yield at 800 °C is 69%. The TGA of complex **2** also shows mainly a four-step gradual weight loss (Figure 6). The first step between 30-132 °C, corresponds to a weight loss of ~2% (loss of non-coordinated two water molecules). The second step (132-168 °C) corresponds to a weight loss ~4% (two noncoordinated and two coordinate water molecules). The third step at (381-457) °C corresponds to a weight loss ~10% (two phosphate ligands). At 800 °C the char yield is 60%. In the case of compound **3** the gradual weight loss from (30-143) °C and at 800 °C the final char yield is 58 %.

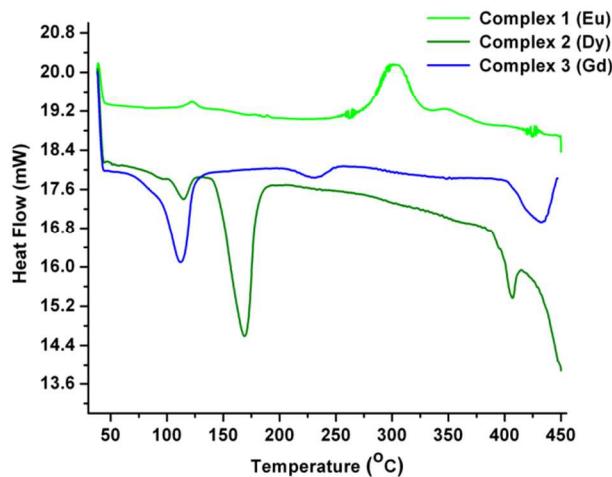


Figure S13. DSC curves for **1-3** (Heating rate: 10° per min)

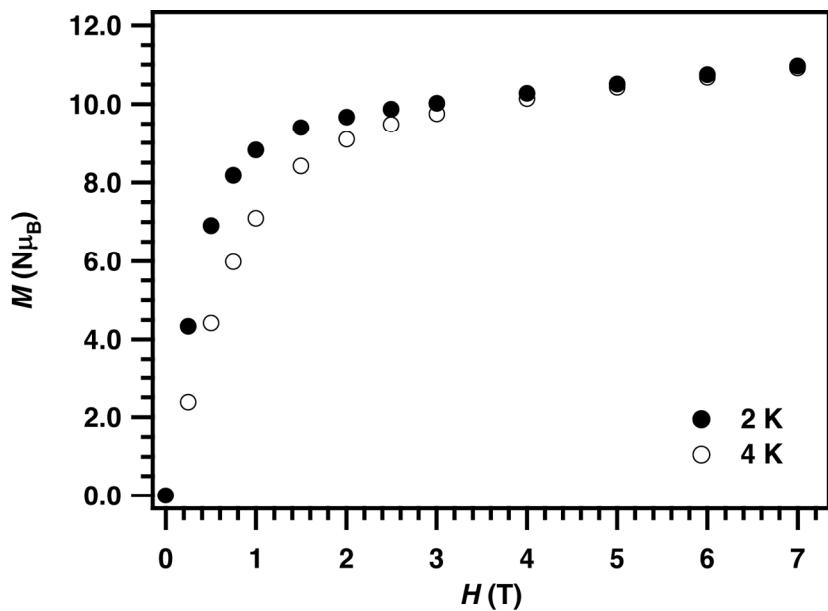


Figure S14. Field-dependent magnetization for compound 2

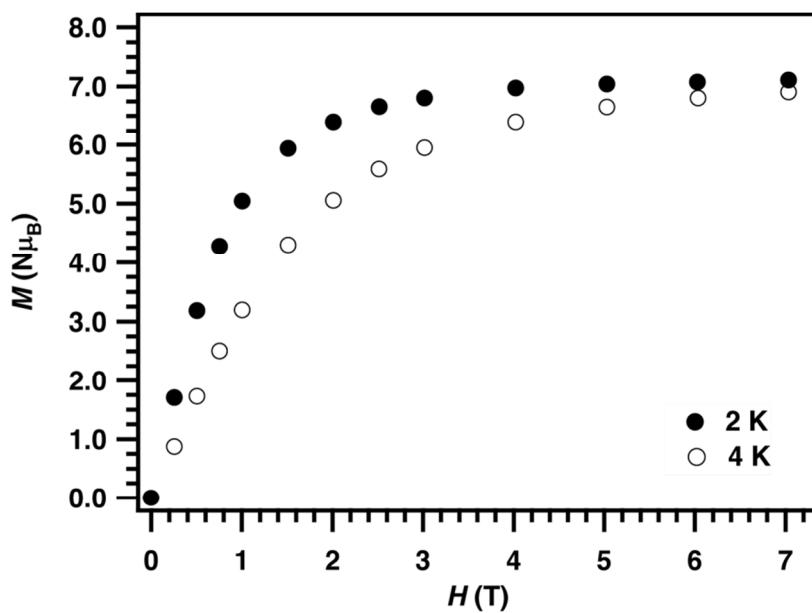


Figure S15. Field-dependent magnetization for compound 3

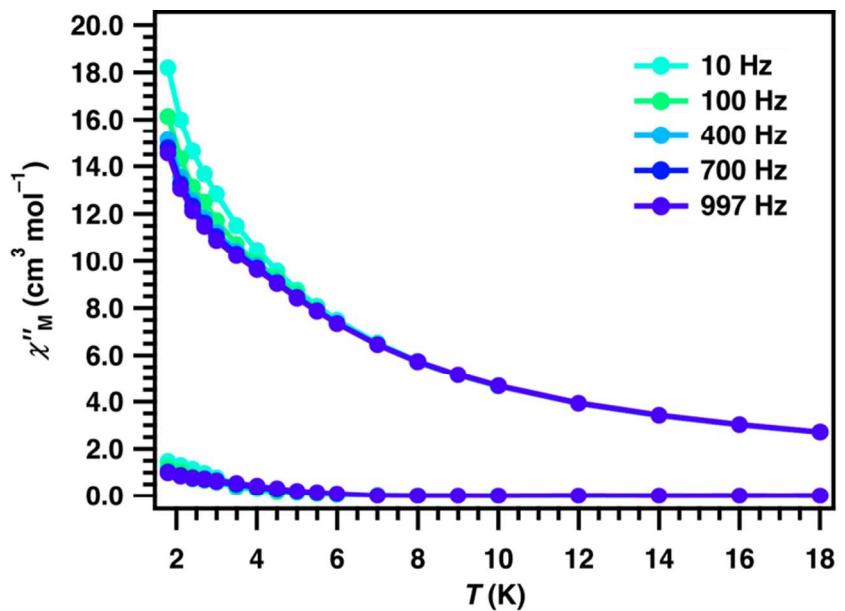


Figure S16. Measure of the in-phase (top) and out-of-phase (bottom) molar magnetic susceptibility for compound 3, measured under a static field of 0.1 T.

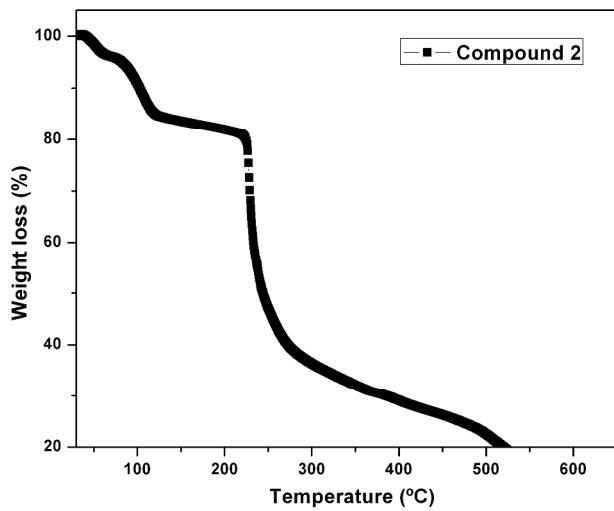


Figure S17. TGA curve for 2

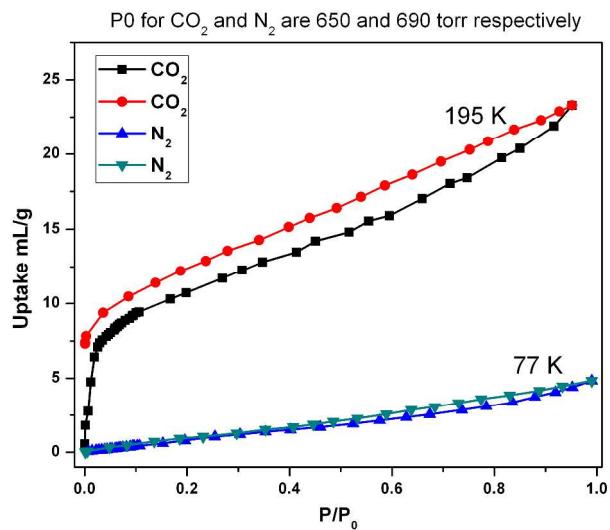
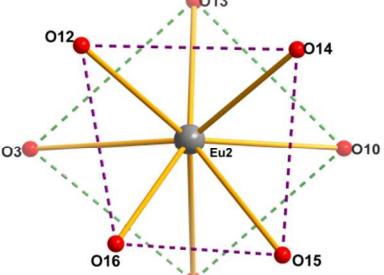


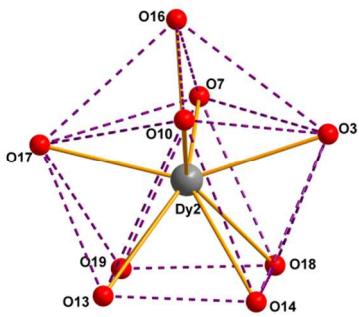
Figure 18. CO₂ and N₂ adsorption isotherms of compound **2**

Table S2. Coordination geometry around the lanthanide ions, bond distances(Å) and bond angles(°) of complexes **1-3**

| Coordination geometry | Bond lengths (Å) | Bond angles (°) | |
|---|---|--|---|
| | | | |
| Distorted monocapped square antiprismatic | Eu(1)-O(19) Eu(1)-O(22) Eu(1)-O(11) Eu(1)-O(6) Eu(1)-O(8) Eu(1)-O(1) Eu(1)-O(7) Eu(1)-O(5) Eu(1)-O(2) | 2.301(7) 2.345(6) 2.365(7) 2.436(7) 2.456(6) 2.492(7) 2.521(7) 2.568(6) 2.638(7) | O(19)-Eu(1)-O(22) O(19)-Eu(1)-O(11) O(22)-Eu(1)-O(11) O(19)-Eu(1)-O(6) O(22)-Eu(1)-O(6) O(11)-Eu(1)-O(6) O(19)-Eu(1)-O(8) O(22)-Eu(1)-O(8) O(11)-Eu(1)-O(8) |
| | | O(6)-Eu(1)-O(8) O(19)-Eu(1)-O(1) O(22)-Eu(1)-O(1) O(11)-Eu(1)-O(1) O(6)-Eu(1)-O(1) O(8)-Eu(1)-O(1) O(19)-Eu(1)-O(7) O(22)-Eu(1)-O(7) O(11)-Eu(1)-O(7) O(6)-Eu(1)-O(7) | 72.2(2) 77.4(2) 72.0(2) 74.1(2) 115.8(2) 137.8(2) 77.9(2) 128.4(2) 80.5(2) 96.4(2) |

| | | | |
|---|---|---|----------|
| | | O(8)-Eu(1)-O(7) | 56.3(2) |
| | | O(1)-Eu(1)-O(7) | 147.2(2) |
| | | O(19)-Eu(1)-O(5) | 72.7(2) |
| | | O(22)-Eu(1)-O(5) | 125.3(2) |
| | | O(11)-Eu(1)-O(5) | 146.0(2) |
| | | O(6)-Eu(1)-O(5) | 55.8(2) |
| | | O(8)-Eu(1)-O(5) | 101.1(2) |
| | | O(1)-Eu(1)-O(5) | 117.8(2) |
| | | O(7)-Eu(1)-O(5) | 74.3(2) |
| | | O(19)-Eu(1)-O(2) | 81.2(2) |
| | | O(22)-Eu(1)-O(2) | 83.9(2) |
| | | O(11)-Eu(1)-O(2) | 127.9(2) |
| | | O(6)-Eu(1)-O(2) | 70.9(2) |
| | | O(8)-Eu(1)-O(2) | 140.9(2) |
| | | O(1)-Eu(1)-O(2) | 54.5(2) |
| | | O(7)-Eu(1)-O(2) | 140.6(2) |
| | | O(5)-Eu(1)-O(2) | 67.7(2) |
|  Distorted square antiprismatic | Eu(2)-O(13) 2.302(6) Eu(2)-O(14) 2.321(7) Eu(2)-O(4)* 2.349(6) Eu(2)-O(10) 2.375(7) Eu(2)-O(12)* 2.404(7) Eu(2)-O(3)* 2.453(6) Eu(2)-O(16) 2.470(7) Eu(2)-O(15) 2.569(7) | O(13)-Eu(2)-O(14) 79.2(2) O(13)-Eu(2)-O(4)* 114.4(2) O(14)-Eu(2)-O(4)* 137.0(2) O(13)-Eu(2)-O(10) 78.3(2) O(14)-Eu(2)-O(10) 70.1(2) O(4)*-Eu(2)-O(10) 73.2(2) O(13)-Eu(2)-O(12)* 81.7(2) O(14)-Eu(2)-O(12)* 79.4(2) O(4)*-Eu(2)-O(12)* 140.6(2) O(10)-Eu(2)-O(12)* 146.0(2) O(13)-Eu(2)-O(3)* 81.4(2) O(14)-Eu(2)-O(3)* 147.2(2) O(4)*-Eu(2)-O(3)* 75.4(2) O(10)-Eu(2)-O(3)* 130.8(2) O(12)*-Eu(2)-O(3)* 71.8(2) O(13)-Eu(2)-O(16) 151.7(2) O(14)-Eu(2)-O(16) 102.8(2) O(4)*-Eu(2)-O(16) 83.7(2) | |

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| | Dy(1)-O(9) 2.266(5) Dy(1)-O(6) 2.319(4) Dy(1)-O(5) 2.329(5) Dy(1)-O(12) 2.350(5) Dy(1)-O(11)* 2.365(5) Dy(1)-O(8)* 2.403(4) Dy(1)-O(2) 2.436(5) Dy(1)-O(1) 2.532(5) | O(9)-Dy(1)-O(6) 111.95(18) O(9)-Dy(1)-O(5) 81.13(17) O(6)-Dy(1)-O(5) 137.88(17) O(9)-Dy(1)-O(12) 77.36(17) O(6)-Dy(1)-O(12) 73.36(16) O(5)-Dy(1)-O(12) 70.90(17) O(9)-Dy(1)-O(11)* 81.33(17) O(6)-Dy(1)-O(11)* 142.00(16) O(5)-Dy(1)-O(11)* 77.69(17) O(12)-Dy(1)-O(11) 144.21(16) O(9)-Dy(1)-O(8)* 80.22(16) O(6)-Dy(1)-O(8)* 74.22(16) O(5)-Dy(1)-O(8)* 147.48(17) O(12)-Dy(1)-O(8)* 129.52(16) O(11)*-Dy(1)-O(8)* 73.35(16) O(9)-Dy(1)-O(2) 152.04(18) O(6)-Dy(1)-O(2) 85.02(17) O(5)-Dy(1)-O(2) 101.43(17) O(12)-Dy(1)-O(2) 130.07(16) O(11)*-Dy(1)-O(2) 72.22(17) O(8)*-Dy(1)-O(2) 83.62(16) O(9)-Dy(1)-O(1) 146.99(16) O(6)-Dy(1)-O(1) 77.62(16) O(5)-Dy(1)-O(1) 72.50(17) O(12)-Dy(1)-O(1) 75.51(16) | |
| Distorted square antiprismatic | | | |

| | | |
|--|---|---|
| | | O(11)*-Dy(1)-O(1) 111.13(17) O(8)*-Dy(1)-O(1) 132.13(16) O(2)-Dy(1)-O(1) 55.86(16) |
|  <p>Distorted monocapped square antiprismatic</p> | Dy(2)-O(3)* 2.265(5) Dy(2)-O(17) 2.305(5) Dy(2)-O(10) 2.320(5) Dy(2)-O(19)* 2.406(5) Dy(2)-O(13) 2.431(5) Dy(2)-O(16) 2.454(5) Dy(2)-O(14) 2.475(5) Dy(2)-O(18) 2.535(4) Dy(2)-O(7)* 2.634(6) | O(3)*-Dy(2)-O(17) 148.34(17) O(3)*-Dy(2)-O(10) 79.72(17) O(17)-Dy(2)-O(10) 87.58(17) O(3)*-Dy(2)-O(19) 128.43(17) O(17)-Dy(2)-O(19)* 71.22(16) O(10)-Dy(2)-O(19) 150.91(16) O(3)*-Dy(2)-O(13) 133.23(17) O(17)-Dy(2)-O(13) 72.28(17) O(10)-Dy(2)-O(13) 82.16(17) O(19)*-Dy(2)-O(13) 72.68(17) O(3)*-Dy(2)-O(16) 76.32(16) O(17)-Dy(2)-O(16) 72.33(16) O(10)-Dy(2)-O(16) 74.23(17) O(19)*-Dy(2)-O(16) 115.66(17) O(13)-Dy(2)-O(16) 137.87(16) O(3)*-Dy(2)-O(14) 77.78(16) O(17)-Dy(2)-O(14) 128.73(16) O(10)-Dy(2)-O(14) 80.47(17) O(19)*-Dy(2)-O(14) 97.14(16) O(13)-Dy(2)-O(14) 56.81(16) O(16)-Dy(2)-O(14) 146.44(17) O(3)*-Dy(2)-O(18) 73.15(16) O(17)-Dy(2)-O(18) 125.71(16) O(10)-Dy(2)-O(18) 146.40(16) O(19)*-Dy(2)-O(18) 56.39(15) O(13)-Dy(2)-O(18) 102.22(16) O(16)-Dy(2)-O(18) 116.80(16) O(14)-Dy(2)-O(18) 74.78(16) O(3)*-Dy(2)-O(7)* 81.37(17) O(17)-Dy(2)-O(7)* 83.90(17) O(10)-Dy(2)-O(7)* 127.80(16) O(19)*-Dy(2)-O(7)* 70.87(16) |

| | | | | |
|--|---|---|--|--|
| | | O(13)-Dy(2)-O(7)* | 141.24(17) | |
| | | O(16)-Dy(2)-O(7)* | 54.10(17) | |
| | | O(14)-Dy(2)-O(7)* | 140.74(16) | |
| | | O(18)-Dy(2)-O(7)* | 67.46(15) | |
| | Gd(1)-O(3)* Gd(1)-O(5) Gd(1)-O(10) Gd(1)-O(9) Gd(1)-O(8)* Gd(1)-O(1) Gd(1)-O(11) Gd(1)-O(2) Gd(1)-O(6)* | 2.343(8) 2.354(8) 2.388(8) 2.419(8) 2.461(8) 2.462(8) 2.482(7) 2.510(7) 2.516(10) | O(3)*-Gd(1)-O(5) O(3)*-Gd(1)-O(10) O(5)-Gd(1)-O(10) O(3)*-Gd(1)-O(9) O(5)-Gd(1)-O(9) O(10)-Gd(1)-O(9) O(3)*-Gd(1)-O(8)* O(5)-Gd(1)-O(8)* O(10)-Gd(1)-O(8)* O(9)-Gd(1)-O(8)* O(3)*-Gd(1)-O(1) O(5)-Gd(1)-O(1) O(10)-Gd(1)-O(1) O(9)-Gd(1)-O(1) O(8)*-Gd(1)-O(1) O(3)*-Gd(1)-O(11) O(5)-Gd(1)-O(11) O(10)-Gd(1)-O(11) O(9)-Gd(1)-O(11) O(8)*-Gd(1)-O(11) O(1)-Gd(1)-O(11) O(3)*-Gd(1)-O(2) O(5)-Gd(1)-O(2) O(10)-Gd(1)-O(2) O(9)-Gd(1)-O(2) O(8)*-Gd(1)-O(2) O(1)-Gd(1)-O(2) O(11)-Gd(1)-O(2) O(3)*-Gd(1)-O(6)* O(5)-Gd(1)-O(6)* O(10)-Gd(1)-O(6)* | 86.4(3) 139.2(3) 82.4(3) 142.5(3) 81.5(3) 74.1(3) 75.5(3) 149.4(3) 95.2(3) 127.4(3) 77.8(3) 125.7(3) 138.9(3) 80.8(3) 74.8(3) 71.4(3) 80.1(3) 68.1(3) 139.6(3) 70.9(3) 138.3(3) 70.2(3) 69.1(2) 138.4(3) 72.3(3) 124.7(2) 56.7(2) 131.4(2) 126.6(3) 146.9(3) 73.1(3) |

| | |
|-------------------|----------|
| O(9)-Gd(1)-O(6)* | 70.7(3) |
| O(8)*-Gd(1)-O(6)* | 57.1(3) |
| O(1)-Gd(1)-O(6)* | 68.0(3) |
| O(11)-Gd(1)-O(6)* | 109.8(3) |
| O(2)-Gd(1)-O(6)* | 116.6(3) |

Table S3. Bond lengths (\AA) found in **1**

| | | | |
|--------------|----------|--------------|----------|
| Eu(1)-O(19) | 2.301(7) | P(1)-O(2) | 1.472(7) |
| Eu(1)-O(22) | 2.345(6) | P(1)-O(1) | 1.477(7) |
| Eu(1)-O(11) | 2.365(7) | P(1)-O(3) | 1.487(7) |
| Eu(1)-O(6) | 2.436(7) | P(5)-O(19) | 1.465(7) |
| Eu(1)-O(8) | 2.456(6) | P(5)-O(20) | 1.468(7) |
| Eu(1)-O(1) | 2.492(7) | P(5)-O(15)* | 1.473(7) |
| Eu(1)-O(7) | 2.521(7) | P(5)-O(16)* | 1.494(7) |
| Eu(1)-O(5) | 2.568(6) | P(3)-O(10) | 1.463(7) |
| Eu(1)-O(2) | 2.638(7) | P(3)-O(9) | 1.467(7) |
| Eu(2)-O(13) | 2.302(6) | P(3)-O(7) | 1.490(7) |
| Eu(2)-O(14) | 2.321(7) | P(3)-O(8) | 1.492(7) |
| Eu(2)-O(4)* | 2.349(6) | P(2)-O(5) | 1.474(7) |
| Eu(2)-O(10) | 2.375(7) | P(2)-O(5)* | 1.474(7) |
| Eu(2)-O(12)* | 2.404(7) | P(2)-O(6)* | 1.482(7) |
| Eu(2)-O(3)* | 2.453(6) | P(2)-O(6) | 1.482(7) |
| Eu(2)-O(16) | 2.470(7) | O(22)-P(4)* | 1.479(7) |
| Eu(2)-O(15) | 2.569(7) | O(12)-Eu(2)* | 2.404(7) |
| P(4)-O(11) | 1.462(7) | O(15)-P(5)* | 1.473(7) |
| P(4)-O(13) | 1.467(7) | O(16)-P(5)* | 1.494(7) |
| P(4)-O(12) | 1.477(7) | O(4)-Eu(2)* | 2.349(6) |
| P(4)-O(22)* | 1.479(7) | O(3)-Eu(2)* | 2.453(6) |
| P(1)-O(4) | 1.471(7) | | |

Table S4. Bond angles ($^{\circ}$) found in **1**

| | | | |
|-------------------|----------|--------------------|------------|
| O(19)-Eu(1)-O(22) | 149.3(2) | O(3)*-Eu(2)-O(15) | 132.0(2) |
| O(19)-Eu(1)-O(11) | 80.0(2) | O(16)-Eu(2)-O(15) | 55.4(2) |
| O(22)-Eu(1)-O(11) | 88.4(2) | O(13)-Eu(2)-P(5)* | 165.47(17) |
| O(19)-Eu(1)-O(6) | 127.4(2) | O(14)-Eu(2)-P(5)* | 87.33(18) |
| O(22)-Eu(1)-O(6) | 71.2(2) | O(4)*-Eu(2)-P(5)* | 79.35(17) |
| O(11)-Eu(1)-O(6) | 151.5(2) | O(10)-Eu(2)-P(5)* | 102.44(16) |
| O(19)-Eu(1)-O(8) | 133.1(2) | O(12)*-Eu(2)-P(5)* | 90.52(17) |
| O(22)-Eu(1)-O(8) | 72.3(2) | O(3)*-Eu(2)-P(5)* | 107.88(15) |
| O(11)-Eu(1)-O(8) | 82.9(2) | O(16)-Eu(2)-P(5)* | 27.76(16) |
| O(6)-Eu(1)-O(8) | 72.2(2) | O(15)-Eu(2)-P(5)* | 27.67(16) |
| O(19)-Eu(1)-O(1) | 77.4(2) | O(13)-Eu(2)-P(1)* | 75.46(17) |
| O(22)-Eu(1)-O(1) | 72.0(2) | O(14)-Eu(2)-P(1)* | 153.80(18) |
| O(11)-Eu(1)-O(1) | 74.1(2) | O(4)*-Eu(2)-P(1)* | 62.31(16) |
| O(6)-Eu(1)-O(1) | 115.8(2) | O(10)-Eu(2)-P(1)* | 110.64(16) |
| O(8)-Eu(1)-O(1) | 137.8(2) | O(12)*-Eu(2)-P(1)* | 90.21(16) |
| O(19)-Eu(1)-O(7) | 77.9(2) | O(3)*-Eu(2)-P(1)* | 20.19(15) |
| O(22)-Eu(1)-O(7) | 128.4(2) | O(16)-Eu(2)-P(1)* | 96.42(15) |
| O(11)-Eu(1)-O(7) | 80.5(2) | O(15)-Eu(2)-P(1)* | 133.84(16) |
| O(6)-Eu(1)-O(7) | 96.4(2) | P(5)*-Eu(2)-P(1)* | 117.02(6) |
| O(8)-Eu(1)-O(7) | 56.3(2) | O(11)-P(4)-O(13) | 110.0(4) |
| O(1)-Eu(1)-O(7) | 147.2(2) | O(11)-P(4)-O(12) | 109.1(4) |
| O(19)-Eu(1)-O(5) | 72.7(2) | O(13)-P(4)-O(12) | 109.8(4) |
| O(22)-Eu(1)-O(5) | 125.3(2) | O(11)-P(4)-O(22)* | 109.7(4) |
| O(11)-Eu(1)-O(5) | 146.0(2) | O(13)-P(4)-O(22)* | 110.4(4) |
| O(6)-Eu(1)-O(5) | 55.8(2) | O(12)-P(4)-O(22)* | 107.8(4) |
| O(8)-Eu(1)-O(5) | 101.1(2) | O(4)-P(1)-O(2) | 111.4(4) |
| O(1)-Eu(1)-O(5) | 117.8(2) | O(4)-P(1)-O(1) | 111.2(4) |
| O(7)-Eu(1)-O(5) | 74.3(2) | O(2)-P(1)-O(1) | 105.7(4) |
| O(19)-Eu(1)-O(2) | 81.2(2) | O(4)-P(1)-O(3) | 108.1(4) |
| O(22)-Eu(1)-O(2) | 83.9(2) | O(2)-P(1)-O(3) | 110.0(4) |
| O(11)-Eu(1)-O(2) | 127.9(2) | O(1)-P(1)-O(3) | 110.4(4) |
| O(6)-Eu(1)-O(2) | 70.9(2) | O(4)-P(1)-Eu(1) | 127.3(3) |
| O(8)-Eu(1)-O(2) | 140.9(2) | O(2)-P(1)-Eu(1) | 55.7(3) |

| | | | |
|--------------------|------------|--------------------|----------|
| O(1)-Eu(1)-O(2) | 54.5(2) | O(1)-P(1)-Eu(1) | 50.0(3) |
| O(7)-Eu(1)-O(2) | 140.6(2) | O(3)-P(1)-Eu(1) | 124.6(3) |
| O(5)-Eu(1)-O(2) | 67.7(2) | O(4)-P(1)-Eu(2)* | 107.5(3) |
| O(19)-Eu(1)-P(3) | 106.18(17) | O(2)-P(1)-Eu(2)* | 135.2(3) |
| O(22)-Eu(1)-P(3) | 100.54(17) | O(1)-P(1)-Eu(2)* | 79.1(3) |
| O(11)-Eu(1)-P(3) | 83.44(17) | O(3)-P(1)-Eu(2)* | 34.7(3) |
| O(6)-Eu(1)-P(3) | 81.09(17) | O(19)-P(5)-O(20) | 107.9(4) |
| O(8)-Eu(1)-P(3) | 28.21(16) | O(19)-P(5)-O(15)* | 111.7(4) |
| O(1)-Eu(1)-P(3) | 156.40(16) | O(20)-P(5)-O(15)* | 111.3(4) |
| O(7)-Eu(1)-P(3) | 28.40(15) | O(19)-P(5)-O(16)* | 110.6(4) |
| O(5)-Eu(1)-P(3) | 85.07(15) | O(20)-P(5)-O(16)* | 111.0(4) |
| O(2)-Eu(1)-P(3) | 148.62(15) | O(15)*-P(5)-O(16)* | 104.4(4) |
| O(19)-Eu(1)-P(2) | 99.73(19) | O(19)-P(5)-Eu(2)* | 125.9(3) |
| O(22)-Eu(1)-P(2) | 97.57(17) | O(20)-P(5)-Eu(2)* | 126.1(3) |
| O(11)-Eu(1)-P(2) | 167.46(16) | O(15)*-P(5)-Eu(2)* | 54.1(3) |
| O(6)-Eu(1)-P(2) | 27.85(16) | O(16)*-P(5)-Eu(2)* | 50.3(3) |
| O(8)-Eu(1)-P(2) | 88.36(18) | O(10)-P(3)-O(9) | 109.4(4) |
| O(1)-Eu(1)-P(2) | 118.17(16) | O(10)-P(3)-O(7) | 112.3(4) |
| O(7)-Eu(1)-P(2) | 87.14(16) | O(9)-P(3)-O(7) | 111.2(4) |
| O(5)-Eu(1)-P(2) | 28.15(15) | O(10)-P(3)-O(8) | 110.3(4) |
| O(2)-Eu(1)-P(2) | 63.99(15) | O(9)-P(3)-O(8) | 109.6(4) |
| O(19)-Eu(1)-P(1) | 78.08(17) | O(7)-P(3)-O(8) | 104.0(4) |
| O(22)-Eu(1)-P(1) | 76.33(17) | O(10)-P(3)-Eu(1) | 132.8(3) |
| O(11)-Eu(1)-P(1) | 100.85(17) | O(9)-P(3)-Eu(1) | 117.7(3) |
| O(6)-Eu(1)-P(1) | 93.60(17) | O(7)-P(3)-Eu(1) | 53.6(3) |
| O(8)-Eu(1)-P(1) | 148.33(16) | O(8)-P(3)-Eu(1) | 51.0(2) |
| O(1)-Eu(1)-P(1) | 27.00(16) | O(5)-P(2)-O(5)* | 113.7(6) |
| O(7)-Eu(1)-P(1) | 155.26(15) | O(5)-P(2)-O(6)* | 111.4(4) |
| O(5)-Eu(1)-P(1) | 93.06(15) | O(5)*-P(2)-O(6)* | 104.9(4) |
| O(2)-Eu(1)-P(1) | 27.46(15) | O(5)-P(2)-O(6) | 104.9(4) |
| O(13)-Eu(2)-O(14) | 79.2(2) | O(5)*-P(2)-O(6) | 111.4(4) |
| O(13)-Eu(2)-O(4)* | 114.4(2) | O(6)*-P(2)-O(6) | 110.5(6) |
| O(14)-Eu(2)-O(4)#1 | 137.0(2) | O(5)-P(2)-Eu(1) | 55.3(2) |
| O(13)-Eu(2)-O(10) | 78.3(2) | O(5)*-P(2)-Eu(1) | 122.9(3) |
| O(14)-Eu(2)-O(10) | 70.1(2) | O(6)*-P(2)-Eu(1) | 131.9(3) |

| | | | |
|--------------------|----------|-------------------|----------|
| O(4)*-Eu(2)-O(10) | 73.2(2) | O(6)-P(2)-Eu(1) | 50.2(3) |
| O(13)-Eu(2)-O(12)* | 81.7(2) | O(5)-P(2)-Eu(1)* | 122.9(3) |
| O(14)-Eu(2)-O(12)* | 79.4(2) | O(5)*-P(2)-Eu(1)* | 55.3(2) |
| O(4)*-Eu(2)-O(12)* | 140.6(2) | O(6)*-P(2)-Eu(1)* | 50.2(3) |
| O(10)-Eu(2)-O(12)* | 146.0(2) | O(6)-P(2)-Eu(1)* | 131.9(3) |
| O(13)-Eu(2)-O(3)* | 81.4(2) | P(4)*-O(22)-Eu(1) | 144.1(4) |
| O(14)-Eu(2)-O(3)* | 147.2(2) | P(3)-O(8)-Eu(1) | 100.7(3) |
| O(4)*-Eu(2)-O(3)* | 75.4(2) | P(3)-O(7)-Eu(1) | 98.0(3) |
| O(10)-Eu(2)-O(3)* | 130.8(2) | P(1)-O(1)-Eu(1) | 103.0(4) |
| O(12)*-Eu(2)-O(3)* | 71.8(2) | P(2)-O(6)-Eu(1) | 102.0(3) |
| O(13)-Eu(2)-O(16) | 151.7(2) | P(1)-O(2)-Eu(1) | 96.8(3) |
| O(14)-Eu(2)-O(16) | 102.8(2) | P(5)-O(19)-Eu(1) | 156.1(4) |
| O(4)*-Eu(2)-O(16) | 83.7(2) | P(4)-O(12)-Eu(2)* | 141.7(4) |
| O(10)-Eu(2)-O(16) | 129.3(2) | P(5)*-O(15)-Eu(2) | 98.2(3) |
| O(12)*-Eu(2)-O(16) | 71.2(2) | P(5)*-O(16)-Eu(2) | 101.9(3) |
| O(3)*-Eu(2)-O(16) | 82.7(2) | P(4)-O(13)-Eu(2) | 166.8(4) |
| O(13)-Eu(2)-O(15) | 146.4(2) | P(1)-O(4)-Eu(2)* | 150.2(4) |
| O(14)-Eu(2)-O(15) | 72.3(2) | P(1)-O(3)-Eu(2)* | 125.1(4) |
| O(4)*-Eu(2)-O(15) | 77.5(2) | P(2)-O(5)-Eu(1) | 96.6(3) |
| O(10)-Eu(2)-O(15) | 75.5(2) | P(3)-O(10)-Eu(2) | 150.5(4) |
| O(12)*-Eu(2)-O(15) | 109.6(2) | P(4)-O(11)-Eu(1) | 158.1(4) |

Table S5. Bond lengths (\AA) found in 2

| | | | |
|--------------|----------|--------------|------------|
| Dy(1)-O(9) | 2.266(5) | P(2)-O(8) | 1.478(5) |
| Dy(1)-O(6) | 2.319(4) | P(2)-O(16)* | 1.489(5) |
| Dy(1)-O(5) | 2.329(5) | P(2)-Dy(2)* | 3.1709(19) |
| Dy(1)-O(12) | 2.350(5) | P(3)-O(15) | 1.462(5) |
| Dy(1)-O(11)* | 2.365(5) | P(3)-O(12) | 1.464(5) |
| Dy(1)-O(8)* | 2.403(4) | P(3)-O(13) | 1.484(5) |
| Dy(1)-O(2) | 2.436(5) | P(3)-O(14) | 1.495(5) |
| Dy(1)-O(1) | 2.532(5) | P(4)-O(9) | 1.460(5) |
| Dy(2)-O(3)* | 2.265(5) | P(4)-O(10) | 1.460(5) |
| Dy(2)-O(17) | 2.305(5) | P(4)-O(11) | 1.472(5) |
| Dy(2)-O(10) | 2.320(5) | P(4)-O(17)* | 1.487(5) |
| Dy(2)-O(19)* | 2.406(5) | P(5)-O(18) | 1.469(5) |
| Dy(2)-O(13) | 2.431(5) | P(5)-O(18)* | 1.469(5) |
| Dy(2)-O(16) | 2.454(5) | P(5)-O(19) | 1.484(5) |
| Dy(2)-O(14) | 2.475(5) | P(5)-O(19)* | 1.484(5) |
| Dy(2)-O(18) | 2.535(4) | O(8)-Dy(1)* | 2.403(4) |
| Dy(2)-O(7)* | 2.634(6) | O(7)-Dy(2)* | 2.634(6) |
| P(1)-O(4) | 1.461(5) | O(11)-Dy(1)* | 2.365(5) |
| P(1)-O(3) | 1.465(5) | O(16)-P(2)* | 1.488(5) |
| P(1)-O(1) | 1.470(5) | O(17)-P(4)* | 1.487(5) |
| P(1)-O(2) | 1.484(5) | O(19)-Dy(2)* | 2.406(5) |
| P(2)-O(7) | 1.455(5) | O(3)-Dy(2)* | 2.265(5) |
| P(2)-O(6) | 1.465(5) | | |

Table S6. Bond angles ($^{\circ}$) found in 2

| | | | |
|--------------------|------------|--------------------|------------|
| O(9)-Dy(1)-O(6) | 111.95(18) | O(19)*-Dy(2)-P(5) | 28.21(11) |
| O(9)-Dy(1)-O(5) | 81.13(17) | O(13)-Dy(2)-P(5) | 89.10(13) |
| O(6)-Dy(1)-O(5) | 137.88(17) | O(16)-Dy(2)-P(5) | 117.80(13) |
| O(9)-Dy(1)-O(12) | 77.36(17) | O(14)-Dy(2)-P(5) | 87.63(12) |
| O(6)-Dy(1)-O(12) | 73.36(16) | O(18)-Dy(2)-P(5) | 28.35(11) |
| O(5)-Dy(1)-O(12) | 70.90(17) | O(7)*-Dy(2)-P(5) | 63.92(12) |
| O(9)-Dy(1)-O(11)* | 81.33(17) | P(3)-Dy(2)-P(5) | 85.16(5) |
| O(6)-Dy(1)-O(11)* | 142.00(16) | O(3)*-Dy(2)-P(2)* | 78.07(13) |
| O(5)-Dy(1)-O(11)* | 77.69(17) | O(17)-Dy(2)-P(2)* | 76.10(12) |
| O(12)-Dy(1)-O(11)* | 144.21(16) | O(10)-Dy(2)-P(2)* | 101.11(12) |
| O(9)-Dy(1)-O(8)* | 80.22(16) | O(19)*-Dy(2)-P(2)* | 93.07(12) |
| O(6)-Dy(1)-O(8)* | 74.22(16) | O(13)-Dy(2)-P(2)* | 148.05(12) |
| O(5)-Dy(1)-O(8)* | 147.48(17) | O(16)-Dy(2)-P(2)* | 27.04(13) |
| O(12)-Dy(1)-O(8)* | 129.52(16) | O(14)-Dy(2)-P(2)* | 155.09(11) |
| O(11)*-Dy(1)-O(8)* | 73.35(16) | O(18)-Dy(2)-P(2)* | 92.43(11) |
| O(9)-Dy(1)-O(2) | 152.04(18) | O(7)*-Dy(2)-P(2)* | 27.07(11) |
| O(6)-Dy(1)-O(2) | 85.02(17) | O(4)-P(1)-O(3) | 107.2(3) |
| O(5)-Dy(1)-O(2) | 101.43(17) | O(4)-P(1)-O(1) | 110.8(3) |
| O(12)-Dy(1)-O(2) | 130.07(16) | O(3)-P(1)-O(1) | 112.0(3) |
| O(11)*-Dy(1)-O(2) | 72.22(17) | O(4)-P(1)-O(2) | 111.5(3) |
| O(8)*-Dy(1)-O(2) | 83.62(16) | O(3)-P(1)-O(2) | 111.4(3) |
| O(9)-Dy(1)-O(1) | 146.99(16) | O(1)-P(1)-O(2) | 104.0(3) |
| O(6)-Dy(1)-O(1) | 77.62(16) | O(4)-P(1)-Dy(1) | 126.1(2) |
| O(5)-Dy(1)-O(1) | 72.50(17) | O(3)-P(1)-Dy(1) | 126.6(2) |
| O(12)-Dy(1)-O(1) | 75.51(16) | O(1)-P(1)-Dy(1) | 53.85(19) |
| O(11)*-Dy(1)-O(1) | 111.13(17) | O(2)-P(1)-Dy(1) | 50.16(19) |
| O(8)*-Dy(1)-O(1) | 132.13(16) | O(7)-P(2)-O(6) | 112.4(3) |
| O(2)-Dy(1)-O(1) | 55.86(16) | O(7)-P(2)-O(8) | 111.5(3) |
| O(9)-Dy(1)-P(1) | 167.12(13) | O(6)-P(2)-O(8) | 108.1(3) |
| O(6)-Dy(1)-P(1) | 80.10(12) | O(7)-P(2)-O(16)* | 104.0(3) |
| O(5)-Dy(1)-P(1) | 86.77(13) | O(6)-P(2)-O(16)* | 110.7(3) |
| O(12)-Dy(1)-P(1) | 102.88(11) | O(8)-P(2)-O(16)* | 110.1(3) |
| O(11)*-Dy(1)-P(1) | 91.83(12) | O(7)-P(2)-Dy(2)* | 55.5(2) |
| O(8)*-Dy(1)-P(1) | 108.39(11) | O(6)-P(2)-Dy(2)* | 128.0(2) |

| | | | |
|--------------------|------------|--------------------|------------|
| O(2)-Dy(1)-P(1) | 27.90(12) | O(8)-P(2)-Dy(2)* | 123.6(2) |
| O(1)-Dy(1)-P(1) | 27.96(12) | O(16)*-P(2)-Dy(2)* | 48.5(2) |
| O(3)*-Dy(2)-O(17) | 148.34(17) | O(15)-P(3)-O(12) | 109.5(3) |
| O(3)*-Dy(2)-O(10) | 79.72(17) | O(15)-P(3)-O(13) | 110.3(3) |
| O(17)-Dy(2)-O(10) | 87.58(17) | O(12)-P(3)-O(13) | 110.1(3) |
| O(3)*-Dy(2)-O(19)* | 128.43(17) | O(15)-P(3)-O(14) | 111.5(3) |
| O(17)-Dy(2)-O(19)* | 71.22(16) | O(12)-P(3)-O(14) | 112.1(3) |
| O(10)-Dy(2)-O(19)* | 150.91(16) | O(13)-P(3)-O(14) | 103.2(3) |
| O(3)*-Dy(2)-O(13) | 133.23(17) | O(15)-P(3)-Dy(2) | 118.0(2) |
| O(17)-Dy(2)-O(13) | 72.28(17) | O(12)-P(3)-Dy(2) | 132.4(2) |
| O(10)-Dy(2)-O(13) | 82.16(17) | O(13)-P(3)-Dy(2) | 51.03(18) |
| O(19)*-Dy(2)-O(13) | 72.68(17) | O(14)-P(3)-Dy(2) | 52.83(19) |
| O(3)*-Dy(2)-O(16) | 76.32(16) | O(9)-P(4)-O(10) | 109.7(3) |
| O(17)-Dy(2)-O(16) | 72.33(16) | O(9)-P(4)-O(11) | 109.5(3) |
| O(10)-Dy(2)-O(16) | 74.23(17) | O(10)-P(4)-O(11) | 109.6(3) |
| O(19)*-Dy(2)-O(16) | 115.66(17) | O(9)-P(4)-O(17)* | 110.5(3) |
| O(13)-Dy(2)-O(16) | 137.87(16) | O(10)-P(4)-O(17)* | 109.7(3) |
| O(3)*-Dy(2)-O(14) | 77.78(16) | O(11)-P(4)-O(17)* | 107.9(3) |
| O(17)-Dy(2)-O(14) | 128.73(16) | O(18)-P(5)-O(18)* | 114.3(4) |
| O(10)-Dy(2)-O(14) | 80.47(17) | O(18)-P(5)-O(19) | 111.7(3) |
| O(19)*-Dy(2)-O(14) | 97.14(16) | O(18)*-P(5)-O(19) | 104.6(3) |
| O(13)-Dy(2)-O(14) | 56.81(16) | O(18)-P(5)-O(19)* | 104.6(3) |
| O(16)-Dy(2)-O(14) | 146.44(17) | O(18)*-P(5)-O(19)* | 111.7(3) |
| O(3)*-Dy(2)-O(18) | 73.15(16) | O(19)-P(5)-O(19)* | 109.9(4) |
| O(17)-Dy(2)-O(18) | 125.71(16) | O(18)-P(5)-Dy(2) | 55.01(17) |
| O(10)-Dy(2)-O(18) | 146.40(16) | O(18)*-P(5)-Dy(2) | 124.08(18) |
| O(19)*-Dy(2)-O(18) | 56.39(15) | O(19)-P(5)-Dy(2) | 131.0(2) |
| O(13)-Dy(2)-O(18) | 102.22(16) | O(19)*-P(5)-Dy(2) | 50.04(19) |
| O(16)-Dy(2)-O(18) | 116.80(16) | O(18)-P(5)-Dy(2)* | 124.09(18) |
| O(14)-Dy(2)-O(18) | 74.78(16) | O(18)*-P(5)-Dy(2)* | 55.01(17) |
| O(3)*-Dy(2)-O(7)* | 81.37(17) | O(19)-P(5)-Dy(2)* | 50.04(19) |
| O(17)-Dy(2)-O(7)* | 83.90(17) | O(19)*P(5)-Dy(2)* | 131.0(2) |
| O(10)-Dy(2)-O(7)* | 127.80(16) | P(1)-O(1)-Dy(1) | 98.2(2) |
| O(19)*Dy(2)-O(7)* | 70.87(16) | P(1)-O(2)-Dy(1) | 101.9(3) |
| O(13)-Dy(2)-O(7)* | 141.24(17) | P(2)-O(6)-Dy(1) | 149.3(3) |

| | | | |
|-------------------|------------|-------------------|----------|
| O(16)-Dy(2)-O(7)* | 54.10(17) | P(2)-O(8)-Dy(1)* | 127.9(3) |
| O(14)-Dy(2)-O(7)* | 140.74(16) | P(2)-O(7)-Dy(2)* | 97.5(3) |
| O(18)-Dy(2)-O(7)* | 67.46(15) | P(3)-O(12)-Dy(1) | 151.3(3) |
| O(3)*-Dy(2)-P(3) | 106.41(13) | P(3)-O(13)-Dy(2) | 100.6(2) |
| O(17)-Dy(2)-P(3) | 100.62(12) | P(3)-O(14)-Dy(2) | 98.4(2) |
| O(10)-Dy(2)-P(3) | 83.11(12) | P(4)-O(9)-Dy(1) | 168.6(3) |
| O(19)*-Dy(2)-P(3) | 81.57(12) | P(4)-O(10)-Dy(2) | 158.5(3) |
| O(13)-Dy(2)-P(3) | 28.34(12) | P(4)-O(11)-Dy(1)* | 143.7(3) |
| O(16)-Dy(2)-P(3) | 156.40(13) | P(2)*-O(16)-Dy(2) | 104.4(3) |
| O(14)-Dy(2)-P(3) | 28.76(11) | P(4)*-O(17)-Dy(2) | 144.1(3) |
| O(18)-Dy(2)-P(3) | 85.86(11) | P(5)-O(18)-Dy(2) | 96.6(2) |
| O(7)*-Dy(2)-P(3) | 149.07(12) | P(5)-O(19)-Dy(2)* | 101.8(2) |
| O(3)*-Dy(2)-P(5) | 100.47(13) | P(1)-O(3)-Dy(2)* | 155.7(3) |
| O(17)-Dy(2)-P(5) | 97.90(13) | | |
| O(10)-Dy(2)-P(5) | 167.80(11) | | |

Table S7. Bond lengths (\AA) found in **3**

| | | | |
|-------------|-----------|-------------|-----------|
| Gd(1)-O(3)* | 2.343(8) | P(1)-O(3) | 1.470(8) |
| Gd(1)-O(5) | 2.354(8) | P(1)-O(2) | 1.479(7) |
| Gd(1)-O(10) | 2.388(8) | P(1)-O(1) | 1.496(8) |
| Gd(1)-O(9) | 2.419(8) | P(2)-O(7) | 1.439(11) |
| Gd(1)-O(8)* | 2.461(8) | P(2)-O(5) | 1.453(8) |
| Gd(1)-O(1) | 2.462(8) | P(2)-O(8) | 1.501(8) |
| Gd(1)-O(11) | 2.482(7) | P(2)-O(6) | 1.531(10) |
| Gd(1)-O(2) | 2.510(7) | O(8)-Gd(1)* | 2.461(8) |
| Gd(1)-O(6)* | 2.516(10) | O(6)-Gd(1)* | 2.516(10) |
| P(1)-O(4) | 1.460(8) | O(3)-Gd(1)* | 2.344(8) |

Table S8. Bond angles ($^{\circ}$) found in **3**

| | | | |
|-------------------|----------|-------------------|------------|
| O(3)*-Gd(1)-O(5) | 86.4(3) | O(8)*-Gd(1)-P(1) | 99.82(19) |
| O(3)*-Gd(1)-O(10) | 139.2(3) | O(1)-Gd(1)-P(1) | 28.46(19) |
| O(5)-Gd(1)-O(10) | 82.4(3) | O(11)-Gd(1)-P(1) | 142.39(19) |
| O(3)*-Gd(1)-O(9) | 142.5(3) | O(2)-Gd(1)-P(1) | 28.23(16) |
| O(5)-Gd(1)-O(9) | 81.5(3) | O(6)*-Gd(1)-P(1) | 92.9(2) |
| O(10)-Gd(1)-O(9) | 74.1(3) | O(3)*-Gd(1)-P(2)* | 102.7(2) |
| O(3)*-Gd(1)-O(8)* | 75.5(3) | O(5)-Gd(1)-P(2)* | 161.07(19) |
| O(5)-Gd(1)-O(8)* | 149.4(3) | O(10)-Gd(1)-P(2)* | 79.9(2) |
| O(10)-Gd(1)-O(8)* | 95.2(3) | O(9)-Gd(1)-P(2)* | 99.9(3) |
| O(9)-Gd(1)-O(8)* | 127.4(3) | O(8)*-Gd(1)-P(2)* | 28.4(2) |
| O(3)*-Gd(1)-O(1) | 77.8(3) | O(1)-Gd(1)-P(2)* | 72.89(19) |
| O(5)-Gd(1)-O(1) | 125.7(3) | O(11)-Gd(1)-P(2)* | 87.11(18) |
| O(10)-Gd(1)-O(1) | 138.9(3) | O(2)-Gd(1)-P(2)* | 129.53(17) |
| O(9)-Gd(1)-O(1) | 80.8(3) | O(6)*-Gd(1)-P(2)* | 29.3(2) |
| O(8)*-Gd(1)-O(1) | 74.8(3) | O(4)-P(1)-O(3) | 109.9(5) |
| O(3)*-Gd(1)-O(11) | 71.4(3) | O(4)-P(1)-O(2) | 111.5(4) |
| O(5)-Gd(1)-O(11) | 80.1(3) | O(3)-P(1)-O(2) | 109.9(5) |
| O(10)-Gd(1)-O(11) | 68.1(3) | O(4)-P(1)-O(1) | 111.0(5) |
| O(9)-Gd(1)-O(11) | 139.6(3) | O(3)-P(1)-O(1) | 109.4(5) |
| O(8)*-Gd(1)-O(11) | 70.9(3) | O(2)-P(1)-O(1) | 105.0(4) |
| O(1)-Gd(1)-O(11) | 138.3(3) | O(4)-P(1)-Gd(1) | 124.7(3) |
| O(3)*-Gd(1)-O(2) | 70.2(3) | O(3)-P(1)-Gd(1) | 125.4(3) |
| O(5)-Gd(1)-O(2) | 69.1(2) | O(2)-P(1)-Gd(1) | 53.4(3) |
| O(10)-Gd(1)-O(2) | 138.4(3) | O(1)-P(1)-Gd(1) | 51.6(3) |
| O(9)-Gd(1)-O(2) | 72.3(3) | O(7)-P(2)-O(5) | 112.0(5) |
| O(8)*-Gd(1)-O(2) | 124.7(2) | O(7)-P(2)-O(8) | 115.2(7) |
| O(1)-Gd(1)-O(2) | 56.7(2) | O(5)-P(2)-O(8) | 109.1(5) |
| O(11)-Gd(1)-O(2) | 131.4(2) | O(7)-P(2)-O(6) | 108.1(7) |
| O(3)*-Gd(1)-O(6)* | 126.6(3) | O(5)-P(2)-O(6) | 108.5(5) |
| O(5)-Gd(1)-O(6)* | 146.9(3) | O(8)-P(2)-O(6) | 103.3(5) |
| O(10)-Gd(1)-O(6)* | 73.1(3) | O(7)-P(2)-Gd(1)* | 117.0(4) |
| O(9)-Gd(1)-O(6)* | 70.7(3) | O(5)-P(2)-Gd(1)* | 130.9(3) |
| O(8)*-Gd(1)-O(6)* | 57.1(3) | O(8)-P(2)-Gd(1)* | 51.2(3) |
| O(1)-Gd(1)-O(6)* | 68.0(3) | O(6)-P(2)-Gd(1)* | 53.4(4) |

| | | | |
|-------------------|-----------|------------------|----------|
| O(11)-Gd(1)-O(6)* | 109.8(3) | P(1)-O(1)-Gd(1) | 99.9(4) |
| O(2)-Gd(1)-O(6)* | 116.6(3) | P(2)-O(8)-Gd(1)* | 100.5(4) |
| O(3)*-Gd(1)-P(1) | 71.0(2) | P(2)-O(6)-Gd(1)* | 97.3(5) |
| O(5)-Gd(1)-P(1) | 97.30(19) | P(1)-O(3)-Gd(1)* | 153.0(5) |
| O(10)-Gd(1)-P(1) | 149.3(2) | P(2)-O(5)-Gd(1) | 149.8(5) |
| O(9)-Gd(1)-P(1) | 75.5(2) | P(1)-O(2)-Gd(1) | 98.4(3) |

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