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# Microwave-assisted large scale synthesis of lanthanide Metal– Organic Frameworks (Ln-MOFs), having preferred conformation and photoluminescence properties

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# Supplimentory Information

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## 1. IR-Spectra



Figure S1. IR spectra of as-synthesized and desolvated samples of 1 and 8.

### 2. <sup>1</sup>H-NMR Spectrum



Figure S2. <sup>1</sup>H-NMR spectrum of ligand (H<sub>3</sub>TTTPC) before MOF preparation in DMSO-*d*<sub>6</sub> solvent.



#### **3. Powder X-Ray Diffraction**

Figure S3. PXRD patterns of Ln-MOF, synthesized by microwave (MW). Here the PXRD patterns show the purity of MOF was obtained perfectly up to 2g scale.



Figure S4. PXRD patterns of 1-9.



Figure S5. PXRD patterns of as-synthesized and desolvated samples of 1 and 8.

4. Thermogravimetric Analysis (TGA)



Figure S6. TGA plots of as-synthesized samples of 1 - 8.



Figure S7. TGA plots of as-synthesized and desolvated samples of 5 and 6.

#### 5. Additional figures





(c)

Figure S8. Packing patterns of LnMOFs. (a) along *b*-axis, (b) along *c*-axis and (c) spacefill model of the packing along *a*-axis.



**Figure S9.** Photography of single crystals, (a) obtained from MWASR (experiment duration was 30 min.) and (b) solvothermal oven (experiment duration was 2 days). Notice that obtained crystals from both processes are single crystal of similar shape and unit cell parameters are also same.



**Figure S10.** Sorption isotherms for 1 and 8. Filled circles represent adsorptions and open circles represent desorption. Po is the saturated vapor pressure at 77K.

6. The solid-state photoluminescence spectra



**Figure S11.** Solid-state excitation spectrum for the **5** (left) and **6** (right) at 298 K(emission monitored at 612 nm).



Figure S12. Photography of 5 (left) and 6 (right) under a UV lamp (excited at 254 nm)

7. Scanning Electron Spectroscopy (SEM) image.



**Figure S13. (a)** Photography of microcrystals obtained from MWASR (experiment duration was 5 min.). SEM image of these microcrystals (b) and (c), where the crystalline property is observed clearly.

#### 8. Selected bond distances and angles

Table S1. Selected bond lengths (Å) and angles (°) for 1.

| La1—O1       | 2.508(4) | La1—O5_e      | 2.492(3) |
|--------------|----------|---------------|----------|
| La1—O2_g     | 2.435(4) | La1—O6_d      | 2.543(4) |
| La1—O3_f     | 2.466(5) | La1—O7        | 2.600(5) |
| La1—O4_c     | 2.528(5) | La1—O8        | 2.544(5) |
| O1—La1—O7    | 72.0(1)  | O3_f—La1—O7   | 72.6(2)  |
| La1—O1—C1    | 129.4(4) | O2_g—La1—O7   | 72.0(1)  |
| O1—La1—O8    | 73.1(1)  | O4_c—La1—O8   | 73.3(2)  |
| La1_g—O2—C1  | 176.6(4) | O6_d—La1—O8   | 140.3(2) |
| O1—La1—O4_c  | 145.2(1) | O5_e—La1—O8   | 74.5(1)  |
| La1_f        | 136.0(4) | O3_f—La1—O8   | 135.8(2) |
| O1—La1—O6_d  | 140.0(1) | O2_g—La1—O8   | 73.3(2)  |
| La1_b04C23   | 134.0(4) | O4_c—La1—O6_d | 74.4(1)  |
| O1—La1—O5_e  | 83.0(1)  | O4_c—La1—O5_e | 79.6(1)  |
| La1_e—O5—C30 | 170.6(4) | O3_f—La1—O4_c | 125.3(1) |
| O1—La1—O3_f  | 75.7(1)  | O2_g—La1—O4_c | 77.9(1)  |
| La1_a—O6—C30 | 106.0(4) | O5_e—La1—O6_d | 121.1(1) |
| O1—La1—O2_g  | 100.7(1) | O3_f—La1—O6_d | 82.5(1)  |
| O7—La1—O8    | 124.1(2) | O2_g—La1—O6_d | 77.9(1)  |
| O4_c—La1—O7  | 136.9(1) | O3_f—La1—O5_e | 71.4(1)  |
| O6_d—La1—O7  | 69.6(1)  | O2_g—La1—O5_e | 144.8(1) |
| O5_e—La1—O7  | 140.1(1) | O2_g—La1—O3_f | 143.7(1) |

Symmetry codes: (i) x,-1+y,-1+z; (ii) x,y,-1+z; (iii) x,y,1+z; (iv) x,1+y,1+z; (v) -x,1-y,1-z; (vi) -x,2-y,1-z; (vii) 1-x,2-y,2-z.

| Ce1—O1       | 2.405(4) | Ce1—O5_d       | 2.456(3) |
|--------------|----------|----------------|----------|
| Ce1—O2_g     | 2.487(4) | Ce1—O6_e       | 2.507(4) |
| Ce1—O3_c     | 2.443(5) | Ce1—O7         | 2.579(5) |
| Ce1—O4_f     | 2.489(5) | Ce1—O8         | 2.513(5) |
| O1—Ce1—O7    | 71.8(2)  | O4_f-Ce1-07    | 136.8(2) |
| Ce1—O1—C1    | 176.6(4) | O2_g—Ce1—O7    | 72.8(1)  |
| O1—Ce1—O8    | 73.4(2)  | O3_c-Ce1O8     | 136.7(2) |
| Ce1_g—O2—C1  | 129.8(4) | O5_d—Ce1—O8    | 74.5(1)  |
| O1—Ce1—O3_c  | 143.2(1) | O6_e-Ce1-08    | 140.1(2) |
| Ce1_bC23     | 135.6(4) | O4_f-Ce1-08    | 73.0(2)  |
| O1—Ce1—O5_d  | 145.2(1) | O2_g—Ce1—O8    | 73.3(2)  |
| Ce1_fO4C23   | 134.8(4) | O3_c-Ce1-O5_d  | 71.5(1)  |
| O1—Ce1—O6_e  | 78.3(1)  | O3_c-Ce1-O6_e  | 81.5(1)  |
| Ce1_a—O5—C30 | 173.1(4) | O3_c-Ce1-O4_f  | 124.7(1) |
| O1—Ce1—O4_f  | 78.6(1)  | O2_g—Ce1—O3_c  | 76.3(1)  |
| Ce1_e—O6—C30 | 107.6(4) | O5_d—Ce1—O6_e  | 120.8(1) |
| O1—Ce1—O2_g  | 100.8(1) | O4_f—Ce1—O5_d  | 79.5(1)  |
| O7—Ce1—O8    | 124.7(2) | O2_g—Ce1—O5_d  | 82.3(1)  |
| O3_c—Ce1—O7  | 72.4(2)  | O4_f—Ce1—O6_e  | 74.4(1)  |
| O5_d—Ce1—O7  | 139.9(1) | O2_g —Ce1—O6_e | 140.4(1) |
| O6_e—Ce1—O7  | 69.4(1)  | O2_g—Ce1—O4_f  | 144.9(1) |

Table S2. Selected bond lengths (Å) and angles (°) for 2.

Symmetry codes: (i) -1+x,-1+y,-1+z; (ii) -1+x,y,-1+z; (iii) 1+x,y,1+z; (iv) 1+x,1+y,1+z; (v) 1-x,1-y,1-z; (vi) 1-x,2-y,1-z; (vii) 1-x,2-y,2-z.

| Pr1—O1       | 2.489(4) | Pr1—O7        | 2.485(5) |
|--------------|----------|---------------|----------|
| Pr1—O2_g     | 2.429(3) | Pr1—O8        | 2.568(5) |
| Pr1—O3_c     | 2.480(5) | Pr1—O5_d      | 2.466(4) |
| Pr1—O4_f     | 2.420(5) | Pr1—O6_e      | 2.386(4) |
| O1—Pr1—O7    | 140.0(2) | O4_f-Pr1-07   | 137.2(2) |
| Pr1—O1—C1    | 108.6(4) | O2_g—Pr1—O7   | 74.4(1)  |
| O1—Pr1—O8    | 69.9(2)  | O3_c—Pr1—O8   | 137.2(2) |
| Pr1_g—O2—C1  | 173.8(4) | O5_d—Pr1—O8   | 72.2(1)  |
| O1—Pr1—O3_c  | 74.2(1)  | O6_e—Pr1—O8   | 71.9(2)  |
| Pr1_b—O3—C23 | 135.3(4) | O4_f-Pr1-08   | 72.2(2)  |
| O1—Pr1—O5_d  | 140.3(1) | O2_g—Pr—O8    | 139.9(1) |
| Pr1_fO4C23   | 135.5(4) | O3_c—Pr1—O5_d | 145.2(1) |
| O1—Pr1—O6_e  | 79.0(1)  | O3_c—Pr1—O6_e | 79.1(1)  |
| Pr1_a—O5—C30 | 130.2(4) | O3_c—Pr1—O4_f | 124.1(1) |
| O1—Pr1—O4_f  | 80.9(1)  | O2_g—Pr1—O3_c | 79.2(1)  |
| Pr1_e—O6—C30 | 176.7(4) | O5_d—Pr1—O6_e | 100.1(1) |
| O1—Pr1—O2_g  | 120.6(1) | O4_f—Pr1—O5_d | 76.8(1)  |
| O7—Pr1—O8    | 124.7(2) | O2_g—Pr1—O5_d | 82.7(1)  |
| O3_c—Pr1—O7  | 73.0(2)  | O4_f-Pr1-O6_e | 143.1(1) |
| O5_d—Pr1—O7  | 73.6(2)  | O2_g—Pr1—O6_e | 144.9(1) |
| O6_e—Pr1—O7  | 73.0(2)  | O2_g—Pr1—O4_f | 71.8(1)  |

Table S3. Selected bond lengths (Å) and angles (°) for 3.

Symmetry codes: (i) x,-1+y,-1+z; (ii) x,-1+y,z; (iii) x,1+y,z; (iv) x,1+y,1+z; (v) 1-x,2-y,-z; (vi) 2-x,2-y,1-z; (vii) 2-x,3-y,1-z.

| Nd1—O1       | 2.460(4) | Nd1—O5_a      | 2.451(4) |
|--------------|----------|---------------|----------|
| Nd1—O3_b     | 2.466(5) | Nd1—O6_g      | 2.378(4) |
| Nd1—O2_e     | 2.409(3) | Nd1—O7        | 2.546(5) |
| Nd1—O4_f     | 2.416(5) | Nd1—O8        | 2.474(5) |
| O1—Nd1—O7    | 70.2(2)  | O4_f-Nd107    | 72.1(2)  |
| Nd1—O1—C1    | 110.0(4) | O6_g—Nd1—O7   | 72.1(2)  |
| O1—Nd1—O8    | 140.2(2) | O5_a—Nd1—O8   | 73.1(2)  |
| Nd1_e—O2—C1  | 174.1(4) | O3_b—Nd1—O8   | 73.5(2)  |
| O1—Nd1—O5_a  | 140.5(1) | O2_e—Nd1—O8   | 74.3(2)  |
| Nd1_cO3C23   | 135.9(4) | O4_f-Nd1O8    | 137.0(2) |
| O1—Nd1—O3_b  | 73.9(1)  | O6_g—Nd1—O8   | 73.1(2)  |
| Nd1_fO4C23   | 135.8(4) | O3_b—Nd1—O5_a | 145.3(1) |
| O1—Nd1—O2_e  | 120.5(1) | O2_e—Nd1—O5_a | 83.0(1)  |
| Nd1_d—O5—C30 | 130.9(4) | O4_f—Nd1—O5_a | 76.9(1)  |
| O1—Nd1—O4_f  | 81.1(1)  | O5_a—Nd1—O6_g | 100.0(1) |
| Nd1_g—O6—C30 | 176.9(4) | O2_e—Nd1—O3_b | 79.1(1)  |
| O1—Nd1—O6_g  | 79.0(1)  | O3_b—Nd1—O4_f | 124.1(1) |
| O7—Nd1—O8    | 124.4(2) | O3_b—Nd1—O6_g | 79.1(1)  |
| O5_a—Nd1—O7  | 71.9(2)  | O2_e—Nd1—O4_f | 72.0(1)  |
| O3_b—Nd1—O7  | 137.3(2) | O2_e—Nd1—O6_g | 144.7(1) |
| O2_e—Nd1—O7  | 139.9(2) | O4_f—Nd1—O6_g | 143.1(1) |

Table S4. Selected bond lengths (Å) and angles (°) for 4.

Symmetry codes: (i) x,-1+y,-1+z; (ii) x,-1+y,z; (iii) x,1+y,z; (iv) x,1+y,1+z; (v) -x,-y,-z; (vi) -x,1-y,-z; (vii) 1-x,1-y,1-z.

| Eu1—O1       | 2.328(4) | Eu1—O5_d      | 2.354(4) |
|--------------|----------|---------------|----------|
| Eu1—O3_c     | 2.365(6) | Eu1—O6_e      | 2.423(5) |
| Eu1—O4_f     | 2.421(6) | Eu1—O7        | 2.502(6) |
| Eu1—O2_g     | 2.411(6) | Eu1—O8        | 2.438(6) |
| O1—Eu1—O7    | 72.1(2)  | O4_f—Eu1—O7   | 137.1(2) |
| Eu1—O1—C1    | 177.8(4) | O2_g—Eu1—O7   | 72.6(2)  |
| O1—Eu1—O8    | 72.7(2)  | O3_c—Eu1—O8   | 138.4(2) |
| Eu1_g—O2—C1  | 131.8(4) | O5_d—Eu1—O8   | 73.8(2)  |
| O1—Eu1—O3_c  | 143.2(2) | O6_e—Eu1—O8   | 140.1(2) |
| Eu1_b—O3—C23 | 136.2(5) | O4_f—Eu1—O8   | 73.8(2)  |
| O1—Eu1—O5_d  | 144.0(2) | O2_g—Eu1—O8   | 73.1(2)  |
| Eu1_fO4C23   | 136.8(5) | O3_c—Eu1—O5_d | 72.7(2)  |
| O1—Eu1—O6_e  | 80.0(2)  | O3_c—Eu1—O6_e | 79.3(2)  |
| Eu1_a—O5—C30 | 176.3(5) | O3_c—Eu1—O4_f | 122.1(2) |
| O1—Eu1—O4_f  | 79.6(2)  | O2_g—Eu1—O3_c | 78.6(2)  |
| Eu1_e—O6—C30 | 112.7(5) | O5_d—Eu1—O6_e | 120.0(2) |
| O1—Eu1—O2_g  | 99.9(2)  | O4_f—Eu1—O5_d | 78.8(2)  |
| O7—Eu1—O8    | 124.6(2) | O2_g—Eu1—O5_d | 82.4(2)  |
| O3_c—Eu1—O7  | 72.4(2)  | O4_f—Eu1—O6_e | 73.1(2)  |
| O5_d—Eu1—O7  | 140.3(2) | O2_g—Eu1—O6_e | 141.4(2) |
| O6_e—Eu1—O7  | 70.8(2)  | O2_g—Eu1—O4_f | 145.3(2) |

Table S5. Selected bond lengths (Å) and angles (°) for 5.

Symmetry codes: (i) -1+x,-1+y,-1+z; (ii) -1+x,y,-1+z; (iii) 1+x,y,1+z; (iv) 1+x,1+y,1+z; (v) 1-x,1-y,1-z; (vi) 1-x,2-y,1-z; (vii) 1-x,2-y,2-z.

| Table S6  | Selected bo  | nd lengths ( | Å) and a | noles (º) f | or <b>6</b>   |
|-----------|--------------|--------------|----------|-------------|---------------|
| Table So. | Selected DOI | iu ienguis ( | A) and a | ingles () I | 01 <b>U</b> . |

| Tb1—O1       | 2.462(4) | Tb1—O5_g      | 2.377(7) |
|--------------|----------|---------------|----------|
| Tb1—O4_b     | 2.467(5) | Tb1—O6_a      | 2.448(4) |
| Tb1—O2_e     | 2.410(3) | Tb1—O7        | 2.468(5) |
| Tb1 -O3_f    | 2.415(5) | Tb1—O8        | 2.547(5) |
| O1—Tb1—O7    | 140.3(2) | O3_f—Tb1—O7   | 137.0(2) |
| Tb1—O1—C1    | 110.1(4) | O5_g—Tb1—O7   | 73.1(2)  |
| O1—Tb1—O8    | 70.1(2)  | O6_a—Tb1—O8   | 71.9(2)  |
| Tb1_e—O2—C1  | 173.8(4) | O4_b—Tb1—O8   | 137.3(2) |
| O1—Tb1—O6_a  | 140.3(1) | O2_e—Tb1—O8   | 140.0(2) |
| Tb1_f—O3—C23 | 135.9(4) | O3_f—Tb1—O8   | 72.1(2)  |
| O1—Tb1—O4_b  | 74.0(1)  | O5_g—Tb1—O8   | 72.1(2)  |
| Tb1_cO4C23   | 135.9(4) | O4_b—Tb1—O6_a | 145.4(1) |
| O1—Tb1—O2_e  | 120.5(1) | O2_e—Tb1—O6_a | 83.2(1)  |
| Tb1_g        | 176.74   | O3_f—Tb1—O6_a | 76.9(1)  |
| O1—Tb1—O3_f  | 81.0(1)  | O5_g—Tb1—O6_a | 100.0(2) |
| Tb1_d        | 130.9(4) | O2_e—Tb1—O4_b | 79.0(1)  |
| O1—Tb1—O5_g  | 78.9(2)  | O3_f—Tb1—O4_b | 124.0(1) |
| O7—Tb1—O8    | 124.3(2) | O4_b—Tb1—O5_g | 79.0(2)  |
| O6_a—Tb1—O7  | 73.2(2)  | O2_e—Tb1—O3_f | 72.1(1)  |
| O4_b—Tb1—O7  | 73.5(2)  | O2_e—Tb1—O5_g | 144.7(2) |
| O2_e—Tb1—O7  | 74.3(2)  | O3_f—Tb1—O5_g | 143.1(2) |

Symmetry codes: (i) x,-1+y,-1+z; (ii) x,-1+y,z; (iii) x,1+y,z; (iv) x,1+y,1+z; (v) -x,-y,-z; (vi) -x,1-y,-z; (vii) 1-x,1-y,1-z.

| Dy1—O1        | 2.316(3) | Dy1—O5_e      | 2.359(4) |
|---------------|----------|---------------|----------|
| Dy1—O4_c      | 2.323(5) | Dy1—O6_d      | 2.291(4) |
| Dy1—O3_f      | 2.378(6) | Dy1—O7        | 2.409(5) |
| Dy1—O2_g      | 2.379(4) | Dy1—O8        | 2.468(5) |
| O1—Dy1—O7     | 73.9(2)  | O3_f—Dy1—O7   | 73.9(2)  |
| Dy1—O1—C1     | 176.9(4) | O2_g—Dy1—O7   | 140.1(2) |
| O1—Dy1—O8     | 140.2(2) | O4_c—Dy1—O8   | 72.2(2)  |
| Dy1_g—O2—C1   | 115.1(4) | O6_d—Dy1—O8   | 71.6(2)  |
| O1—Dy1—O4_c   | 72.5(1)  | O5_e—Dy1—O8   | 73.1(2)  |
| Dy1_fO3C23    | 137.50   | O3_f—Dy1—O8   | 136.8(2) |
| O1—Dy1—O6_d   | 144.8(1) | O2_g—Dy1—O8   | 70.4(2)  |
| Dy1_bO4C23    | 137.5(4) | O4_c—Dy1—O6_d | 142.5(1) |
| O1—Dy1—O5_e   | 82.4(1)  | O4_c—Dy1—O5_e | 78.5(2)  |
| Dy1_e—O5 —C30 | 131.9(4) | O3_f—Dy1—O4_c | 121.4(2) |
| O1—Dy1—O3_f   | 78.6(2)  | O2_g—Dy1—O4_c | 78.8(2)  |
| Dy1_a—O6—C30  | 179.2(4) | O5_e—Dy1—O6_d | 99.8(2)  |
| O1—Dy1—O2_g   | 119.4(1) | O3_f—Dy1—O6_d | 80.7(2)  |
| O7—Dy1—O8     | 125.5(2) | O2_g—Dy1—O6_d | 80.3(2)  |
| O4_c—Dy1—O7   | 138.5(2) | O3_f—Dy1—O5_e | 145.7(2) |
| O6_d—Dy1—O7   | 73.2(2)  | O2_g—Dy1—O5_e | 141.4(2) |
| O5_e—Dy1—O7   | 73.5(2)  | O2_g—Dy1—O3_f | 72.8(2)  |

Table S7. Selected bond lengths (Å) and angles (°) for 7.

Symmetry codes: (i) -1+x,-1+y,-1+z; (ii) x,-1+y,z; (iii) x,1+y,z; (iv) 1+x,1+y,1+z; (v) 2-x,2-y,-z; (vi) 2-x,2-y,1-z; (vii) 2-x,3-y,1-z.

Table S8. Selected bond lengths (Å) and angles (°) for 8.

| Ho1—O1       | 2.364(3) | Ho1—O5_d      | 2.350(4) |
|--------------|----------|---------------|----------|
| Ho1—O3_c     | 2.374(4) | Ho1—O6_e      | 2.275(4) |
| Ho1—O4_f     | 2.316(4) | Но1—О7        | 2.381(5) |
| Ho1—O2_g     | 2.296(3) | Но1—О8        | 2.451(5) |
| O1—Ho1—O7    | 139.8(2) | O4_f—Ho1—O7   | 138.8(2) |
| Ho1—O1—C18   | 116.4(3) | O2_g—Ho1—O7   | 73.7(2)  |
| O1—Ho1—O8    | 70.7(2)  | O3_c—Ho1—O8   | 136.9(2) |
| Ho1_g—O2—C18 | 176.5(4) | O5_d—Ho1—O8   | 72.9(2)  |
| O1—Ho1—O3_c  | 72.6(1)  | O6_e—Ho1—O8   | 71.8(2)  |
| Ho1_b—O3—C24 | 137.8(4) | O4_f—Ho1—O8   | 71.9(2)  |
| O1—Ho1—O5_d  | 141.5(1) | O2_g—Ho1—O8   | 140.1(2) |
| Ho1_f—O4—C24 | 137.4(3) | O3_c—Ho1—O5_d | 145.9(1) |
| O1—Ho1—O6_e  | 80.6(1)  | O3_c—Ho1—O6_e | 80.8(1)  |
| Ho1_a—O5—C30 | 132.8(4) | O3_c—Ho1—O4_f | 121.3(1) |
| O1—Ho1—O4_f  | 78.6(1)  | O2_g—Ho1—O3_c | 78.5(1)  |
| Ho1_e—O6—C30 | 179.60   | O5_d—Ho1—O6_e | 99.8(1)  |
| O1—Ho1—O2_g  | 119.1(1) | O4_f—Ho1—O5_d | 78.6(1)  |
| O7—Ho1—O8    | 126.0(2) | O2_g—Ho1—O5_d | 82.5(1)  |
| O3_c—Ho1—O7  | 73.5(2)  | O4_f—Ho1—O6_e | 142.5(1) |
| O5_d—Ho1—O7  | 74.0(2)  | O2_g—Ho1—O6_e | 144.7(1) |
| O6_e—Ho1—O7  | 73.2(2)  | O2_g—Ho1—O4_f | 72.7(1)  |

Symmetry codes: (i) x,-1+y,-1+z; (ii) x,-1+y,z; (iii) x,1+y,z; (iv) x,1+y,1+z; (v) -x,2-y,-z; (vi) 1-x,2-y,1-z; (vii) 1-x,3-y,1-z.

| Yb1—O1       | 2.400(4) | Yb1—O5_d       | 2.448(4) |
|--------------|----------|----------------|----------|
| Yb1—O2_g     | 2.487(4) | Yb1—O6_e       | 2.506(5) |
| Yb1—O3_c     | 2.442(6) | Yb1—O7         | 2.576(6) |
| Yb1—O4_f     | 2.490(6) | Yb1—O8         | 2.504(6) |
| O1—Yb1—O7    | 72.0(2)  | O4_f—Yb1—O7    | 136.9(2) |
| Yb1—O1—C1    | 176.7(4) | O2_g—Yb1—O7    | 72.9(2)  |
| O1—Yb1—O8    | 73.4(2)  | O3_c—Yb1—O8    | 136.7(2) |
| Yb1_g—O2—C1  | 129.6(4) | O5_d—Yb1—O8    | 74.4(1)  |
| O1—Yb1—O3_c  | 143.1(2) | O6_e—Yb1—O8    | 140.3(2) |
| Yb1_b—O3—C23 | 135.5(5) | O4_f—Yb1—O8    | 73.0(2)  |
| O1—Yb1—O5_d  | 145.1(2) | O2_g—Yb1—O8    | 73.2(2)  |
| Yb1_f        | 135.1(5) | O3_c—Yb1—O5_d  | 71.8(2)  |
| O1—Yb1—O6_e  | 78.3(2)  | O3_c-Yb1-O6_e  | 81.4(2)  |
| Yb1_a—O5—C30 | 173.0(5) | O3_c—Yb1—O4_f  | 124.9(2) |
| O1—Yb1—O4_f  | 78.4(2)  | O2_g—Yb1—O3_c  | 76.2(2)  |
| Yb1_e—O6—C30 | 107.9(5) | O5_d—Yb1—O6_e  | 120.9(2) |
| O1—Yb1—O2_g  | 101.1(2) | O4_f—Yb1—O5_d  | 79.5(2)  |
| O7—Yb1—O8    | 124.8(2) | O2_g—Yb1—O5_d  | 82.2(2)  |
| O3_c—Yb1—O7  | 72.1(2)  | O4_f—Yb1—O6_e  | 74.5(2)  |
| O5_d—Yb1—O7  | 139.9(2) | O2_g —Yb1—O6_e | 140.4(2) |
| O6_e—Yb1—O7  | 69.4(2)  | O2_g—Yb1—O4_f  | 144.7(2) |

Table S9. Selected bond lengths (Å) and angles (°) for 9.

Symmetry codes: (i) -1+x,-1+y,-1+z; (ii) -1+x,y,-1+z; (iii) 1+x,y,1+z; (iv) 1+x,1+y,1+z; (v) 1-x,1-y,1-z; (vi) 1-x,2-y,1-z; (vii) 1-x,2-y,2-z.

## 9. Element analyses of compounds

| Num. | Formula                                       |        | C (%) | H (%) | N (%) |
|------|---|--------|-------|-------|-------|
| 1.   | $[La(TTTPC)(NO_2)_2(Cl)]\bullet(H_2O)_{10}$   | Calcd. | 34.78 | 4.57  | 6.76  |
|      |   | Found  | 34.94 | 4.76  | 6.85  |
| 2.   | $[Ce(TTTPC)(NO_2)_2(Cl)]\bullet(H_2O)_{10}$   | Calcd. | 34.74 | 4.57  | 6.75  |
|      |   | Found  | 34.39 | 4.42  | 7.10  |
| 3.   | $[Pr(TTTPC)(NO_2)_2(Cl)]\bullet(H_2O)_{10}$   | Calcd. | 34.71 | 4.56  | 6.75  |
|      |   | Found  | 34.74 | 4.36  | 7.15  |
| 4.   | $[Nd(TTTPC)(NO_2)_2(Cl)] \bullet (H_2O)_{10}$ | Calcd. | 34.71 | 4.56  | 6.75  |
|      |   | Found  | 34.78 | 4.41  | 6.85  |
| 5.   | $[Eu(TTTPC)(NO_2)_2(Cl)]\bullet(H_2O)_{10}$   | Calcd. | 34.34 | 4.52  | 6.68  |
|      |   | Found  | 34.44 | 4.51  | 6.65  |
| 6.   | $[Tb(TTTPC)(NO_2)_2(Cl)] \bullet (H_2O)_{10}$ | Calcd. | 34.12 | 4.49  | 6.63  |
|      |   | Found  | 34.52 | 4.55  | 6.75  |
| 7.   | $[Dy(TTTPC)(NO_2)_2(Cl)] \bullet (H_2O)_{10}$ | Calcd. | 34.00 | 4.47  | 6.61  |
|      |   | Found  | 34.30 | 4.50  | 6.73  |
| 8.   | $[Ho(TTTPC)(NO_2)_2(Cl)] \bullet (H_2O)_{10}$ | Calcd. | 33.93 | 4.46  | 6.59  |
|      |   | Found  | 34.14 | 4.66  | 6.75  |
| 9.   | $[Yb(TTTPC)(NO_2)_2(Cl)] \bullet (H_2O)_{10}$ | Calcd. | 33.67 | 4.43  | 6.54  |
|      |   | Found  | 34.02 | 4.56  | 6.69  |

## Table S10. Element analyses of 1-9