# Electronic supporting information (ESI)

# Hybrid Materials Based on Novel 2D Lanthanide Coordination Polymers Covalently Bonded to Amine-Modified SBA-15 and MCM-41: Assembly, Characterization, Structural Features, Thermal and Luminescence Properties

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Synthesis of 5-methoxy-(4-benzaldehyde)-1,3-benzenedicarboxylic acid (H<sub>2</sub>L).



Scheme S1 Synthetic procedure for H<sub>2</sub>L.

#### Trimethyl 1,3,5-benzenetricarboxylate (1).

Benzenetricarboxylic acid (8.0 g, 38 mmol) was dissolved in methanol (140 mL). Concentrated sulfuric acid (2 mL) was added. The solution was refluxed for 24 h. Solvent was removed under reduced pressure, the residue was dissolved in chloroform (150 mL) and washed with saturated bicarbonate (200 mL), and the solvent was removed under reduced pressure to give the desired product **1** as a white powder (9.15 g, 95%).<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.86 (s, 2H), 3.98 (s, 6H) ppm; <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  165.31, 134.49, 131.08, 52.56 ppm.

#### Dimethyl 5-hydroxymethylbenzene-1,3-dicarboxylate (2).

Trimethyl-1,3,5-tricarboxybenzoate (5 g, 19.82 mmol) was placed, along with a stirring bar, in a two-neck 100 ml flask equipped with a reflux condenser and a 20 ml addition

funnel. While maintaining a dry nitrogen atmosphere, anhydrous THF (15 ml) was added to the reaction flask causing dissolution of the white solid. NaBH<sub>4</sub> (0.9 g, 23.79 mmol) was then added to this solution thus forming a suspension which was stirred continuously at room temperature. While stirring, a mixture of THF/MeOH (12.5 ml / 3.7 ml) was added dropwise via the addition funnel. The reaction mixture was then refluxed for 30 min; during this time the reaction mixture changed from a transparent solution to a light yellow and back to transparent again. After cooling, the reaction was quenched with 20 ml of 1 N HCl (note: should be added slowly as the evolution of  $H_2$  gas occurs). The product was then extracted with EtOAc (3 x 25 ml). The organic phase was dried over Na<sub>2</sub>SO<sub>4</sub> and the solvent was removed in vacuo affording a white solid. The crude mixture was purified by silica gel column chromatography with EtOAc to give 2.53 g of 2 (11.30 mmol, yield 57 %) as a white solid.  $R_f = 0.46$ . <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.59 (t, 1H, J = 1.6Hz), 8.23 (t, 2H, J = 0.8Hz), 4.81 (d, 2H, J = 5.9Hz), 3.95 (s, 6H), 1.86 (s, 1H) ppm; <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 166.49, 142.28, 132.37, 130.93, 129.84, 64.32, 52.59 ppm.

### Dimethyl 5-chloromethyl-1,3-benzenedicarboxylate (3).<sup>1</sup>

To dimethyl 5-hydroxymethylbenzene-1,3-dicarboxylate (2.0 g, 8.9 mmol) was added SOCl<sub>2</sub> (1.3 mL). The solution was refluxed under an argon atmosphere for 1 h. CHCl<sub>3</sub> was added to the resulting solution, and the organic phase was washed with 0.1 M NaOH (2 $\beta$  50 mL) and brine (50 mL). Solvent was removed under reduced pressure to yield **4** as a white powder (2.0 g, 93%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): 8.64 (t, 1H, *J* = 1.3Hz), 8.26 (t, 2H, *J* = 1.3Hz), 4.65 (s, 2H), 3.96 (s, 6H) ppm; <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  165.85, 138.57, 133.99, 131.31, 130.68, 52.68, 44.98 ppm.

#### Dimethyl 5-methoxy-(4-benzaldehyde)-1,3-benzenedicarboxylate (4).

4-Hydroxybenzaldehyde (2.78 g, 22.73 mmol) and K<sub>2</sub>CO<sub>3</sub> (5.7 g,17.4 mmol) were dissolved in DMF (30 mL). The solution was heated at 110 °C under an argon atmosphere for 2 h. Then, dimethyl 5-chloromethyl-1,3-benzenedicarboxylate (5.0 g, 20.66 mmol) was added to the mixture, which was heated at 110 °C under an argon atmosphere for 24 h. After cooling, the reaction mixture was poured into water, and extracted with Et<sub>2</sub>O. The organic layer was washed with saturated aqueous NaCl, dried over anhydrous MgSO<sub>4</sub>, and concentrated under reduced pressure to give a white powder of **4** (6.0 g, 89%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  9.91 (s, 1H), 8.67 (t, 1H, *J* = 1.3 Hz), 8.32 (d, 2H, *J* = 0.8Hz), 7.86 (d, 2H, *J* = 8.8 Hz), 7.10 (d, 2H, *J* = 8.8 Hz), 5.23 (s, 2H), 3.97 (s, 6H) ppm; <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  190.87, 165.99, 163.25, 137.22, 132.70, 132.16, 131.25, 130.62, 130.54, 115.20, 69.19, 52.65 ppm.

#### 5-Methoxy-(4-benzaldehyde)-1,3-benzenedicarboxylic acid (5) (H<sub>2</sub>L).

Dimethyl 5-methoxy-(4-benzaldehyde)-1,3-benzenedicarboxylate (5.0 g, 15.24 mmol) and NaOH (1.5 g, 38.1 mmol) was dissolved in a mixture of H<sub>2</sub>O/MeOH (50 mL / 50 mL), heated at 80 °C for 5 h. After cooling, the solution was acidified with concentrated HCl until pH < 2. A light yellow precipitate was collected by filtration, washed with deionized water, and dried under vacuum (4.4 g, 96%). <sup>1</sup>H NMR (400 MHz, d-DMSO)  $\delta$  13.36 (s, 1H), 9.88 (s, 1H), 8.44 (s, 1H), 8.26 (d, 2H, *J* = 1.3 Hz), 7.89 (d, 2H, *J* = 8.7 Hz), 7.24 (d, 2H, *J* = 8.8 Hz), 5.41 (s, 2H) ppm. <sup>13</sup>C NMR (101 MHz, d-DMSO)  $\delta$  191.56, 166.43, 163.01, 137.92, 132.51, 131.65, 130.03, 129.75, 115.62, 115.12, 68.58 ppm.

Synthesis of the amine-modified mesoporous materials SBA-15-NH<sub>2</sub> and MCM-41-

 $NH_{2.}^{2}$  3-aminopropyltrimethoxysilane (ATPES) (4.0 g) and the calcined mesoporous materials SBA-15 or MCM-41 (4.0 g) were dispersed in toluene (100 ml), heated to reflux for 6 h, followed by distilling out the methanol-toluene mixture (10 ml). After refluxing another 6 h, 10 ml methanol-toluene solution was distilled again. The mixture was then refluxed again for 12 h. Powder samples of the amine-functionalized mesoporous materials were collected by centrifugation, washed with the mixture of toluene-menthol (1:1), and dried under vacuum to give the final SBA-15-NH<sub>2</sub> and MCM-41-NH<sub>2</sub> matrices.

Th-L					
10-11					
Tb1—Tb2	3.8398 (4)	Tb1—O2AA	2.350 (4)	Tb1—O18	2.362 (4)
Tb1—C38	3.023 (6)	Tb1—O39	138.34(6)	Tb1—O0AA <sup>i</sup>	2.293 (4)
Tb1—O3 <sup>ii</sup>	2.321 (4)	Tb1—O5AA <sup>ii</sup>	2.336 (4)	Tb1—O1	2.378 (4)
Tb2—O3AA	2.279 (4)	Tb2—O2AA	2.638 (5)	Tb2—O6AA	2.316 (4)
Tb2—C25	2.904 (8)	Tb2—O29	2.397 (5)	Tb2—O4AA <sup>iii</sup>	2.269 (4)
Tb2—O4 <sup>ii</sup>	2.391 (5)	Tb2—C5 <sup>ii</sup>	2.911 (7)	Tb2—O5AA <sup>ii</sup>	2.674 (5)
Tb2—O1 <sup>iv</sup>	2.555 (4)	Tb2—O2	2.468 (5)	Tb1—O0AA <sup>i</sup>	2.293 (4)
Tb1—O3 <sup>ii</sup>	2.321 (4)	Tb1—O5AA <sup>ii</sup>	2.336 (4)	Tb1—O1	2.378 (4)
Tb2—O3AA	2.279 (4)	Tb2—O2AA	2.638 (5)	Tb2—O6AA	2.316 (4)
Tb2—C25	2.904 (8)	Tb2—O29	2.397 (5)	Tb2—O4AA <sup>iii</sup>	2.269 (4)
Tb2—O4 <sup>ii</sup>	2.391 (5)	Tb2—C5 <sup>ii</sup>	2.911 (7)	Tb2—O5AA <sup>ii</sup>	2.674 (5)
Tb2—O1 <sup>iv</sup>	2.555 (4)	Tb2—O2	2.468 (5)	O2AA—Tb1—Tb2	42.50 (11)
O2AA—Tb1—O18	78.59 (16)	O2AA—Tb1—C38	69.48 (16)	O2AA—Tb1—O1	150.70 (16)
O18—Tb1—Tb2	79.77 (11)	O18—Tb1—C38	22.88 (16)	O18—Tb1—O1	123.25 (16)
C38—Tb1—Tb2	58.53 (12)	O39—Tb1—Tb2	94.24 (11)	O39—Tb1—O2AA	133.95 (16)
O39—Tb1—O18	78.44 (18)	O39—Tb1—C38	73.94 (18)	O39—Tb1—O1	73.69 (16)
O0AA <sup>i</sup> —Tb1—Tb2	131.98 (11)	O0AA <sup>i</sup> —Tb1— O2AA	91.64 (16)	O0AA <sup>i</sup> —Tb1—O18	76.95 (16)
O0AA <sup>i</sup> —Tb1—C38	98.26 (17)	O0AA <sup>i</sup> —Tb1—O39	120.85 (17)	O0AA <sup>i</sup> —Tb1—O3 <sup>ii</sup>	88.32 (16)
O0AA <sup>i</sup> —Tb1— O5AA <sup>ii</sup>	162.79 (17)	O0AA <sup>i</sup> —Tb1—O1	76.70 (15)	O3 <sup>ii</sup> —Tb1—Tb2	90.00 (10)
O3 <sup>ii</sup> —Tb1—O2AA	73.67 (15)	O3 <sup>ii</sup> —Tb1—O18	148.11 (17)	O3 <sup>ii</sup> —Tb1—C38	142.68 (16)
O3 <sup>ii</sup> —Tb1—O39	132.79 (17)	O3 <sup>ii</sup> —Tb1—O5AA <sup>ii</sup>	75.77 (15)	O3 <sup>ii</sup> —Tb1—O1	79.13 (15)
5AA <sup>ii</sup> —Tb1—Tb2	43.32 (11)	O5AA <sup>ii</sup> —Tb1— O2AA	7.77 (17)	O5AA <sup>ii</sup> —Tb1—O18	113.46 (15)
O5AA <sup>ii</sup> —Tb1—C38	90.75 (16)	5AA <sup>ii</sup> —Tb1—O39	75.70 (18)	O5AA <sup>ii</sup> —Tb1—O1	106.07 (16)

Table S1. Selected Bond Lengths (Å) and Angles (°) for Tb-L and Nd-L.

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01 Th1 Th2	140.20 (11)	O1 Th1 C29	120 16 (17)	$O^2$ A A Th 2 Th 1	105 20 (10)
01—101—102	149.39 (11)	01-101-038	138.16 (17)	03AA—102—101	105.29 (10)
O3AA—Tb2— O2AA	77.69 (14)	O3AA—Tb2—O6AA	82.86 (16)	O3AA—Tb2—C25	74.89 (17)
O3AA—Tb2—O29	77.82 (17)	O3AA—Tb2—O4 <sup>ii</sup>	153.51 (17)	O3AA—Tb2—C5 <sup>ii</sup>	158.82 (17)
O3AA—Tb2— O5AA <sup>ii</sup>	142.06 (14)	O3AA—Tb2—O1 <sup>iv</sup>	77.28 (15)	O3AA—Tb2—O2	125.89 (16)
O2AA—Tb2—Tb1	37.01 (10)	O2AA—Tb2—C25	26.14 (17)	O2AA—Tb2—C5 <sup>ii</sup>	82.48 (16)
O2AA—Tb2— O5AA <sup>ii</sup>	67.24 (14)	O6AA—Tb2—Tb1	50.37 (12)	O6AA—Tb2—O2AA	69.44 (15)
O6AA—Tb2—C25	95.03 (19)	O6AA—Tb2—O29	120.19 (17)	O6AA—Tb2—O4 <sup>ii</sup>	120.54 (16)
O6AA—Tb2—C5 <sup>ii</sup>	97.17 (18)	O6AA—Tb2— O5AA <sup>ii</sup>	71.88 (15)	O6AA—Tb2—O1 <sup>iv</sup>	83.42 (15)
O6AA—Tb2—O2	75.09 (16)	C25—Tb2—Tb1	59.42 (15)	C25—Tb2—C5 <sup>ii</sup>	84.03 (18)
O29—Tb2—Tb1	81.49 (12)	O29—Tb2—O2AA	51.33 (15)	O29—Tb2—C25	25.28 (18)
O29—Tb2—C5 <sup>ii</sup>	84.03 (18)	O29—Tb2—O5AA <sup>ii</sup>	90.70 (16)	O29—Tb2—O1 <sup>iv</sup>	142.78 (16)
O29—Tb2—O2	154.99 (17)	O4AA <sup>iii</sup> —Tb2—Tb1	151.10 (11)	O4AA <sup>iii</sup> —Tb2—O3AA	90.40 (16)
O4AA <sup>iii</sup> —Tb2— O2AA	129.52 (17)	O4AA <sup>iii</sup> —Tb2— O6AA	158.04 (16)	O4AA <sup>iii</sup> —Tb2—C25	103.4 (2)
O4AA <sup>iii</sup> —Tb2—O29	78.27 (18)	O4AA <sup>iii</sup> —Tb2—O4 <sup>ii</sup>	71.99 (16)	O4AA <sup>iii</sup> —Tb2—C5 <sup>ii</sup>	96.56 (18)
O4AA <sup>iii</sup> —Tb2— O5AA <sup>ii</sup>	122.86 (15)	O4AA <sup>iii</sup> —Tb2—O1iv	74.70 (15)	4AA <sup>iii</sup> —Tb2—O2	92.42 (17)
O4 <sup>ii</sup> —Tb2—Tb1	84.18 (11)	O4 <sup>ii</sup> —Tb2—O2AA	98.05 (16)	O4 <sup>ii</sup> —Tb2—C25	89.76 (19)
O4 <sup>ii</sup> —Tb2—O29	79.27 (19)	O4 <sup>ii</sup> —Tb2—C5 <sup>ii</sup>	24.57 (17)	O4 <sup>ii</sup> —Tb2—O5AA <sup>ii</sup>	50.88 (14)
O4 <sup>ii</sup> —Tb2—O1 <sup>iv</sup>	115.09 (17)	O4 <sup>ii</sup> —Tb2—O2	75.77 (18)	C5 <sup>ii</sup> —Tb2—Tb1	60.77 (13)
O5AA <sup>ii</sup> —Tb2—Tb1	36.82 (9)	O5AA <sup>ii</sup> —Tb2—C25	79.54 (17)	O5AA <sup>ii</sup> —Tb2—C5 <sup>ii</sup>	26.31 (16)
O1 <sup>iv</sup> —Tb2—Tb1	131.86 (10)	O1 <sup>iv</sup> —Tb2—O2AA	144.95 (14)	O1 <sup>iv</sup> —Tb2—C25	152.09 (16)
O1 <sup>iv</sup> —Tb2—C5 <sup>ii</sup>	123.85 (17)	O1 <sup>iv</sup> —Tb2—O5AA <sup>ii</sup>	125.50 (15)	O2—Tb2—Tb1	97.41 (10)
O2—Tb2—O2AA	134.12 (14)	O2—Tb2—C25	154.24 (17)	O2—Tb2—C5 <sup>ii</sup>	73.93 (17)
O2—Tb2—O5AA <sup>ii</sup>	74.79 (15)	O2—Tb2—O1 <sup>iv</sup>	51.85 (15)	C5AA—O3AA—Tb2	140.8 (4)
Tb1—O2AA—Tb2	100.49 (17)	C25—O2AA—Tb1	140.9 (4)	C25—O2AA—Tb2	88.6 (4)
C38—O6AA—Tb2	143.9 (5)	C38—O18—Tb1	109.8 (4)	O2AA—C25—Tb2	65.3 (4)
O29—C25—Tb2	54.3 (4)	C65—C25—Tb2	171.1 (5)	C25—O29—Tb2	100.4 (5)

O6AA
C38
Tb1
74.9 (4)
O18
C38
Tb1
47.3 (3)
C71
C38
Tb1
166.7 (4)

C7BA
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Symmetry codes: (i) x+1, y, z; (ii) x+1/2, -y+1/2, z-1/2; (iii) x-1/2, -y+1/2, z-1/2; (iv) x-1, y, z; (v) x-1/2, -y+1/2, z+1/2; (vi) x+1/2, -y+1/2, z+1/2.

Nd-L					
Nd1—O1 <sup>i</sup>	2.367 (5)	Nd1—O2 <sup>ii</sup>	2.372 (5)	Nd1—O4 <sup>iii</sup>	2.427 (4)
Nd1—O3	2.454 (5)	Nd1—O8	2.537 (5)	Nd1—O2W	2.542 (5)
Nd1—O1W	2.545 (5)	Nd1—07	2.558 (5)	Nd1—O4	2.815 (5)
Nd1—C17	2.913 (7)	Nd1—C8	3.017 (7)	O1—Nd1 <sup>iv</sup>	2.367 (5)
O2—Nd1 <sup>ii</sup>	2.372 (5)	O4—Nd1 <sup>iii</sup>	2.427 (4)	O1 <sup>i</sup> —Nd1—O2 <sup>ii</sup>	101.15 (18)
O1 <sup>i</sup> —Nd1—O4 <sup>iii</sup>	84.71 (17)	O2 <sup>ii</sup> —Nd1—O4 <sup>iii</sup>	149.61 (19)	O1 <sup>i</sup> —Nd1—O3	142.91 (19)
O2 <sup>ii</sup> —Nd1—O3	73.98 (18)	O4 <sup>iii</sup> —Nd1—O3	118.39 (16)	01 <sup>i</sup> —Nd1—O8	77.28 (19)
O2 <sup>ii</sup> —Nd1—O8	127.83 (19)	O4 <sup>iii</sup> —Nd1—O8	82.56 (16)	O3—Nd1—O8	77.62 (18)
O1 <sup>i</sup> —Nd1—O2W	138.6 (2)	O2 <sup>ii</sup> —Nd1—O2W	78.23 (18)	O4 <sup>iii</sup> —Nd1—O2W	77.95 (17)
O3—Nd1—O2W	77.50 (19)	O8—Nd1—O2W	135.74 (17)	O1 <sup>i</sup> —Nd1—O1W	70.58 (19)
O2 <sup>ii</sup> —Nd1—O1W	72.71 (19)	O4 <sup>iii</sup> —Nd1—O1W	81.51 (17)	O3—Nd1—O1W	137.05 (18)
O8—Nd1—O1W	145.17 (18)	O2W—Nd1—O1W	69.81 (18)	O1 <sup>i</sup> —Nd1—O7	69.75 (18)
O2 <sup>ii</sup> —Nd1—O7	78.84 (18)	O4 <sup>iii</sup> —Nd1—O7	130.30 (16)	O3—Nd1—O7	73.26 (17)
O8—Nd1—O7	51.21 (16)	O2W—Nd1—O7	146.77 (18)	O1W—Nd1—O7	124.64 (16)
O1 <sup>i</sup> —Nd1—O4	138.95 (16)	O2 <sup>ii</sup> —Nd1—O4	117.31 (15)	O4 <sup>iii</sup> —Nd1—O4	69.68 (16)
O3—Nd1—O4	48.76 (14)	O8—Nd1—O4	68.27 (16)	O2W—Nd1—O4	67.76 (16)
O1W—Nd1—O4	132.46 (16)	O7—Nd1—O4	102.71 (15)	O1 <sup>i</sup> —Nd1—C17	70.61 (19)
O2 <sup>ii</sup> —Nd1—C17	103.5 (2)	O4 <sup>iii</sup> —Nd1—C17	106.55 (18)	O3—Nd1—C17	74.86 (18)
O8—Nd1—C17	25.90 (17)	O2W—Nd1—C17	150.51 (19)	O1W—Nd1—C17	139.32 (18)
O7—Nd1—C17	25.36 (17)	O4—Nd1—C17	86.18 (17)	O1 <sup>i</sup> —Nd1—C8	149.74 (19)
O2 <sup>ii</sup> —Nd1—C8	94.78 (18)	O4 <sup>iii</sup> —Nd1—C8	94.43 (16)	O3—Nd1—C8	23.97 (16)
O8—Nd1—C8	72.64 (18)	O2W—Nd1—C8	69.77 (18)	O1W—Nd1—C8	139.32 (18)

O7—Nd1—C8	88.60 (17)	O4—Nd1—C8	24.86 (15)	C17—Nd1—C8	80.78 (18)
Nd1—O1W—H1WA	109.40	Nd1—O1W—H1WC	109.40	H1WA—O1W— H1WC	109.50
C1—O1—Nd1 <sup>iv</sup>	171.6 (5)	C1—O2—Nd1 <sup>ii</sup>	138.0 (5)	Nd1—O2W—H2WA	109.30
Nd1—O2W—H2WB	109.40	O1 <sup>i</sup> —Nd1—O2 <sup>ii</sup>	101.15 (18)	O1 <sup>i</sup> —Nd1—O4 <sup>iii</sup>	84.71 (17)
O2 <sup>ii</sup> —Nd1—O4 <sup>iii</sup>	149.61 (19)	O1 <sup>i</sup> —Nd1—O3	142.91 (19)	O2 <sup>ii</sup> —Nd1—O3	73.98 (18)
O4 <sup>iii</sup> —Nd1—O3	118.39 (16)	O1 <sup>i</sup> —Nd1—O8	77.28 (19)	O2 <sup>ii</sup> —Nd1—O8	127.83 (19)
O4 <sup>iii</sup> —Nd1—O8	82.56 (16)	O3—Nd1—O8	77.62 (18)	O1 <sup>i</sup> —Nd1—O2W	138.6 (2)
O2 <sup>ii</sup> —Nd1—O2W	78.23 (18)	O4 <sup>iii</sup> —Nd1—O2W	77.95 (17)	O3—Nd1—O2W	77.50 (19)
O8—Nd1—O2W	135.74 (17)	Oli—Nd1—O1W	70.58 (19)	O2 <sup>ii</sup> —Nd1—O1W	72.71 (19)
O4 <sup>iii</sup> —Nd1—O1W	81.51 (17)	O3—Nd1—O1W	137.05 (18)	O8—Nd1—O1W	145.17 (18)
O2W—Nd1—O1W	69.81 (18)	O1 <sup>i</sup> —Nd1—O7	69.75 (18)	O2 <sup>ii</sup> —Nd1—O7	78.84 (18)
O4 <sup>iii</sup> —Nd1—O7	130.30 (16)	O3—Nd1—O7	73.26 (17)	O8—Nd1—O7	51.21 (16)
O2W—Nd1—O7	146.77 (18)	O1W—Nd1—O7	124.64 (16)	O1 <sup>i</sup> —Nd1—O4	138.95 (16)
O2 <sup>ii</sup> —Nd1—O4	117.31 (15)	O4 <sup>iii</sup> —Nd1—O4	69.68 (16)	O3—Nd1—O4	48.76 (14)
O8—Nd1—O4	68.27 (16)	O2W—Nd1—O4	67.76 (16)	O1W—Nd1—O4	132.46 (16)
O7—Nd1—O4	102.71 (15)	O1 <sup>i</sup> —Nd1—C17	70.61 (19)	O2 <sup>ii</sup> —Nd1—C17	103.5 (2)
O4 <sup>iii</sup> —Nd1—C17	106.55 (18)	O3—Nd1—C17	74.86 (18)	O8—Nd1—C17	25.90 (17)
O2W—Nd1—C17	150.51 (19)	O1W—Nd1—C17	139.32 (18)	O7—Nd1—C17	25.36 (17)
O4—Nd1—C17	86.18 (17)	O1 <sup>i</sup> —Nd1—C8	149.74 (19)	O2 <sup>ii</sup> —Nd1—C8	94.78 (18)
O4 <sup>iii</sup> —Nd1—C8	94.43 (16)	O3—Nd1—C8	23.97 (16)	O8—Nd1—C8	72.64 (18)
O2W—Nd1—C8	69.77 (18)	O1W—Nd1—C8	139.32 (18)	O7—Nd1—C8	88.60 (17)
O4—Nd1—C8	24.86 (15)	C17—Nd1—C8	80.78 (18)	Nd1—O1W—H1WA	109.40
Nd1—O1W—H1WC	109.40	H1WA—O1W— H1WC	109.50	C1—O1—Nd1 <sup>iv</sup>	171.6 (5)
C1—O2—Nd1 <sup>ii</sup>	138.0 (5)	Nd1—O2W—H2WA	109.30	Nd1—O2W—H2WB	109.40
C17—O7—Nd1	93.4 (4)	C17—O8—Nd1	93.8 (4)		

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



Fig. S1 PXRD patterns of Eu-L, Nd-L, and simulated Nd-L.



Fig. S2 PXRD patterns of Tb-L, Eu-L, Nd-L and derived hybrid materials.



Fig. S3 FT-IR spectra of the SBA-15 type matrices and hybrid materials.



Fig. S4 FT-IR spectra of the MCM-41 type matrices and hybrid materials.



Fig. S5 TGA curves of (a) Tb-L, (b) Eu-L, (c) Nd-L, and derived hybrid materials.



**Fig. S6** Excitation and emission spectra: (a) Tb-L, Tb-SBA-15, and Tb-MCM-41; (b) Eu-L, Eu-SBA-15, and Eu-MCM-41; (c) Nd-L, Nd-SBA-15, and Nd-MCM-41. All samples underwent heating treatment at 200 °C; spectra were then measured at room temperature. Inset photographs in (a) and (b) show solid samples of free coordination polymers (left), SBA-15-type (middle), and MCM-41-type hybrid materials (right) under UV irradiation (365 nm).



**Fig. S7** PXRD patterns of coordination polymers and hybrid materials after undergoing the heating treatment at 200 °C.



**Fig. S8** PXRD patterns of coordination polymers and hybrid materials after being soaked in boiling water for 1 day.



**Fig. S9** Excitation and emission spectra: (a) Tb-L, Tb-SBA-15, and Tb-MCM-41; (b) Eu-L, Eu-SBA-15, and Eu-MCM-41; (c) Nd-L, Nd-SBA-15, and Nd-MCM-41. All samples were soaked in boiling water for 1 day; spectra were then measured at room temperature.



**Fig. S10** PXRD patterns of (a) Tb-L, (b) Tb-SBA-15, and (c) Tb-MCM-41 after being soaked in aqueous solutions at different pH (pH = 3-11) for 12 h.



**Fig. S11** PXRD patterns of (a) Eu-L, (b) Eu-SBA-15, and (c) Eu-MCM-41 after being soaked in aqueous solutions at different pH (pH = 3-11) for 12 h.



**Fig. S12** (a) Scatter diagram of luminance changes over time for various samples. (b) Images of commercial available UV-LED cells ( $\lambda_{em} = 365-370$  nm) coated with (from left to right): Tb-MCM-41, Tb-SBA-15, Tb-L, blank UV-LED, Eu-L, Eu-SBA-15, and Eu-MCM-41-NH<sub>2</sub>. (c) The same images as in (b) but after being illuminated. (d) The same images as in (b) but after being illuminated for 240 h.

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