Supplementary Material

DMF/H$_2$O Volume-Ratio-Controlled Assembly of 2D and 3D Ln-MOFs with 5-(Pyridin-4-yl)isophthalic Acid Ligand

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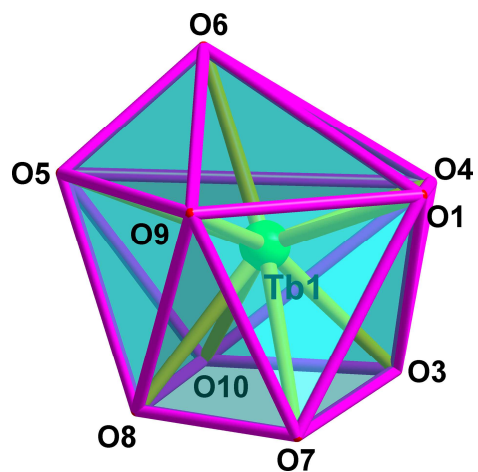[B]Corresponding authors. E-mail: zhangqingfu@lcu.edu.cn; jmdou@lcu.edu.cn.

Fig. A1. A distorted monocapped square-antiprism geometry of Tb1 in complex **1**.
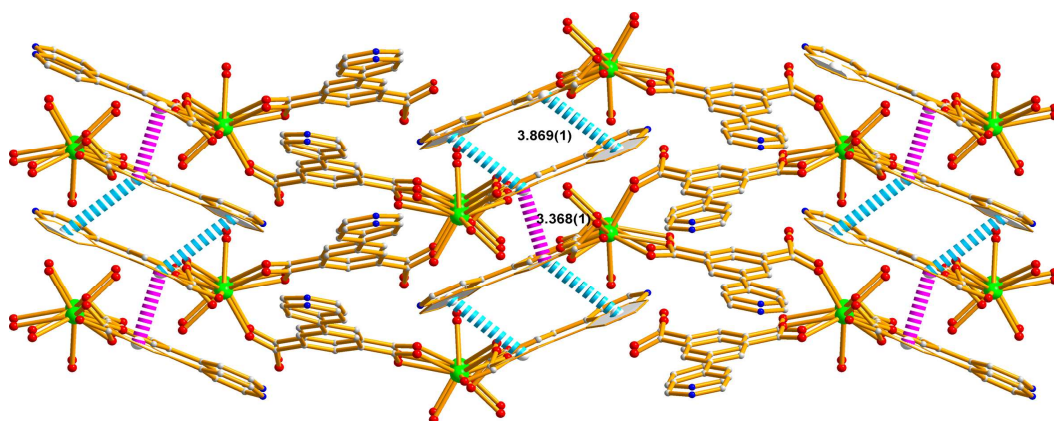

Fig. A2. A 3D supramolecular framework of complex **1** linked by π-π stacking interactions (3.368(1) Å & 3.869(1) Å).
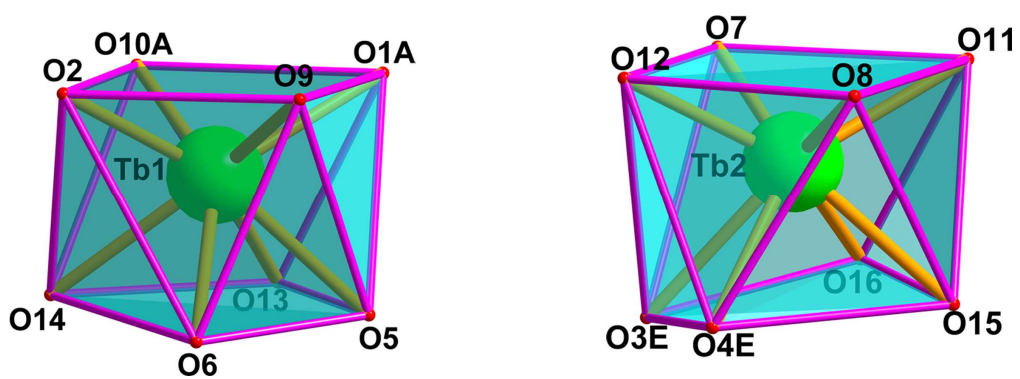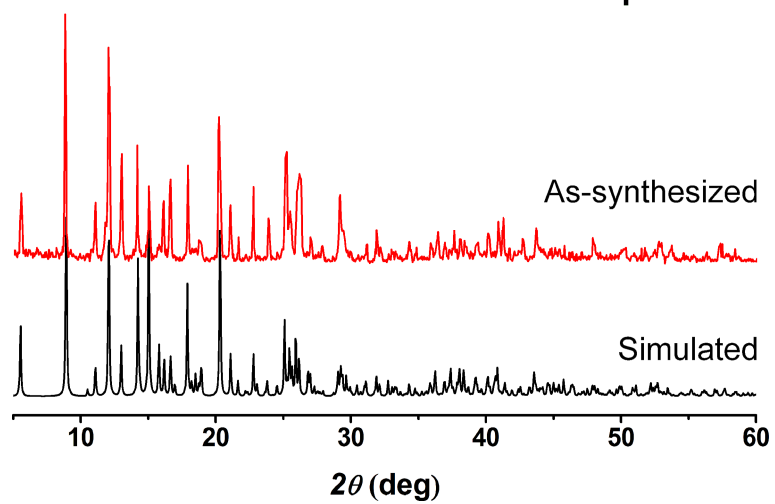

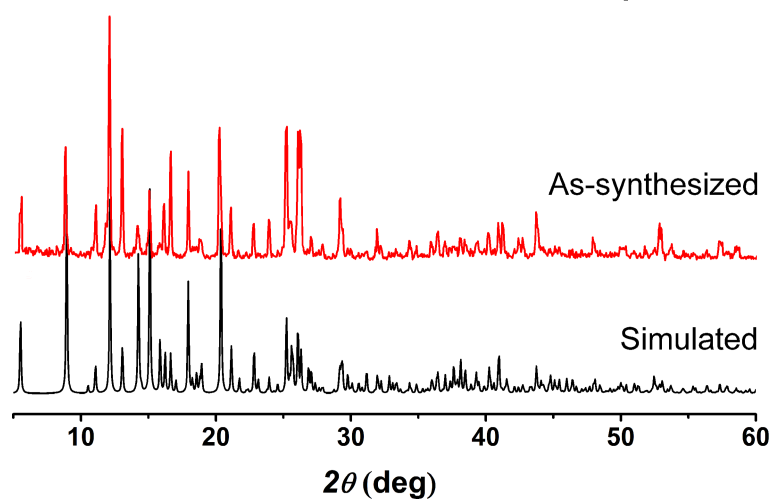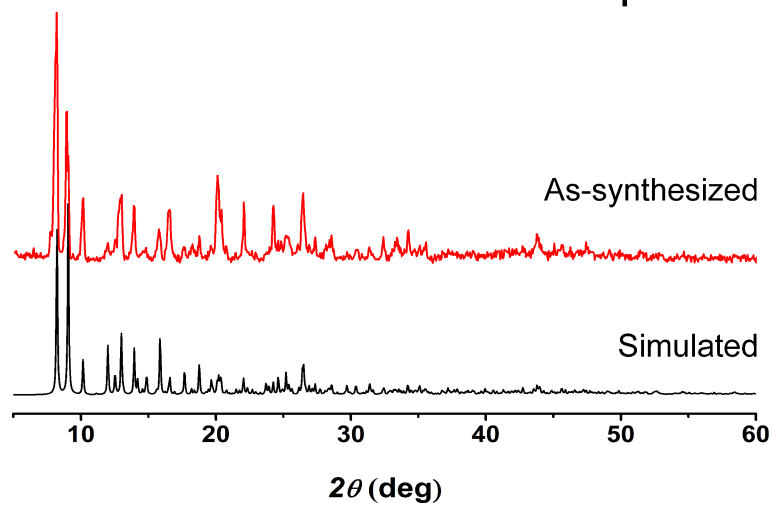**Fig. A3.** Distorted square-antiprism geometries of Tb1 and Tb2 in complex **3**.

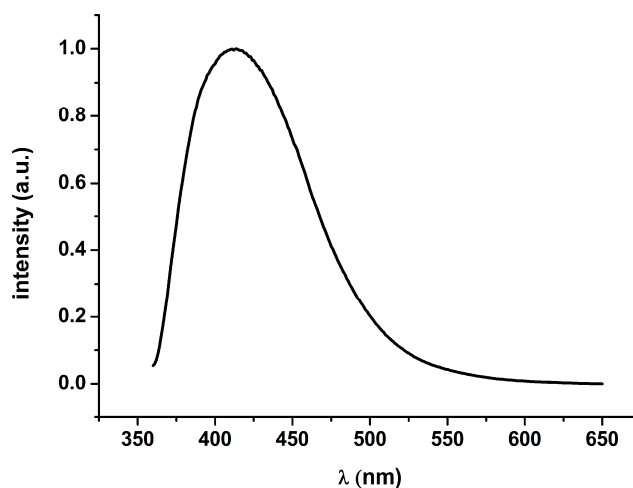**Fig. A4.** PXRD patterns of complexes **1**-**3**.

Fig. A5. The solid-state emission spectrum of free ligand H$_2$PIA at room temperature, upon excited by 330 nm.

Table A1. Selected bond lengths (Å) for complexes 1-3

Complex **1**[a]			
Tb(1)-O(1)	2.321(5)	Tb(1)-O(7B)	2.505(9)
Tb(1)-O(3A)	2.427(6)	Tb(1)-O(8B)	2.400(7)
Tb(1)-O(4A)	2.639(6)	Tb(1)-O(9)	2.446(5)
Tb(1)-O(5)	2.482(7)	Tb(1)-O(10)	2.493(9)
Tb(1)-O(6)	2.415(11)		
Complex **2**[b]			
Eu(1)-O(1)	2.306(4)	Eu (1)-O(7B)	2.514(6)
Eu (1)-O(3A)	2.652(5)	Eu (1)-O(8B)	2.384(5)
Eu (1)-O(4A)	2.436(5)	Eu (1)-O(9)	2.459(4)
Eu (1)-O(5)	2.506(5)	Eu (1)-O(10)	2.452(5)
Eu (1)-O(6)	2.429(7)		
Complex **3**[c]			
Tb(1)-O(1A)	2.363(10)	Tb(2)-O(3E)	2.485(10)
Tb(1)-O(2)	2.301(11)	Tb(2)-O(4E)	2.462 (12)
Tb(1)-O(5)	2.456 (10)	Tb(2)-O(7C)	2.311 (11)
Tb(1)-O(6)	2.444 (10)	Tb(2)-O(8B)	2.274 (10)
Tb(1)-O(9)	2.327 (12)	Tb(2)-O(11D)	2.348 (11)
Tb(1)-O(10A)	2.306 (10)	Tb(2)-O(12)	2.280 (12)
Tb(1)-O(13)	2.425 (12)	Tb(2)-O(15)	2.463 (14)
Tb(1)-O(14)	2.466 (11)	Tb(2)-O(16)	2.415 (12)

[a] Symmetry codes A: -x+1/2, y-1/2, z; B: x, y, z+1; [b] Symmetry codes A: -x+3/2, y+1/2, z; B: x, y, z+1; [c] Symmetry codes A: -x+1, -y, -z; B: -x+1/2, y+1/2, -z+1/2; C: x+1/2, -y-1/2, z+1/2; D:-x+1, -y, -z+1; E:-x+1/2, y-1/2, -z+1/2.