

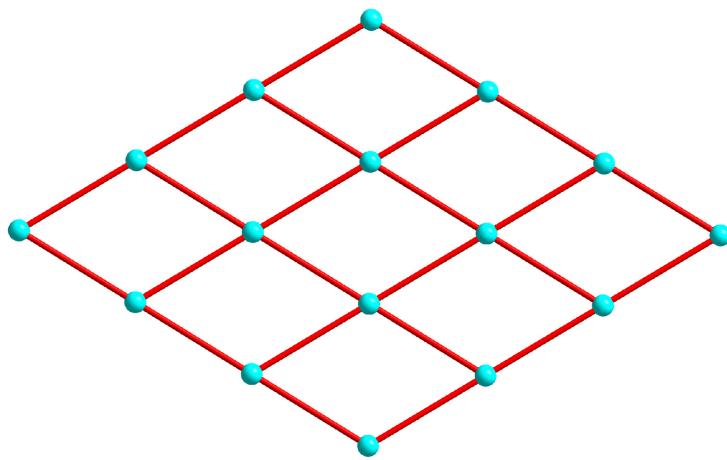
## ***Electronic Supplementary Information (ESI)***

**Controllable assembly of copper(I)-iodide coordination polymers by tecton design of benzotriazol-1-yl-based pyridyl ligands: from 2D layer to 3D self-penetrating or homochiral networks†**

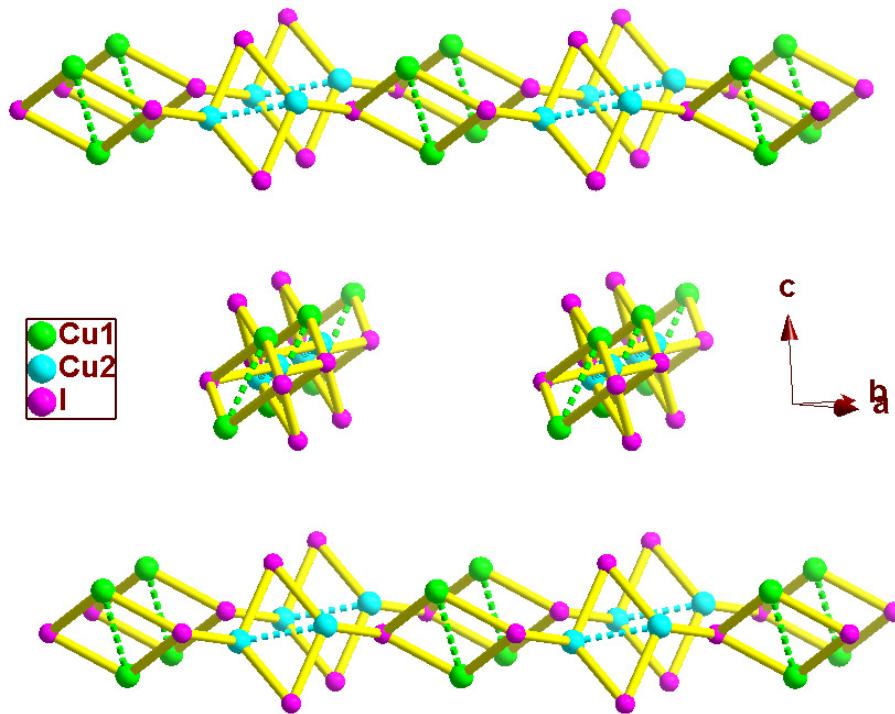
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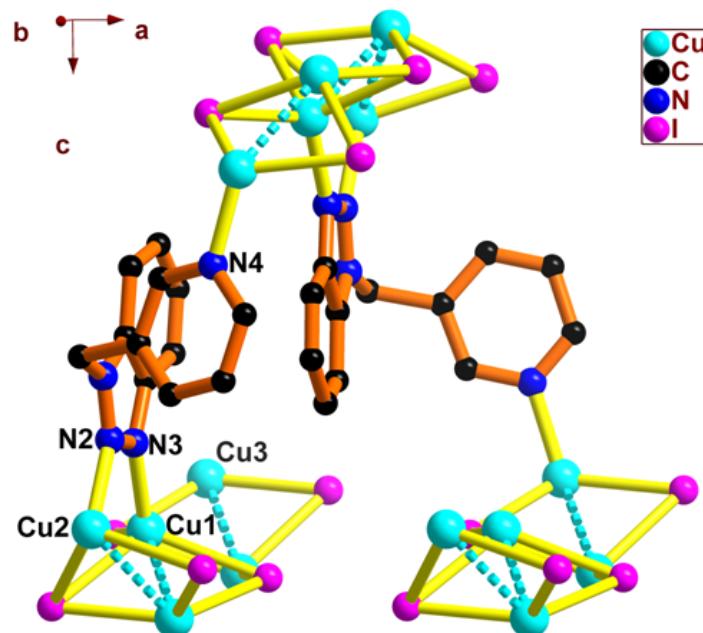
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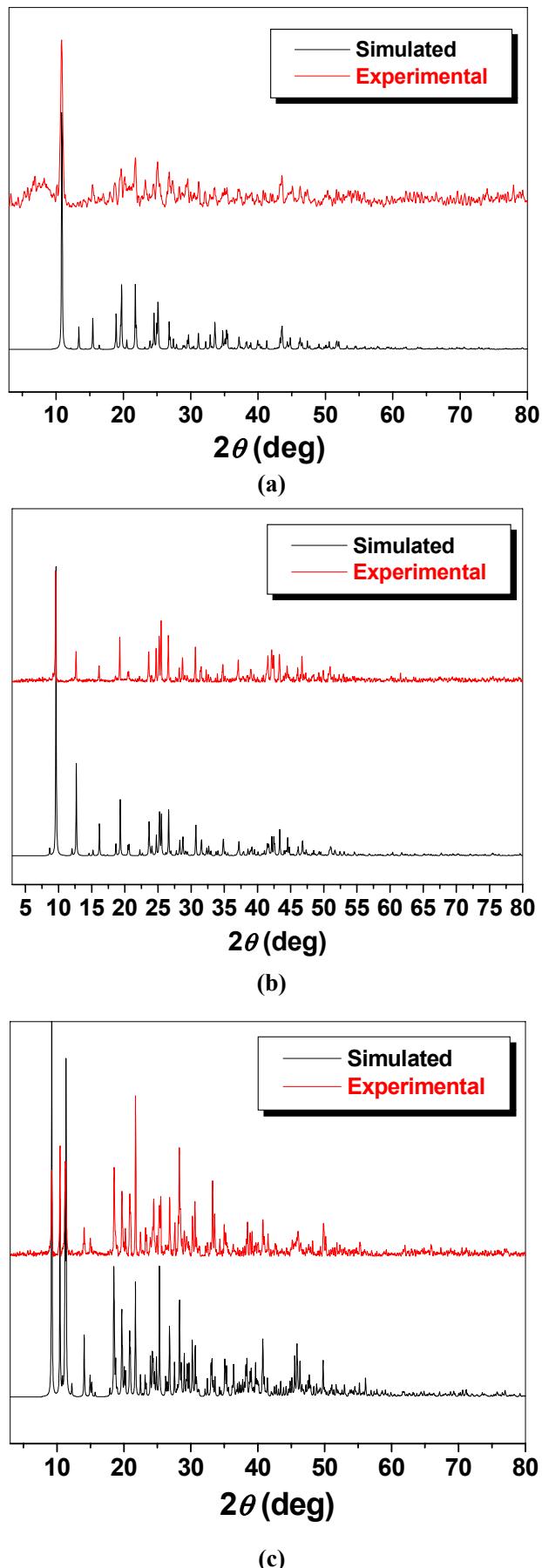
**Fig. S1** A schematic representation of the 2D (4,4) network in **1** (red rods: 2-pbt ligands; cyan spheres: rhomboid  $[Cu_2I_2]$  dimeric units).



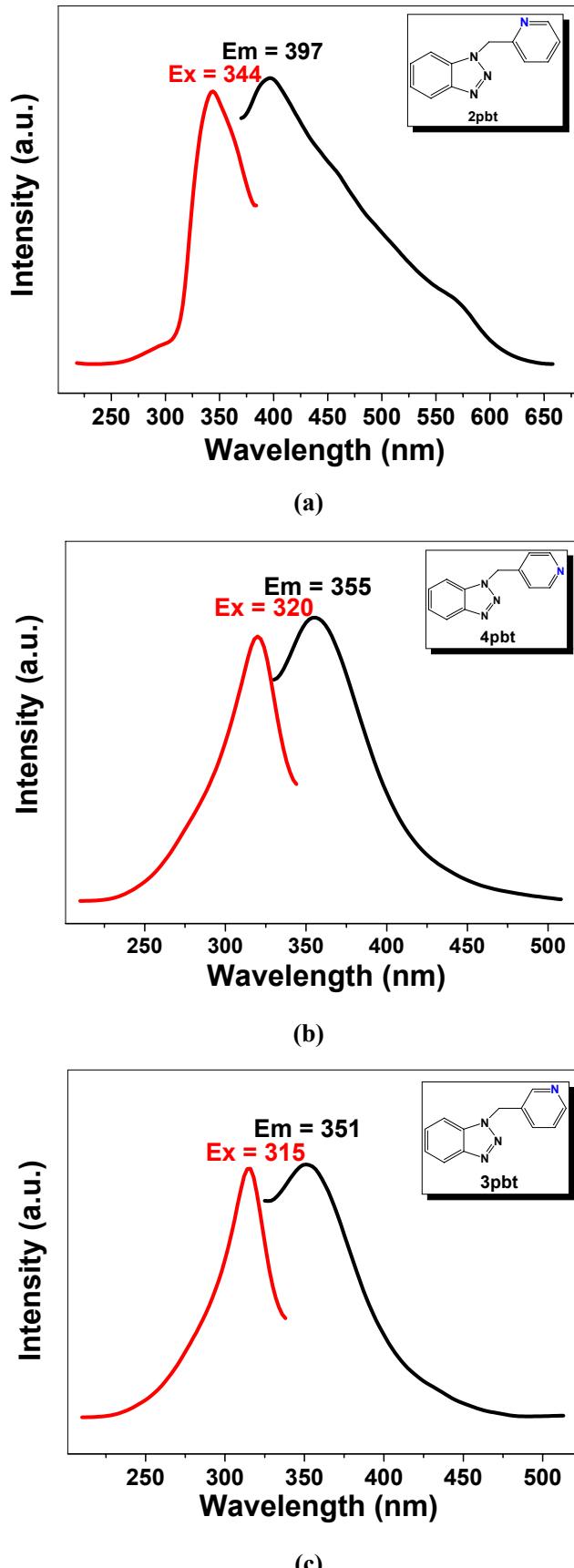
**Fig. S2** View of the perpendicularly arrangement of the (1D)  $[\text{Cu}_2\text{I}_2]_n$  skeletons, extracted from the 3D framework in **2**, running along the  $[110]$  and  $\bar{[110]}$  directions, respectively.



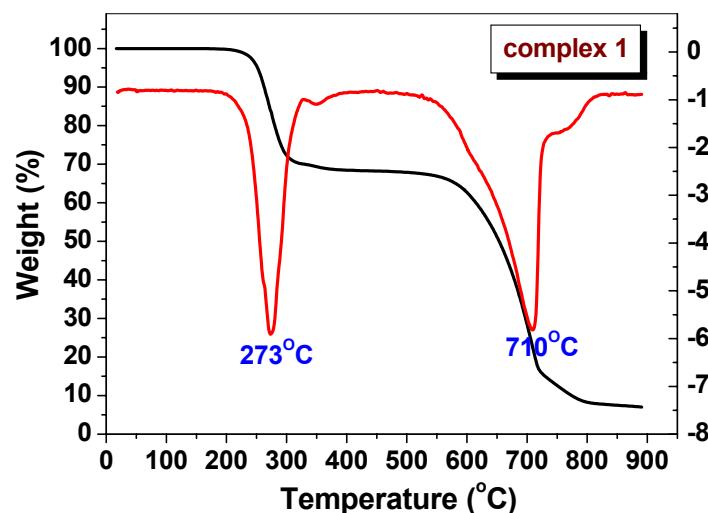
**Fig. S3** View of the coordination connectivity of 3-pbt between the adjacent  $[Cu_3I_3]_n$  skeletons in **3**, running along the crystallographic [010] direction.



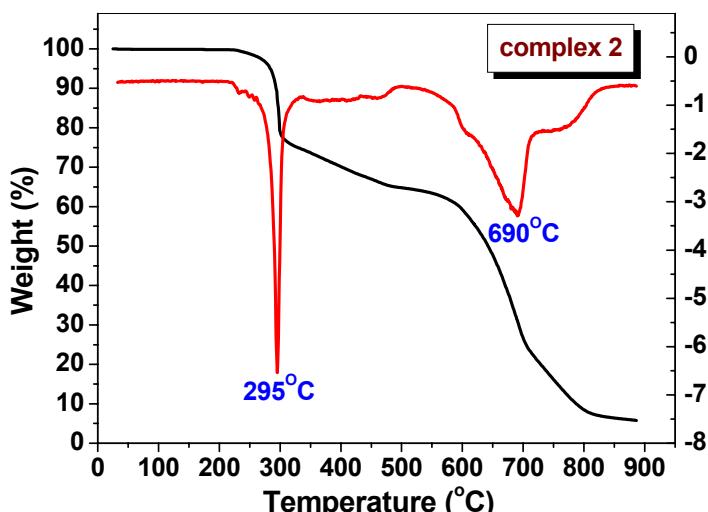
**Fig. S4** XRPD patterns of (a) **1**, (b) **2**, and (c) **3**.



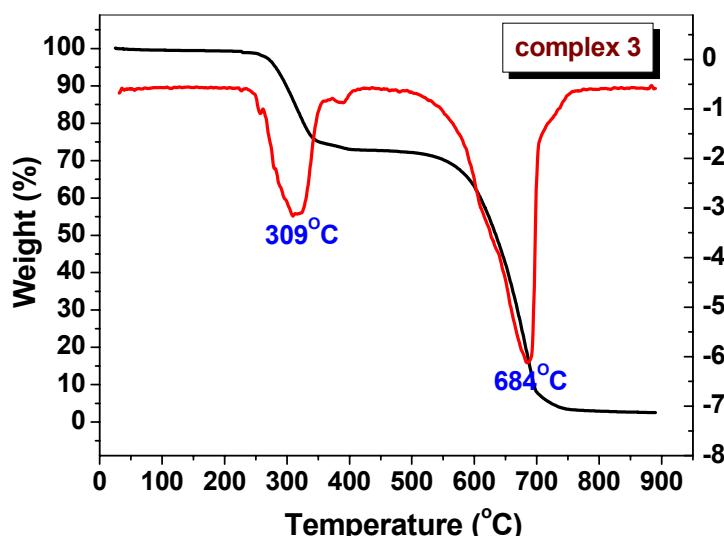
**Fig. S5** Excitation and emission spectra of the free ligands in the solid state at room temperature: (a) for 2-bpt, (b) for 4-bpt, and (c) for 3-bpt.



(a)



(b)



(c)

**Fig. S6** Thermogravimetric (TG) analysis plots of (a) 1, (b) 2, and (c) 3.