## **Electronic Supplementary Information**

## The first three-fold interpenetrated framework with two different four-connected uniform nets: 6<sup>6</sup> dia net and new chiral 8<sup>6</sup> mdf net

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**Figure S1.** Each  $Dy^{3+}$  and  $Cu^+$  ion could be considered as a four-connected node.

**Optical properties:** The photoluminescence property of **1**, the L<sup>-</sup> ligand and **2** has been explored at room temperature in the solid state under excitation of 405 nm, 329nm and 484nm, respectively.



Fig. S2 (a) Emission spectrum of 1 and the L'ligand. (b) Emission spectrum of 2

**Thermal Analysis**: The thermal analysis was performed on Netzsch STA449C under the flowing nitrogen atmosphere. **1** and **2** was heated from room temperature to 1000 °C at a heating rate of 10 °C/min.



Figure S3. The TGA diagram of 1 and 2.

There is one step weight loss from 360 to 440°C. Assuming that the residue corresponds to  $Ln_2O_3$  and CuO the observed weight (26.3% for 1, 26.8% for 2) is in good agreement with the calculated value (26.1% for 1, 26.4% for 2).

**X-ray powder diffraction**: X-ray powder diffraction experiments were performed on A PANaly tical X'pert PRO diffractometer with Cu Ka radiation (40 kV, 40 mA) was used to identify the crystal structure of the sample. The measurement was conducted in the continuous scanning mode. The 2 $\theta$  scanning range was from 5° to 50° in steps of 0.02° with a collection time of 12 s per step.



Figure S4. Simulated PXRD patterns of 1 and experimental PXRD patterns of 1 and 2.

**IR spectroscopy:** The IR spectra of **1** and **2** are similar. The characteristic features of the L<sup>-1</sup> ligand dominate the IR spectrum. The strong vibrations appearing at 1589 and 1418 cm<sup>-1</sup> correspond to the asymmetric and symmetric stretching vibrations of the carboxylate group, respectively. The absence of strong bands in the range 1690–1730 cm<sup>-1</sup> indicate IN ligands are deproponated



Figure S5. IR spectra of 1 and 2.