

The first 2D→3D polycatenation array built on (3, 4)-connected bilayer nets

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Materials and Physical Measurements.

The acylamide ligand of **1** is synthesized according to literature method.¹ Others were analytically pure from commercial sources and used without further purification. Elemental analyses were performed on a Vario EL-II analyzer. Single crystal X-ray diffraction is carried out on Bruker Apex II. X-ray powder diffraction (XRPD) data were recorded in a Bruker D8 ADVANCE diffractometer. Thermal analyses were carried out in air atmosphere using SETARAM LABSYS equipment with a heating rate of 5°C/min. Steady-state photoluminescence spectra were measured on a SHIMADZU RF-5301PC spectrofluorophotometer. The sample used for measurement of photoluminescence spectra is solid and treated by tablet machine to give suitable piece. Then, this piece is fixed on a bracket of spectrofluorophotometer.

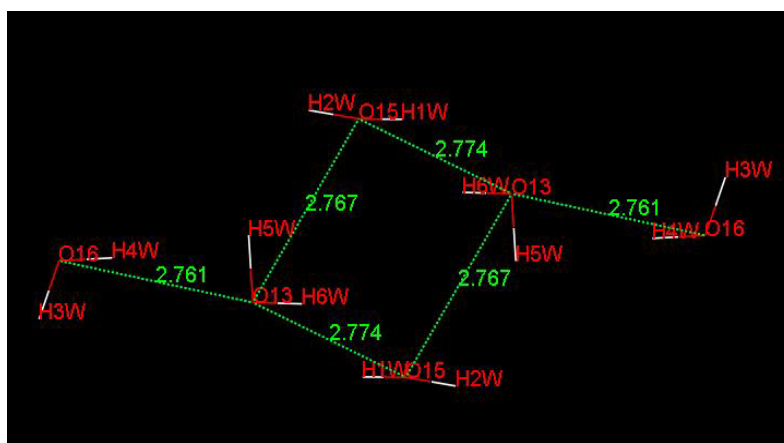


Figure S1. View of the atom-labelled six-nuclear water cluster: the O...O distances are highlighted in the color of green.

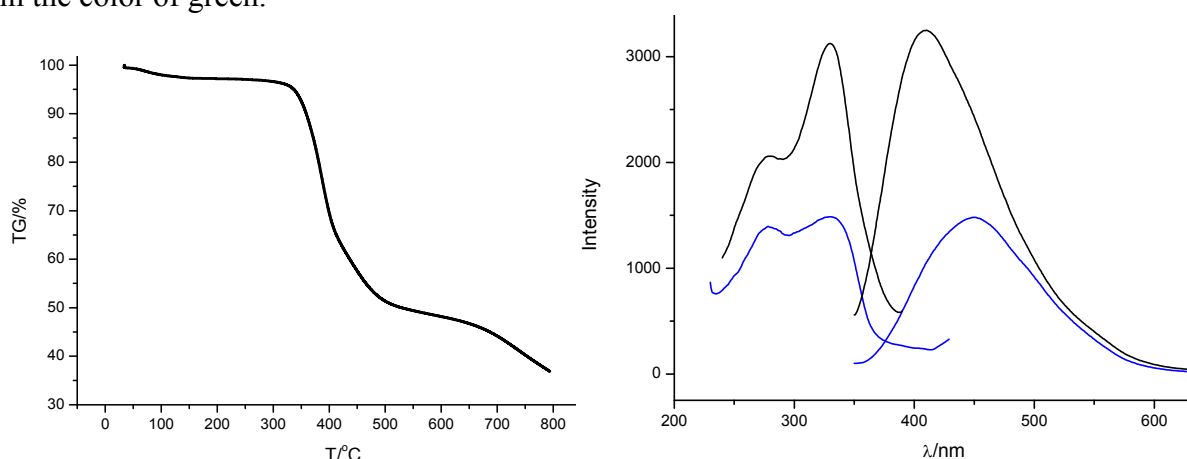


Figure S2. The TG and luminescence studies of **1**: the excitation spectrum and emission spectrum for **1** and L ligand is highlighted by black and blue.

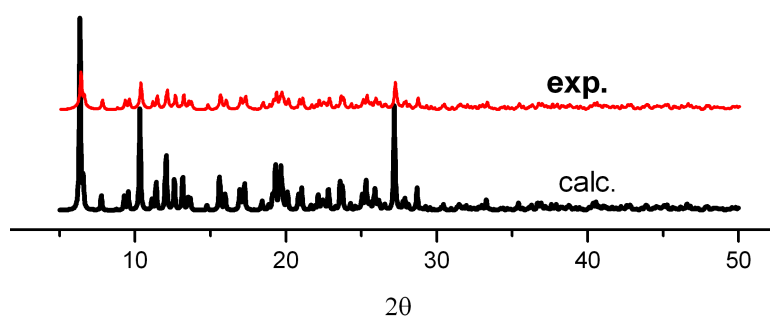


Figure S3. The experimental XRD pattern and the simulated XRD pattern from the single crystal data.