Electronic Supplementary Information (ESI) for

Synthesis and gas sorption property of a homochiral metal–organic framework with octahedral cages

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Experimental Section

General information: All of the reagents and solvents used in reactions were purchased commercially and used without purification, unless otherwise indicated. The chiral ligands (S)-H_3PIA and (R)-H_3PIA were synthesized from L-proline, D-proline and benzene-1,3,5-tricarboxylic acid by our group. Elemental analyses were carried out by the analysis center of our institute. All Powder X-ray diffraction (PXRD) analyses were recorded on a Rigaku Dmax2500 diffractometer with Cu K α radiation (λ = 1.54056 Å). Thermal stability studies were performed on a NETSCHZ STA-449C thermoanalyzer with a heating rate of 10°C/min under an N_2 atmosphere in the range of 30~800°C.

Synthesis of [Cu_3((*S*)-PIA)$_2$(pyridine)$_3$(H_2O)]·THF·6.25H_2O (1-L): A mixture of (S)-H_3PIA (31mg, 0.1 mmol) and $Cu(NO_3)_2$·2.5H_2O (47 mg, 0.2 mmol) was dissolved in a mixed solvent of THF and H_2O (3mL/1mL) with two drops of pyridine in a Teflon reactor. The reaction mixture was heated at 100℃ for 48 hours and then cooled to room temperature. Green-blue block crystals (32 mg, 60%, based on (S)-H_3PIA) were obtained after filtration. Elemental analysis calcd (%) for **1-L**: C 47.35, H 3.97, N 5.26; found C 46.48, H 4.12, N 5.45

Synthesis of [Cu_3((*R*)-PIA)$_2$(pyridine)$_3$(H_2O)]·THF·6.25H_2O (1-D): The same procedure as **1-L**, except (R)-H_3 PIA was used. Green-blue powdery crystals (19 mg, 35%, based on (R)-H_3PIA) were obtained after filtration. Elemental analysis calcd (%) for **1-D**: C 47.35, H 3.97, N 5.56; found C 46.71, H 4.11, N 5.31.

Measurements of solid CD spectra: The mixture of sample and 50 mg dry KCl powder was well grounded and then pressed into a disk for the CD measurement with a MOS-450 spectropolarimeter.

Gas adsorption measurement: A freshly prepared sample of **1-L** was also soaked in acetone at 40℃ for three days to exchange guest molecules. Subsequently, the sample was degassed under dynamic vacuum at 80℃ for 5 h to activate the sample. Finally, gas adsorption measurements was performed in the ASAP (Accelerated Surface Area and Porosimetry) 2020 System.

Table S1. Summary of Crystal Data and Refinement Results

Compound reference	1-L
Chemical formula	$C_{38}H_{30}Cu_3N_4O_{15}$•$C_4H_8O$•6.25($H_2O$)
Formula Mass	1160.00
Crystal system	Monoclinic
a/Å	20.7929(8)
b/Å	12.4871(7)
c/Å	19.5025(9)
α/°	90.00
β/°	97.329(4)
γ/°	90.00
Unit cell volume/Å^3	5022.3(4)
Temperature/K	293(2)
Space group	*C*2
No. of formula units per unit cell, *Z*	4
Radiation type	CuKα
Absorption coefficient, *μ*/mm^{-1}	2.204
No. of reflections measured	9300
No. of independent reflections	6627
R_{int}	0.0323
Final R_1 values ($I > 2\sigma(I)$)	0.0549
Final $wR(F^2)$ values ($I > 2\sigma(I)$)	0.1330
Final R_1 values (all data)	0.0668
Final $wR(F^2)$ values (all data)	0.1418
Goodness of fit on F^2	1.108
Flack parameter	-0.03(4)

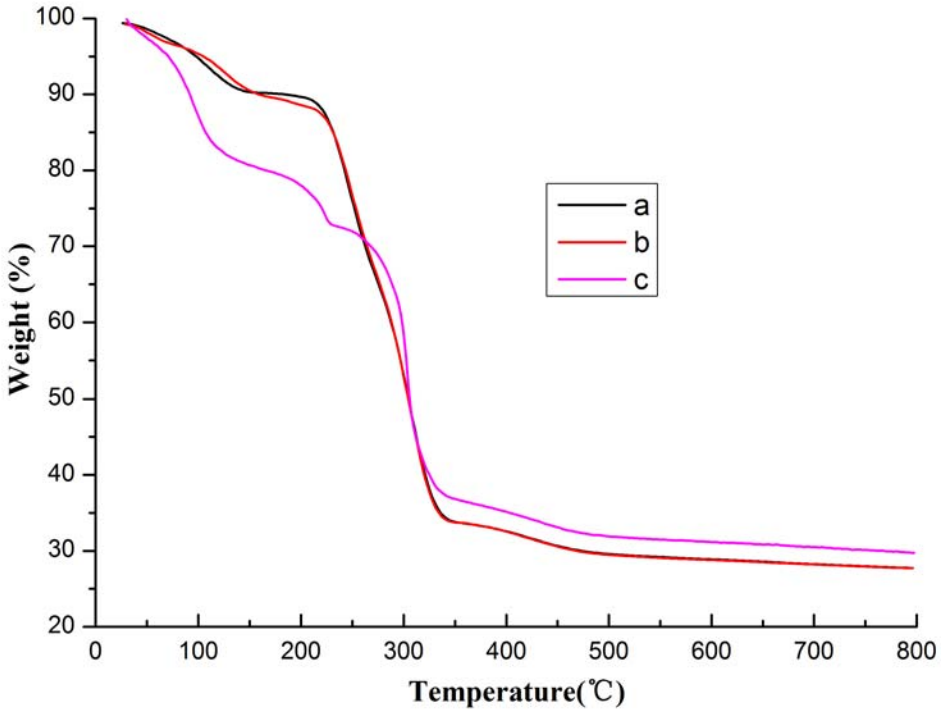

Figure S1. The TGA diagrams of **1-L** (a), the TGA diagrams of **1-D** (b) and the TGA diagrams of **1-L** soaked in acetone (c).

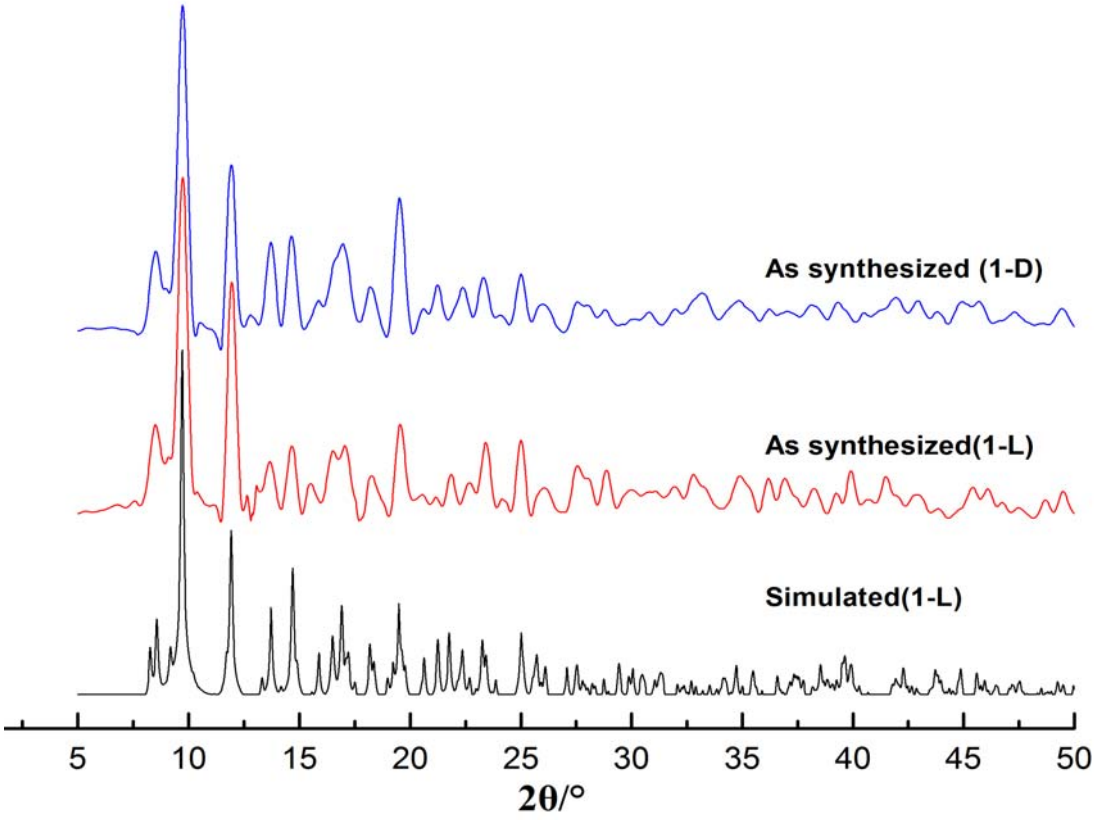

Figure S2. XRPD patterns of **1-D** and **1-L**.

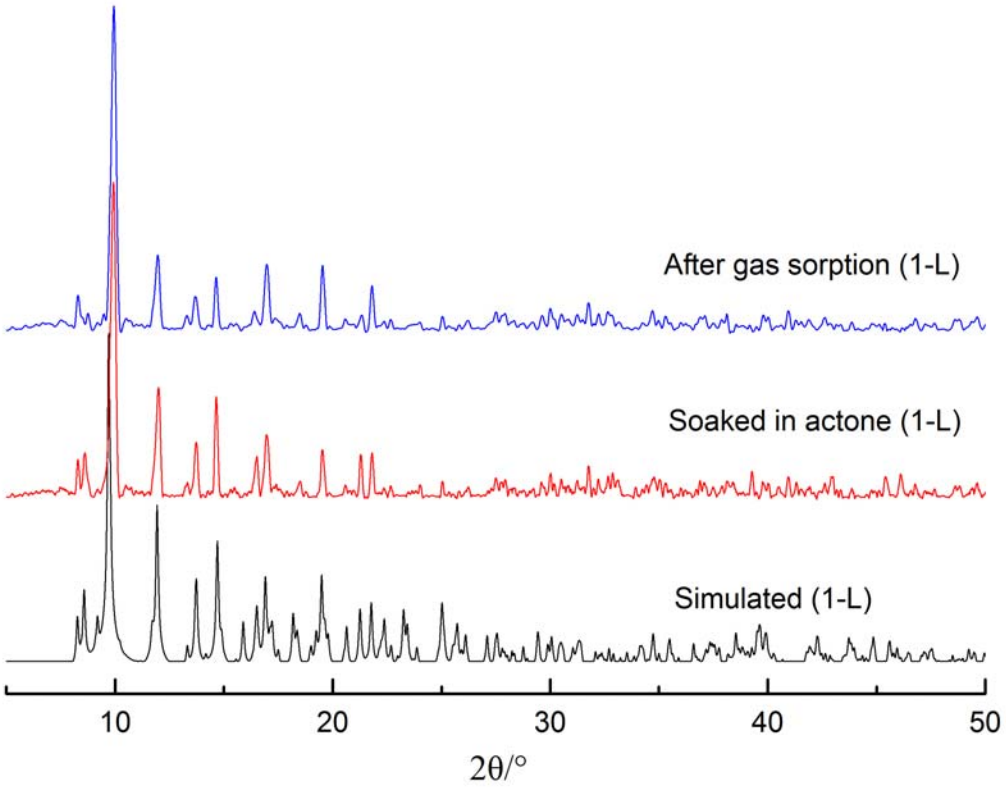

Figure S3. XRPD patterns of **1-L**.