

Supporting information

“Systematic mechanochemical preparation of a series of coordination pillared layer frameworks”

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Experimental Section

Materials

Solvent and starting materials for synthesis were purchased commercially, and were used as received. Preparations of CPLs in solution process were carried out according to the literature procedures.

Physical measurements

X-ray powder diffraction was carried out on a Rigaku RINT-2000 Ultima diffractometer with CuK α radiation ($\lambda = 1.54056 \text{ \AA}$).

The sorption isotherm measurements were performed by using an automatic volumetric adsorption apparatus (BELSORP-18; Bel Japan, Inc.). A known weight (100-200 mg) of the as-synthesized sample was placed in the quartz tube, then, prior to measurements, the sample was dried under high vacuum ($<10^{-2} \text{ Pa}$) at 383 K for 8 h to remove the solvated water molecules. The adsorbate was placed into the sample tube, then the change of the pressure was monitored and the degree of adsorption was determined by the decrease of the pressure at the equilibrium state.

Observation of time course of the transition, **M to **CPL-1****

To investigate the transition behavior from **M** to **CPL-1**, time course of XRPD patterns of **M** was sequentially measured for 12 hours as shown in Fig. S1. As the measurements were proceeding, the peaks of the initial pattern were gradually decaying, and simultaneously the peaks of **CPL-1** were emerging. Although the transition had not completed after 12 h of the measurement, the sample quickly changed its color when taken out from the chamber of the powder diffractometer, and showed clear patterns of **CPL-1** within several hours left in air. After the sample was left in air for further 12 h, no more changes were observed in the patterns. We assumed that the difference in the transition rate was attributed to the humidity in the chamber or in air.

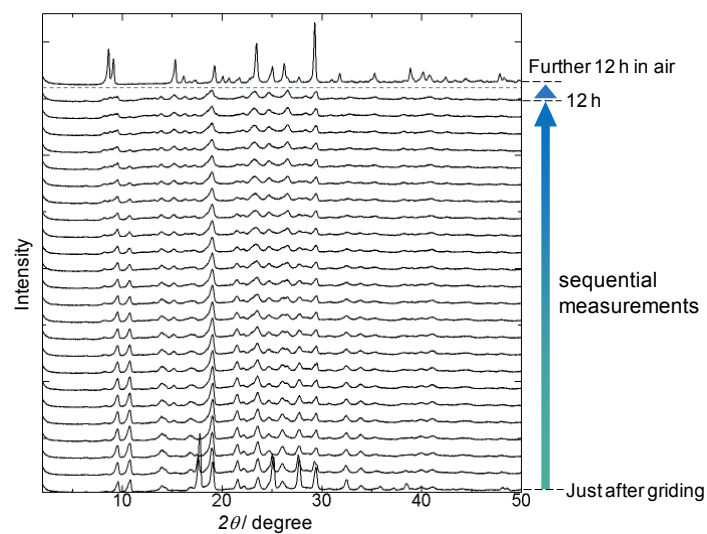


Fig. S1 Time course of XRPD patterns of **M** sequentially measured for 12 h (30 min per one measurement).

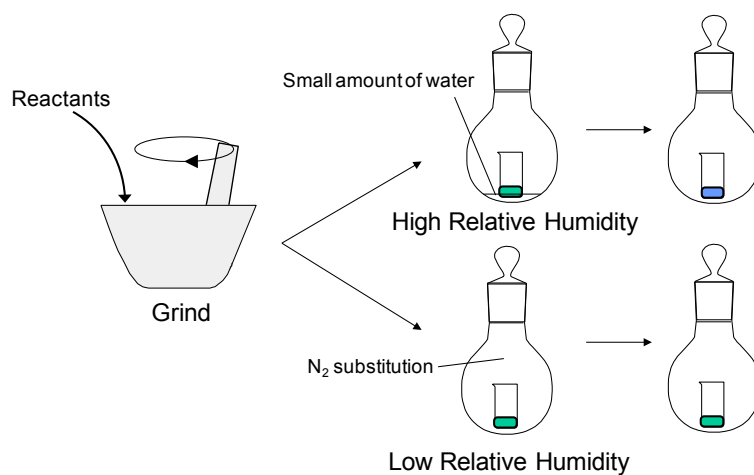


Fig. S2 Schematic illustration of the protocol in the humidity control experiment