Supporting Information

A Triply Interpenetrated Microporous Metal-Organic Framework for Selective Sorption of Gas Molecules

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Experimental Section

Materials and Methods: All reagents and solvents employed were commercially available and used as supplied without further purification. TGA data were obtained on an TGA G500 V5.3 Build 171 instrument with a heating rate of 5 deg/min under N₂ atmosphere. Powder XRD patterns were obtained with a Scintag X1 powder diffractometer system using K_{α} radiation with a variable divergent slit and a solid-state detector. The routine power was 1400 W (40 kV, 35 mA). Low-background quartz XRD slides (Gem Depot, Inc., Pittsburgh, PA) were used. For analyses, powder samples were dispersed on glass slides.

Single-crystal X-ray Crystallography: Intensity data for **1** were collected using Bruker X8 APEX II diffractometer (Mo radiation) in a cold nitrogen stream. Data collection and reduction were done using Bruker Apex2 software package. Structures were solved by direct methods and refined by full-matrix least squares, using SHELXL97. All non-hydrogen atoms were refined anisotropically.

Gas sorption measurements: A Beckman Coulter SA3100 surface area analyzer was used to measure gas adsorption. In order to remove guest solvent molecules in the

framework, the fresh sample soaked in methanol was filtered and vacuumed at temperature 150°C overnight to form MOF **1a**. Before the measurement, the sample was vacuumed again by using the "outgas" function of the surface area analyzer for 2 hours at 150 °C. A sample of 90.0 mg was used for sorption measurement, and was maintained at 77K with liquid nitrogen, and at 195 K with acetone/dry ice slush, respectively.



Figure S1. Crystal structure of **1** within the unit cell indicating the local coordination environments around Zn.



Figure S2. Powder X-ray diffraction (PXRD) patterns of the simulated MOF 1 and the as-synthesized 1.



Figure S3. TGA trace of as-synthesized **1**. (Note: TGA indicates that the fresh-prepared MOF **1** loses solvent molecules in two steps with total weight loss of 32.94 % that is slightly higher than that calculated (30.20%) based on $Zn(ADC)(4,4'-Bpe)_{0.5}(DMF)_{2.5}(H_2O)_{0.5}$ for the sample dried in the air for several hours. Such difference is because the as-synthesized **1** lost part of the solvent molecules in the air).