Supplementary Material (ESI) for Chemical Communications

SUPPLEMENTARY INFORMATION

Structure Determination Directly from Powder X-ray Diffraction Data of a Metal-Organic Framework Material Prepared by Solvent-Free Grinding

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Experimental Details

The material **1-M-P** was prepared by grinding together $Zn(OAc)_2.2H_2O$ (87.6 mg, 0.40 mmol), H_2fma (46.4 mg, 0.40 mmol) and bipy (31.2 mg, 0.20 mmol) in a 25 ml steel jar containing a 32 g steel ball bearing using a Retsch MM301 mixer mill at 25 Hz for 20 mins. Because no product purification was to be carried out, the purity levels and hydration states of the starting materials required careful consideration. As ^{1}H NMR and microanalysis indicated that the commercial bipy sample contained variable amounts of water, it was dried under vacuum for 8 hours before use, after which no detectable amount of water was present. The fumaric acid was analytically pure and was used as received. The product **1-M-P** was obtained as a free-flowing white powder.

Desolvation of **1-M-P** to produce **1-M-D** was carried out by heating a sample of **1-M-P** in an oven at 150 °C for 20 hrs.

A sample of **1-S-DMF** was prepared using the published solvothermal method[16] with $Zn(NO_3)_2.6H_2O$, fumaric acid and bipy heated in DMF-water-ethanol for 2 days at 60 °C.

For structure determination of **1-M-D**, powder XRD data were recorded at ambient temperature on a Bruker D8 diffractometer ($CuK_{\alpha 1}$) with a linear position-sensitive VANTEC detector covering 12° in 2θ. The data were recorded in transmission mode using a rotating foil sample holder (2θ range, 4° – 70°; step size, 0.0167°; data collection time, 17 hours).

High-resolution solid-state ^{13}C NMR spectra of **1-M-P** and **1-M-D** were recorded on a Varian VNMRS spectrometer (40.53 MHz) under conditions of $^{13}C \leftarrow ^{1}H$ cross-polarization (CP), magic angle spinning (MAS) and high-power ^{1}H TPPM decoupling (CP contact time, 3 ms; recycle delay, 1 s; MAS frequency, 6.8 kHz).

Figure S1 SEM image of **1-M-P**.

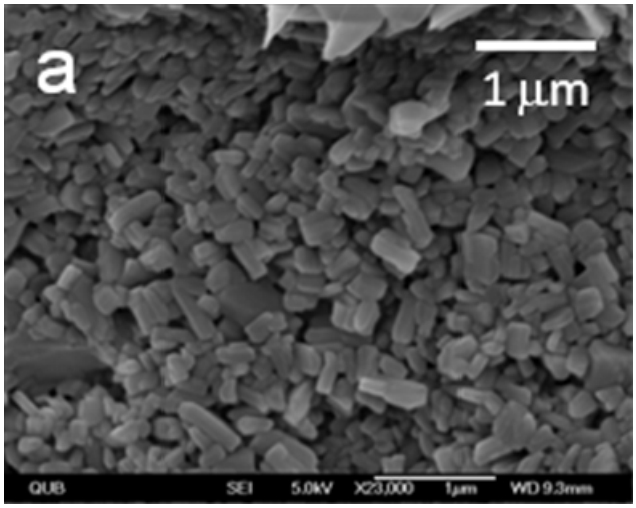

Figure S2 Le Bail fitting of the powder XRD pattern of **1-M-D**, showing the experimental (+), calculated (–) and difference (bottom) powder X-ray diffraction profiles. Reflection positions are marked.