Electronic Supplementary Material (ESI) for Physical Chemistry Chemical Physics.

SUPPLEMENTARY INFORMATION

Simultaneous Neutron Diffraction and Microwave Characterisation at Elevated Temperatures

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Simulated Neutron Powder Diffractogram

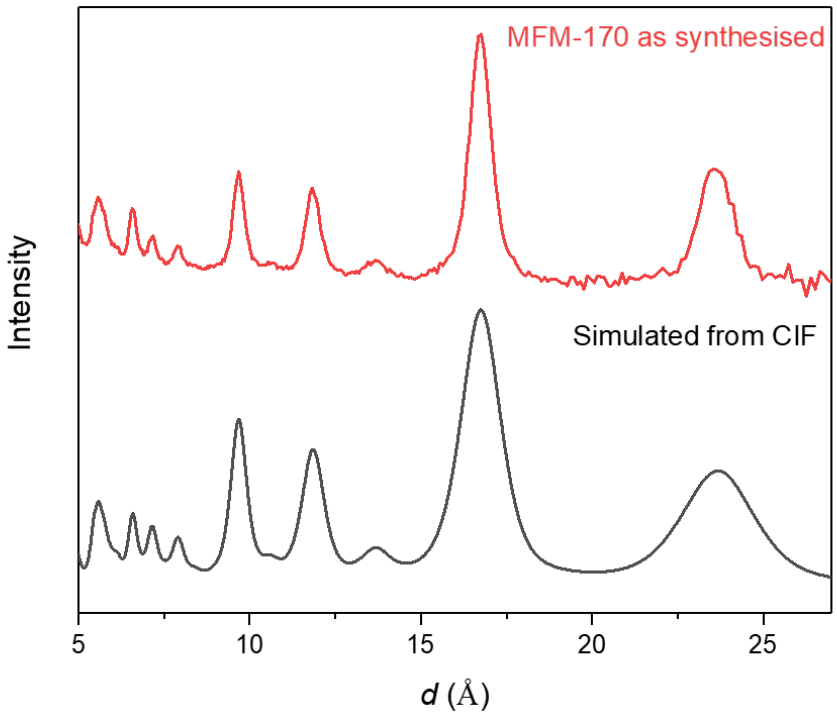

Figure S1. As synthesised NPD plot for MFM-170 from bank 1 of POLARIS (red) and the simulated NPD pattern for MFM-170 generated from the crystal structure (black). The poor signal-to-noise ratio is due to the fast collection time of the NPD data (15 x 2-minute runs summed).

PID value for temperature controllers

The heating coils were initially set to 60 °C during optimisation of PID values, which are shown in Table S1.

Table S1. PID values of the heating coils set during Section A.

Heating Coil	P	I	D
Top	100	120	20
Bottom (0-15 minutes)	100	600	100
Bottom after 15 minutes*	100	360	60

*The temperature of the top heating coil stabilised within 15 minutes, however the temperature of the bottom coil initially fluctuated by approximately 20 °C, thus PID values for the bottom coil were altered to correct for this.

Once PID values had been changed the temperature of both coils stabilised within 20 minutes. PID values given should be used as representative values as subtle changes in sample environment would require changes in PID values.

TGA-MS Data

Thermal Gravimetric Analysis–Mass Spectrometry (TGA-MS) was conducted *ex situ* on an air-dried as synthesised sample of MFM-170. TGA-DSC were performed under a flow of air (50 mL/min) on a Discovery SDT 650 DSC/TGA coupled with a mass spectrometer to analyse the off-gas from the exhaust port. The sample was heated 20-450 °C at a ramp rate of 1°C/min (Fig S2). Intensity trends were monitored for m/z 43 and 18 for acetone and water, respectively, using the SEM detection mode.

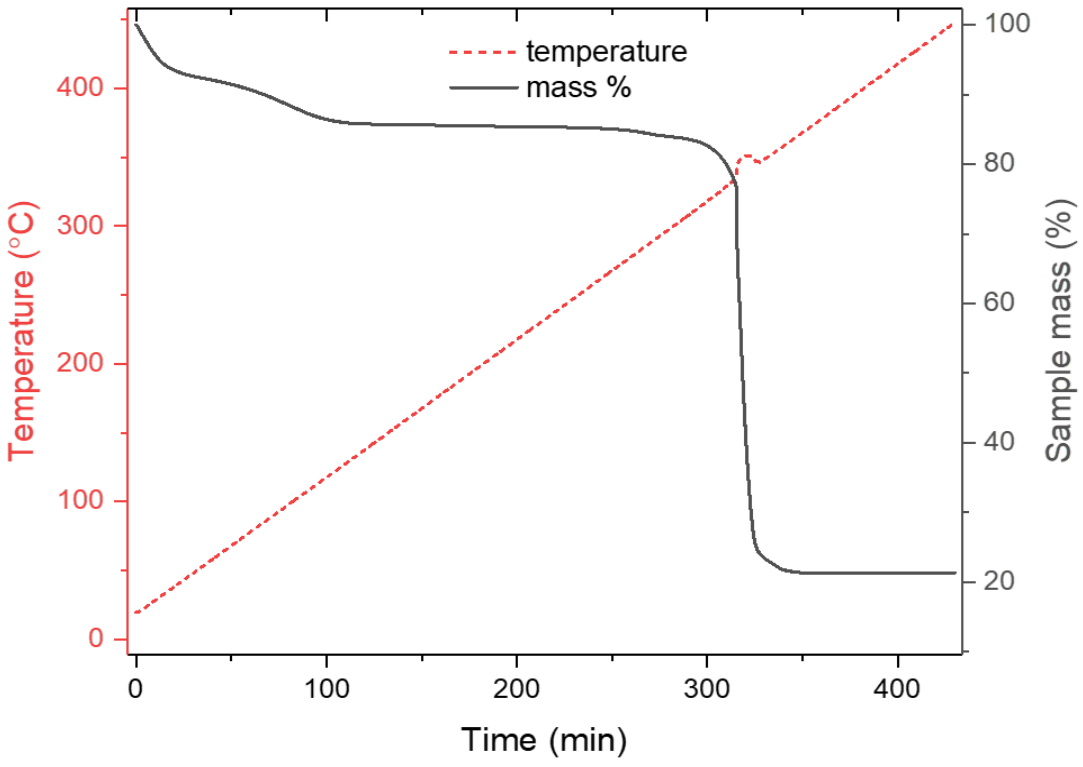

Figure S2. Full TGA plot of an air-dried as-synthesised sample of MFM-170 heated 20-450 °C at a ramp rate of 1°C/min, showing sample decomposition past 310 °C.

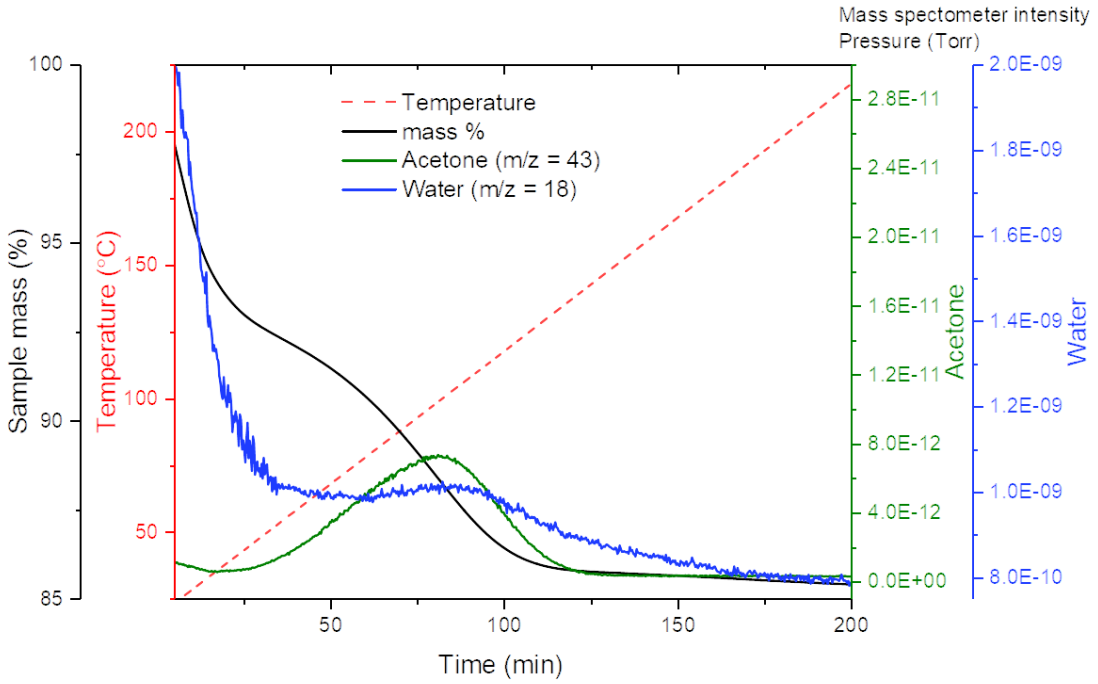

Figure S3. TGA-MS plot of an air-dried as-synthesised sample of MFM-170, heated at a ramp rate of 1°C/min. The plot has been cropped to 25-225 °C to show solvent loss on heating.