

## Supporting information:

### Three isolated structural motifs in one crystal: Penetration of two 1D chains through large cavities within 2D polymeric sheets

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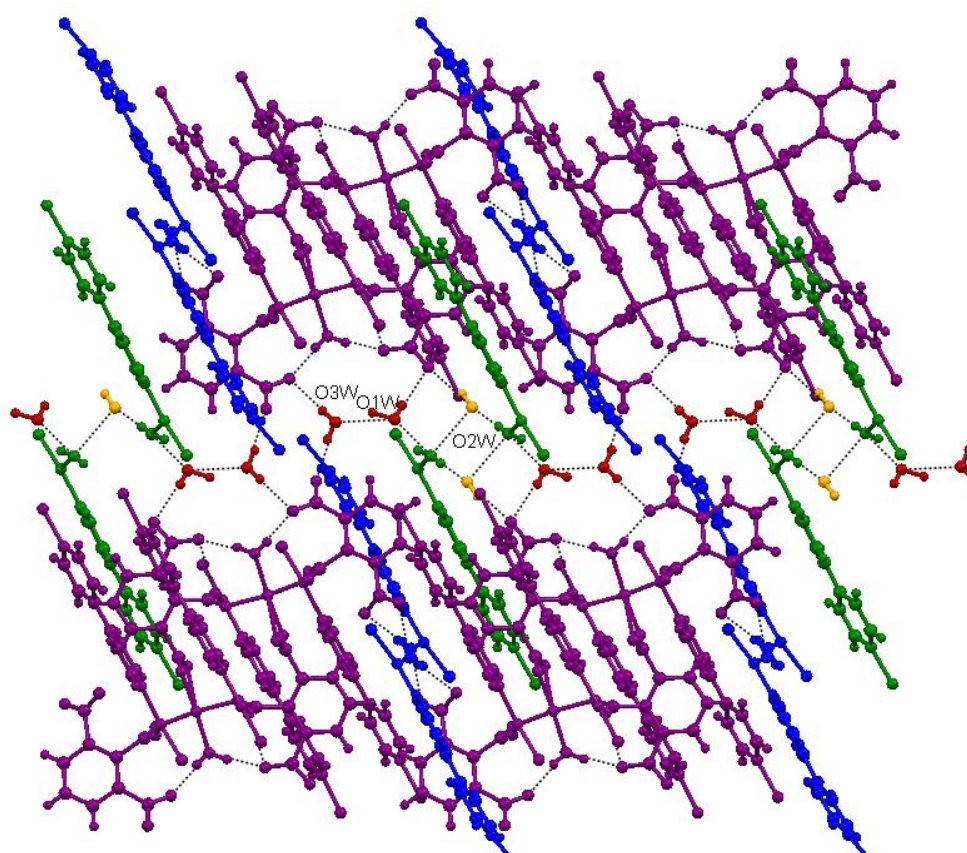


Figure S1: Detail of hydrogen-bonding between the 2D layers, generating the pseudo-3D structure. Polymer A (2D) shown in purple and the two 1D polymers, **B** and **C**, in green and blue respectively. Lattice water molecules are shown in red, whilst hydroxide ions ( $\text{OH}^-$ ) in yellow.

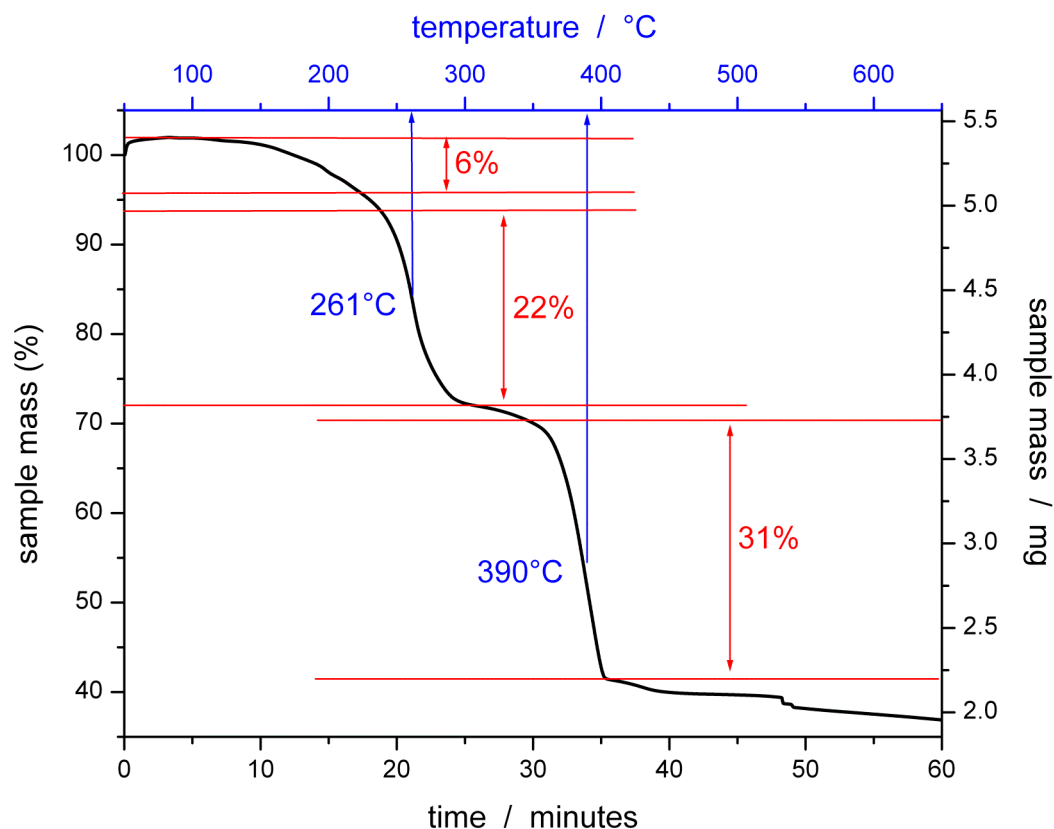


Figure S2: Thermogravimetric analysis of  $\text{Cu}_3(\text{Hbtc})(\text{btc})(\text{bpy})_2$ . Three distinct regions of mass loss were observed: (i) 6% over the temperature range of 150 - 220°C corresponding to the loss of water molecules (both coordinated and non-coordinated), *theoretical*: 9.8%; (ii) 22% around 261°C corresponding to the loss of the organic components of the 1D polymers **B** and **C**, *theoretical*: 21.3%; and (iii) 31% at 390°C corresponding to sample degradation. There is an additional small mass loss around 530°C. All samples were run under a flow of nitrogen at a heating rate of 10°C/min.