

**MS30-2-8 Structure Determination of a Mechanochemically Synthesised Metal-Organic Framework Using 3D Electron Diffraction**  
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**Abstract**

3D Electron Diffraction (3D ED) is an emerging technique for structural characterization of crystals of up to ten's of nanometres in size thanks to the strong interaction between electrons and matter, thus overcoming the main limitation of Single Crystal X-Ray Diffraction (SCXRD)<sup>1</sup>.

Metal-Organic Frameworks (MOFs) on the other hand, are hybrid porous materials that have been receiving much attention in the recent years by reason of their versatility as well as tunability, leading to a wide variety of applications such as catalysis, separation, sensing, storage, amongst others. The crystal structure of the MOFs plays an important role on their properties and applications. However, it is often very difficult to grow crystals large enough for conventional SCXRD, making them great candidates for characterization by 3D ED<sup>2</sup>.

In this work, we describe the structure solution of a MOF with particle size of approximately 250nm synthesized through an environmentally friendly technique: Liquid Assisted Grinding mechanosynthesis, using water as solvent and biocompatible precursors. Using protocatechuic acid as ligand we have been able to obtain a mixed-metal MOF containing both Mn and Cu in the structure. The structure solution using 3D ED data was obtained ab-initio and required a low dose experiment to avoid amorphization. The structure, which is monoclinic exhibits a relevant porosity. Interestingly, in the vacuum of the microscope, the channels seem to be completely solvent free.

**References**

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2. Huang, Z., Grape, E. S., Li, J., Inge, A. K. & Zou, X. 3D electron diffraction as an important technique for structure elucidation of metal-organic frameworks and covalent organic frameworks. Coord. Chem. Rev. 427, 213583 (2021).