3D ED: Bridging the gap between bulk powders and pristine single crystals

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The importance of structure determination in the study of functional crystalline materials and in synthetic chemistry more broadly is well accepted, with powder X-ray diffraction allowing a powerful tool to study bulk synthetic outcome and structural response to environment. However, *ab initio* structure solution of novel materials from powder diffraction is often prohibitively difficult, with the loss of 3D spatial information reducing scattering vectors to their magnitudes only often prohibiting even indexation of the unit cell. In this context, single-crystal X-ray diffraction has long been the gold-standard for structure solution, but requires reasonable quality single crystals of dimensions typically exceeding the micron regime, even when state of the art synchrotron beamlines are utilised. Where such crystals are not reasonably obtained, often the case for Metal Organic Frameworks (MOFs) and functional metal oxides, for example, this presents a clear gap in capability. Here, the advent of commercial, lab-based instrumentation for single-crystal structure determination by electron diffraction (i.e. 3D ED) has offered a breakthrough technique to complement the study of sub-micron crystallites.

In partnership with the UK's National Crystallographic Service at the University of Southampton, the University of Warwick has now been running the world's first national 3D ED facility for three years. Rather than taking on specific exceptional and methodological studies, we instead aim to provide the technique as a service to local, national, and international users to expedite and elevate their science through access to this powerful technique in a more routine fashion. Here, our work has covered a broad range of chemical systems, from molecular pharmaceuticals to organo-metallic framework materials and functional metal oxides, and applied to crystallites ranging from 10s of nm to the X-ray transitional micron limit. I will introduce 3D ED and the UK's National Electron Diffraction Facility, and highlight with select examples how the technique may be of meaningful and complementary use to a community that is already making exceptional use of existing tools.