MS23-P10

Electron crystallography greatly expands organic and inorganic X-ray crystal structure determination

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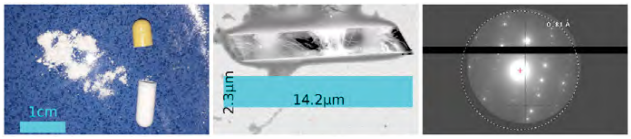
X-ray crystallography is the main discipline in chemistry that provides the 3D coordinates of molecules. The crystal structure is an essential aspect in many fields in chemistry. Electrons offer a fascinating extension to X-rays. Crystals smaller than the grains in icing sugar can be investigated too small for X-rays. 3D single crystal structures can be determinaed from powder, including caseswhere crystal size affects the chemistry, like for catalysts and MOFs[1].

This application of electron crystallography (EC) is young and was made possible with the adaption of the rotation method to[2]. Many publications proof the reliability of the coordinates from EC. Despite all these advantages, EC only plays a niche role in structural chemistry: it accounts for much less than 1% of all published 3D single crystal. This is in stark contrast to the number of crystalline powders, that, like in the case of Novartis, exceeds the number of available single crystals by a factor of 3-4.

One of the main reasons for this discrepancy could be the difference in methods and software that are available to process and refine EC data. The combination of PEDT and Jana2006 can improve the model R1-factor down to X-ray levels, and produce the hydrogen atoms in a heavy atom structure with striking clarity[3]. However, this level of accuracy is not required for the average crystal structure. In this presentation I report on our success to copy X-ray crystallography onto EC. We mounted an EIGER X 1M hybrid pixel detector from Dectris Ltd. to an electron microscope, collected data like with an inhouse X-ray machine and used the methods many structural chemist are well familiar with. We solved the structure of a new organic methylene blue derivative with a unit cell volume of 9,000A^3. From 17 fragments of a long needle-like crystal we collected 60 degree wedges and processed all data sets within a few hours. The structure solved with direct methods with default options. The data are accurate enough to complete the model and even model a disordered BF4- solvent molecule. We believe it is time to strongly advertise EC in all its flavours to the Structural Chemistry community.



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Keywords: (in)organic structural chemistry, electron crystallography

MS23-P11

Pushing the limits of material characterization using transmission electron microscopy at the university of oviedo

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Electron microscopy is at the forefront of many characterization methodologies used nowadays to obtain valuable structural information for different types of materials down to the atomic scale. In particular, our facility JEOL JEM-2100F provides a platform for combining different characterization techniques, such as high-resolution transmission electron microscopy (HR-TEM); electron diffraction techniques (selected area electron diffraction: SAED, nano-beam electron diffraction: NBD and 3D precession electron diffraction tomography); scanning transmission electron microscopy (STEM) in both bright-field (BF) and high-angle annular dark-field (HAADF) modes; energy dispersive X-ray spectroscopy (EDX) and electron energy loss spectroscopy (EELS). In this communication, we shade the light on outstanding examples that show the beauty of using our facility to effectively characterize inorganic, hybrid inorganic-organic, and biological samples.

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Keywords: HR-TEM, Electron diffraction, EDX