

MS37-02 Electron Crystallography on porous materials
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The structure determination of porous materials is always a challenging problem, especially when big high quality crystals are not available. Electron crystallography is a unique technique to tackle this problem since it can provide structural information from tiny crystals.¹ High resolution transmission electron microscope (HRTEM) images can be extremely valuable for the determination of pore evolutions. For example, for the mesoporous materials IBN-9, the images from three zone axes showed the clear contrast which can further be reconstructed into 3D space. And the structure of IBN-9 turns out to be the first tricontinuous mesoporous materials.² In many cases, the atomic resolution can be reached for the HRTEM images of porous materials, like high silica zeolites, and they can also be used to produce the 3D potential map. Using the chemical information from other related materials, atomic positions can be obtained from the potential map.^{3,4} In many studies, porous materials are too beam sensitive to be used for HRTEM imaging. Electron diffraction needs much less electron dose and can be used in this case. In the studies of the ITQ-37 structure, the electron diffraction intensities were used to pre-partition the overlapped reflections in powder X-ray diffraction, which significantly increases the convergence of the charge-flipping iterations and the chance of obtaining informative electron density maps⁵. The recently developed 3D electron diffraction even widened the application of electron crystallography on porous materials, cause it further reduced the beam damage and increased the data completeness. Using this new technique, a number of known/unknown porous materials have been determined to the atomic resolution and the least square refinement based on this kind of datasets showed comparable results with single crystal X-ray diffraction.

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MS37-03 Structure investigation of nanocrystalline small-molecule organics and MOF by ADT and simulated annealing. Enrico Mugnaioli,^a Ute Kolb,^a Giovanni Luca Cascarano,^b Corrado Cuocci,^b ^a*Johannes Gutenberg-Universität Mainz, Germany,* ^b*Istituto di Cristallografia CNR Bari, Italy*
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Information derived from structure characterization is fundamental for understanding and engineering new material properties. One of the most challenging demands for nowadays crystallography is the investigation of nanocrystalline phases, common among catalysts, high-tech materials, pigments and pharmaceuticals. In the last years automated diffraction tomography (ADT) [1] is emerging as a powerful and quick tool for a reliable and reproducible electron diffraction data collection from single nanocrystals. ADT data sets allow a 3D reconstruction of reciprocal space, unambiguous cell parameter determination and rich quasi-kinematical reflection intensity collection [2,3]. Direct methods proved able to deliver ab-initio structure solution by ADT data for a number of inorganic phases, eventually quite complex [4,5]. On the contrary, structure determination of organic or hybrid organic-inorganic phases is still a challenging demand. Typical problems arise from fast beam deterioration under the electron beam, mosaicity and limited long range periodicity of the sample [6]. Cooling the sample is of undoubted benefit for many compounds, particularly if water containing. Nevertheless, perturbations in the collected intensity data significantly hamper ab-initio structure solution routines, especially for weak scattering atoms like carbon, nitrogen and oxygen. Often only the overall position and the envelope of the organic molecule can be detected, without a proper atomic resolution. Even when a complete solution is achieved, this is affected by high structural residual and important distortions [7,8]. On the other hand, the presence of well-known rigid groups in organic molecules (eg. flat benzene rings and functional groups), make it possible to take this a-priori information into consideration and to restrict the number of variables to be determined. Simulated annealing already proved to be a valid method for structure determination of organic phases when only poor X-ray powder data are available [9]. Recently, simulated annealing has been implemented in the package SIR2011 [10], which includes atomic scattering for electrons and well tested routines for dealing with electron diffraction data. Exemplary known and unknown, organic and hybrid organic-inorganic phases were selected for ADT data collection. Such data sets were used for structure determination of metal-organic frameworks (MOF) [11] and organic compounds [6], either by direct methods and simulated annealing, and a critical comparison is here proposed.

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