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X-ray diffraction is an important analytical method to visualise the atomic positions of many chemical compounds. It is applied in many disciplines in Chemistry, Materials Science, Pharmacology, and many others. Recently, electron diffraction has attracted great attention [1], known as 3D ED[2], or microED[3] . 3D ED provides the same type of information as an X-ray structure, including the atom coordinates, lengths of bonded and non-bonded contacts, differentiation of elements, and determination of the absolute configuration of chiral compounds. Where X-ray diffraction requires crystals with at least a few µm in length, 3D ED determines the crystal structure from submicrometer sized crystals, even smaller than individual grains in powder. This is useful in cases where large crystals cannot be grown.
Instrumentation for 3D ED is based on transmission electron microscopes. Academic studies had to provide work-arounds, because the control software lacks features essential for crystallographic data acquisition. There are now two commercial systems (ELDICO and RIGAKU), that change the situation, and provide a user interface optimised for crystallography.
This talk will focus on our realisation for an instrument, in particular the use of the JUNGFRAU detector [4]. Originally designed for X-ray Free Electron studies, this detector reveals an enhanced sensitivity, and provides us with great level of details. Our examples includes organic compounds, metal organic framework, and zeolites.

The zeolite A, and also the aluminosilicate albite acted as benchmark for the JUNGFRAU detector. Our data differentiated aluminium from silicon in two aluminosilicates, zeolite A and albite, a result which is difficult to achieve even with X-ray data.
Our latest study benefited from the superior quality of the JUNGFRAU detector. We localised coke in the pores of ZSM5, and we monitored the process of coking in the micropores.

Finally, I will also share my experience from sample preparation. Because samples are not visible with light-microscopes, one has to prepare them blindly, until the TEM reveals the quality of the grid. This might be a bottleneck, but is mainly a matter of experience.

(1) Gruene, T.; Wennmacher, J. T. C.; Zaubitzer, C.; Holstein, J. J.; Heidler, J.; Fecteau-Lefebvre, A.; De Carlo, S.; Müller, E.; Goldie, K. N.; Regeni, I.; Li, T.; Santiso-Quinones, G.; Steinfeld, G.; Handschin, S.; van Genderen, E.; van Bokhoven, J. A.; Clever, G. H.; Pantelic, R. Rapid Structure Determination of Microcrystalline Molecular Compounds Using Electron Diffraction. *Angew. Chem., Int. Ed.* **2018**, *57*, 16313–16317. https://doi.org/10.1002/anie.201811318.

(2) Gemmi, M.; Mugnaioli, E.; Gorelik, T. E.; Kolb, U.; Palatinus, L.; Boullay, P.; Hovmöller, S.; Abrahams, J. P. 3D Electron Diffraction: The Nanocrystallography Revolution. *ACS Cent. Sci.* **2019**, *5* (8), 1315–1329. https://doi.org/10.1021/acscentsci.9b00394.

(3) Jones, C. G.; Martynowycz, M. W.; Hattne, J.; Fulton, T. J.; Stoltz, B. M.; Rodriguez, J. A.; Nelson, H. M.; Gonen, T. The CryoEM Method MicroED as a Powerful Tool for Small Molecule Structure Determination. *ACS Cent. Sci.* **2018**, *4* (11), 1587–1592. https://doi.org/10.1021/acscentsci.8b00760.

(4) Fröjdh, E.; Wennmacher, J. T. C.; Rzepka, P.; Mozzanica, A.; Redford, S.; Schmitt, B.; van Bokhoven, J. A.; Gruene, T. Discrimination of Aluminum from Silicon by Electron Crystallography with the JUNGFRAU Detector. *Crystals* **2020**, *10*, 1148. https://doi.org/10.3390/cryst10121148.

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