Combining Electron and X-ray Crystallography for Structure Characterization

**A perfect liaison: combining crystal mapping with PXRD**

J. Merkelbach¹, C. Jandl¹, G. Steinfeld¹, D. Stam¹, P. Simoncic¹

¹ELDICO Scientific AG, Park Innovaare, 5234 Villigen, Switzerland

merkelbach@eldico.ch

**Keywords:** crystal mapping, electron diffraction/3DED/microED, PXRD, polymorphism, liquid assisted grinding

Electron diffraction as a tool for single crystal structure analysis of nanocrystals is well known in the crystallographic community.¹ In recent months we assisted in a range of projects discovering the opportunities of a dedicated electron diffractometer with crystal mapping² capabilities outside of that restricted purpose of structure elucidation. And most projects start with X-ray powder diffraction (PXRD) profiles not fully understood.

PXRD is the preferred technique of chemists to screen different batches for phase purity and crystallisation quality. It is good practice to fully assign every signal in the profile. But this can be a challenge with mixtures of three or more components or phases and/or broadened peaks due to nanosized crystallinity. By combining the imaging feature (STEM mode) with the diffraction mode of an electron diffractometer, crystal mapping of dozens to hundreds of nanosized crystals can provide the missing information needed to fully understand the PXRD profiles. This has shown to be especially useful in areas where the powder is the final product and further purification or crystallisation is not intended, like for liquid assisted grinding (LAG) experiments.³ For a quantitative analysis of the crystal mapping results one has to come back to the PXRD pattern and therefore close the circle of this perfect liaison.

![Figure 1. Asymmetric unit of tyramine hydrochloride. Crystals were found as a side product of a sample from a liquid assisted grinding experiment to synthesise tyramine cocrystals. The structure was solved from one ED dataset routinely recorded during a crystal mapping experiment.][1]

