

## A Perfect Liaison: Combining Microed With PXRD

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Electron diffraction as a tool for single crystal structure analysis of nanocrystals is well known in the crystallographic community.[1] In recent time we assisted in a range of projects discovering the opportunities of a dedicated electron diffractometer with crystal mapping[2] 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 features (STEM mode) with the diffraction mode of an electron diffractometer, crystal mapping of dozens to hundreds of nanosized single 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] Additional benefits of a single crystal analysis via microED are the possibility to find and refine hydrogen positions and to determine the absolute structure of enantiopure samples. For a quantitative analysis of the crystal mapping results and refinement of unit cell parameters one must come back to the PXRD results and therefore close the circle of this perfect liaison.

{1} Gemmi, M., Mugnaioli, E., Gorelik, T. E., Kolb, U., Palatinus, L., Boullay, P., Hovmöller, S. & Abrahams, J. P. (2019). *ACS Cent. Sci.*, 5, 1315.

{2} Rauch, E. F., & Véron, M. J. M. C. (2014). *Mater. Charact.*, 98, 1.

Days, L. C., Mahler, J., Pagola, S., Holder, A. A. & Merkelbach, J., in preparation.

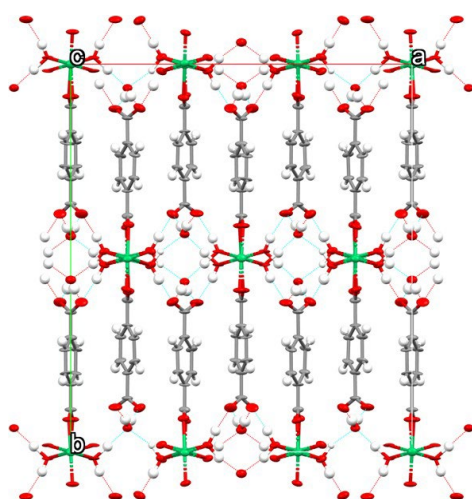


Figure 1

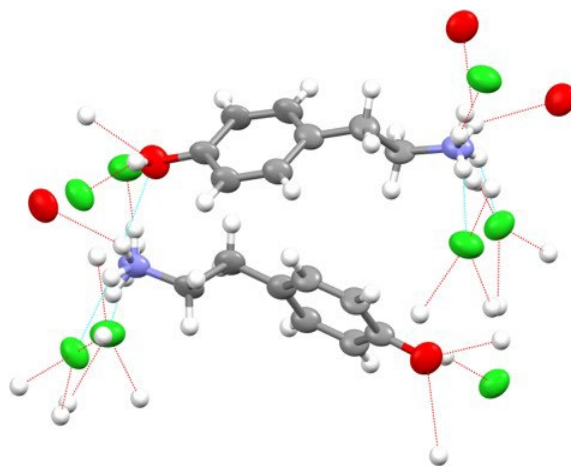


Figure 2