Combining Techniques for Increased Accuracy of Crystal Structures from Powder Diffraction Data

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Powder X-Ray Diffraction (PXRD) is one of the most powerful and widespread tools for structural analysis of micro- and nanocrystalline materials in the bulk. As such, it shows continuous growth in number of crystal structures deposited in structural databases. With this in mind, it is crucial to ensure accuracy of submitted structures.
However, the path to crystal structure determination is paved with sources of errors. While some of them include the usual suspects (e.g. data resolution), the others are sometimes overlooked. First, the process requires a significant users' input throughout the process.
Moreover, a lack of exact evaluation criteria makes it difficult to monitor the progress of structure determination and Rietveld refinement, and to check the quality of the final structure. Finally, the Rietveld method is not capable of "repairing" a wrong structure. A group of materials that is particularly prone to such errors are small molecules, due to their limited scattering power, relatively high structural flexibility and a low degree of crystallinity [1,2]. Nonetheless, the power of PXRD for structural analysis of small-molecules is not questionable. But, how to recognise its limits? How to overcome them? How to utilise the power of PXRD the best?
This presentation focuses on increasing accuracy of small-molecule structures by highlighting potential sources of errors and proposing ways to reduce them. Using a series of molecular structures, the audience will take a journey through sources of errors and see their effect on crystal structure accuracy. Depending on the source of error and the quality of the PXRD data, each case will be complemented with an appropriate method to improve accuracy (reduce errors). This includes use of difference Fourier maps, complementary analytical techniques, use crystal structure databases, as well as an approach to search the parameter space to avoid local minima in testing different sets of geometry restraints [1].
The set of selected examples is suitable for both beginners and advanced PXRD practitioners.

Reference
{1} Sisak Jung, D., Lukin, S., Halasz, I. (2023). Helv. Chim. Acta 106, e202200087.
{2} Altomare, A. (2022). IUCrJ 9, 403–