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# Reducing errors and increasing accuracy of small-molecule crystal structures

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Powder X-Ray Diffraction (PXRD) is one of the most powerful tools for structural analysis of micro- and nanocrystalline materials in the bulk. As such, its use and the number of published crystal structures is expected to continuously grow. Populating databases with accurate crystal structures is crucial for two reasons: chemical sensibility of structures and the fact that the PXRD technique heavily relies on chemical (and crystallographic) information stored in databases.

However, the path to an accurate 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. To start with, the method requires significant users' input. Furthermore, 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 (organic) 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 recognize its limits? How to overcome them? How to utilize 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 (with different quality of PXRD data), 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 associated data, each case will be complemented with appropriate method to improve accuracy (reduce errors). This includes use of difference Fourier maps, complementary analytical techniques, 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. The audience is expected to leave with two ideas: importance of obtaining the most accurate structures possible, and some practical advice on how ensure this accuracy.

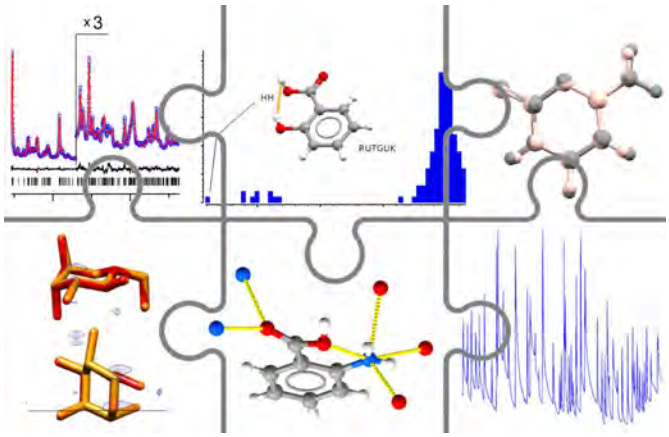


**Figure 1**. Increasing accuracy of small-molecule structures obtained by PXRD data can be sometimes be achieved by using PXRD data only. However, the use of information and data obtained by different sources are usually needed.

[1] Sisak Jung, D., Lukin, S., Halasz, I. (2023). *Helv. Chim. Acta* **106**, e202200087.
[2] Altomare, A. (2022). *IUCrJ* **9**, 403–405.