

# Powder X-ray Diffraction: direct space, reciprocal space, and between the two

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Considering the problems inherent to crystals of small organic molecules, it is rather surprising that their structures are almost routinely determined from Powder X-ray diffraction (PXRD) data. At this point, there seem only a couple questions to answer:

- Why are some structures difficult?
- How to deal with structures that cannot be (routinely) solved?
- What accuracy of organic structures can be obtained?

This work unpacks examples where routine processes are not sufficient for structure determination, and provides some tips on tinkering with data collection strategies and structure determination methods.

Data collection strategies will be discussed as a tool for obtaining the highest possible resolution, as well as in context of potentially overlooked challenges, such as radiation damage and data statistics [1, 2].

Structure determination will cover routine processes and problems encountered in direct and dual-space methods. Using the direct-space platform FOX [3] and charge-flipping program Superflip [4,5], this work presents how the calculations can be optimized to facilitate structure determination of difficult molecules.

As last, this work proposes a methodology to facilitate the phasing process in Superflip by introducing a partial or incorrect structure obtained in FOX [5]. The method relies on freeware and does not require reprogramming or modifications of existing algorithms.

Problems presented in this work include organic structures whose molecular structure was completely unknown, or exhibited ambiguities such as disorder, isomerism or cocrystal vs. salt. The presented examples will also be used to highlight the level of accuracy that is obtainable for crystal structures derived from PXRD data. This includes restraint-free Rietveld refinement and modeling disorder based on examination of difference Fourier maps.

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[2] Šišak Jung, D. *et al.* (2014). *J. Appl. Cryst.* 47, 1569-74

[3] Favre-Nicolin, V., Cerný, R. (2004). *Z. Kristallogr.* 219(12), 847-856.

[4] Oszlanyi, G., Sütő, A. (2004). *Acta Cryst.* A60, 4-141

[5] Palatinus, L., Chapuis, G. (2007). *J. Appl. Cryst.* 40, 786-790