STRUCTURAL SCIENCE
CRYSTAL ENGINEERING
MATERIALS

Volume 78 (2022)

Supporting information for article:

FOX-7 high-energy-density material: thermal expansion and phase transitions revisited

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Process for producing Gandolfi powder patterns:

First a single crystal dataset is collected. In this case we used a standard single crystal diffraction strategy of 360-degree rotations around one axis, ω, at two settings of κ, and three settings of ϕ with exposures of 0.1 s per 0.1 degree. This gave 14400 frames per dataset to sum together.

Run	Omega	Phi	Kappa (α = 24°)	Chi equivalent
1	0-360	0	0	0
2	0-360	0	54	21.3
3	0-360	90	54	21.3
4	0-360	180	54	21.3

This number of frames is not required nor the small step size but with modern area detectors there is little cost in producing this number of frames initially. Satisfactory results can be obtained with a single ω rotation; however, this could lead to lower than ideal completeness for the single crystal analysis.

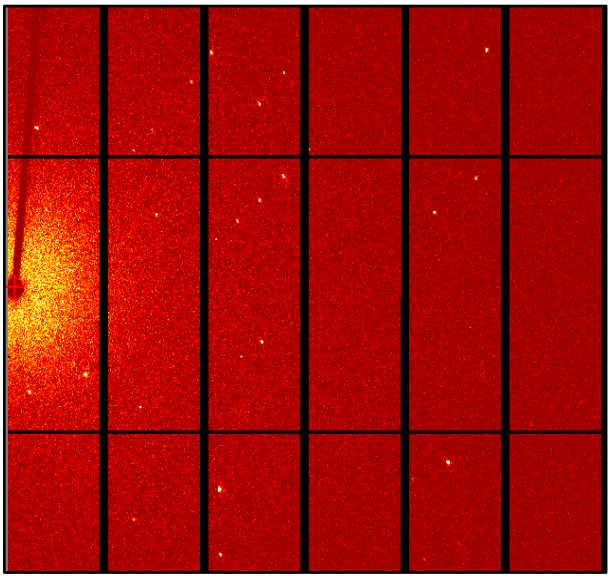

Figure 1 – Extract of a typical image from a single crystal data set

All the images were then summed together using the Converter tool within SNBL Toolbox to output a Gandolfi diffractogram, .cbf file. (Dyadkin *et al.*, 2016) https://soft.snbl.eu/toolbox.html

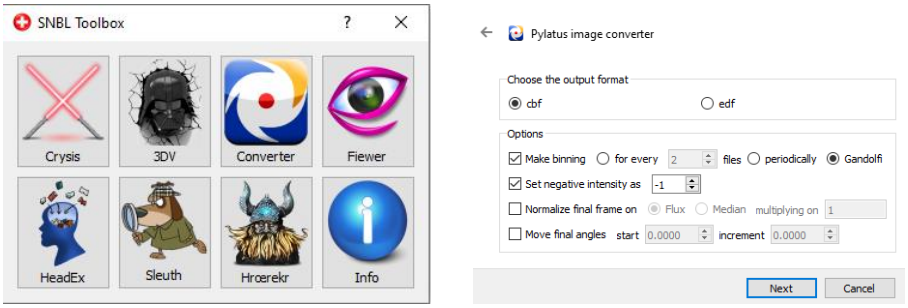

Figure 2 – Screen shots of the SNBL Toolbox and Converter tool.

The Gandolfi image is then azimuthally integrated to generate a powder pattern using Bubble. (Dyadkin *et al.*, 2016) https://soft.snbl.eu/bubble.html Rietveld refinement is possible for these data however the intensity are not accurate as not all orientations of the crystal were sampled equally. These data are more than good enough for unitcell dimensions.

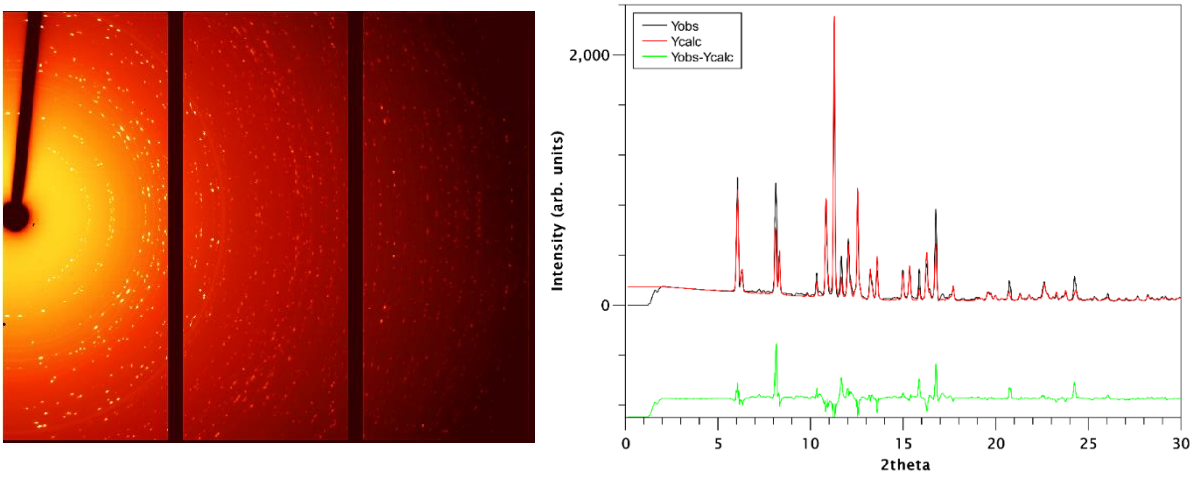

Figure 3 – Extract of a typical Gandolfi diffractogram and the Rietveld refinement of α-FOX-7 at 274 K. Observed intensity, black, calculated intensity, red, and the difference, green.

Multiple datasets

In the case multiple data sets such as variable temperature, it is helpful to view all these data sets together in one place. MEDVED is a useful tool for this. (Chernyshov *et al.*, 2016) https://soft.snbl.eu/medved.html

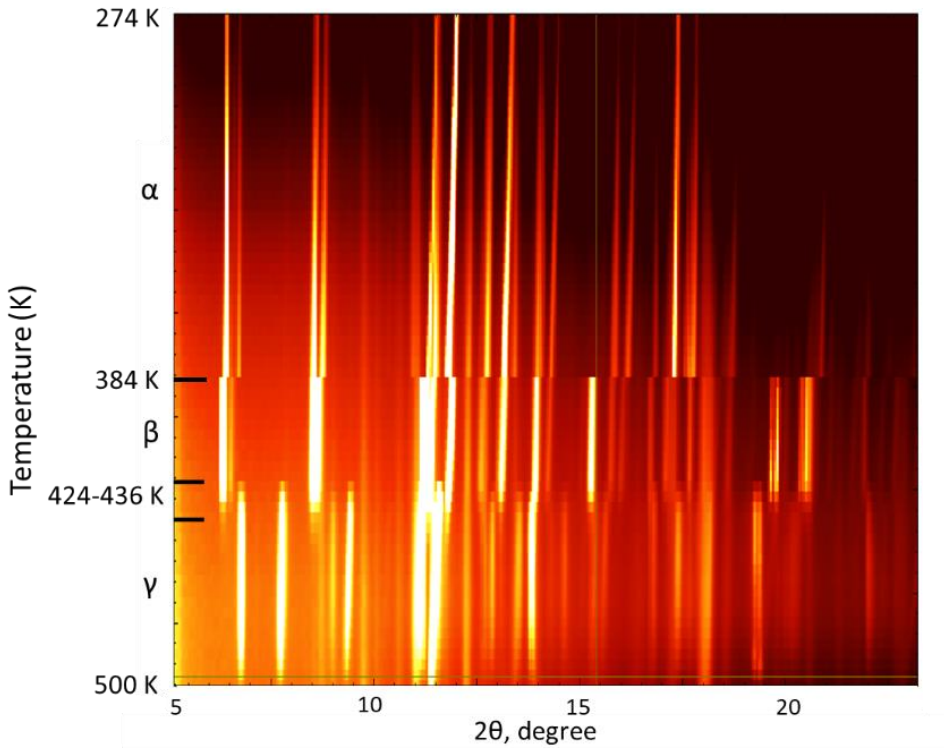

Figure 4 – FOX-7 with variable temperature as displayed by MEDVED clearly showing the α-β and β-γ phase transitions.

Sequential refinement of the unit cell parameters can be carried with powder diffraction software to examine the changes in unit cell with temperature. The raw images can be used for single crystal analysis to inform the powder refinements, particularly useful for indexing after a phase change. Likewise, the Gandolfi powder patterns allow for quick inspection of all the data collected that can inform the most efficient use of more labour-intensive single crystal reduction and refinement.

References

Chernyshov, D., Dyadkin, V., van Beek, W. & Urakawa, A. (2016). *Acta Crystallogr. Sect. A* **72**, 500-506.
Dyadkin, V., Pattison, P., Dmitriev, V. & Chernyshov, D. (2016). *Journal of synchrotron radiation* **23**, 825-829.