

MS42-P05 | SEQUENTIAL SHELXL REFINEMENT OF CONSECUTIVE DATASETS: A TOOL TO PROBE DYNAMICALLY EVOLVING SINGLE CRYSTAL STRUCTURES

Chernyshov, Dmitry (SNBL at the ESRF, Grenoble, FRA); Dyadkin, Vadim (Swiss-Norwegian Beam Lines, Grenoble, FRA); Törnroos, Karl Wilhelm (Department of Chemistry, University of Bergen, Bergen, NOR)

Modern area detectors combined with bright synchrotron radiation allow for collection of powder or even single crystal data with high time resolution, while external conditions (e.g. P or T) are changing at a certain rate. Such experiments are quite common for powder diffraction where crystal structure analysis for many datasets is conveniently done with sequential or parametric refinement [FullProf & TOPAS]. As far as we are aware, a similar generic option for single crystals experiments is still absent.

If the temperature change or any other external stimuli step is small, and there is no rapid phase transition, one may consequently expect that the inherited parameters, e.g. atomic positions and ADPs, would serve as a good starting model for the succeeding data point. Starting from this idea, we have developed a tool for a sequential SHELXL refinement of the evolution of a crystal structure documented in a set of closely time spaced temperature steps as well as the automated extraction of any parameter from the resulting output.

The software was tested against a series of data collected upon slow cooling for a spin crossover compound. The analysis uncovered that in the temperature range 300-80 K the compound possesses a crossover from high spin (HS) to a mixed HS-LS state. The later state is intrinsically disordered and the gain in entropy seems to be enough to overcome the thermal contraction upon cooling: we observe an increase of the unit cell volume at low temperatures.