Single-crystal quality data from polycrystalline samples: finding the needle in the haystack

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In recent years [1-4], our laboratory has been investigating non-covalent interactions in co-crystals comprising liquid co-formers such as C₆F₆. This type of sample needs to be handled in X-ray capillaries due to the volatile nature of the components and cooled to the solid phase. While it is possible in theory to grow a single-crystal at low temperature in the capillary, it is experimentally demanding to get just one crystal. This led us to the hypothesis: can recent advances in laboratory single-crystal hardware and software be used to determine crystal structures from a polycrystalline sample in much the same way that crystal structures are determined from high-pressure data using a multigrain approach rather than a traditional single-crystal approach? The concept of a multigrain approach is not in itself novel, at least with regard to X-ray data collected at the synchrotron on polycrystalline samples. While the concept of processing and solving structures from data exhibiting twinning due to the presence of two or more crystals goes back decades, the development of “multigrain crystallography” at synchrotron sources, where there is the potential to study multiple crystals under pressure, has been more recent [e.g. 5-7].

In this presentation, we will discuss a number of systems studied using the multigrain approach in which we start with a liquid sample in an X-ray capillary, freeze it to the solid state, and determined the structure from what initially looks like very poor data (see Fig. 1).

![Figure 1](image_url)

**Figure 1.** From polycrystalline sample in capillary to multigrain data to a reliable crystal structure.

The reliability of the structures is evident from the fact the geometry of the molecules is as expected. We have used this approach for the last few years and have noted what works and what does not work with modern laboratory hardware and software. One of the crucial steps is identifying the correct unit cell, which is a bit like looking for the “proverbial needle in a haystack” but not knowing the shape of the needle. The second issue is knowing how good the data has to be for the structures to be solved when solid-solid phase transitions occur, something that happens often in the systems we are studying. This presentation will address some of these issues. The methodology of our approach is currently the subject of a paper in preparation (and two further papers on non-covalent interactions in which our approach has been exploited) and may be of interest to those using high pressure.