Making the Most out of Non-Ideal Samples: When and How to Use Problem Datasets

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Single crystal diffraction methods have always been limited by the need to grow suitable crystals, which can only be obtained through recrystallizations which must be empirically optimized through a frequently lengthy, mass-intensive process. Recrystallizations may even be practically impossible for many systems at the cutting edge of chemical research. For example, highly complex supramolecular systems or coordination polymers may be irreversibly destroyed upon dissolution, and there may be no safe way to handle the amounts of certain highly bioactive or radioactive compounds in the amounts needed to do parallel crystallizations. For systems like these, the best possible effort in growing a crystal may still give a problem dataset – data which, due to problems such as polycrystallinity or small crystal size, results in a model with unresolvable artifacts.

Modern in-house single crystal diffractometers and software suites such as SHELX are often credited for facilitating an exponential increase in the throughput of crystal structure determinations by automating many formerly demanding tasks in structure determination. What may be less well known is how versatile these tools are for treating data sets that fail to refine properly. When no further effort can conceivably lead to a better crystal, that effort may be better spent on careful experimental design and model building using these tools. This talk will explore topics including rationales for using problem datasets, how to diagnose artifacts that may be resolvable, and how to extract precise and meaningful chemical information from a problem dataset while remaining transparent about the limits of its interpretation.