Integrated solutions for most efficient in house high-pressure single crystal experiments

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Within the last decade high pressure studies have received significant increase of interest. Present typical applications range from the investigation of solid-state organics as part of the pharmaceutical drug development to the study of rocks and minerals with applied pressures of up to 50 GPa. While in the past it has been almost impossible to accommodate and extend standard in-house solutions, i.e. hardware and software, to the needs of high-pressure crystallography, today this picture has changed completely. Modern high-performance software, such as APEX3, has been developed to address the intrinsic challenges of high pressure crystallography, particularly with respect to sample alignment, indexing, data integration and scaling. This complements the significantly higher flexibility available in the hardware setup. Current instrumentation can easily be used for routine low temperature data collections, fully optimized with respect to minimizing air scattering and achieving full completeness and desired data multiplicity. Within just a few minutes the same instrument can be converted into an optimized solution for high pressure data collection, e.g. for the investigation of organic solid state material in a diamond anvil cell (DAC). Electronic component recognition ensures automated reconfiguration and validation of the instrument. This approach avoids invalid and useless configurations on the one hand, while on the other hand taking the special requirements of a high-pressure experiment into account. For example, even with the limitations implied by the DAC best possible, completeness and data redundancy is automatically ensured.

Using a D8 VENTURE with a fixed-chi stage we will demonstrate the advanced hardware capabilities and processing methods based on the monoclinic polymorph of the sulfonium ylid.

The high flexibility of the D8 VENTURE design will be further demonstrated in the second part of the presentation: For the investigation of miniscule mineral crystals enclosed in diamond anvil cells, functionality typically only available at synchrotron facilities has been added to the in-house system. These features include highly accurate, motorized sample positioning and the ability to monitor intensity of the X-ray beam passing through diamond anvil cell. The latter is used both for DAC alignment, as well as for correcting measured intensity data. The availability of online pressure measurements based on ruby fluorescence completes the extension of the D8 VENTURE, making the system “a little synchrotron at home”.

Lab-based experiments up to 50 GPa with synchrotron-sized high-pressure single-crystal samples of even triclinic symmetry will be presented.