Increasing Completeness in Single-Crystal High-Pressure Diffraction Experiments by Pre-Orienting Crystals

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Diamond anvil cells (DACs) exert high pressure while allowing light to access the sample. They enable single crystals to be investigated under extreme conditions by means of X-ray diffraction (XRD). Structure factors collected this way provide unique structural information, but their quality is never on par with routine experiments.[1,2] A typical 35° opening angle of modern DACs renders up to 97% of the limiting sphere inaccessible. Resulting diffraction patterns are thus systematically incomplete, especially in samples with low internal symmetry, which impedes the crystal structure solution and affects the applicability of techniques that require high reciprocal space coverage.[3,4]

The completeness of high-pressure XRD experiments can be increased either by merging datasets originating from multiple crystals or by utilizing the internal symmetry of a single crystal. The first approach requires meticulous processing protocols or dedicated equipment to warrant sufficient precision.[5] Meanwhile, employing inherent crystal symmetry calls for precise control over its orientation, but otherwise can be performed in virtually any laboratory.[6]

During my presentation, I will show the impact of crystal orientation, DAC opening angle, resolution, and radiation wavelength on maximum attainable completeness in XRD experiments. I will evaluate common experimental setups and draw general conclusions. I will present the immense effect of sample orientation on the reciprocal space coverage, as even cubic samples can offer fewer data than orthorhombic ones when misoriented. I will demonstrate nearly-complete XRD datasets for monoclinic and orthorhombic 1,3-diacetylpyrenes collected under high pressure and refined using the TAAM approach. I will spotlight my dedicated Python tools, hikari-toolkit and dtools.pl, which allow others to evaluate their specific systems.

References:
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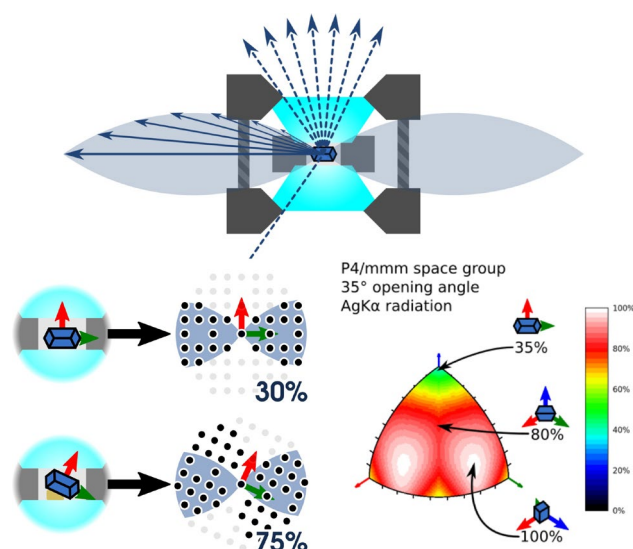*{6}D. Tchoń, A. Makal, IUCrJ, 8 (2021) 1006–1017.*

Figure 1