A47

Ultra-high resolution protein crystallography: concanavalin A to 0.98 Ångstrom and beyond

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Many years ago the idea of collecting voluminous quantities of weak reflection intensities from a protein crystal, at high resolution, was a particular challenge [1]. The combination of insertion devices with very high X-ray fluxes at short X-ray wavelengths, sensitive CCD detectors and freezing of crystals have provided the means to certainly match those best hopes. So much so that the data can best be described as ultra-high resolution, at least as evidenced in our studies on the plant protein concanavalin A of 25000 molecular weight. [The intrinsic property of this protein is to bind sugar molecules; it is implicated in cell to cell recognition processes and is widely used as a laboratory diagnostic tool.] At CHESS we have used a 0.9 Ångstrom wavelength beam on station A1, fed by a 24 pole multipole wiggler. Image plate scanning (Fuji) and a CCD detector (Princeton, [2]) have been used on this experimental set up to collect diffraction data sets from frozen concanavalin A crystals (saccharide free crystal form). The rapid read out of the CCD was most convenient compared with the image plate and associated scanning. Moreover the data processing results towards the edges of the diffraction, 0.98 Ångstrom, for the two detector media, show that the CCD is much better at recording these weaker data than the image plate (Rmerge(I) 13% versus 44% respectively). The poor performance of the image plate with weak signals has of course been documented by the Daresbury detector group [3]. However, the aperture of the CCD used was limiting, and the need for a bigger CCD detector confirmed. Very recently, in another run at CHESS with the CCD on A1, we have been able to record diffraction data to 0.94 Ångstrom by further offsetting the detector off-center, but again find that the reflections are still strong at the edge. Clearly the use of even shorter wavelengths than 0.9 Ångstrom would be very useful in matching the solid angle of the diffraction pattern to the available detector aperture, for a reasonable crystal to detector distance. In addition absorption errors in the data can be simultaneously removed by such a strategy. Indeed, finely focussed X-ray beams of, say 0.5 Ångstrom wavelength, are well suited to high energy SR machines. Hence, the diffraction resolution limits seen already in our concanavalin A studies can be further enhanced, and are important for much more detailed molecular model refinement (and testing structure solving strategies), as well as opening up novel spectroscopic and theoretical studies, in conjunction with the structural results.

1. J. R. Helliwell (1979) DL/SCI/R13,1-6.

2. S. Gruner et al.

3. R. Lewis (1984) J Synchrotron Rad 1, 43-53 (figure 8).