ESRF

Experiment title: MAD experiments on the AOPS, a protein protein invloved in biotin synthesis

Experiment number: LS-964

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Report:

Attempts were made to freeze crystals of the AOPS, in order to perform the experiments at 100K. Unfortunately we were unable to reproduce the cryo-conditions that had previously given good results.

Previous experiments had shown that the crystals were prone to radiation damage, and did not survive more than a few minutes in the intense X-ray beam. It was therefore decided to abandon the experiment on this protein.

The backup project was that of the cytochrome-c552 from Pseudomonas nautica. A MAD experiment was performed exploiting the K-edge anomalous dispersion effects of the iron atom present in the heme group. The entire experiment was performed using one single crystal frozen at 100K.

The precise energies were obtained from the X-ray fluorescence and transmission measurements from the crystal sample, see figure 1. The rod-shaped morphology allowed different regions of the crystal to be exposed to the X-rays by successive translations of the spindle axis. In this manner, a fresh region of the crystal was available after the fluorescence measurements and each data collection.

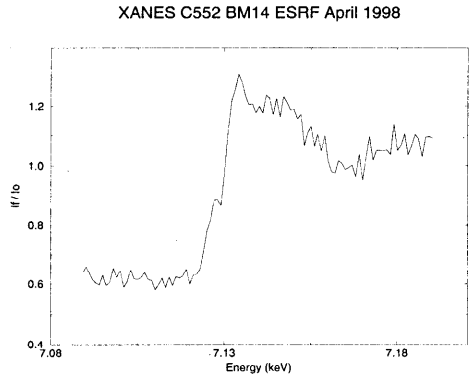

Figure 1

Data were first collected at the maximum off' (7132 eV, λ=1.734Å). The second data collection was performed at the minimum of f″ (7123 eV, λ=1.740Å), and lastly at the remote energy (11278 eV, λ=1.090Å).

Before calculating Patterson maps, the cumulative intensity distribution was calculated, from which it was concluded that the crystals were hemihedrally twinned. This is relatively common in trigonal space groups, and arises from the 'mixing' of two sub-lattices within the same crystal domain. A consequence of this twinning was that the structure solution could not be persude, and unfortunately the data collected during this experiment could not be used.