Report of experiment MX497 on beamline ID23.2

The three shifts assigned at ID23.2 were the first in a microfocusing beamline. The beam is really intense and powerful. The beamline is very well setup and maintained, and there is also enough space for mounting the crystals in the non-automated way. We used magnetic pins but not the automated sample changer since we are waiting for the gun to be delivered.

On the other hand we experienced a few problems in centring the crystals, and operation which was fine whenever the three dimensions were similar, but proved to be very hard when there was a lot of difference. In fact most of the time of each data collection was used to centre the crystal.

The separate large screen with the diagnostic of the beam was extremely useful, giving us another way to check that everything was going alright and letting us know when to stop and to re-start a data collection for a sudden instability or drop. In all but one data collection we had to attenuate the beam, since crystals were decaying very fast.

During the 24h we had to re-focus the beam only twice after the beamline scientist set up the beamline for use, a thing that we particularly appreciated.

A total of 12 data sets on five projects have been collected and 20 crystals tested for diffraction. All the data sets were indexed and scaled during the shifts, using our personal licence of HKL.

One project proved particularly difficult given the large unit cell (a=75.48Å, b=177.63Å, c=129.43Å, beta=96.24°), the low symmetry (P21) and the high sensitivity to radiation damage (2.3Å dropped after a few frames into 2.7Å, with 60% attenuation of the beam). On the other hand the needle-like shape of the crystals helped us collecting in slices. Final merging statistics were good to try a molecular replacement solution with a model of a different organism, and the structure is under refinement.

Further three data collections have been performed on three different crystal forms of an already solved protein form Schistosoma mansoni. The structures are under refinement and we hope to have all the intermediates of the fully oxidised to fully reduced protein. The space groups varied from $P2_12_12_1$ (a=33.93Å, b=51.99Å, c=59.31Å) to $P3_2$ (a=b=62.14Å, c=58.29Å) to P6 (a=b=90.63Å, c=45.5Å).

Another project, for which we tested a lot of crystals before finding two suitable ones, happened to have a large cell and in both cases the crystal orientation was so unlucky that even after 360° data were complete only up to 90% at 2.7Å. The cell parameters were as follows: $C222_1$, a=118.75Å, b=141.90Å, c= 89.0Å. This was the first time we

were able to collect data on such tiny crystals. In the next assigned shifts we shall try to perform a MAD experiment to the Se-Met derivatives of the protein, which grew slightly bigger.

The last two projects were solvable by MR, but we were seeking in both cases for inhibitors bound to the active site. Unfortunately, the first omit maps produced on the beamline were unsuccessful, but we improved at least the resolution up to 1.6Å, despite the tiny size.