



<b>Experiment title:</b> Towards Sub-Picosecond temporal resolution in x-ray diffraction experiments.	<b>Experiment number:</b> LS-506	
<b>Beamline:</b> ID09	<b>Date of experiment:</b> from: 24 Oct 96                      to: 28 Oct 96	<b>Date of report:</b> 24 Feb 97
<b>Shifts:</b> 12	<b>Local contact(s):</b> Michael Wulff	<i>Received at ESRF:</i> <b>03 MAR 1997</b>

**Names and affiliations of applicants** (\* indicates experimentalists):

Richard Neutze\*, Janos Hajdu\*: Department of Biochemistry, Uppsala University, Uppsala, Sweden

Inger Andersson: Department of Molecular Biology, Swedish University of Agricultural Sciences, Uppsala, Sweden

Ian J. Clifton\*, Rupert Wilmouth\*: Oxford Center of Molecular Sciences, Oxford OX1 3QY, U.K.

Michael Wulff\*: ESRF, Grenoble, France

Meinhard Kocsis\*: ESRF, Grenoble, France

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**Report:**

**Summary:**

Topographic images of elastase were recorded at ID09 when the torridoral mirror was removed, thereby allowing a broad and low divergence beam to enter the experimental hutch. Unfortunately the on-line CCD detector had insufficient spatial resolution for topography applications and we had to use high resolution X-ray film instead.

This experiment represented the first steps towards developing a new technique, crossed beam topography, through which it may be possible to achieve sub-picosecond temporal resolution in X-ray exposures of picosecond or longer duration [1]. The experiment was successful in that it demonstrated that topogrammes of protein crystals can be recorded on ID09 although no full data sets could be collected due to the insufficient spatial resolution of the available image intensified CCD detector.

## Details:

The technique itself requires that the sample is immersed in a broad beam and a topogramme of the entire crystal recorded for every diffraction spot. For crystal topography three requirements must be achieved:

i) A low divergence, homogeneous beam must enter the experimental hutch. By removing the torridoral mirror and using the wiggler as an insertion device, such a beam was available at ID09. Intensity profiles of this beam were recorded through two dimensional lattice scans using a pinhole mounted on the goniometer. When a short exposure of the direct beam was recorded on very slow x-ray film it was noted that the beam was “cracked”, being partially blocked upstream, providing an unwanted feature in its intensity profile (figure 1).

ii) A high spatial resolution detector is necessary to record a topogramme of the crystal for each diffraction spot. Unfortunately the pixel size and the point spread function of the online CCD detector turned out to be unsuitable for resolving the shape of a  $0.2 \times 0.2 \times 0.6 \text{ mm}^3$  crystal. Eventually it was decided to use high resolution X-ray film with a spatial resolution of 5 microns. Using this film, topogrammes from single crystals of native elastase were recorded (figure 2) clearly showing a projection of the crystal (figure 3) convoluted with the intensity profile of the beam.

iii) Crystals must be of sufficiently low mosaicity so as not to smear out the topogramme itself. Our observation of good quality topogrammes for macromolecular crystals indicates that the applicability of this technique may well be quite broad. Of particular interest is the projection of the crack within the X-ray beam onto the topogrammes. This indicates that the combined effects of beam divergence and crystal mosaicity lead to smearing of less than -10 microns.

We conclude primarily from these experiments that, with improvements of the spatial resolution of the detector (several candidates are available) and in the quality of the beam, X-ray topography of protein crystals is viable at the high flux beamlines of the ESRF. This motivates continuation of the development of this technique.

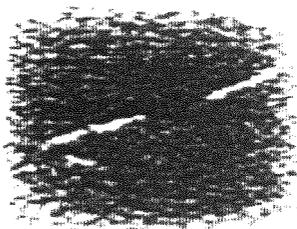


Figure 1: Beam profile

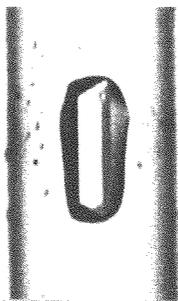


Figure 2: Crystal of elastase



Figure 3 : Topogramme

## References:

[1] On femtosecond time resolution in X-ray diffraction experiments, R. Neutze and J. Hajdu, submitted PNAS.