ESRF	**Experiment title:** Seryl-tRNA synthetase revisited. Phasing Bragg reflections of its uranium derivative by MAD at wavelengths near the M_V edge.	**Experiment number:** LS 1130
Beamline: ID1	**Date of Experiment:** from: 10 Dec. 1998 to: 15 Dec. 1998	**Date of Report:** 24. Febr. 1999
Shifts: 12	**Local contact(s):** Dominique Thiaudière (since 1.Nov.1998) Stéphane Lequien (till 31. Oct. 1998)	*Received at ESRF :* 01 MAR. 1999

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Report:

Anomalous dispersion of X-ray diffraction from single crystals of the uranium derivative of asparaginyl tRNA synthetase has been measured at 4 wavelengths near the M_V absorption edge at $\lambda = 3.5$ Å. The penetration depth of this radiation in organic matter is about 80 µm. This is the size of the protein crystals used. The protein crystal was cooled by a stream of cold nitrogen gas. This very fact excluded the possibility of an evacuated beam path from the source to the detector as it is available at ID1. A helium path constructed at IBS has been inserted to bridge the distance of 1.6 m between the beryllium window and the sample with the mirror in between. Remaining absorbers are: A kapton foil of 240 µm thickness (part of the system to control the beam intensity leaving the monochromator) reducing the intensity of the beam by a factor 20. It has been removed most of the time. An air gap of 50 mm near the sample (beam attenuation by a factor 3.0). The Be window of 125 µm thickness is fairly transparent to 3.5 Å photons. The sensitivity of the CCD camera to 3.5 Å photons is of the order of a few percent, at most. Hence, the overall sensitivity of the set up to 3.5 Å photons varies between 10^{-2} to 10^{-3}.

The beam time was used for:

- test experiments with tetragonal lysozyme, native and uranium derivative
  - using a CCD camera

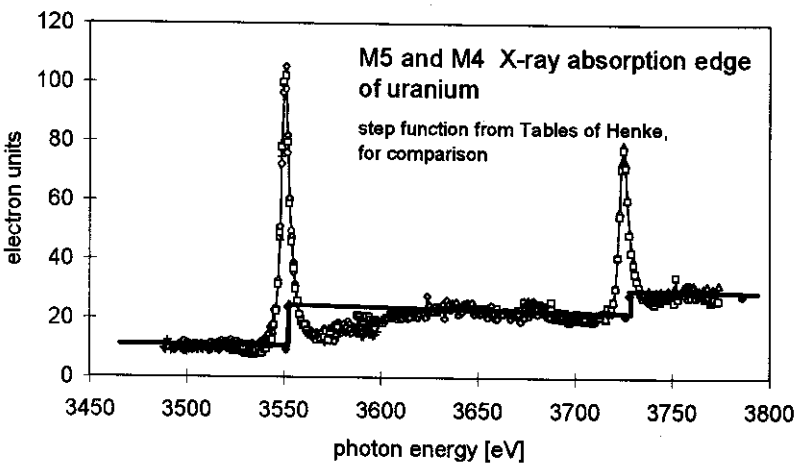
- using an image plate (quite successful, very desirable with online readout)

■ diffraction from crystals of asparaginyl tRNA synthetase, U derivative, using the CCD camera, at

- 4 wavelengths *) , angular range: $0° \leq 2\theta \leq 60°$, crystal rotation: $12 \times 1°$.
- 3 wavelengths, angular range : $-30° \leq 2\theta \leq 30°$, crystal rotation: $48 \times 1°$.

*This report is preliminary as it addresses to the measurements marked by *) only.*

Any experiment of anomalous dispersion starts from the absorption spectrum, from which the imaginary part of the atomic form factor of the anomalous scatterer can be deduced.



Anomalous dispersion of the imaginary part of the atomic form factor of uranium. The corresponding absorption spectrum has been taken at ID1, using various gaps of the undulator.

The measured diffraction peaks from crystals of the Asn-tRNA synthetase were weak and not numerous. Their intensity did not change with the duration of irradiation. Radiation damage was insignificant. The peak width at half maximum was about three pixels (=0.128 mm) and the peak height exceeded 1000 counts per pixel for some reflections close to the φ -axis. There is a high non-uniform background scattering, the intensity of which varies between 1000 to 5000 counts per pixel. The dark current (including offset) varies between 280 to 360 counts per pixel. The peaks have been indexed and their dispersion is presently being analysed. Expected $\Delta I/I = 50\%$. Max. attenuation by dispersion: 14%.

This first experiment opened the door to the use of the MAD method at wavelengths near the M_V edge of uranium. The requirements for the measurement of a complete data are listed together with the gain factors involved

	background reduction	intensity increase at the sample
new kapton foil of 12 μm (done recently)		20
collimator (+ beam stop)	10	
focusing mirrors		10
reduction of the air gap		2

A hardware synchronisation has bee set up for the experiment. Its principle is similar to the one developed by the CRG-D2AM.