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Undulator QEXAFS at the ESRF beamline ID26

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Undulator Q-EXAFS with continued scanning of undulator gap is routinely performed at the ESRF beamline ID26. The undulator gap is changed at constant speed while the monochromator angle is changed at variable speed. The flux loss when doing this, if any, can be kept below 10 % of the photon flux one would obtain when moving the undulator step by step. Spectra reproducibility is better than 0.1 eV. The acquisition time of a 1 keV scan goes from seconds to minutes as function of energy, harmonic selected and resolution required. Present limits of the technique and its applications are presented.

Keywords: Q-EXAFS, Biological EXAFS, Time-resolved XAS

1. Introduction

Undulators are very attractive insertion devices. Their brightness make them the best choice for microfocus beamlines but their relatively narrow emission bands has always been an inconvenient for using them on long range energy scanning experiments (i.e. EXAFS). In order to obtain the highest flux, the gap has to be changed relatively often during a scan and this procedure is in most cases time consuming (Rogalev et al., 1998).

We have found out that good quality spectra can be recorded by changing simultaneously and in a continued way the undulator gap and the Bragg angle of the monochromator. The angle is always set to the energy of the emission maximum of the undulator. This implies that for a constant opening speed of the undulator gap the monochromator angle has to be changed at variable speed. The flux loss when doing this, if any, is well below 10 % of the flux obtained by a conventional step by step movement. Previous attempts to perform QEXAFS (Frahm, 1989) experiments at undulator beamlines (Als-Nielsen et al., 1995) made use of wide harmonics (typically second harmonic) without changing the gap during the scan. Our method allows to use any harmonic order as well as it permits to have the maximum flux at each point of the spectrum.

2. Experimental Setup

The measurements have been performed at the European Synchrotron Radiation Facility (ESRF) beamline ID26. We used one of the three existing 42 mm magnetic-period 1.65 m long undulators as source.

The beamline monochromator is a fixed exit Kohzu monochromator operated with a Mc Lennan DC motor and a Mc Lennan PM304 motor controller. Two pairs of Si crystals (220 and 111) cooled at -140 degrees are permanently installed. PIN Photodiodes operated in photovoltaic mode are used as intensity monitors as well as fluorescence detectors. Each diode is associated to a low-noise current-to-voltage amplifier and then to a high-linearity voltage-to-frequency converter. The output signal is fed into a gated integrator (ESRF VDL board, Goujon, 1992). These cards provide two data per period of a periodic gating signal and several cards can be synchronized in such way that the acquisition is simultaneous to all of them. The monochromator encoder signals are measured in these channels allowing an automatic reconstruction of the energy scale. At fixed total scanning time, the number of points in the spectrum is determined by the frequency of the gating signal.

Scanning speed is conditioned by the undulator harmonic chosen. The undulator gap can be continuously moved at a minimum speed of 0.03 mm/s and a maximum speed of 0.5 mm/s. This roughly corresponds to a maximum time of 2 minutes and a minimum time of 10 seconds for a 1 keV spectrum using the first (fundamental) harmonic. Obviously, using a higher harmonic the time is reduced due to a faster change of the energy. Below 7 keV only the fundamental harmonic can be used at our beamline.

Fig 1 shows a single 10 second fluorescent-EXAFS spectrum of a 7 mM Carbonmonoxy-Myoglobin sample. No filter was used. The collection time of each of the 1000 points of the spectrum was 10 ms. The sample stayed in air at room temperature and several spectra could be taken without noticeable radiation damage. Fig 2 shows the XANES region of a single 1 keV scan of the same sample collected in 40 seconds with 10 ms acquisition time per point. The acquisition time corresponding to the region shown in the figure is less than 3 seconds. Both spectra were taken in fluorescence mode using the third undulator harmonic.

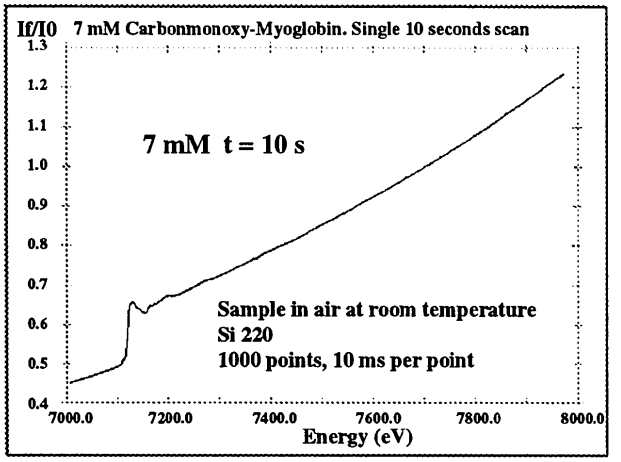

Figure 1
Fe K-edge spectrum of 7 mM Carbonmonoxy-Myoglobin recorded in 10 seconds

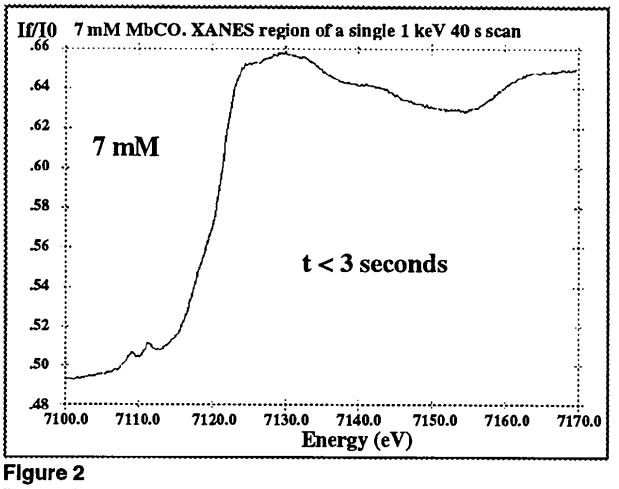

Figure 2
Fe K-edge XANES spectrum of 7 mM Carbonmonoxy-Myoglobin recorded in 2.95 seconds

3. Conclusion

We have demonstrated that by synchronising the movements of monochromator and undulator gap, high quality spectra can be recorded with a minimum lost of time. This enables us to perform a wide range of experiments, from mapping to slow kinetics experiments, as well as to minimise radiation damage to the samples.

4. Acknowledgements

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5. References

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