



Experiment title:
High resolution x-ray absorption measurements

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MI-83

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Report:

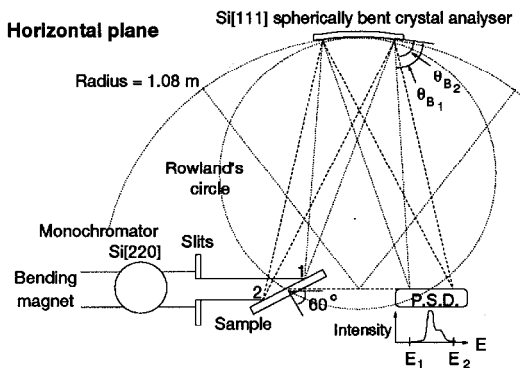


Figure 1: Schematic diagram of experimental setup implemented on BM29 for this experiment. The setup exploits the wide horizontal radiation fan from the dipole magnet to create an extended source so that several eV of scattered radiation are simultaneously imaged onto the position sensitive detector. This distinguishes it from existing configurations on undulator or wiggler sources where the analyser crystal has to be scanned.

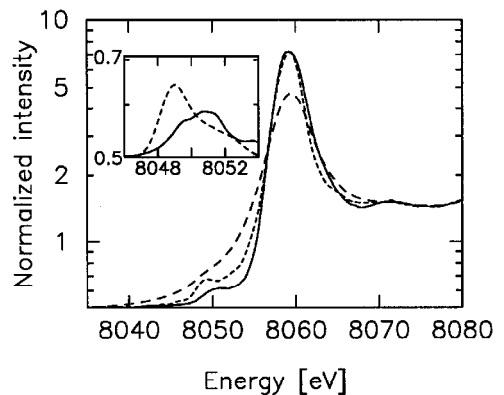


Figure 2: Inelastic x-ray scattering (IXS) measurement (self absorption corrected) of $\text{Ho}(\text{NO}_3)_3$ analysing a constant emission energy (solid line) coplotted with raw XAS data (long dashes) and the XAS spectrum deconvoluted by a Lorentzian of $\Gamma = 2.3$ eV according to the procedure described in ref. [1]. The pre-edge feature is attributed to $2p - 4f$ quadrupolar resonances and the offset between the pre-edge features are discussed fully in ref.[1]. The inset shows the calculated $2p - 4f$ XAS spectrum (dashed) compared to the calculated quadrupolar part of the IXS spectrum. All plots are shown in log scale (after an intensity offset of 0.5) for clarity of presentation.

We attempted to examine the Resonant X-ray Raman Spectrum (RXRS) of Xe above the K-edge employing (17,17,17) planes from silicon in near back reflection geometry to image the Xe $K\alpha_2$ line. The particular geometry chosen is worthy of note (see Figure 1) in that it differs from geometries used elsewhere to perform similar studies. In particular, three atmospheres of Xe gas have a substantial attenuation length at the K-edge ($\approx 3\text{cm}$) and this forms an extended source which can be wavelength resolved on the Rowland's circle with a position sensitive detector. Thus we have a parallel detection scheme with a static analyser crystal, rather than a step scanning system. Our geometry is particularly useful for solid specimen when working with a bending magnet. A line secondary source can be produced by tilting the specimen to grazing incidence, in contrast to the point source produced from an undulator or wiggler.

The system was setup in a short time of less than 1 day. It was aligned optically using a fibre optic point source fed by a laser to produce a reflected image on the PSD. Despite the care taken, the RXRS was not observed. Calculations showed that some signal should have been observed and it has subsequently been discovered that the monochromator was particularly inefficient at the Xe K-edge at that time and was delivering $\approx 10^{-2}$ of published flux, which may account for this.

However, the secondary spectrometer was successfully tested using an extended solid specimen containing strontium by bringing the X-ray beam onto the sample at an angle of incidence of 60° . The image, which evolves into the Sr $K\beta_1$ fluorescence was recorded and an excitation scan was measured using the (8,8,8) planes of Si.

Having proved the secondary spectrometer, in house research time has subsequently been used to record RXRS of Ho compounds (see Figure 2). To accompany this, we have made advances in general x-ray absorption technique by finding a numerical route to overcoming the lifetime difficulties. A comparison of RXRS and a lifetime removed XAS spectrum was thus possible for the first time (Fig.2). The performance of the analyser has also been assessed using SHADOW and this is also submitted for publication. The title and abstracts of these papers is given below [1, 2].

References

- [1] **Deconvolution of lifetime broadening at rare earth L_{III} edges compared to resonant inelastic x-ray scattering measurements.**

P. W. Loeffen, R.F. Pettifer, S. Müllender, M.A. van Veenendaal, J. Röhler, and D.S. Sivia, Phys. Rev. B 54, 14877 (1996)

The lifetime broadening of some rare earth L_{III} edge x-ray absorption spectra (XAS) is partially removed by deconvolving high quality data. The ligand field splitting of dipolar resonances are clearly resolved. Exposed pre-edge structure is compared to atomic multiplet calculations of the $2p \rightarrow 4f$ quadrupolar excitations. Ratios are thereby determined for the quadrupolar/dipolar oscillator strengths. Deconvolved XAS data of $\text{Ho}(\text{NO}_3)_3$ are compared to inelastic x-ray scattering measurements showing experimentally that the latter cannot reproduce a narrowed XAS.

- [2] **Inelastic x-ray scattering measurements at BM29**

P. W. Loeffen, R.F. Pettifer, S. Müllender, and D. Hill, J. de Physique, accepted (1997)

An inelastic x-ray scattering spectrometer has been implemented on the bending magnet beamline BM29 at the ESRF. It comprises a spherically bent perfect crystal as a Bragg analyser in near back reflection and a linear position sensitive detector (PSD) in Rowland's geometry. The setup exploits the wide horizontal radiation fan from a dipole magnet to create an extended source so that several eV of scattered radiation are simultaneously imaged onto the PSD with a static analyser crystal. This is in contrast to existing configurations using undulator or wiggler sources where one component of the emission spectrum is imaged and the analyser has to be scanned in angle. Results are shown at the holmium L_{III} edge where the resolution is better than -0.3 eV throughout the whole spectrum.