



Experiment title: High Energy Resonant X-ray Powder Diffraction

Experiment number:
HS-31

Beamline:
BL 1.5

Date of experiment:
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9

Local contact(s): Eric Dooryhee

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Names and affiliations of applicants (* indicates experimentalists):

*Dr. J. P Attfield, *Mr. A. M. T. Bell, *Ms. L. M. Rodriguez-Martinez

Department of Chemistry, University Of Cambridge, Lensfield Road, Cambridge CB2 1EW, UK.

Report:

One of the principal advantages of synchrotron radiation over characteristic X-rays is that a large wavelength range is available. This enables resonant (anomalous dispersion) effects to be exploited by selecting a wavelength at an elemental absorption edge. The advent of the ESRF enables absorption edges at much higher energies to be accessed for resonant studies. This simple experiment was performed to test the utility of resonant powder X-ray diffraction experiments at high energy absorption edges and over wide $\sin\theta/\lambda$ ranges.

A simple solid, CsI, was studied at the Cs K and I K edges at 33.2 and 36.0 keV, respectively, on powder diffractometer D16. The magnitude and possible $\sin\theta/\lambda$ dependence of f and f' at the Cs and I K edges were to be determined accurately. Several I doped CsBr samples were also studied at I K edge and off-edge wavelengths in order to determine the concentration of I from its resonant scattering.

Data were successfully collected and analysed by the Rietveld method. f' values up to -8.3 electrons/atom were observed for I at the K edge showing that large anomalous dispersion values can be observed at high energy K edges, comparable to those observed for lighter elements with K edges in the 5-20 eV region.

However, the $\sin\theta/\lambda$ dependence off and the detection of small amounts of I doped into CsBr could not be determined due to the Debye-Waller factors limiting data collection at high angles. For these room temperature measurements with $\lambda = 0.37 \text{ \AA}$, diffraction peaks were not observable above $2\theta = 3.5^\circ$. Low temperature measurements will be required to fully exploit high energy anomalous scattering effects.