

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

<http://193.49.43.2:8080/smis/servlet/UserUtils?start>

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.


Experiment title:

Time-Resolved QUEXAFS for in-situ characterisation of chalcogenide-based semiconductor nanoparticle formation using a combined biological/chemical approach

Experiment number:

MA 430

Beamline: ID26	Date of experiment: from: 31/10/07 to: 06/11/07	Date of report: 01/09/08
Shifts: 18	Local contact(s): Dr. Tsu-Chien WENG	<i>Received at ESRF:</i>

Names and affiliations of applicants (* indicates experimentalists):

*Dr Carolyn Isobel PEARCE (University of Manchester)

*Dr. John M. CHARNOCK (University of Manchester)

*Ms Victoria COKER (University of Manchester)

Dr Jon LLOYD (University of Manchester)

Dr. Frederick MOSSELMANS (Diamond Light Source)

*Prof Richard PATTRICK (University of Manchester)

Prof. Gerrit VAN DER LAAN (Diamond Light Source)

Report:

This was an *in situ*, time resolved experiment, in an injection cell system, to compare the manufacture of cadmium selenide quantum dots from biogenic versus inorganic starting materials. The dilute XAS capabilities of ID26 were used to analyse the different aqueous hydrogen selenide (5mM) starting materials produced by (i) the biological reduction of sodium selenite using *Veillonella atypica*, and (ii) by the chemical reduction of elemental selenium using sodium borohydride. Fig. 1 shows that there are significant differences between the two materials. The biogenic selenide was stable in the X-ray beam and the EXAFS showed four oxygens at a distance of 3.32 Angstroms, indicating completely dissociated Se anions surrounded by water. The inorganic selenide oxidised almost instantaneously in the beam and the EXAFS was dominated by Se-Se interactions.

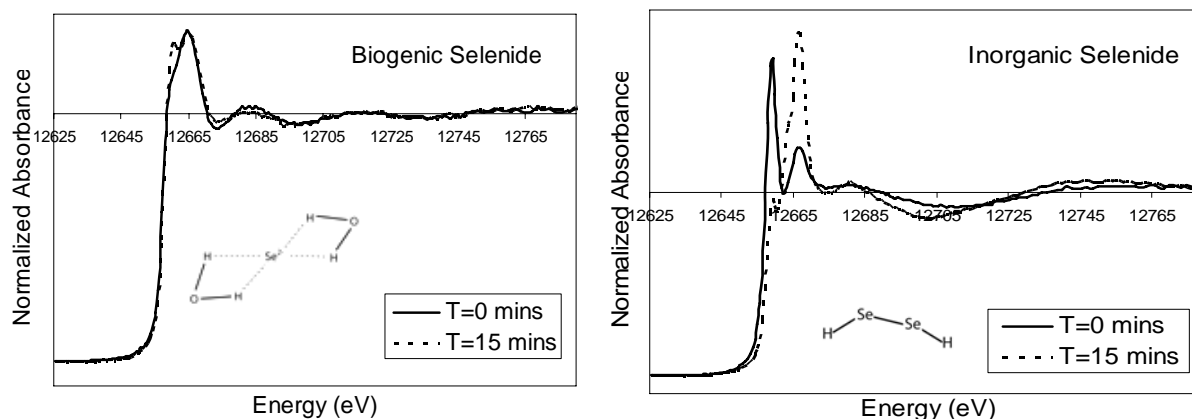


Fig. 1: Beam stability of biogenic vs inorganic selenide

The precipitation of the cadmium selenide quantum dots, upon addition of a cadmium perchlorate solution containing a thiol capping agent, was too rapid to be followed using X-ray Absorption Spectroscopy (XAS). Therefore, a novel technique involving using time scans to follow the absorbance at particular energies was employed. The energies selected to correspond to shifts in the absorbance as a result of the transition from aqueous hydrogen selenide to cadmium selenide colloids (Fig. 2)

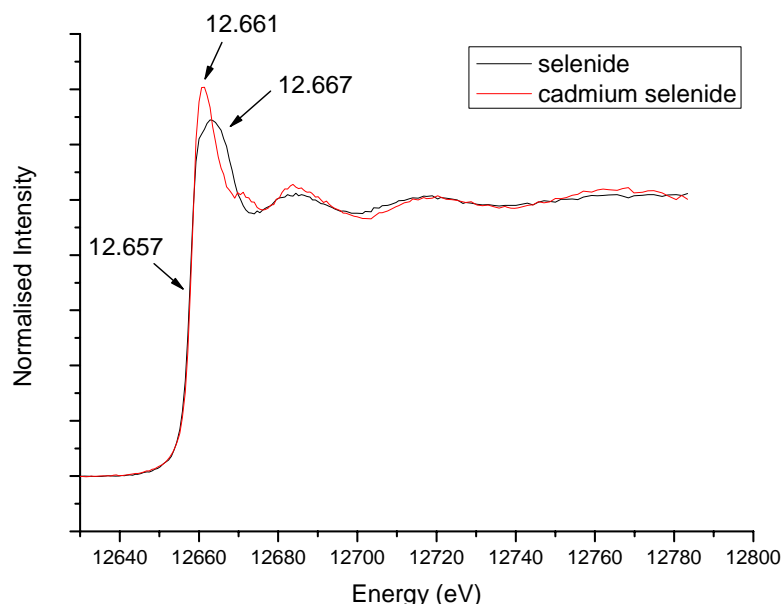


Fig. 2: XANES spectra for starting point (selenide) and end point (cadmium selenide) showing energies selected for time scans

The results from the time scans (Fig. 3) allowed the reaction to be followed and highlighted that the biogenic selenide reacted with the cadmium perchlorate at a slightly slower rate, giving greater control over the size of the precipitated cadmium selenide quantum dots.

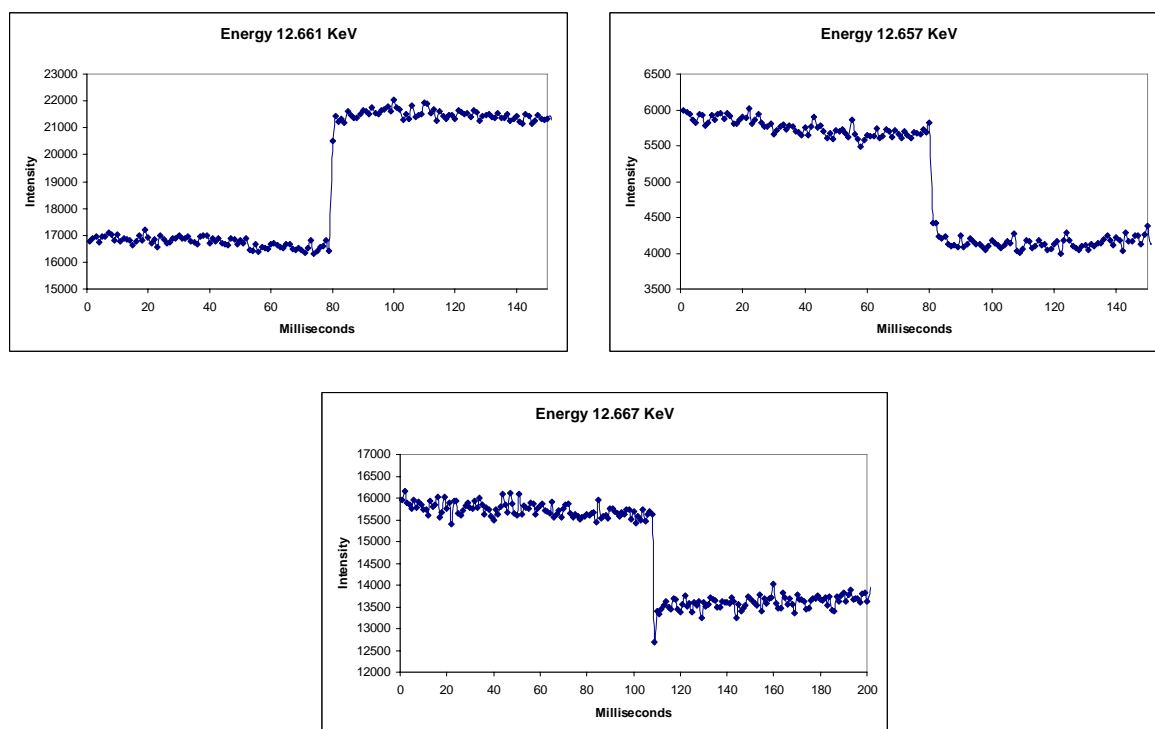


Fig. 3: Time scans to show precipitation of CdSe from a biogenic aqueous hydrogen selenide starting material

Pearce, C.I., Coker, V.S., Cutting, R., Patrick, R.A.D., Lloyd, J.R. and Prakash, N.T. (2008) Bion-nano-engineering: From clean up to catalysis. *Geochimica et Cosmochimica Acta. Suppl.* 72(12S), p. A730