

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:**

XAS characterization of Au-Fe nanoparticles functionalised with neoglycoconjugate molecules

Experiment number:

CH1601

Beamline:

BM29

Date of experiment:

from: 03-11-2003 to: 07-11-2003

Date of report:**Shifts:**

9

Local contact(s): Olivier MATHON*Received at ESRF:***Names and affiliations of applicants (* indicates experimentalists):**

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Report:

Nanoparticles, sufficiently small to form stable colloidal aqueous solutions, can be functionalised with different biomolecules, providing the possibility of recognising or binding to different targets, such a tissues or cells (1). Very recently we have developed a simple and versatile approach by which neoglycoconjugates of biologically significant carbohydrates are linked covalently to gold clusters, as a method for tailoring stable and water-soluble glyconanoparticles (2). By the same procedure, we have also prepared magnetic glyconanoparticles with a Au-Fe core of around 2 nm in diameter. These glyconanoparticles were the object of the experiment CH1601 carried out at the BM29 line.

In a first step we have carried out the study of the microstructure and electronic structure of pure gold nanoparticles functionalised either with neoglycoconjugates (Au-SRmaltose) or with alkane chains (Au-SR). Average particle sizes of 1.8 and 1.4 nm have been respectively found for these samples. In figure 1a, we have included the Au-L₃ XANES spectra where an increase in intensity of the white line at this L₃ resonance was found for the smaller sulphur functionalised gold nanoparticles (Au-SR sample).

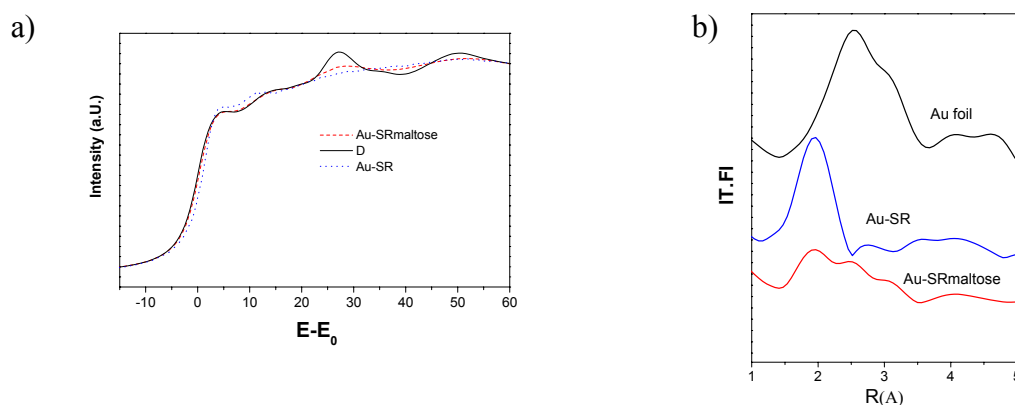


Figure 3: Au-L₃ edge XANES spectra of the different capped gold nanoparticles compared to bulk gold (a), and Modulus of the Fourier transform of the Au-L₃ edge of the different capped gold nanoparticles compared to bulk gold (b).

This behaviour indicates a charge transfer from the 5d gold states to sulphur atoms which produces a higher density of holes (3). The Fourier Transform of the EXAFS oscillations (figure 1.b) shows large Au-S and low Au-Au coordination numbers for the Au-SR nanoparticles. Taking into account the TEM images obtained for these samples, a gold core surrounded by a gold sulphide-like shell has been proposed to explain XANES and EXAFS data.

In a second step we have carried out the study of two samples constituted by an Au/Fe core functionalised with two different neoglycoconjugate molecules: a) 1-Au-Fe: Au/Fe nanoparticles functionalised with the disaccharide maltose, with a Au:Fe atomic ratio of 5: 0.1 and approximated formula $\text{Au}_{100}\text{Fe}_2(\text{C}_{23}\text{H}_{43}\text{O}_{11}\text{S})_{32}$ and b) 2-Au-Fe: Au/Fe nanoparticles functionalised with the disaccharide lactose, with a Au:Fe atomic ratio of 5:1.0 and approximate formula $\text{Au}_{100}\text{Fe}_{20}(\text{C}_{20}\text{H}_{37}\text{O}_{14}\text{S})_{32}$. Fe-K and Au-L₃ edges were measured, respectively in fluorescence and transmission modes, for solid samples supported on cellulose filters. Figure 2 shows the XANES at the Fe-K edge for the samples 1-Au-Fe and 2-Au-Fe (figure 2 a and 2.b respectively) in comparison to bulk iron and iron oxide ($\gamma\text{-Fe}_2\text{O}_3$), used as references. Just by comparison of the spectra it is obvious that the samples contain Fe in a oxidised state. As the beam time was allocated in November 2003, we are still undertaken a complete data analysis.

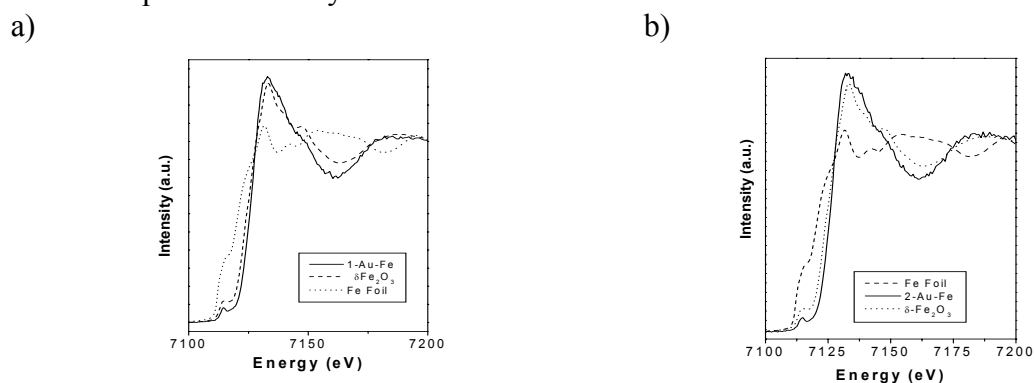


Figure 1: K-Fe edge XANES spectra for: a) 1-Au-Fe in comparison to the bulk iron and $\gamma\text{-Fe}_2\text{O}_3$; and b) 2-Au-Fe in comparison to the bulk iron and $\gamma\text{-Fe}_2\text{O}_3$.

In connection with this research, Palladium nanoparticles protected with alkylammonium salts showing a singular ferromagnetic behaviour (4) were also investigated in a preliminary study at the allocated beam time in BM29 last November. These samples will be the object of a new application for beam time.

References:

1. a) J. J. Schneider, *Adv. Mater.* **13** (2001) 529; b) A. Jordan, R. Scholz, P. Wust, H. Fahling, R. Felix, *J. Magn. Magn. Mater.* **201** (1999) 414
2. a) T. C. Rojas, J. M. de la Fuente, A. G. Barrientos, S. Penadés, A. Fernández, *Adv. Mater.* **14** (2002) 585; b) J. M. de la Fuente, A. G. Barrientos, T. C. Rojas, J. Cañada, A. Fernández, S. Penadés, *Angew. Chem. Int. En. Ed.* **40** (2001) 2257
3. P. Zhang, T. K. Sham, *Phys. Rev. Lett.* **90** (2003) 245502
4. B. Sampedro, P. Crespo, A. Hernando, R. Litrán, J. C. Sánchez-López, C. López-Cartes, A. Fernandez, J. Ramírez, J. Gonzalez Calbet, M. Vallet, *Phys. Rev. Lett.* **91** (2003) 237203