EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

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 via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can 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.

ESRF	**Experiment title:** Mechanistic study of solvent extraction of rare earths with sterically hindered alkyl phosphonic acids	**Experiment number:** **26-01-1040**
Beamline:	**Date of experiment:** from: 01-02-2015 to: 05-02-2015	**Date of report:** 08-02-2016
Shifts:	**Local contact(s):** Dipanjan Banerjee	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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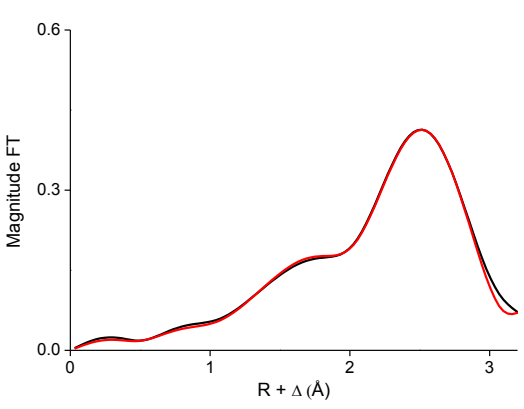
As new users, we have succeeded in measuring approximately 60 different samples during 9 shifts of 8 h. The edges of 6 different elements were measured: Br, In, Nd, Eu, Dy and Lu. About 80% of these measurements gave valuable and useful data. Bad data sets were often obtained in case of samples with low metal concentrations. All results will be used for publication (6 manuscripts) and as input for future proposals at DUBBLE. We were satisfied with the beamline performance and the help of the beamline scientist.

Bromine

A few samples HBr extracted to the organic compound trihexyltetradecylphosphonium bromide were measured and gave very good data (Figure 1, left). The data will be used in a manuscript about the extraction of acids towards quaternary compounds. Tests on the stability and quality of the data on a tribromide compounds were performed as well. The EXAFS function was of high quality and the compound seems to be stable. This type of compounds and measurements is calling for a new proposal (Figure 1, right).



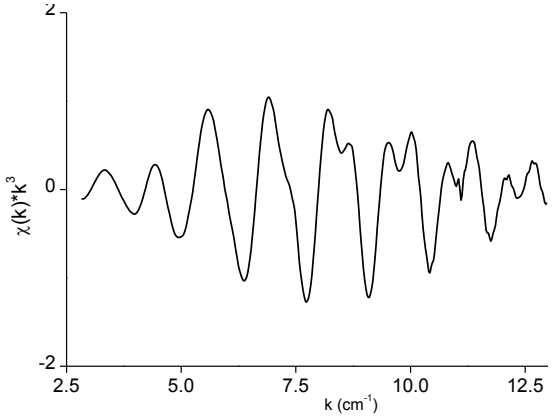


Figure 1. Left: Fourrier transform and model of Br, hydrated by two water molecules in trihexyltetradecylphosphonium bromide. Right: EXAFS function of Br_3^-.

Indium

The speciation of indium into trihexyltetradecylphosphonium chloride was studied as well and will be incorporated into a manuscript about the extraction of this metal towards quaternary compounds. The spectra were of very good quality because of the high energy K-edge of indium. The indium complex has allready been modelled and seemed to be $InCl_4^-$ (Figure 2).

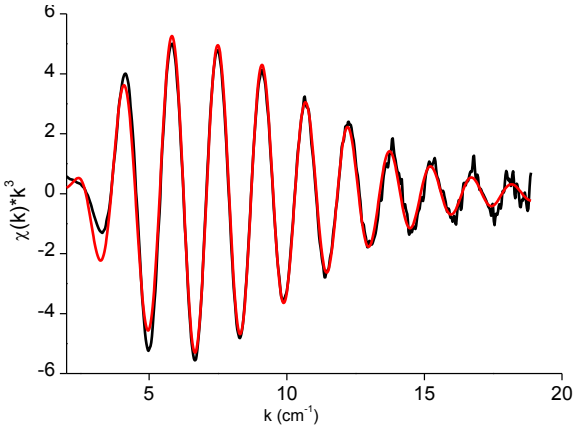


Figure 2. EXAFS function (black) and model (red) of $InCl_4^-$ extracted towards trihexyltetradecylphosphonium chloride.

Rare earths (Nd, Eu, Dy, Lu) (1)

The extraction of the rare earths towards sterically sterically hindered alkyl phosphonic acids was studied as well and compared to an industrially used extractant. The data quality was rather poor, as the concentrations were low. However, the XANES part of the spectrum (and part of the EXAFS) could be used and showed that the complexes formed with sterically hindered alkyl phosphonic acids showed no difference with the complexes formed by the industrially used extractants. This information can be used further to make even more sterically hindered extractants.

Rare earths (Nd, Eu, Dy, Lu) (2)

The extraction of the rare earths towards the quaternary compound trihexyltetradecylphosphonium nitrate and trihexyltetradecylphosphonium chloride was studied by XAFS. The data are of good quality and will be used in a manuscript that studies the speciation of the rare earths extracted to these type of compounds. An example of a typical EXAFS spectrum and its FT can be found in Figure 3.

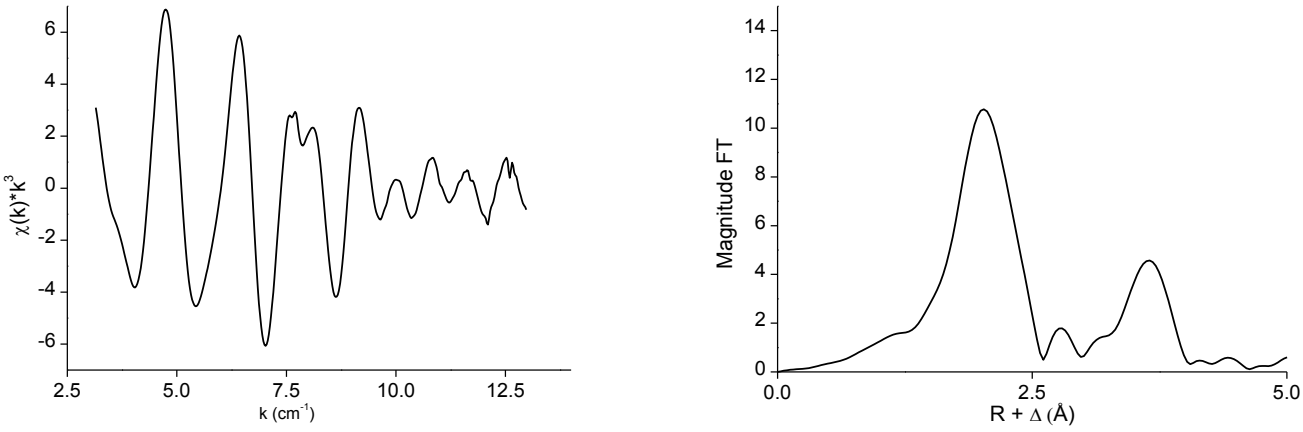


Figure 3. EXAFS function and FT of $Lu(NO_3)_3$ extracted into trihexyltetradecylphosphonium nitrate.