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:** Transitions in the coordination of cobalt and nickel based liquid metal salts upon heating	**Experiment number**: 26-01 1075
Beamline:	**Date of experiment**: from: 13-05-2015 to: 15-05-2015	**Date of report**: 17-05-2016
Shifts:	**Local contact(s)**: Dipanjan Banerjee	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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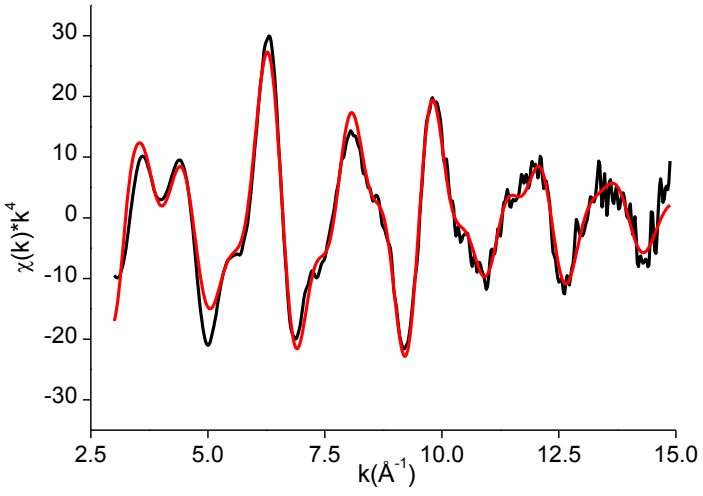
We have succeeded in measuring approximately 60 different samples during 9 shifts of 8 h. The edges of 12 different elements were measured: 6 transition metals: Mn, Fe, Ni, Co, Cu, Zn and 6 lanthanides Gd, Tb, Ho, Er, Tm and Yb. We succesfully installed and measured samples on the heating stage up to a temperature of 150 °C. In all cases, excellent data were obtained. We were satisfied with the beamline performance, the help of the beamline scientist and the help of the ESRF when we required suddenly dry ice for cooling our samples.

Transition metals

Metal containing ionic liquids of Mn, Fe, Ni, Co, Cu, Zn were measured at room temperature and at different temperatures. The example below shows the data and the model for the copper comples $[Cu(HeIm)_4][NO_3]_2$ (Figure 1a and 1b). Similar data and graphs are obtained for the other transition metals.



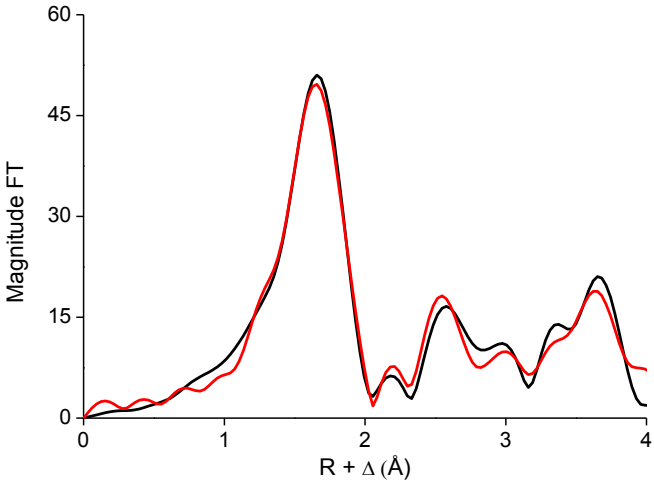


Figure 1a. EXAFS function (black) and model (red) of $[Cu(HeIm)_4][NO_3]_2$ fitted in R space between 0 and 4 Å.

Figure 1b. FT (black) and model (red) of $[Cu(HeIm)_4][NO_3]_2$ fitted in R between 0 and 4 Å.

In addition, we observed changes in structure as a function of the temperature. Figure 2 shows for instance the copper edge for $[Cu(BuIm)_4][Cl]_2$. There is a transition at 58 °C, followed by an equilibrium between 2 species at higher temperatures. Scheme 1 shows the phase transitions and equilibria based on different techniques as function of the temperature present in $[Cu(BuIm)_4][Cl]_2$.

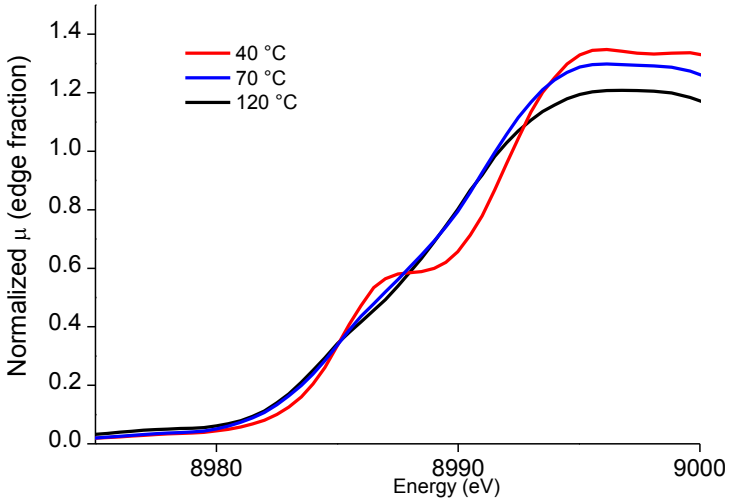
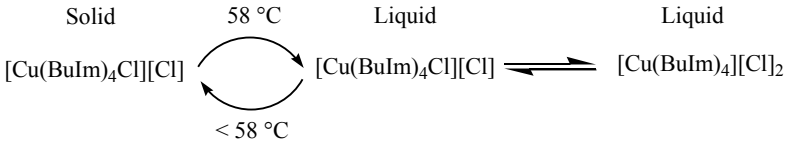


Figure 2. Absorption edges of Cu present in $[Cu(BuIm)_4][Cl]_2$ at different temperatures.



Scheme 1. Phase transitions and equilibria present in $[Cu(BuIm)_4][Cl]_2$.

Lanthanides

We succeeded in measuring 6 other lanthanides than the ones measured during our beam time at DUBBLE in february 2016. In total, we have now measured 10 lanthanides on the same system. The data (bond distances, coordination number, etc) will be used in combination with data obtained from other techniques such as IR, RAMAN, UV/VIS, Luminescence and NMR and published by the end of this year. Measurements on lanthanide pentanitrate complexes were performed. Figure 3a and 3b show a quick analysis (data and model) of $Yb(NO_3)_5^{2-}$.

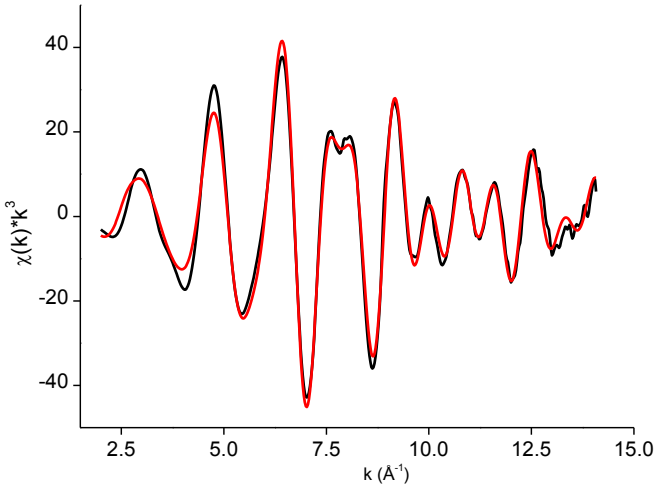


Figure 3a. EXAFS function (black) and model (red) of $Yb(NO_3)_5^{2-}$ fitted in R space between 0 and 4.6 Å.

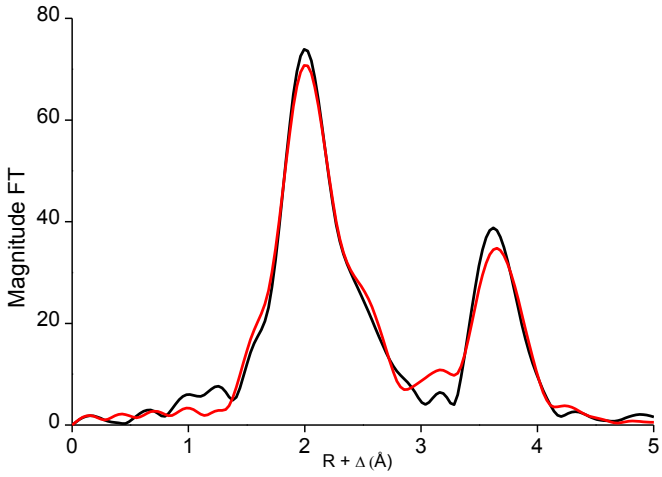


Figure 3b. FT (black) and model (red) of $Yb(NO_3)_5^{2-}$ fitted in R between 0 and 4.6 Å.