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 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.

ESRF	**Experiment title: In situ study of Zr complexation in aqueous fluids at high P and T**	**Experiment number**: EC-654
Beamline: ID26	**Date of experiment**: from: 13/07/2010 to: 20/07/2010	**Date of report**: 15/09/10
Shifts: 18	**Local contact(s)**: Kristina Kvashnina	*Received at ESRF:*

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Report:

The aim of the experiment is to study Zr complexation in aqueous fluids containing dissolved silicate components at conditions of the deep Earth. XANES and RIXS measurements at high temperature and pressure are used to provide further insight to the nearest and next-nearest neighbor elements surrounding Zr in the fluid. Temperature and pressure conditions are achieved by using hydrotheral diamond anvil cells.

RIXS and XANES spectra in fluorescence mode were recorded in fluorescence mode, due to the low concentrations of Zr in the fluids, using a high-resolution wave-length dispersive spectrometer. Several spectra on model compounds were also recorded in transmission mode. The measurements were done using different monochromator crystals, Si311 to collect high resolution spectra, and Si111 for higher intensitites especially for samples with low Zr concentrations.

Fig.1 shows XANES spectra collected on zirconosilicates, oxide and sodium aluminosilicate glasses model compounds. Fig.2 shows the comparison of XANES spectra of Zr in various fluids at high P & T. While the spectra of the silicate bearing fluids are very similar to those of zirconosilicates models in which Zr is 6-coordinated, those of HCl and NaOH solutions distinctly differs. This observation clearly points to the formation of different complexes as function of the melt composition.

The results clearly demonstrate that it is possible to obtain information on the complexation of Zr in aqueous fluids at high temperature and pressure using XAFS techniques. The spectra already indicate that Zr-complexation is very sensitive to the chemical composition of the system.

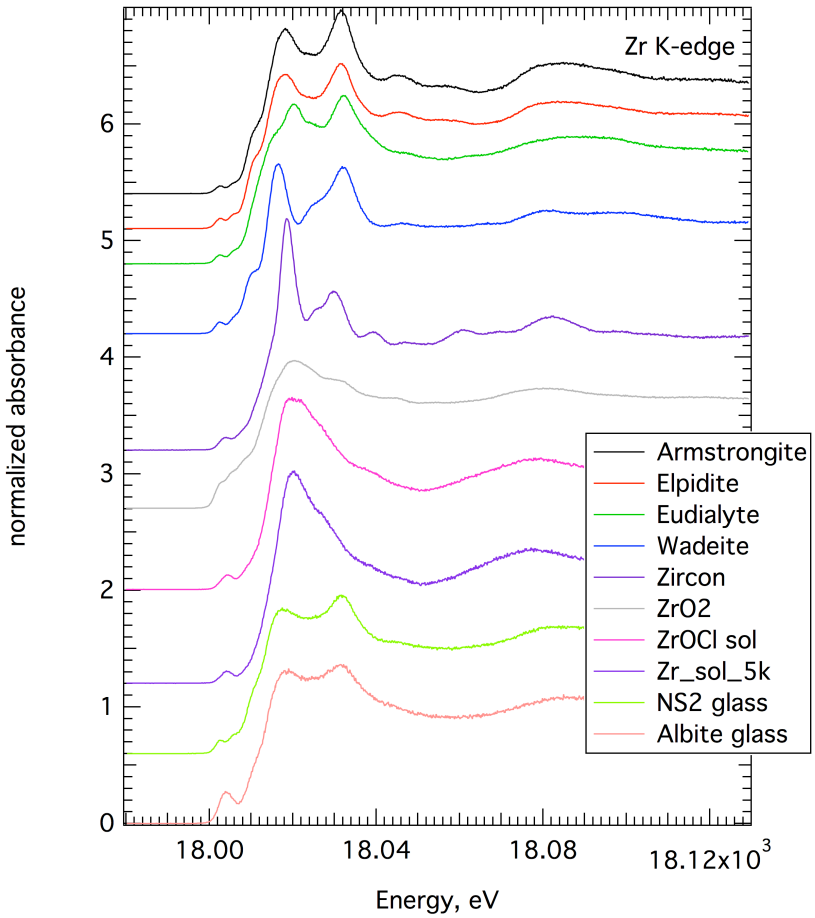

Fig. 1: Zr K-edge high-resolution XANES spectra for model compounds in which Zr is 6-coordinated (upper 4 spectra), 7-coordinated (zircon, baddeleyite) and 8-coordinated ()

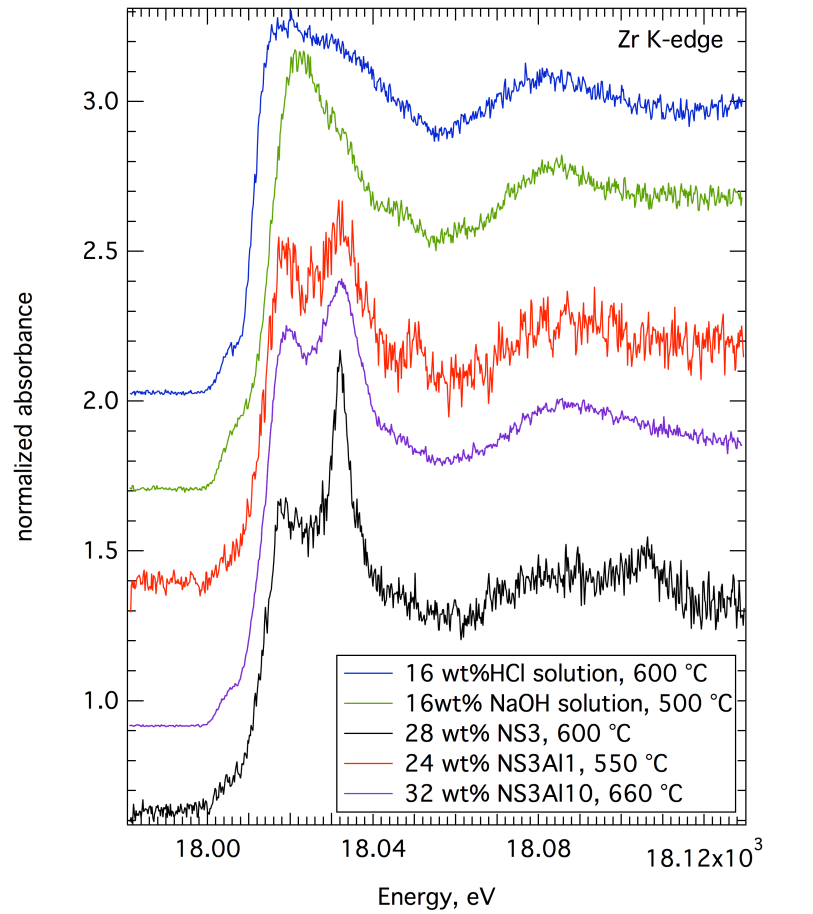

Fig. 2: comparison of high-resolution XANES spectra of Zr in various fluids at high P & T.