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:** Separating the twins: the hydrothermal geochemistry of Nb and Ta	**Experiment number**: ES-1266
Beamline: BM-16	**Date of experiment**: from: 11 July 23 to: 17 July 23	**Date of report**: 07 Sep 23
Shifts: 18	**Local contact(s)**: Denis Testemale	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Joel Brugger*, Barbara Etschmann*, Bin Hu* Monash University

Richen Zhong*, Huan Chen*, Beijing University of Science & Tech.

Aim

The aim of this experiment was to measure the coordination structure and solubility of Nb in hydrothermal fluoride, chloride and carbonate solutions at up to 500 °C and 800 bar to better understand the transport and deposition properties of Nb and its fractionation from its geochemical twin tantalum (Ta) in hydrothermal systems. This study complemented our previous experiment on the hydrothermal speciation of Ta.

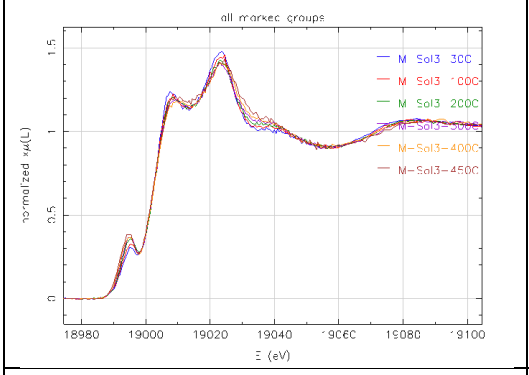
Experimental

Data were collected at the Nb K-edge (18986 eV) at the BM-16 (FAME-UHD) beamline, using the high T-P autoclave developed by the Institut Neel.

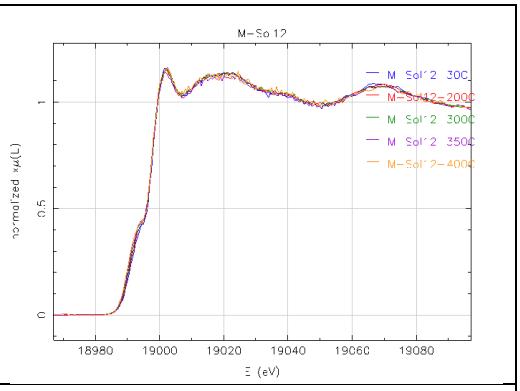
Sample	Conditions
Nb_2O_5	Pellet
Sol1: Nb_2O_5 in 2% HF	30-400 °C, 800 bar
Sol3: NbF_5 in 5% HF	25-450 °C, 800 bar
Sol4: Nb_2O_5 in 5 m K_2CO_3	25-500 °C, 800 bar
Sol6: Nb_2O_5 in 2.5 m K_2CO_3	25-450 °C, 800 bar
Sol7: Nb_2O_5 in 5 m NaOH	25-450 °C, 800 bar
Sol9: Nb_2O_5 in 1.2 m HCl	100-450 °C, 800 bar
Sol11: Nb_2O_5 in K_2CO_3 + NaF	200-500 °C, 800 bar
Sol12: $NbCl_5$ in 16 m HCl	25-400 °C, 800 bar

General observations

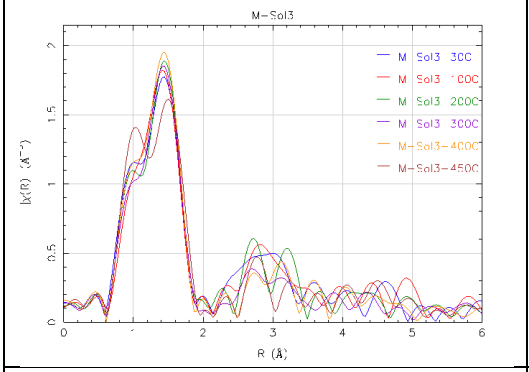
1. Nb had low solubility in carbonate solutions – this does seem to be a good ligand for tranporting Nb.
2. Nb is soluble in fluorine solutions (2 and 5%). The higher the fluorine concentration, the higher the temperature at which it was possible to measure Nb XAS.
3. Require high Cl concentrations to be able to measure a Nb-Cl complex.



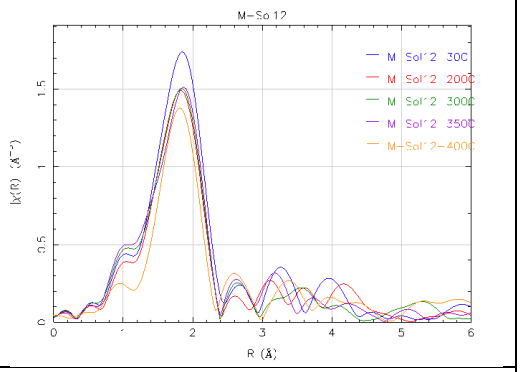
Sol3: NbF_5 in 5% HF



Sol12: $NbCl_5$ in 16 m HCl



Sol3: NbF_5 in 5% HF



Sol12: $NbCl_5$ in 16 m HCl

Impact

These data will form part of Bin Hu's phd thesis and will also be published as a paper.

Previous work on critical metals, with data collected at ESRF.

*Louvel, M., Etschmann, B., Guan, Q., Testemale, D. and Brugger, J. (2022) Carbonate complexation enhances hydrothermal transport of rare earth elements in alkaline fluids. Nature Communications 13, 1-11.

* Guan, Q.S., Mei, Y., Etschmann, B., Testemale, D., Louvel, M. and Brugger, J. (2020) Yttrium complexation and hydration in chloride-rich hydrothermal fluids: A combined ab initio molecular dynamics and in situ X-ray absorption spectroscopy study. *Geochimica Et Cosmochimica Acta* 281, 168-189.

*Guan, Q., Mei, Y., Etschmann, B., Louvel, M., Testemale, D., Bastrakov, E. and Brugger, J. (2022) Yttrium speciation in sulfate-rich hydrothermal ore-forming fluids. *Geochimica et Cosmochimica Acta* 325, 278-295

*Guan, Q., Mei, Y., Etschmann, B., Louvel, M., Testemale, D., Spezia, R. and Brugger, J. (2022b) Speciation and thermodynamic properties of La (III)-Cl complexes in hydrothermal fluids: a combined molecular dynamics and in situ X-ray absorption spectroscopy study. *Geochimica et Cosmochimica Acta*, 330, 27-46.

*Liu, Etschmann, Hazemann, Testemale, Migdisov and Brugger, 2017. Revisiting the hydrothermal geochemistry of europium(II/III) in light of new in-situ XAS spectroscopy results. *Chemical Geology*, 459, 61–74.