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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:** **An XAS study of pressure effect on aqueous nickel chloride complexes in hydrothermal brine**	**Experiment number**: ES 77
Beamline: BM30B	**Date of experiment**: from: 27 Nov, 2013 to: 03 Dec, 2013	**Date of report**: 22/08/14
Shifts:	**Local contact(s)**: Jean-Luis Hazemann, Denis Testemale	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Synchrotron XAS measurements at the Nikel *k*-edge for Ni bearing chloride solutions have been conducted at the FAME beamline (BM30B) at ESRF on 27 Nov-03 Dec 2013. The aim of this project was to investigate the impact of pressure on the speciation of aqueous Ni chloride complexes in hydrothermal brines.

Afer solving some technical problems (leaking Be windows, testing of new heater) that accoured in the first three days, Ni *k*-edge EXAFS spectra for 8 sample solutions were then collected with sodium chloride concentration ranging from 0 to 6 m and temperature range from 35 ^{o}C to 500 ^{o}C, and pressure ranging from 300-1500 bar. XAS data have also been collected for 0.05 m NaBr solution and $NiCl_2$ in concetrated LiCl solutions.

We found that Pressure has opposite impact on the speciation of nickel chloride complexes: while increasing tempertures and chloride concentration enhance the transition from octahedral (e.g., $Ni(H_2O)_6^{++}$) to tetrahedral species (e.g., $NiCl_3(H_2O)^-$), increasing pressure cause the formation of octahedral species as shown in Fig. 1 and Fig. 2a, 2b). However the impact is not as significant as temperature does (Fig 2c, 2d). We attribute these changes to the change of entropy that drive the speciation change.

Preliminary data have been presented as part of an invited talk at 2014 Goldschmidt conference in Sacramento, USA in August 2014. A manuscript including thise work is under prepration.

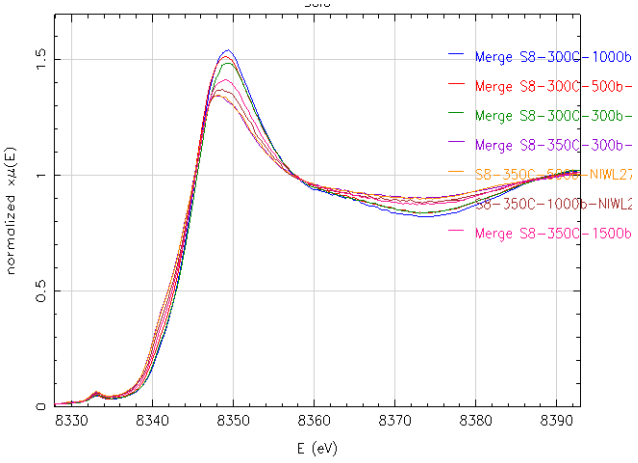

Fig. 1 XANES spectra change as a funtion of pressure at 300 and 350^{o}C for 0.07 m NiCl2 in 6 m NiCl soltuion

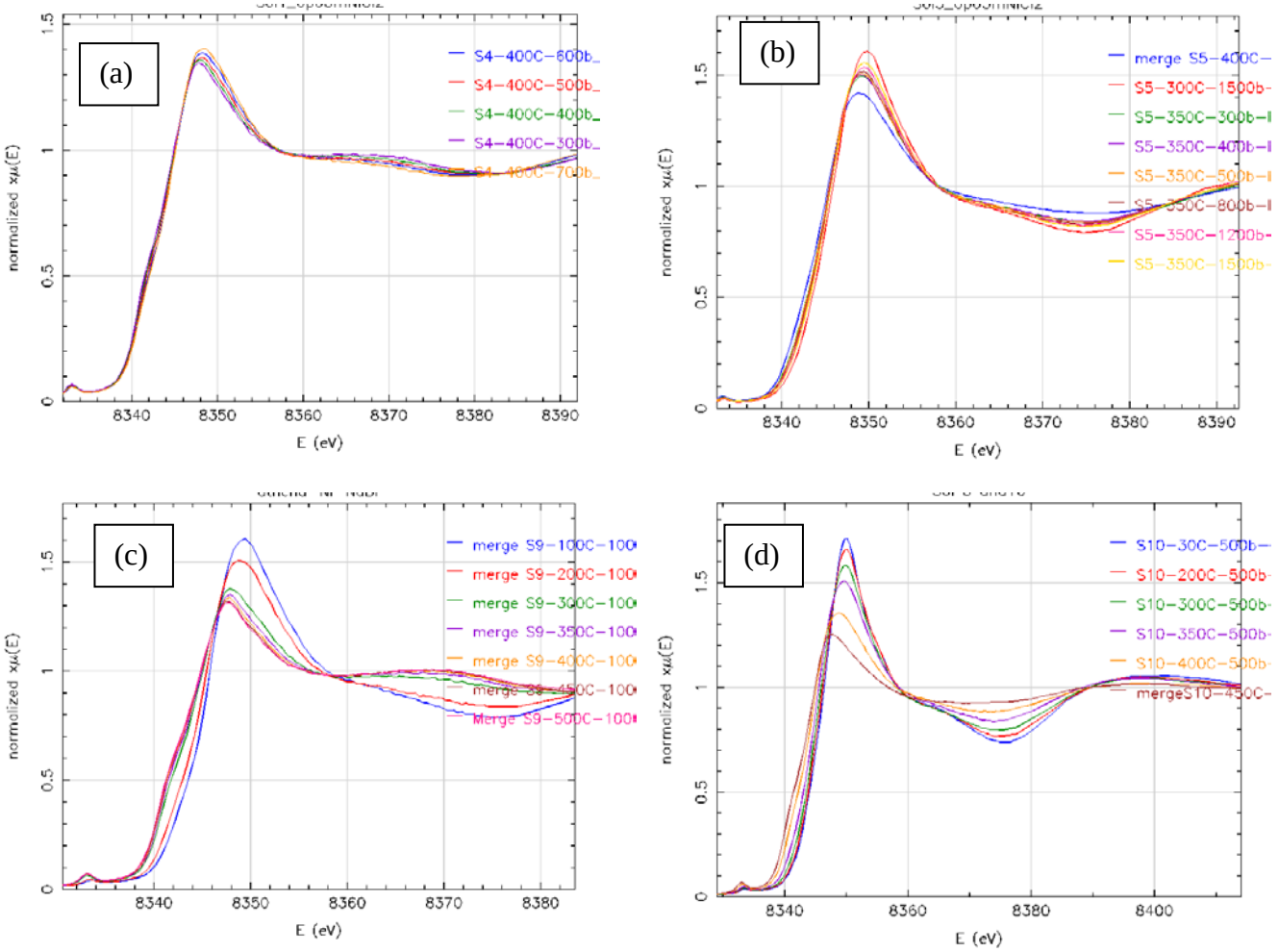

Figure 2. Nikel K-edge XANES spectra of sample solutions: (a) 0.05 $NiCl_2$; (b) 0.07 m $NiCl_2$ in 5 m NaCl; (c) 0.07 m NiCl2 in 15 m LiCl; and (d) 0.07 m $NiBr_2$ in 0.5 m NaBr.