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:** Probing the dynamics of the thermal quenching process with XAS	**Experiment number**: 26-01 1017
Beamline: BM26A	**Date of experiment**: from: 5/11/2014 to: 10/11/2014	**Date of report**:
Shifts: 15	**Local contact(s)**: Dipanjan Banerjee	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Philippe Smet

Dirk Poelman

Katleen Korthout

Jonas Joos

Heleen Sijbom

Katrien Meert

Report:

a) XANES

XANES spectra will help us determining the oxidation state of Mn in CaZnOS. Figure 1 depicts the normalized XANES spectra at the Mn K edge of the two investigated samples, together with some XANES spectra of reference materials. This figure demonstrates the presence of Mn^{2+} in both samples.

b) Concentration of Mn

Although we cannot determine the concentration of the samples quantitatively, we can determine it qualitatively. The setup was not changed, i.e. the distance between the fluorescence detector and the sample was the same, for the different samples. As such we can compare the signals of both and look at the difference in x-ray absorption coefficient. Figure 2 shows the spectra of the two samples after a merge of 4 spectra. As can be seen from this Figure, the signal in CaZnOS (Ang) is twice the signal in CaZnOS (Jonas), showing that the concentration of Mn in CaZnOS (Ang) is twice the concentration in CaZnOS (Jonas).

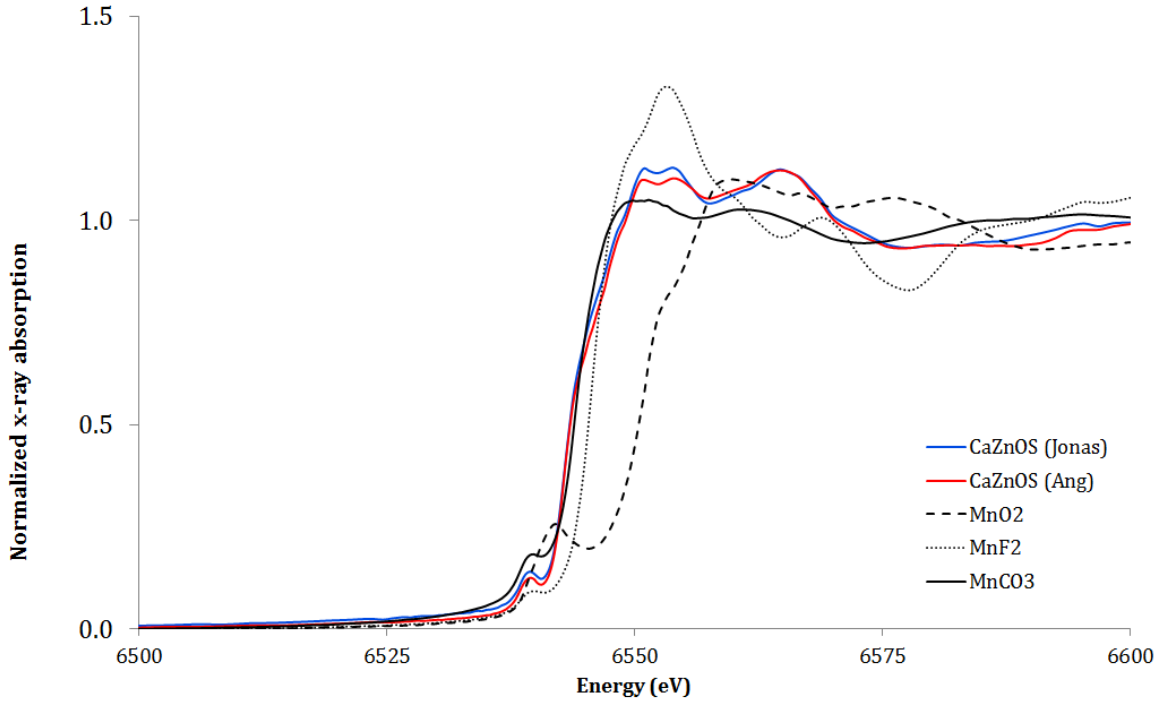

Figure 1: ***Normalized XANES spectra at the Mn K edge.***

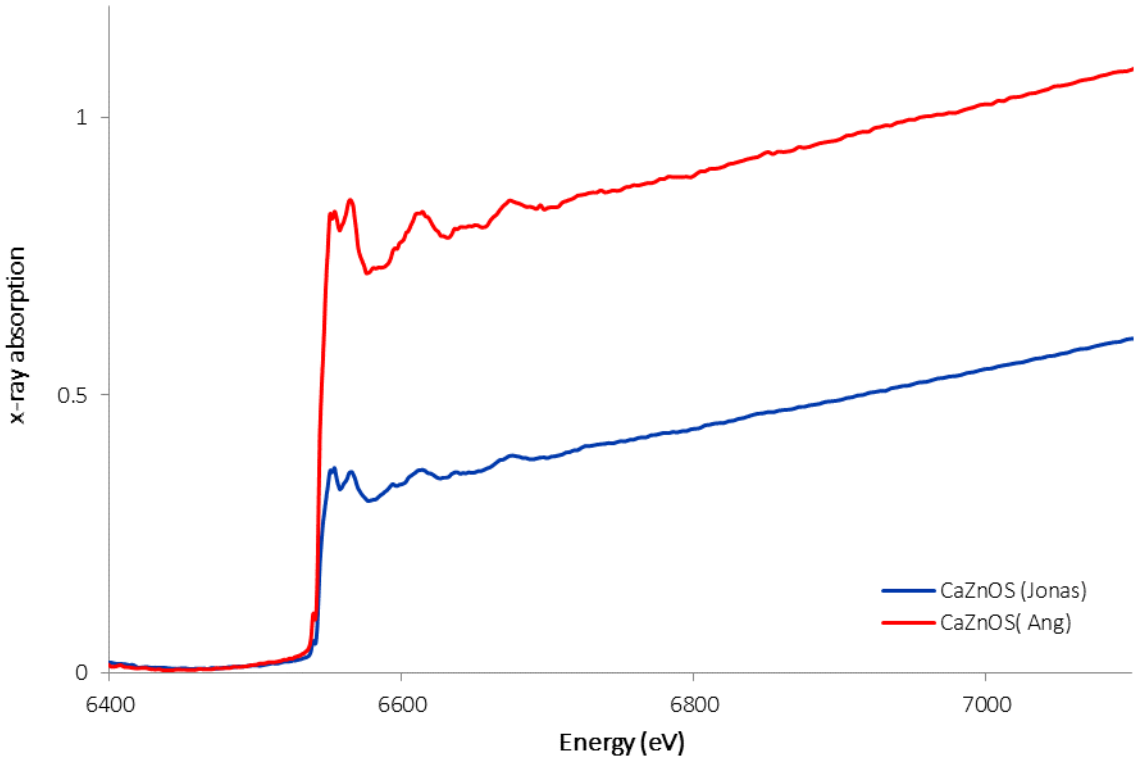

Figure 2: ***X-ray absorption spectra as measured (for each sample 4 spectra were merged).***

c) EXAFS

To start the EXAFS analysis both the substitution at the Ca site and the Zn site were evaluated (see Figure 3), this figure clearly shows that the tetrahedral Zn site coincides with the measured spectrum.
Subsequently the spectrum was analysed using the Zn site model as a starting point. We were able to decide that Mn ions in CaZnOS will occupy the Zn sites in the lattice.
These results were published as:

"Charge transfer induced energy storage in CaZnOS:Mn – insight from experimental and computational spectroscopy", Jonas J. Joos, Kurt Lejaeghere, Katleen Korthout, Ang Feng, Dirk Poelman and Philippe F. Smet, Phys. Chem. Chem. Phys., 2017, 19, 9075.

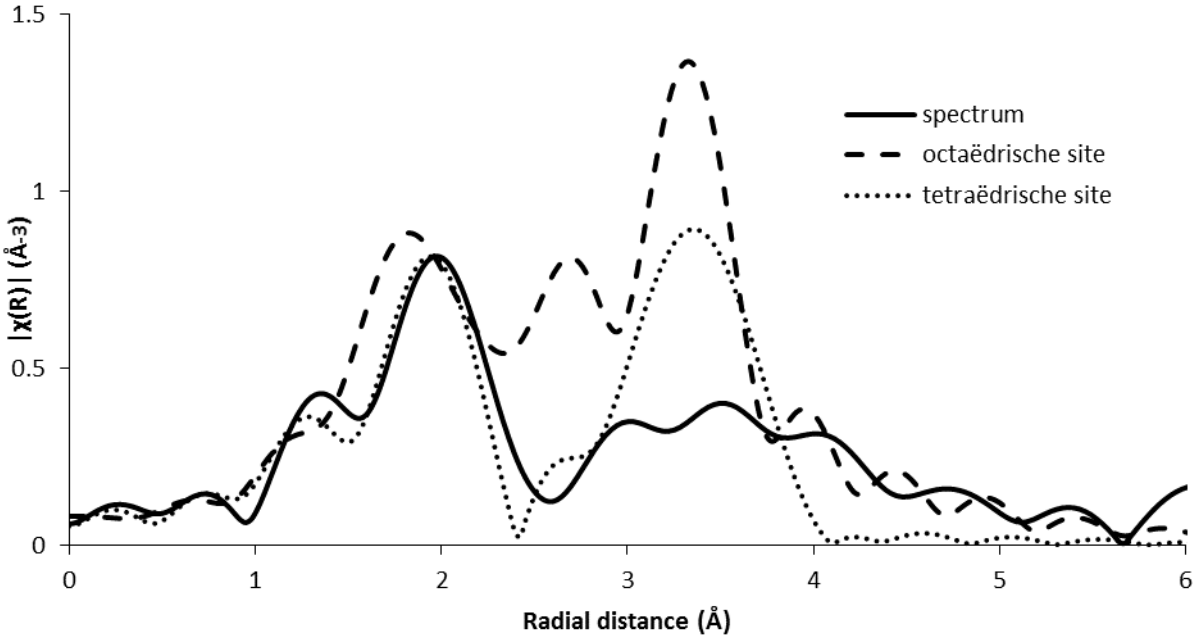

Figure 3: ***Comparison of the measured spectrum (solid line), with the theoretical spectra of the Ca site (dashed lines) and the*** *Zn site* ***(dots).***

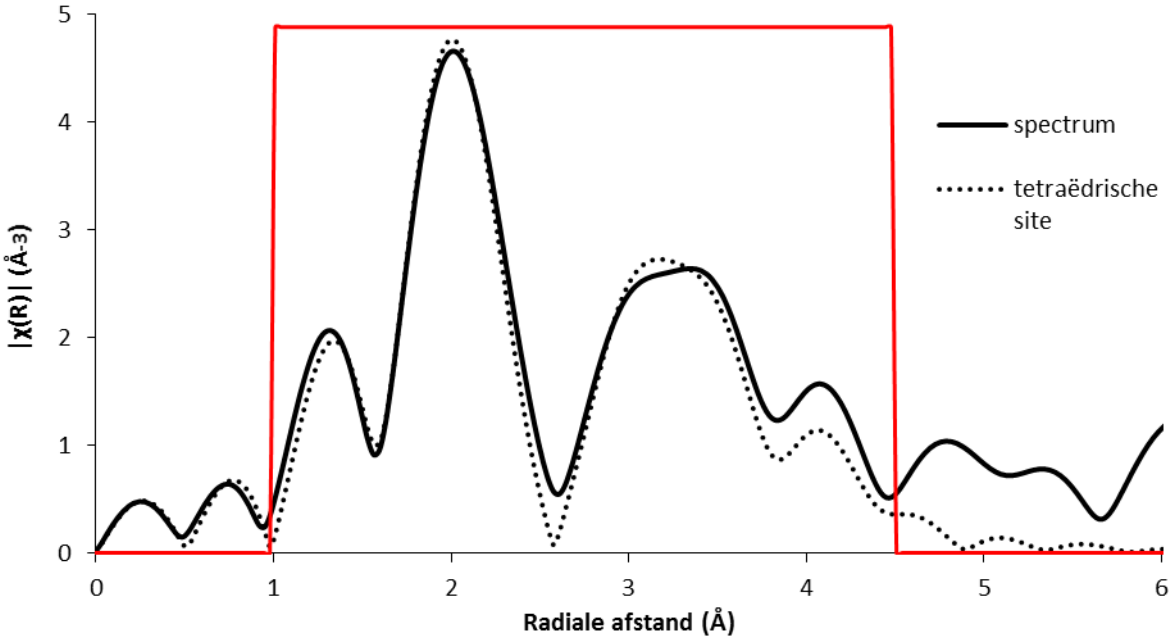

Figure 4: ***Result of the fitting of the experimental spectrum where the Mn ions occupy the Zn sites in the lattice. The parameters of the fit are listed below***

Parameter	01.1	01.3	S1.1	S1.2	Ca1.1	Ca1.2	Zn1.1
amp	0.534(25)						
ΔE0	4.23(36)						
ΔR	0.101(16)	-0.022(18)	0.0730(51)	0.107(27)	0.0093(76)	0.096(11)	-0.1589(92)
σ^2	0.0013(18)		0.00278(75)		0.00057(73)		0.0079(12)
Reff	1.900	4.21	2.372	4.443	3.401	3.768	3.757
N	1	6	3	3	3	3	6