

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://www.esrf.fr/misapps/SMISWebClient/protected/welcome.do>

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal (“relevant report”)

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a “*preliminary report*”),
- even for experiments whose scientific area is different from the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as “relevant report(s)” in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

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.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number 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.



	Experiment title: At the dawn of Zr-MOF formation	Experiment number: 20210416 (bm311144)
Beamline:	Date of experiment: from: 16.04 - to: 19.04	Date of report: 13.09
Shifts:	Local contact(s): Wouter VAN BEEK <wouter@esrf.fr>	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Olena Zavorotynska (University of Stavanger) Sachin Maruti Chavan (University of Stavanger)		

Report:

Due to the travel restrictions, it was not possible to perform the in-situ experiments suggested in the proposal. Instead, the ex-situ samples were synthesised and sent to the local contact who performed in situ XAS and PXRD measurements together with 2 in-situ PXRD experiments.

In total, we have measured 27 samples at Zr- and Ce k-edges and at Ce L-edge, also at Ni and Co k-edge (XAS) plus the same samples in XRD. All the measurements were performed on MOFs so the experiment was related to the proposal although we were not able to carry out the in situ experiments.

The experiments are divided into 4 groups where each is expected to result in a publication:

1) Parent compounds (4 samples): effect of synthesis parameters and post-synthetic treatment on structure of Zr-MOFs. Additional characterisation is being performed now in-house (TGA, Raman, proton-NMR, BET surface area measurements). In-situ synchrotron PXRD was performed on one of these samples.

2) Zr/Ce-mixed MOFs : physical mixture – comparison of Zr-Ce XAS data of our preparation method to the method reported in literature. 3 samples are included in this groups

3) Zr/Ce-mixed MOFs: synthesis of the mixed MOFs. These MOFs were synthesised (in house) adding Ce-precursor at selected points in time determined in our previous synchrotron experiment in BALDER beamline at MX-IV. This experiments includes 8 samples. The TGA data collected in-house indicate some differences in the resulting samples. Here, the local structures around Ce and Zr are being compared along with the synchrotron X-ray diffraction data.

4) Metal loading (12 samples): Ni and Co metals were loaded in different conditions on Zr-MOF. The data were obtained on Co- and Ni k-edges along with Zr edge. At present, the XAS data at the metal edges indicated a very low presence of these metals in the samples (Figure 1).

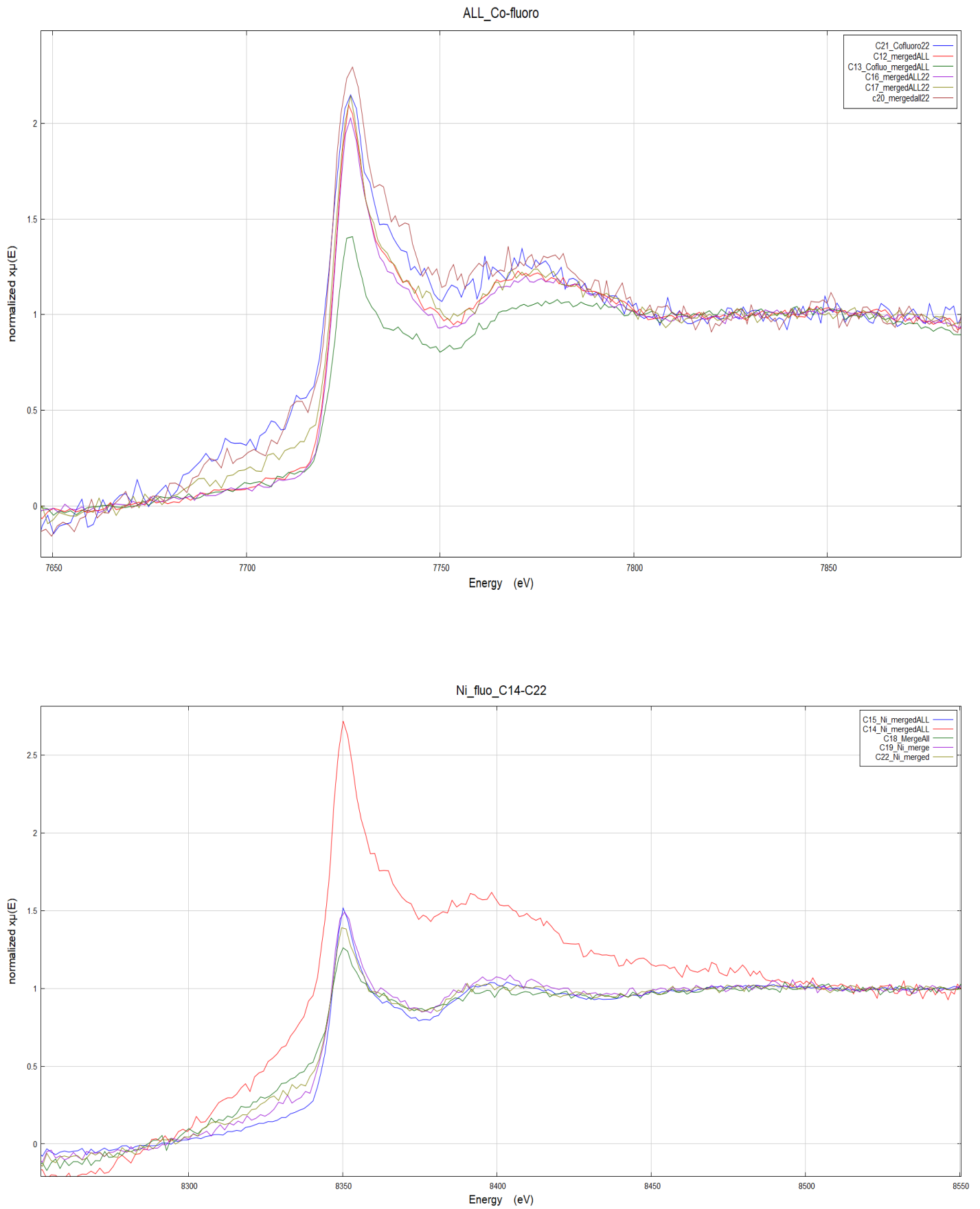


Figure 1. XAS data (in fluorescence) at Co- and Ni k-edges for the metal uptake in MOFs. For all the samples, at the moment data analysis and additional characterisation is in process.

