



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:**

Searching for primordial metabolisms on >3.3 Gy microfossils from Barberton

Experiment number:

ES999

Beamline:

BM31

Date of experiment:

from: 30 Apr 2021 to: 02 May 2021

Date of report:

13/09/21

Shifts:

6

Local contact(s):

Wouter Van Beek

*Received at ESRF:***Names and affiliations of applicants** (* indicates experimentalists):

Lemelle, L. ; Bonneviot L.; Simionovici, A.; Maldanis L., Van Beek W.

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Laurence.lemelle@ens-lyon.fr; laurent.bonneviot@ENS-Lyon.fr*ISTerre, Univ. Grenoble Alpes, CNRS, CS 40700, 38058 Grenoble Cedex 9, France, Institut Universitaire de France (IUF),alexandre.simionovici@univ-grenoble-alpes.fr, lara.maldanis@univ-grenoble-alpes.fr**Report:**

This XANES experimental work was successfully carried out in full remote mode applying the automated acquisition procedure of the beamline.

The 6 allocated shifts were split into two sessions (May and June) to optimize the fairly long (3-4 weeks) chemical syntheses of the samples, the sample preparations, and the Noise-over-signal ratio. 27 pellets were prepared with cellulose binding ligand with a weight of compounds predicted by Athena software. Other were mineral references lent by Alain Manceau.

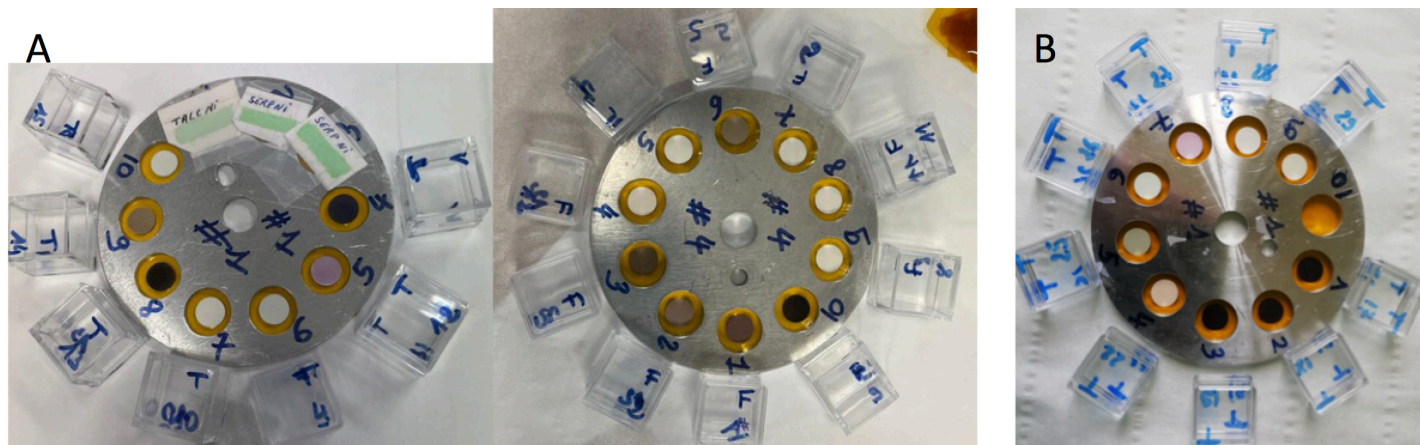


Figure 1: (A) Photographs of the sample holders prepared for transmission XANES measurements (#1) and for the fluorescence XANES measurements (#4) in the May session. (B) Photograph of the sample holder prepared for the definitive transmission XANES spectra acquisition.

The first exploratory session was carried out on 17 pellets + 3 standards on tape+ the calibrating Nickel foil of the BM31 beamline. This session displayed that all the applied thermal annealing of the Ni-complexes under air severely oxidized the synthesized Ni-porphyrin complexes, even if encapsulated in Silica. Besides, while the statistics of the fluorescence spectra were entirely satisfactory, those of the spectra acquired in transmission mode could be improved.

The final second session was carried out in June. New Ni-porphyrin complexes were synthesized and incorporated in silica. Thermal annealing was carried out under Argon. All the spectra were acquired in transmission mode with a fully satisfactory statistical quality.

Based on these two experimental sessions, the first set of spectra of inorganic Ni-porphyrinoid complex, thermally annealed or not, is fundamental for constraining the interpretation of the nano-XANES recorded on the >3.3 Ga microfossils is now acquired.

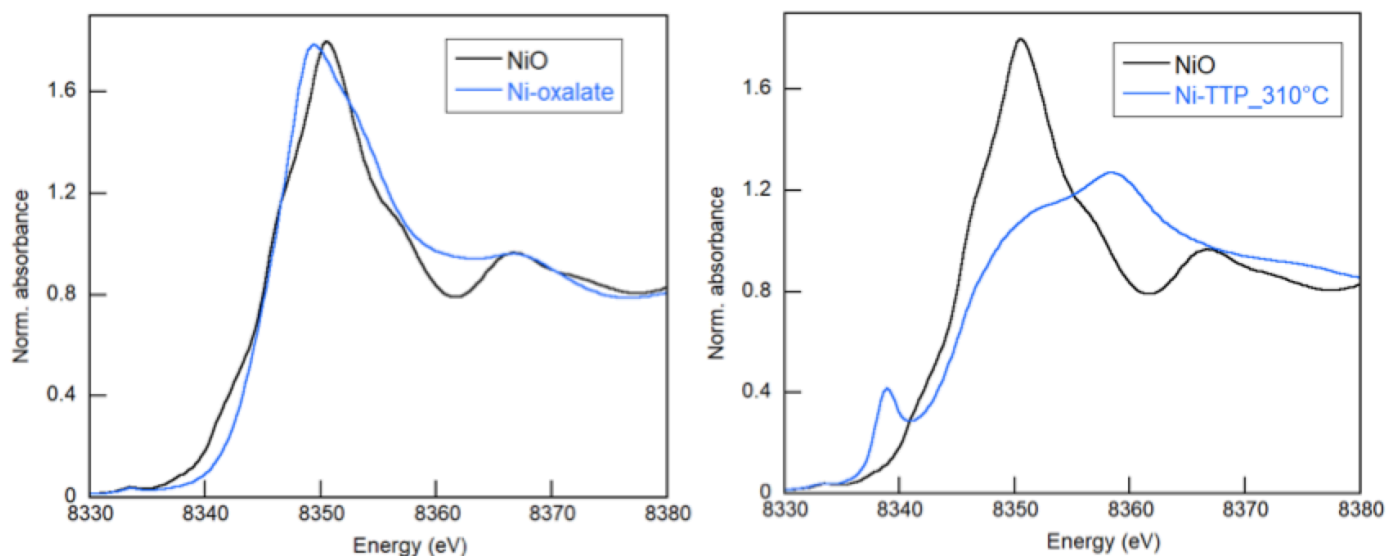


Figure 2: Synchrotron radiation XANES spectra at the Ni K-edge to display the quality of the statistics of the spectra acquired on the pellet prepared with the synthesized powder of Ni-porphyrinoid complex (triphenylporphyrin) annealed under reducing atmosphere at 310°C (right) and two reference compounds: Ni-oxalate complex (left) and commercial NiO and synthesized (left and right).