EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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

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 form 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:	Experiment number: ES934
Beamline: ID16B	**Date of experiment**: from: 21-01-2021 to: 26-01-2021	**Date of report**:
Shifts: 12	**Local contact(s)**: Remi TUCOULOU TACHOUERES	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Cinzia Bottini, Università di Milano, Dipartimento di Scienze della Terra*

Monica Dapiaggi, Università di Milano, Dipartimento di Scienze della Terra*

Nicola Rotiroti, Università di Milano, Dipartimento di Scienze della Terra*

Report:

During the experiment ES934 we perfomed XRF analyses, in very high resolution, of isolated coccoliths (average size comprised between 2 and 8 μm, depending on the species) taken from geological samples of early Cretaceous age colected in different localities. Coccoliths are produced by phytoplanktonic algae called coccolithophores and are made of calcite. The main objective of our analyses was to map the concentration and distribution of trace elements (Sr and lighter elements) in coccoliths formed during peculiar paleoenvironmental conditions (extreme warmth, high fertility, and/or high concentration of trace elements) and to compare the results with those published in Bottini et al. 2020 referring to cultured specimens in normal VS stress conditions (high CO_2 and metal concentrations). The fossil specimens analysed belonged to six different species and this allowed us to compare the element distribution not only through time and among paleolocalities but also among species.

The experiment was very successful as we managed to analyze a relatively large number of samples, ca. 50, with a resolution of 50 x 50 nm, and a few larger ones with a resolution of 100 x 100 nm. This was, to our knowledge, the first time that a large number of fossil coccoliths were analysed in one session.

The experiment took tresure of experiment ES-841 particularly for what concerns the sample preparation procedure which was improved. Specifically, we used a TEM sample holder with a silicon nitride grid. This was a winning choice for what refers to finding the specimens and to get a good signal.

The results obtained are stunning. For instance we were able to appreciate differences in the Ca and Sr/Ca distribution in the various species analysed (Fig. 1) as well as the distibution of Mn which largely differ among specimens in relation to coccolith preservation (Figs. 1 and 2). We were also able to differenciate among elements included in the calcite and others related to diagenetic coating. The first publication of the results is in preparation and will be submitted soon.

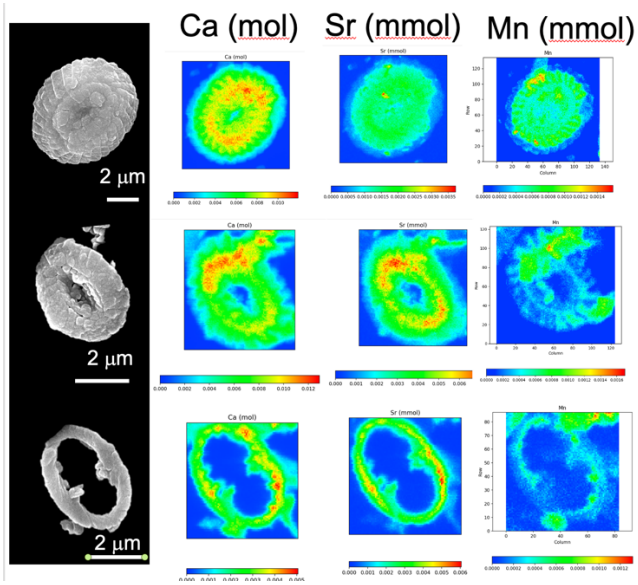

Fig. 1 shows high-resolution images at 50 nm resolution of three fossil coccoliths of about 3 to 6 µm of mid-Cretaceous ag(ca. 120-113 Ma). From top: *Watznaueria, Biscutum constans* and *Zeughrabdotus erectus*. The maps report the concentrations of Ca, Sr (that enters the calcite structure) and Mn.

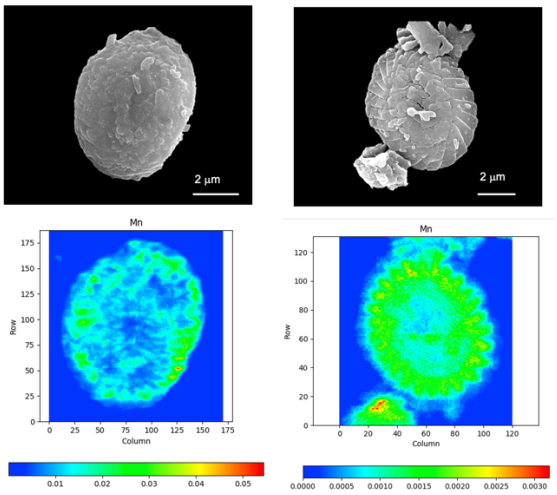

Fig. 2 shows the concentration and distribution of Mn of two specimens of *Watznaueria*. The left one is more diagenetically altered compared to the right one and show different Mn distribution patterns.

References

Bottini C., Dapiaggi M., Erba E., Faucher G., Rotiroti N. (2020). Scientific Reports. 10:9825 | https://doi.org/10.1038/s41598-020-66503-x