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.

ESRF	**Experiment title:** Nanoscale carbonate-oxalate mineralogy from the Great Barrier Reef: understanding pollution and climate change impacts during coral and sediment formation	**Experiment number**: EV-449
Beamline: ID21	**Date of experiment**: PART 1: from: 4 Nov 2021 to: 8 Nov 2021 PART 2: from: 28 Jan 2022 to: 31 Jan 2022	**Date of report**: 5 September 2022
Shifts: Part 1: 12 Part 2: 9	**Local contact(s)**: Edgar Eduardo Villalobos Portillo Hiram Castillo Michel	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Please note: EV-449 was undertaken in two parts as described above.

Parts 1 and 2:

Dr. Stuart Mills, Museums Victoria

Owen Missen, Monash University (co-affiliated with Museums Victoria)

Part 2 only:

Prof. Joël Brugger & Dr. Barbara Etschmann, Monash University

Aim

To study the correlation between the mineralogical, geochemical and microbial characteristics of sediments and (mineralised) corals from the Low Isles reef near Port Douglas, in the Great Barrier Reef, Queensland, Australia. We aim to characterise the interfaces between different calcium carbonate and oxalate minerals and also analyse trace element signatures in the corals, particularly phosphorus and potassium.

Experimental

Our experiment encompassed the measurement of standards of calcium (Ca) carbonates and oxalates (calcite, aragonite and whewellite in different crystallographic orientations) to then correlate these standards with the mineralogical compositions of the corals.

Calcium was analysed at the *K*-edge (4038 eV) for X-ray absorption spectrscopy (XAS), and all elements from magnesium to calcium were analysed in the X-ray fluorescence (XRF) maps. The samples benefited from the use of high energy XAS for analysis of trace elements. Linear combination fitting of processed data has been employed to analyse the mineralogical composition of the corals.

Our thanks to beamline scientists Eduardo and Hiram for their tireless efforts to do all on-site tasks as this was a remote beamtime due to the pandemic. Without them a successful beamtime would have been impossible.

General Observations

- Calcium carbonates undergo transformations to calcium oxalates in mineralised corals.
- Differences between phosphorus and potassium hot-spots from bleached and non-bleached corals shows that contamination from fertilizer run-off from land-based agriculture results in changes to the trace element signatures in coral.

Impact

This study is one of the first to use a minerlogical lens to analyse transformations in the corals of the Great Barrier Reef. Most studies use ecological or climatic indicators only.

We submitted an abstract to the *GeoMinKöln* conference 2022: Villalobos Portillo, E.E., Missen, O.P., Castillo Michel, H. & Mills, S.J. Great Barrier Reef nanoscale carbonate-oxalate mineralogy: pollution and climate change impacts on the coral reef ecosystem.

We intend to publish the results from this experiment following further data analysis and incorporation of additional mineralogical and potentially climatic information, linking mineralogy with the direct impacts of changing climate on corals.

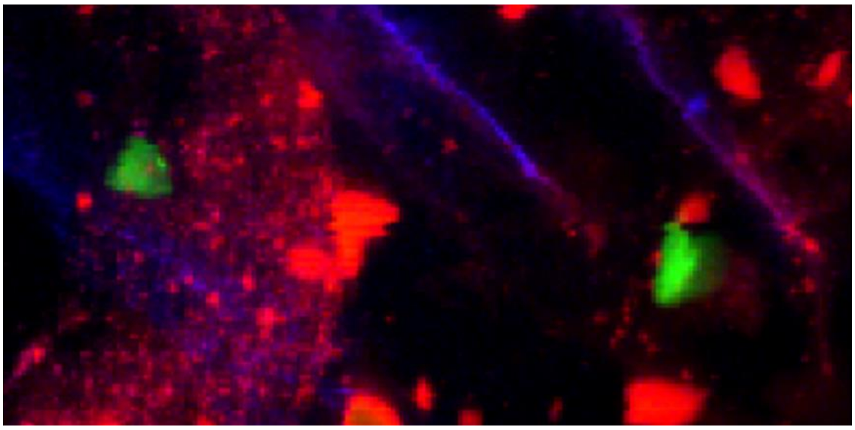

Figure 1: Calcium-Potassium-Phosphorus (RGB map, listed elements in red, green and blue respectively) developed from an XRF map. Phosphorus shows streaking (blue lines, perhaps cavities in the coral filled by P-rich fluids) while the potassium (green) forms hot-spots separate from the calcium. The porous structure of the coral is shown by the varying distribution intensiyu of calcium (red).

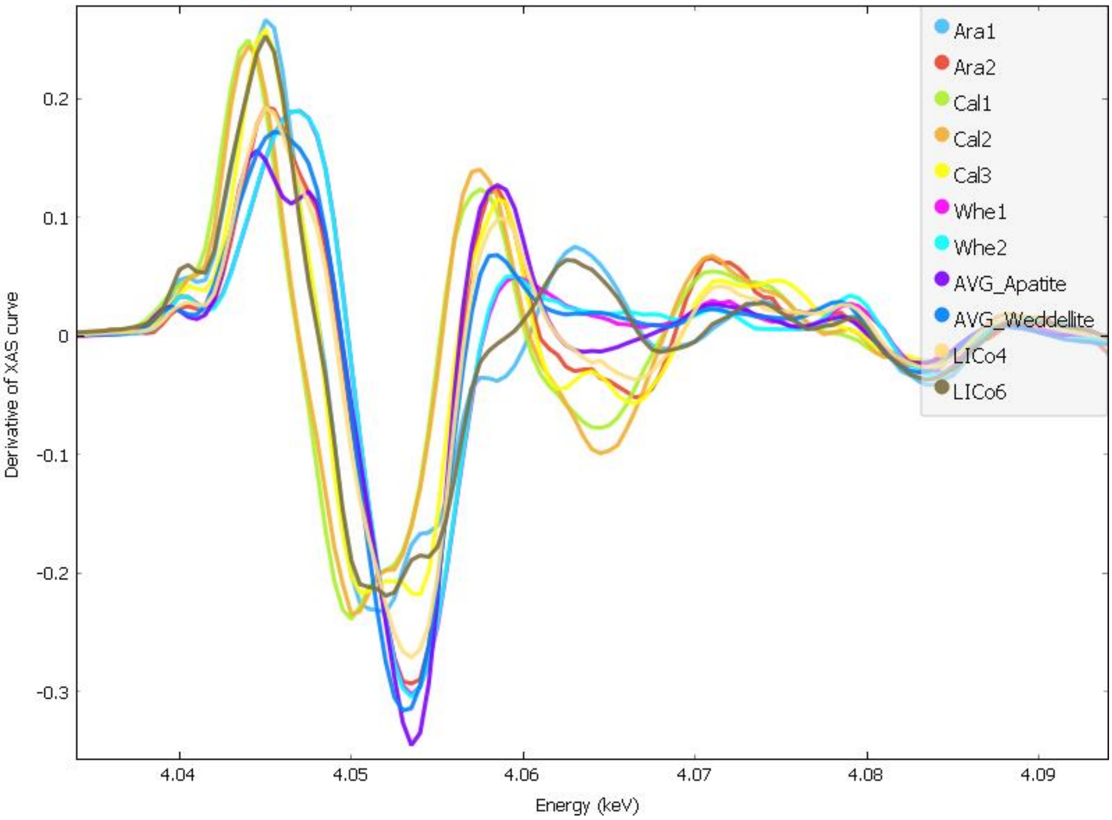

Figure 2: Comparison of first derivatives of averaged XAS spectra, showing that the local minimum is shifted to the right for the oxalate spectra. Mineralised Low Isles Coral samples LICo4 nd LICo6 fall between the standard spectra of pure carbonate and oxalate spectra, with LICo4 having mainly oxalate character, and LICo6 carbonate character.