![ESRF]	**Experiment title:** Micro-XRF geochemical characterisation of speleothem layers at/near hiatuses: a novel approach to the investigation of abrupt climate changes	**Experiment number**: EC-807
Beamline: ID-22	**Date of experiment**: from: 28/04/11 08:00 to: 03/05/11 08:00	**Date of report**:
Shifts: 15	**Local contact(s):** Remi TUCOULOU	*Received at ESRF:*

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Report:

1. **General remarks: details concerning time loss** (see also the Evaluation Form):

In the 16 bunch mode the acquisitions had to be bracketed between refills because the I0_flux normalization didn't correct completely the fluctuations of the flux during the refill. Because of this, in the 6-hour time between refills it wasn't possible to acquire long high-resolution maps. We also lost several hours the first day due to the initial utilization of a Molibdenum detector that induced a false Mo-peak it all the spectra.
The major problem was related to the map acquisitions that SYSTEMATICALLY CRASHED after some tens of lines (between 10 and 60). This forced us to run and/or re-run several parts of the same maps up to 5-6 times with a considerable loss of time. To avoid the problem we also run some maps in the mesh mode (instead of the continuous acquisition) but this almost doubled the acquisition times due to the dead time related to the motor movements between successive points.
Another cause of time loss was related to the necessity of re-create the correct mounting angle and the coordinate system of a specific sample every time we shifted from one sample to the other. This is because the sample holders are attached to the goniometric head with a screw and the positioning of the sample at 45° with respect to the incoming beam had to be judge approximately and successively carefully checked with the focus system on the entire surface of the samples.
For all these reasons several scheduled samples or intervals in the samples couldn't be analysed also because the initial 18 shift allocated to the experiment had to be reduced to 15 due to the tight schedule of ID22.

2. Sample description and results (analyzed at 21 KeV).

Sample Null 370_FM13 (Nullarbor - Australia).

The map depicts an area characterized by high Sr, Br, U and Rb sandwiched between two layers with much lower concentration in these elements. The feature is that of a complex crystal termination area strongly affected by diagenetic modifications that caused the partial loss of the original growth morphology.
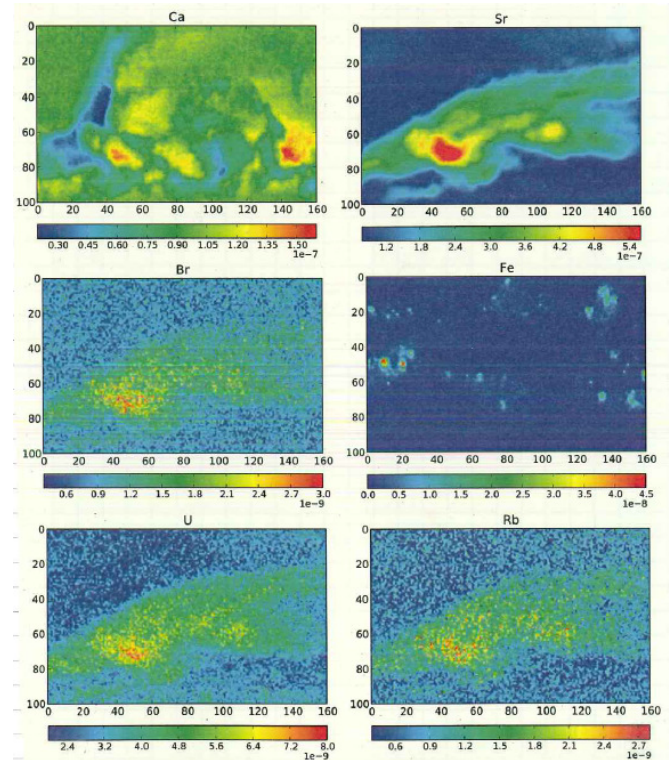

Map Null 370_FM13-2: h= 320 μm; v= 200 μm, pixel size= 2 μm, dwell time 0.8s.

Sample Sv7-Em2 (Savi Cave, Northern Italy) and sample Sp1 (Spiller Cave, Northern Italy).

The elemental concentration of the more interesting elements (Sr, Br, Fe) were very low (i.e. long acquisition times) and the fluctuations in the line-scans quite small and muted. For this reason, and being the time lost discussed above, we decide not to proceed in the analysis.

### Sample Julia (Nullarbor - Australia).

In this freatic sample the final map obtained is a collage of 2 successive parts. On the map it's possible to observe the final growth stage of a single calcite crystal characterized by high U and Sr concentrated especially in the upper part of the crystal and along the crystal boundaries, and low Br and Fe content. The successive growth phase, possibly following a hiatus, is characterized by low Sr and U, high Br and relatively high Fe. The shape of the discontinuity is rounded and rugged suggesting a possible dissolution phase.
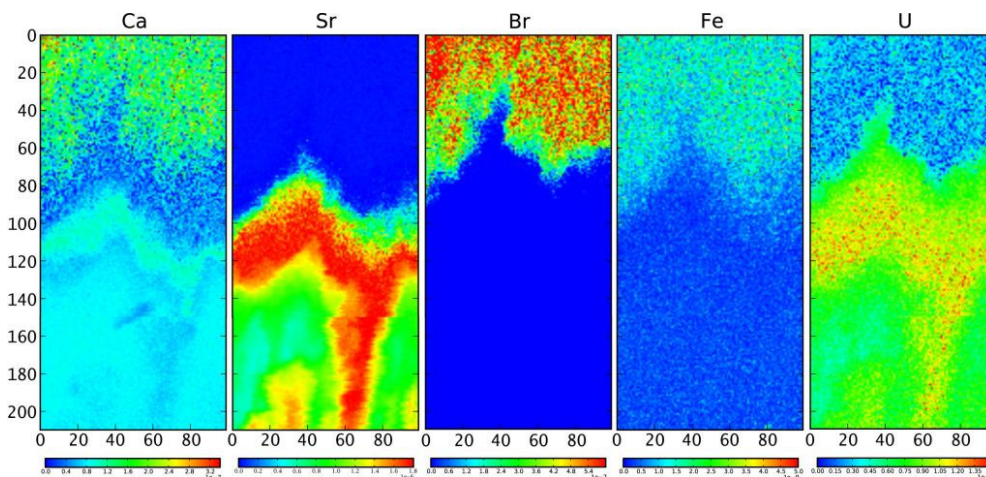
Map Julia #7 and #10 combined: h= 200 μm; v= 420 μm, pixel size= 2 μm, dwell time 0.8s.
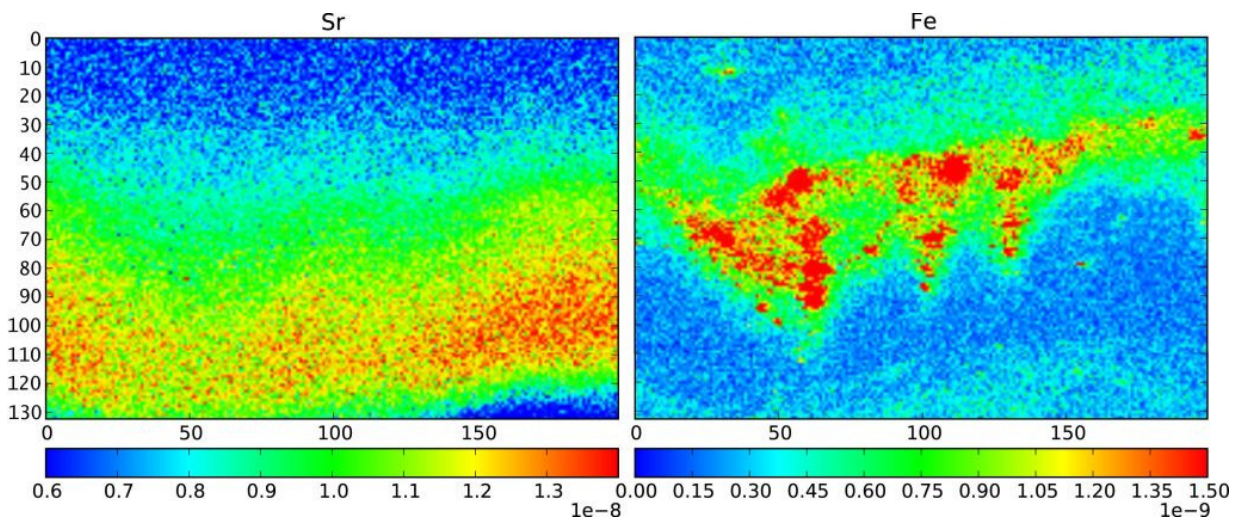
Sample Null 645_MO1t (Nullarbor - Australia).

The map obtained is a collage of 4 successive aborted maps. On the map it's possible to observe the final growth stage of a single calcite crystal characterized by high Fe and Sr and low Br content. The successive growth phase, possibly following an hiatus, is characterized by low Fe, progressively decreasing Sr and relatively high Br content. The shape of the discontinuity is rounded and rugged suggesting a possible dissolution phase.
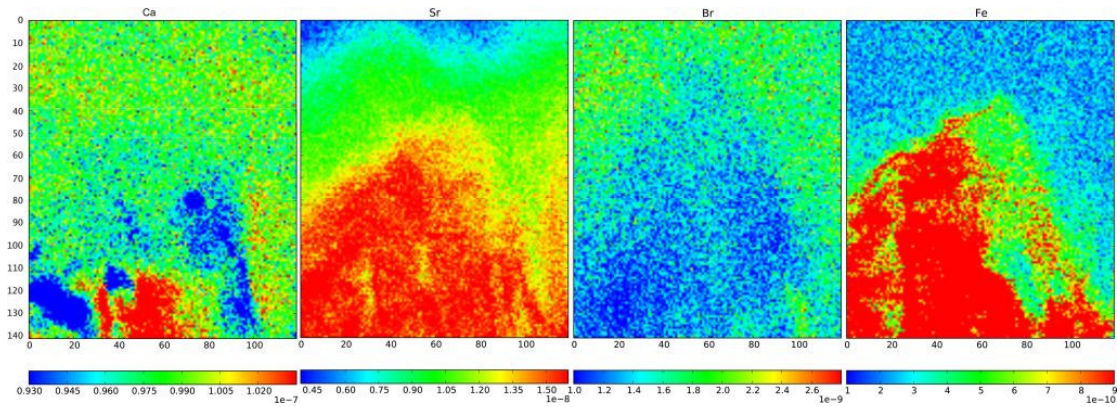
Map Null 645_MO1t #1 to #4 combined: h= 240 μm; v= 300 μm, pixel size= 2 μm, dwell time 0.8s.

Sample Mol-7 (Moline Cave, Northern Italy).

Several attempts were made to obtain a good quality map from this calcite stalagmite, focusing along a hiatus of several thousand years. The final map below depicts a quite intriguing and articulated situation with two possible hiatuses that braked a short phase of growth characterized by high Sr and low Fe. Fe is particularly concentrated along the hiatuses and marks the rounded terminations of the crystals. The higher Fe concentration is recorded after the hiatus inside the troughs at the top of the crystal boundaries.

Map MOL7-9: h= 400 μm; v= 270 μm, pixel size= 2 μm, dwell time 0.6s.

Map MOL7-10: h= 600 μm; v= 390 μm, pixel size= 3 μm, dwell time 0.6s.

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