

## 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>

### ***Reports supporting requests for additional beam time***

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

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.

### **Deadlines for submission of Experimental Reports**

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

### **Instructions for preparing your Report**

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



	<b>Experiment title:</b> High spatial resolution mapping of elemental distributions within calcrete secondary carbonate archives to improve dating and palaeoclimate reconstruction	<b>Experiment number:</b> ES 348
<b>Beamline:</b>	<b>Date of experiment:</b> from: 22/09/2016 to: 25/09/2016	<b>Date of report:</b>
<b>Shifts:</b>	<b>Local contact(s):</b> Bernhard Hesse	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants</b> (* indicates experimentalists): Dr Kathryn Adamson* - Senior Lecturer Dr Leon Clarke – Senior Lecturer Both at: School of Science and the Environment Manchester Metropolitan University Manchester M1 5GD		

### Report:

Experiment ES 348 allowed us to successfully analyse five samples of secondary carbonate (calcrete) from Montenegro. Such material is typically used to date geological sequences through uranium series (U-series) analysis. The samples had previously been bulk U-series dated and span an age range of 350,000 years to 65,000 years BP. The beamline data effectively identified different phases of formation within the calcretes, and allowed us to explore their elemental composition. In particular, we established the dominant detrital components of the different formation phases, and were able to explore the distribution of uranium across the samples. Understanding the way that uranium is stored within the calcrete crystal matrix is important for our ability to effectively target individual crystal phases for accurate uranium-series dating. This is especially significant because bulk U-series dating is standard procedure, but it may be obscuring considerable variation in the timing of calcrete formation phases, and/or incorporating detritally contaminated crystals.

Having established the elemental content and distribution of the sample, we have recently applied for, and been successfully granted, a bid to the UK Natural Environment Research Council (NERC) isotope laboratory (NIGL) for high-resolution, in-situ U-series analysis of the calcrete samples, so that the ages of the individual formation phases can be compared with their elemental composition. The ESRF data was a key factor in the success of the proposal, and will allow us to progress with the research with a view to analyse a larger dataset at the ESRF, and at the NIGL, in future. The applicants will arrange laboratory analysis at NIGL for 2017. The additional U-series data will allow us to target a higher profile journal for the publication of the XRF results from the ESRF. It is therefore

anticipated that the data from the ESRF will be published or, at least in review, by the end of 2017.

The applicants have also applied to the Natural Environment Research Council (NERC) ion microprobe facility, for in-situ analysis of stable carbon and oxygen isotope analysis of the samples. This will allow us to examine the atmospheric conditions at the time of calcrete formation, and tie this with the U-series ages and XRF data. It has been requested that the applicants resubmit their proposal once the U-series analysis has been completed.