



## 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 using the **Electronic Report Submission Application:**

*<http://193.49.43.2:8080/smis/servlet/UserUtils?start>*

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

Reports can now 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> The nature of uranium in shales	<b>Experiment number:</b> ES-1032
<b>Beamline:</b> BM-16	<b>Date of experiment:</b> from: 15 Feb 22 to: 21 Feb 22	<b>Date of report:</b> 31 Aug 22
<b>Shifts:</b> 15	<b>Local contact(s):</b> Olivier Proux	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants (* indicates experimentalists):</b> Joel Brugger* and Barbara Etschmann, Monash University Evgeniy Bastrakov, GeoScience Australia		

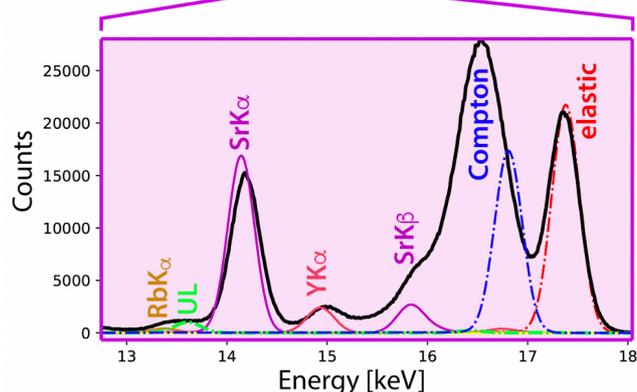
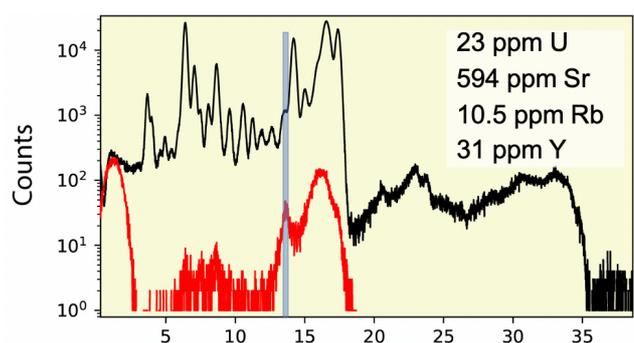
### Aim

Throughout geological history, organic-rich shales show systematic enrichment in uranium compared to other crustal rocks. Uranium concentrations vary significantly (< 5 to > 500ppm) among these shales in both time and space, as a result of global changes in the chemistry of the atmosphere and oceans through geological time, as well as local physical and chemical conditions of sedimentation and diagenetic processes. This experiment will be the first to use high resolution XANES spectroscopy to characterize U speciation in marine sediments. The aim of the study is to check the hypothesis that sorption rather than simple reduction might control the primary correlation between organic carbon content and U in shales.

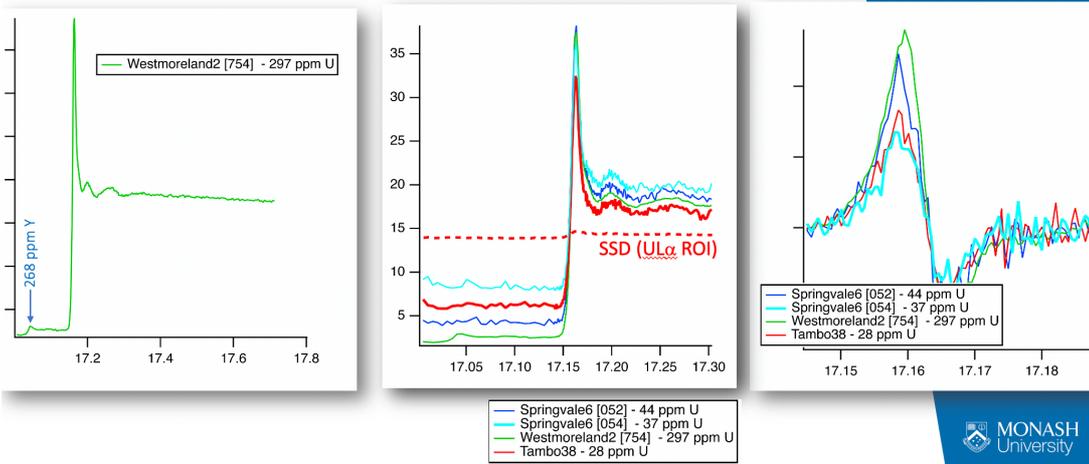
### Experimental

Ambient data for 15 samples were collected at the U L<sub>3</sub>-edge (17166 eV) at the BM-16 (FAME-UHD) beamline. As these samples had a maximum U concentration of ~300 ppm, the data benefitted from the use of the high resolution XAS measurements.

The XANES data has been processed (fitting peaks/edge to determine U<sup>4+</sup>/U<sup>6+</sup> ratio). Complementary SEM imaging and nano-sims data have been collected.

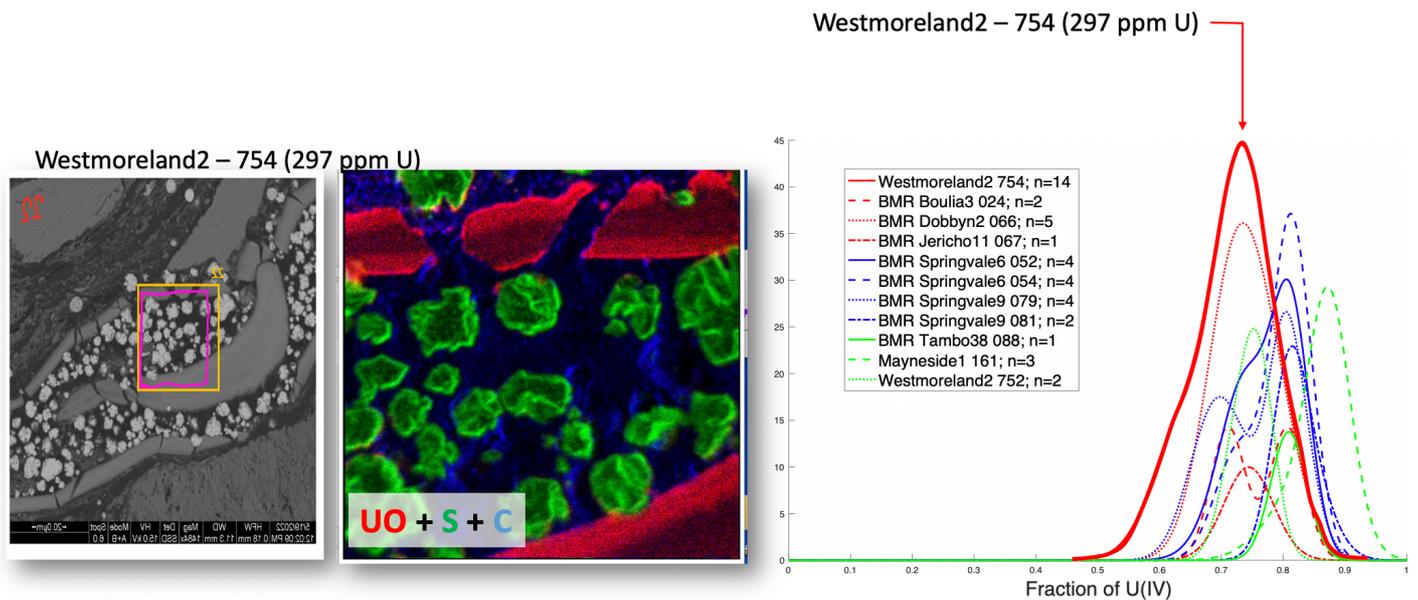


# HERFD-XAS UL3 data on shales



## General observations

1. Most samples were quite reduced, however 3 of them had ~50%  $U^{6+}$ .
2. SEM was used to determine areas of interest, ie where U could possible occur in these samples.
3. These areas were mapped with nano-sims



## Impact

- The results were presented at XAFS2022 as an invited talk [Brugger, J., Etschmann, B., Bazarkina, E.F., Proux, O., Testemale, D.; Bastrakov, E. Liu W. *High-Energy Resolution Fluorescence Detected X-Ray Absorption Spectroscopy (HERFD-XAS): Perspective from a New User and Exciting Times Ahead!* XAFS2022, 18<sup>th</sup> International Conference on X-Ray Absorption and Fine Structure, 10-15 July 2022, Sydney, Australia.]
- More nano-sims data will be collected and then we aim to write a paper.