Experiment Report Form

The double page inside this form is to be filled in for each experiment at the Rossendorf Beamline (ROBL). This double-page report will be reduced to a one page, A4 format, to be published in the Bi-Annual Report of the beamline. The report may also be published on the Web-pages of the HZDR. If necessary, you may ask for an appropriate delay between report submission and publication.

Should you wish to make more general comments on the experiment, enclose these on a separate sheet, and send both the Report and comments to the ROBL team.

Published papers

All users must give proper credit to ROBL staff members and the ESRF facilities used for achieving the results being published. Further, users are obliged to send to ROBL the complete reference and abstract of papers published in peer-reviewed media.

Deadlines for submission of Experimental Report

Reports shall be submitted not later than 6 month after the experiment.

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 / experiment 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.
- bear in mind that the double-page report will be reduced to 71% of its original size, A4 format. A type-face such as ”Times” or ”Arial” , 14 points, with a 1.5 line spacing between lines for the text produces a report which can be read easily.

Note that requests for further beam time must always be accompanied by a report on previous measurements.

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HZDR HELMHOLTZ ZENTRUM DRESDEN ROSSENDORF ROBL-CRG	**Experiment title:** Structural characterisation of uranyl aluminate nanoparticles	**Experiment number**: 20-01 741
Beamline: BM 20	**Date of experiment**: from: 21.06.2014 to: 23.06.2014	**Date of report**:
Shifts: 6	**Local contact(s)**: Andreas Scheinost	*Received at ROBL:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Ultrasonically assisted hydrolytic precipitation of U(VI) in the presence of mesoporous alumina followed by thermal treatment of solid precursor enabled the formation of crystallized uranyl aluminate (URAL) nanoparticles (NPs) dispersed in alumina matrix. Effect of U(VI) concentration and calcination temperature on the yield of URAL NPs was studied using XRD, XAFS and HRTEM techniques. At 800°C, URAL NPS (d≈5 nm) are formed only for low uranium loading of about 5 wt% whereas for higher content of uranium, larger U_3O_8 NPs (d≈20 nm) were identified as a principal uranium specie. At 500°C, URAL NPs are formed even for 25 wt% of uranium. U L_{III} edge EXAFS spectra pointed out that uranyl cation in URAL is coordinated by bidentate aluminate groups (Figure 1). Presumably URAL is formed during the heating of $2UO_3{\cdot}NH_3{\cdot}2H_2O$/AlO(OH) precursor. However, high temperature and larger content of uranium promote URAL transformation to more thermodynamically stable U_3O_8. This

process is accompanied by uranium NPs growing via Ostwald ripening mechanism as it shown in Figure 2. These results have been published in the article:

Chave T., Le Goff X., Scheinost A. C., Nikitenko S. I. Insights into the structure and thermal stability of uranyl aluminate nanoparticles, New J. Chem. 2017, DOI: 10.1039/c6nj02948e.

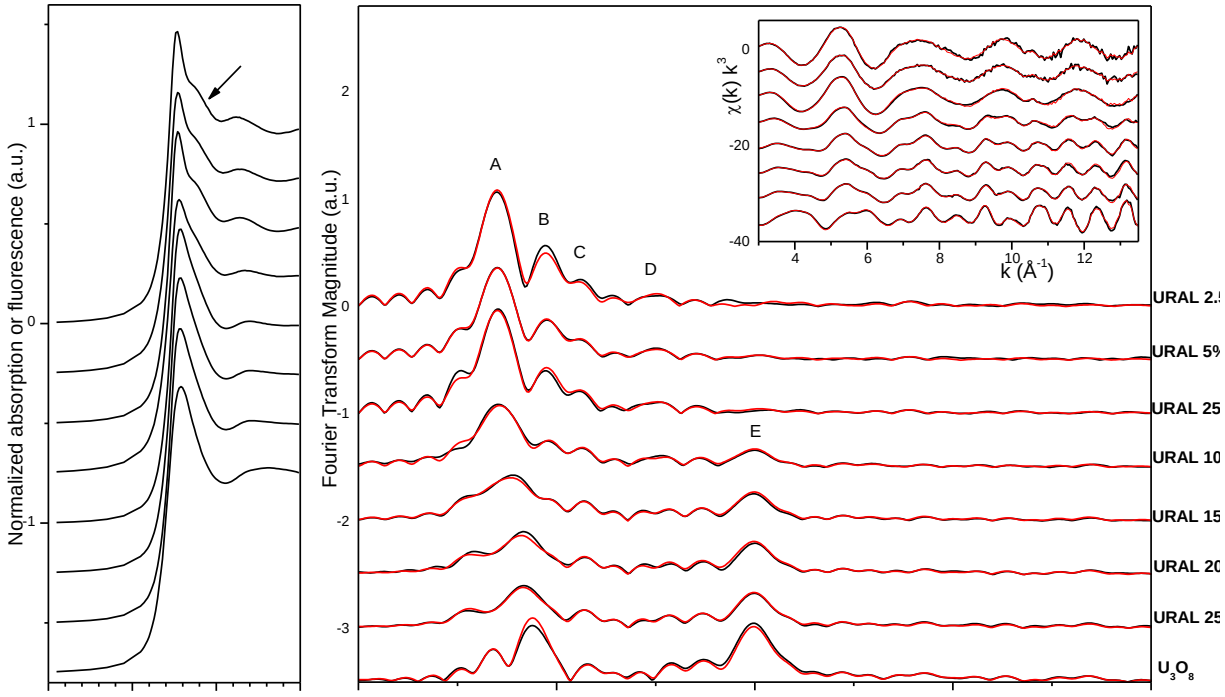

Figure 1. U-L_{III} edge XANES (left) and the Fourier transforms uncorrected for the phase shift (right) of the k^3 weighted EXAFS spectra of URAL samples shown in the inset. Black lines are experimental data, red lines are reconstructed EXAFS data using two principal components.

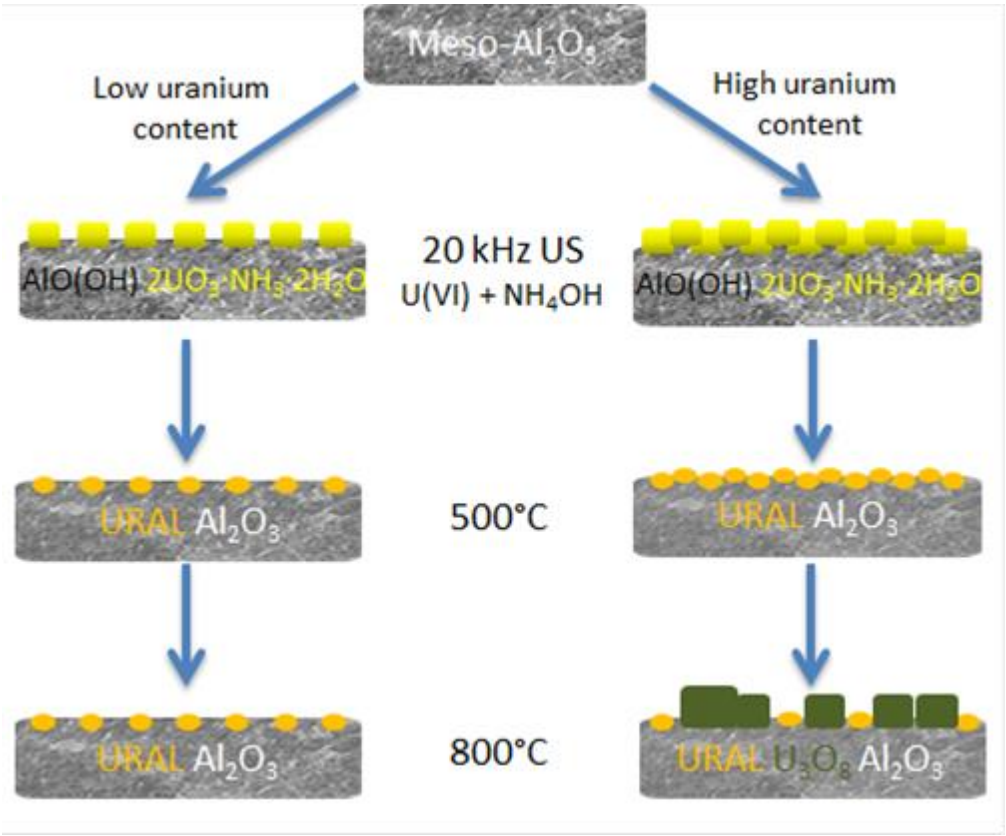

Figure 8. Schematic representation of the URAL formation mechanism.