# 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: <a href="https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do">https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do</a>

### **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

- ➤ 1<sup>st</sup> March Proposal Round 5<sup>th</sup> March
- ➤ 10<sup>th</sup> September Proposal Round 13<sup>th</sup> 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 <u>each project</u> 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.

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# **Experiment title:**

Sorption and Reduction of Plutonium in the Environment: Effects of Mineral Complexity

**number**: A20-1-850

**Experiment** 

Beamline: Date of experiment: Date of report:

BM20 from: 04 October 2022 to: 12 October 2022 11 January 2023

Shifts: Local contact(s): Received at ESRF:

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Names and affiliations of applicants (\* indicates experimentalists):

Prof. Amy Hixon (University of Notre Dame, USA)

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#### Report:

We report the successful measurements of plutonium (Pu) at the M<sub>4</sub>-edge in HERFD-XANES mode at beamline BM20. The samples contained ~1000 ppm of Pu sorbed to the surfaces of iron oxide minerals: hematite, goethite, and magnetite. The mineral substrates comprised aluminium-doped minerals, minerals with decreased crystallite sizes, and "reference" minerals that were more chemically pure and with crystal sizes in the micrometre range. These experiments were carried out to determine the effects of mineral complexity. impurities (Al-substitution) and particle size (nanoscale crystallites), on sorption and redox behaviour of Pu. Pu was introduced to the mineral aqueous suspensions as Pu(V) in the laboratories at HZDR. The successful measurements revealed that in some cases, Pu is present in more than one oxidation state from a qualitative comparison with reference compounds of known oxidation states. In essence, our results suggest that some samples contained a mixture of Pu(IV) and Pu(V). Picture on the left below shows a qualitative comparison of a Pu + mineral sample and two reference compounds, suggesting contributions from more than oxidation state of Pu. Picture on the right below shows a preliminary (not final) quantitative assessment of contributions from Pu(IV) and Pu(V) likely to be present in the sample. Data analysis in ongoing for a quantitative determination of the oxidation states present in the samples and their fractions. We also report the successful measurements of Pu at the L<sub>3</sub>-edge in HERFD-XANES mode at beamline BM20. M<sub>4</sub> and L<sub>3</sub>-edge measurements will be compared to determine potential correlations between the M and L edges with respect to oxidation state and/or speciation of Pu.



