



## 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>

### 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 from 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 each project 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.



	<b>Experiment title:</b> Monitoring degradation of hybrid nanostructures in situ using X-ray photon-correlation spectroscopy	<b>Experiment number:</b> SC-5389
<b>Beamline:</b> ID02	<b>Date of experiment:</b> from: 14.04.2023 to: 17.04.2023	<b>Date of report:</b> 17.07.2023
<b>Shifts:</b> 9	<b>Local contact(s):</b> William Chevremont	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants</b> (* indicates experimentalists):  <b>Dageförde N., University of Hamburg</b> <b>Otange B., University of Hamburg</b> <b>Otto F., University of Hamburg</b> <b>Schulz F., University of Hamburg</b>		

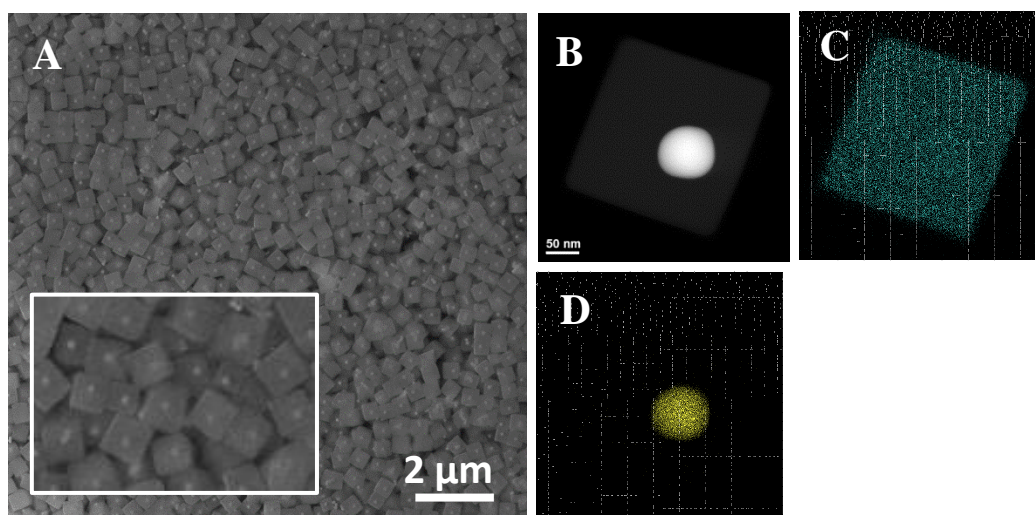
## Report:

The degradation and dissolution of a hybrid nanoparticle was observed in regard of their chemical and colloidal stability in different aquatic environments for potential use in drug delivery applications. The particle system consisted of a Zeolitic Imidazolate Framework-8 shell as a porous metal organic framework with potential drug loading capability. Encapsulated inside were gold nanoparticle cores as tracers for successfully dissolved particles. The resulting hybrid particle is shown in Figure 1.

In order to monitor the particle dynamics including the hydrodynamic radius and changes in the crystal structure X-ray photon correlation spectroscopy (XPCS) measurements combined with wide-angle X-ray scattering (WAXS) measurements were used. The hybrid nanoparticles were exposed to the following aquatic solutions: H<sub>2</sub>O, Dulbecco's Modified Eagle Medium, phosphate buffered saline 1x, different HCl concentrations (0.1%, 0.05%, 0.01%, 0.001% (pH=4), 0.0001% (pH=5)) and different Ethylenediaminetetraacetic acid concentrations (0.5 M, 10 mM, 1 mM).

Overall, the particles showed high longtime stability in H<sub>2</sub>O and in the tested mild HCl concentrations in regards of colloidal and chemical stability. In EDTA, PBS and DMEM the particles tend to aggregate before the dissolution of the ZIF-8 shell takes place. This showed in an unchanged intensity of the ZIF-8 WAXS signal combined with a decrease of the diffusion constant. After some time those samples lost their diffusive behavior but their crystal structure remained intact.

Under harsh degradation conditions with 0.5 M EDTA a decreasing WAXS signal intensity of the ZIF-8 crystal structure was determined before the colloidal stability was lost and the particles' diffusive behavior was lost. However, the remaining gold nanoparticle cores were not colloidal stable in solution and formed aggregates which lost their diffusive behavior. Therefore the biological application does not seem reasonable with this hybrid particle.



*Figure 1: Characterization of the examined hybrid particle. A) scanning electron microscopy image showcasing the shape and size control. B) Transmission electron microscopy image of a single hybrid particle. C) and D) Energy dispersive X-ray spectroscopy of a single hybrid particle Zinc signal in C) and Gold signal in D).*

### **Experimental Setup:**

For both, WAXS and SAXS experiments, X-rays were monochromatized to a photon energy of 12.2 keV. For WAXS measurements the Rayonix LX 170HS detector was used and for XPCS measurements the Eiger500K detector was used. WAXS was measured in a distance of 15 m, covering the  $q$ -range of 7 to  $41 \text{ nm}^{-1}$ . The XPCS detector was at the maximum distance of 31 m and covered the  $q$ -range from 0.003 to  $0.03 \text{ nm}^{-1}$ . All experiments were performed at room temperature using a 1 mm capillary. In order to avoid local beam damage of the sample, the sample was moved in between measurements. The XPCS measurements were performed in 8-bit mode, taking 10000 frames at 10 kHz with an exposure time of  $7 \times 10^{-5}$  s each. Additional slower XPCS measurements were taken in 16-bit mode with an exposure time of 0.002 s at 250 Hz. WAXS and complementary SAXS measurements with an exposure time of 1 s were taken in 32-bit mode.

Samples were synthesized locally and washed with water to remove any excess reactants. They were then transferred into their respective medium by centrifugation and redispersion in the final degradation medium. The particle concentration is estimated to be roughly  $0.5 \text{ nm}^{-1}$ .  $10 \mu\text{L}$  of the sample is then transferred to a capillary and measured. Thereby changes in the samples happening within the first minutes could not be monitored completely due to the time it takes to place the capillary, clear the room and start the experiment.