



## 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> Operando XAS study of Cu-In nanoparticles as catalysts for electrochemical CO <sub>2</sub> reduction	<b>Experiment number:</b> CH-6621
<b>Beamline:</b> BM31	<b>Date of experiment:</b> from: 07.06.23 to: 12.06.23	<b>Date of report:</b> 07.09.23
<b>Shifts:</b> 14	<b>Local contact(s):</b> Dragos Stoian	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants</b> (* indicates experimentalists): Valery Okatenko <sup>1</sup> * Dr. Mark A. Newton <sup>1</sup> * Gaetan Ramona <sup>1</sup> * Dr. Dragos Stoian <sup>2</sup> * Prof. Raffaella Buonsanti <sup>1</sup>  <sup>1</sup> Laboratory of Nanochemistry for Energy Research, Institute of Chemical Sciences and Engineering, Ecole Polytechnique Fédérale de Lausanne, Sion, CH-1950, Switzerland <sup>2</sup> Swiss-Norwegian Beamlines, European Synchrotron Radiation Facility, 38000 Grenoble, France		

## Report:

Understanding metal surface reconstruction during operation is of the uttermost importance in heterogeneous catalysis as it directly affects the available active sites. However, surface reconstruction is notoriously difficult to study because of the dynamic nature of the phenomena behind it. Here, we report on the mechanism and the intermediates, which drive the rearrangement of copper catalysts during the electrochemical CO<sub>2</sub> reduction reaction. In-situ methods, including mass spectrometry and fluorescence spectroscopy, evidence a dissolution – redeposition process mediated by transient species containing copper in +1 oxidation state. Theory identifies copper-adsorbate complexes which form in solution under operating conditions. Copper carbonyls and oxalates emerge as the major reaction-specific species driving copper reconstruction. This work motivates future studies to specifically target these compounds to improve the catalyst operational stability in the electrochemical CO<sub>2</sub> reduction reaction.

The preprint of this work is available as:

Jan Vavra, Gaetan Ramona, Federico Dattila, Attila Kormányos, Tatiana Priamushk, Petru Albertini, Anna Loiudice, Serhiy Cherevko, Núria Lopéz, Raffaella Buonsanti. Cu<sup>+</sup> transient species mediate Cu catalyst reconstruction during CO<sub>2</sub> electroreduction. DOI: [10.26434/chemrxiv-2022-3cr9k-v2](https://doi.org/10.26434/chemrxiv-2022-3cr9k-v2).

The paper is submitted to Nature Catalysis and is currently under revision.