EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

# 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://wwws.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 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** |
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- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th 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.

| ESRF | **Experiment title:** Operando XAS insights on Cu-Ga nanoparticles regeneration in electrochemical CO2 reduction | **Experiment number**: CH-6130 |
|---|---|---|
| **Beamline**: BM31 | **Date of experiment**: from: 02.10.21 to: 05.10.21 | **Date of report**: 09.06.22 |
| **Shifts:** 9 | **Local contact(s)**: Dragos Stoian | *Received at ESRF:* |


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

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**Report:**

Liquid metals (LMs) have been used in electrochemistry since the 19th century, but it is only recently that they have emerged as electrocatalysts with unique properties, such as inherent resistance to coke poisoning, which derives from the dynamic nature of their surface. The use of LM nanoparticles (NPs) as electrocatalysts is highly desirable to enhance any surface-related phenomena. However, LM NPs are expected to rapidly coalesce, similarly to liquid drops, which makes their implementation in electrocatalysis hard to envision. Herein, we demonstrate that liquid Ga NPs (18 nm, 26 nm, 39 nm) drive the electrochemical $CO_2$ reduction reaction ($CO_2$RR) while remaining well-separated from each other. CO is generated with a maximum faradaic efficiency of around 30% at −0.7 $V_{RHE}$, which is similar to that of bulk Ga. The combination of electrochemical, microscopic, and spectroscopic techniques, including *operando* X-ray absorption, indicates that the native oxide skin of the Ga NPs is still present during $CO_2$RR and provides a barrier to coalescence during operation. This discovery provides an avenue for future development of Ga-based LM NPs as a new class of electrocatalysts.

The work is published as:


Valery Okatenko, Laia Castilla-Amorós, Dragos Constantin Stoian, Jan Vávra, Anna Loiudice, and Raffaella Buonsanti. The Native Oxide Skin of Liquid Metal Ga Nanoparticles Prevents Their Rapid Coalescence during Electrocatalysis. *J. Am. Chem. Soc.* 2022, 144, 22, 10053–10063.