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:
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

- 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:** Investigation of visible-light-promoted photoredox catalysis of lanthanide complex with pump-probe x-ray liquidography	**Experiment number**: CH-6315
Beamline: ID09	**Date of experiment**: from: 2022.3.28 to: 2022.4.3	**Date of report**: 2023.9.11
Shifts: 18	**Local contact(s)**: Alix VOLTE	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

(Notes: The originally allocated beamtime under proposal number CH-6315 was planned for an experiment on the photoredox reaction of the europium complex. However, at that time, the synthesis of the europium complex was not completed on schedule. So, we asked for permission from the beamline manager and experimented with the wavelength-dependent photodissociation dynamics of CF_2I_2, originally planned as a backup sample.)

The goal of our experiment was to study the photodissociation pathways of diiododifluoromethane (CF_2I_2) and how they depend on excitation wavelength. In previous spectroscopic studies, it has been known that CF_2I_2 exhibits two-body, three-body, or a mixture of both photodissociation pathways depending on the excitation wavelength. (**Figure 1**) Depending on the photodissociation pathway, CF_2I_2 can release one or both iodine atoms, and it is anticipated that different intermediates will be captured in these two pathways.

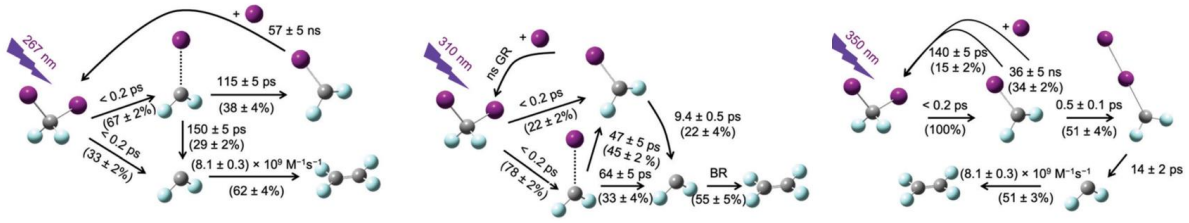

Figure 1. Reported photodissociation pathways and their time constants for each excitation wavelength revealed by previous time-resolved IR spectroscopy studies.

In this work, we utilized time-resolved X-ray solution scattering (TRXSS), a prominent tool to visualize the structural dynamics in liquids and solutions, to precisely extract the structure of each intermediate. We conducted the TRXSS measurement of 30 mM CF_2I_2 @ cyclohexane in three different wavelengths: 267, 310,

and 350 nm. The X-ray scattering signals from heated solvents were measured in a separate experiment using ferrocene solution, a well-known heating dye.

We azimuthally integrate the scattering images to obtain a set of static 1D curves that have momentum (q) as the x-axis. Then, we subtracted the curves collected at positive time delays from those at negative time delays to generate difference x-ray scattering curves. Also, we removed the contribution of the solvent heating signal by using structural components extracted from the separate experiment using ferrocene solution. Finally, we performed a singular value decomposition (SVD) to decompose the collected data $\Delta S(q,t)$ into time-independent structural components ($U(q)$), their temporal evolutions ($V(t)$) and relative importance in the data (S).

The experimental results of the TRXSS data of CF_2I_2 at 267, 310, and 350 nm in cyclohexane (**Figure 2-3**) were summarized. We concluded that structure components are similar regardless of the excitation wavelength but different in their temporal evolution. Further analysis is required to clarify the wavelength-dependent photodissociation pathways and time profile variations. Details of the analysis are still ongoing, and we plan to publish the results in a scientific paper in the future.

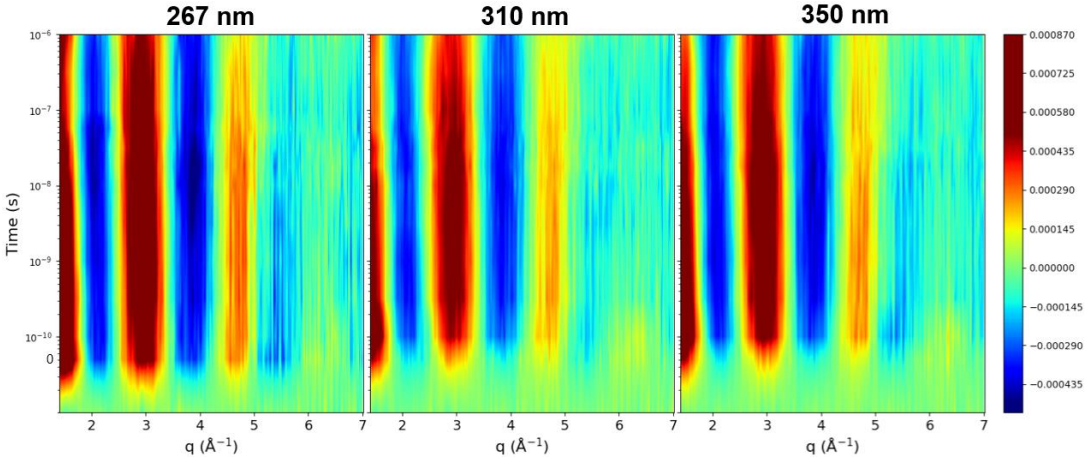

Figure 2. Time-resolved X-ray scattering signals obtained from CF_2I_2 @ cyclohexane using excitation wavelengths of 267, 310, and 350 nm, respectively. The contribution of the solvent heating signal was removed.

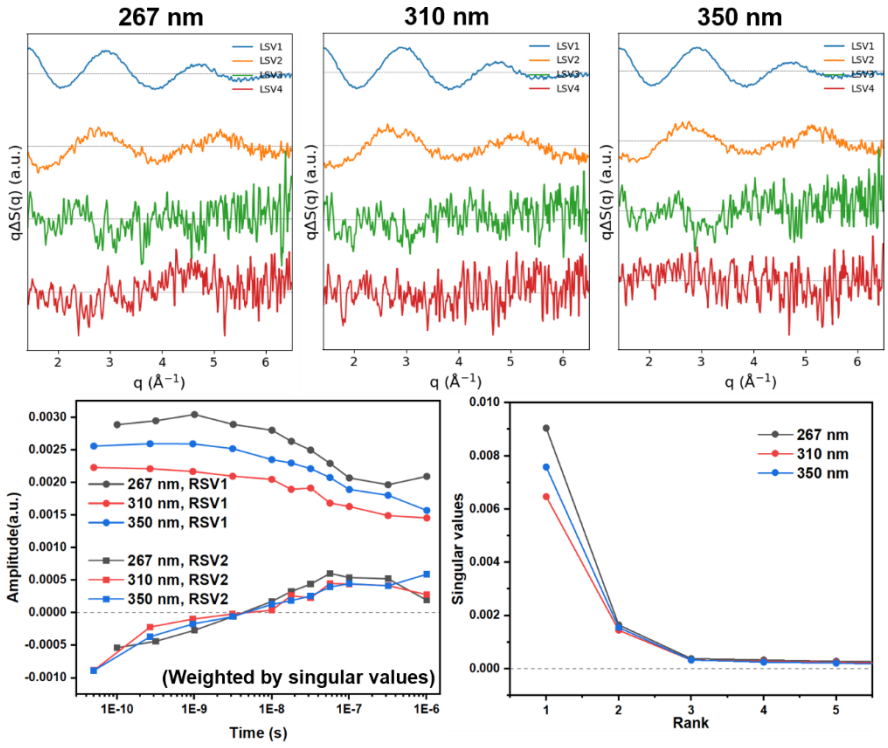

Figure 3. SVD results of data which is shown in **Figure 2.**