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

- 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:** Anomalous dispersion values from diffraction data at the L edges of gold compounds	**Experiment number:** MI-1445
Beamline: BM-20	**Date of experiment:** from: 07 March 2023 to: 13 March 2023	**Date of report:** 09 Sep 2023
Shifts: 18	**Local contact(s):** Christoph Hennig, Volodymyr Svitlyk	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

In this experiment, we extended our endeavors to refine the anomalous dispersion correction values from single-crystal X-ray diffraction (SC-XRD) experiments and validate them using X-ray near edge absorption spectroscopy (XANES) and extended X-ray absorption fine structure (EXAFS) spectroscopy, to molecular gold compounds. Fig. 1 shows the systematic series of structural motifs investigated during this beamtime.
The aim was to determine the influence of different halogens attached to the gold metal as well as multiple halogens attached to gold in different oxidation states is visible in the refined anomalous dispersion parameters. To investigate this, the recorded X-ray diffraction (XRD) data at several energies around the absorption edges for each compound as well as X-ray absorption spectroscopy (XAS) measurements to compare our refined values for anomalous dispersion parameters against.

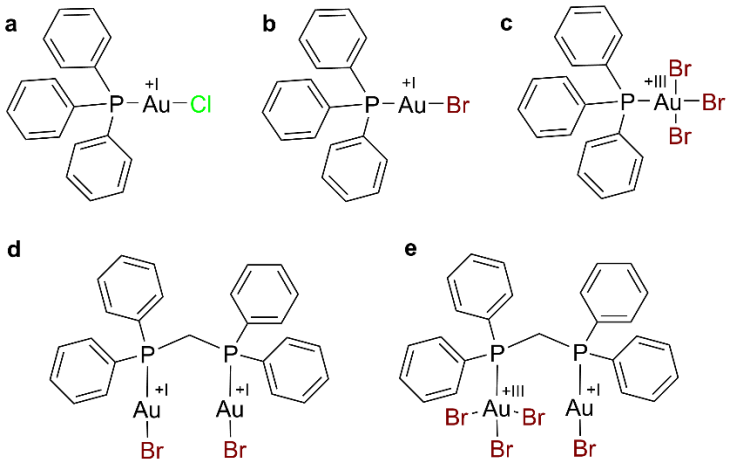
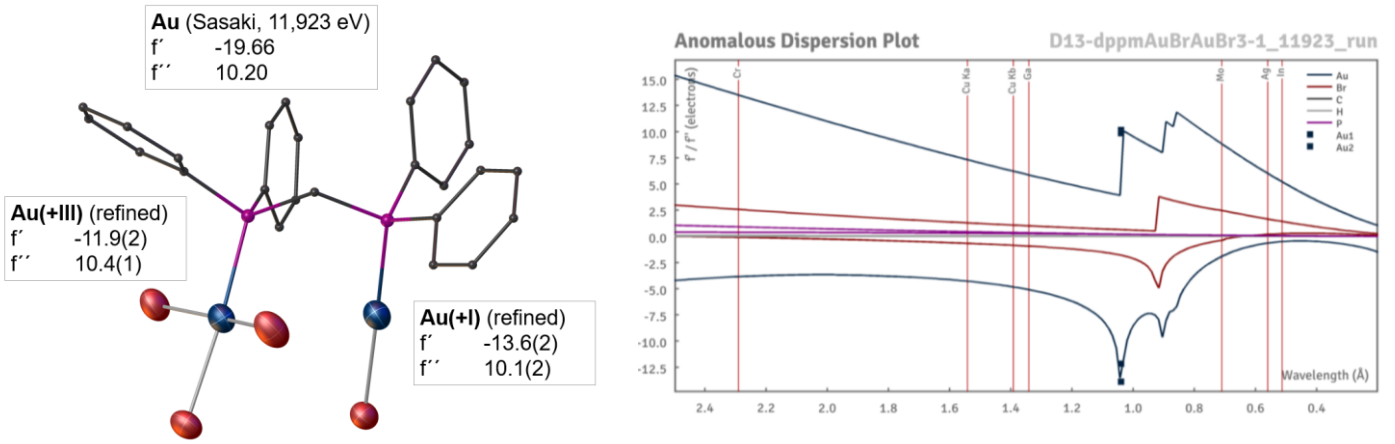


Figure 1: Compounds investigated using our established anomalous dispersion refinement in *Olex2*.[1]

While the full evaluation of the in total five XAS spectra and 45 XRD data sets is still ongoing, there are promising preliminary results.
Especially compound **e** of Fig. 1 is of special interest: Bivalent positions cannot easily be distinguished by X-ray absorption spectroscopic methods as the received signal consists of a superposition of both metal positions. This is not the case for the individual refinement of anomalous dispersion parameters in the XRD experiment, as both sites contribute differently to different reflections in the diffraction patter. In this case, both gold positions show a significant difference of 1.7 *e* for *f″*. The values for both Au positions differ strongly from the tabulated values by Sasaki (Fig. 2 a) which further supports our efforts to refine these values in the close vicinity of absorption edges.[2]
This could help to improve the oxidation state determination of bi- or multivalent compounds in small molecule crystallography. For protein crystallography, this method has been established by Einsle *et al.* and has been used with great effect.[3]
Further examples are needed to validate this approach, as small molecular chemistry is has a facetted range of different compounds with various elements of interest.



a **b**

Figure 2: Comparison of the refined dispersion values for the two bivalent gold positions in DPPM(Au[I]Br)(Au[III]Br_3) compared to the tabulated values according to Sasaki shown with a structure representation (**a**, H-atoms omitted for clarity) as well as the tabulated progression of anomalous dispersion values with X-ray wavelength (**b**).

Literature:

[1] F. Meurer, F. Kleemiss, O. Dolomanov, N. Peyerimmhoff, H. Puschmann, M. Bodensteiner, *IUCrJ* **2022**, *9*, 604.
[2] S. Sasaki, *KEK Report* **1989**, *88*, 1.
[3] O. Einsle, et al., *J. Am. Chem. Soc.*, **2007**, *129*, 2210; T. Spatzal et al., *Nat. Commun.*, **2016**, *7*, 10902.