

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 using the **Electronic Report Submission Application:**

<http://193.49.43.2:8080/smis/servlet/UserUtils?start>

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

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.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal 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.



	Experiment title: Combined diffraction and XANES investigation of photoinduced structural changes	Experiment number: 01-02-840
Beamline: BM01A	Date of experiment: from: 29/10/08 to: 2/11/09	Date of report: 26 February 2009
Shifts: 9	Local contact(s): Phil Pattison	<i>Received at ESRF:</i>

Names and affiliations of applicants (* indicates experimentalists):

Gervais Chapuis, Laboratoire de Cristallographie, Ecole Polytechnique Federale de Lausanne

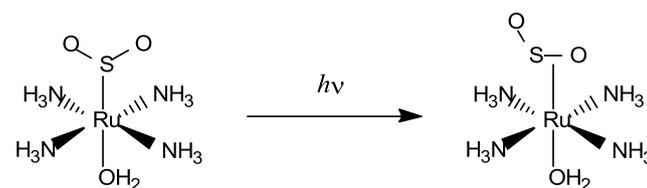
*Jacqueline Cole, Cavendish Laboratory, University of Cambridge

*Thierry d'Almeida, Cavendish Laboratory, University of Cambridge

*Anthony Phillips, Cavendish Laboratory, University of Cambridge

Report:

The aim of this experiment was to exploit anomalous X-ray scattering to draw out high Bragg intensity contrast in a difference experiment where small structural changes arise from photoexcitation. We focus on photoinduced structural changes of a metastable light-activated complex, $[\text{Ru}(\text{NH}_3)_4(\text{H}_2\text{O})(\text{SO}_2)](\text{tosylate})_2$, where the SO_2 ligand photoisomerises from η^1 (end-bound) to η^2 (side-bound) bonding to the Ru metal. (See Scheme 1).



Scheme 1: Photoisomerism of the SO_2 ligand

We performed full structure collections on both the ground state and photoexcited structures, each both using standard 0.7 \AA radiation and at the Ru K edge to capture the anomalous X-ray scattering. We also measured the XANES spectra of each structure *in situ*.

Preliminary data analysis indicates that the experiment was a great success. The photoinduced η^2 state is clearly identifiable (Fig. 1). Moreover, the high-resolution data obtained are of sufficient quality for charge-density analysis (Fig. 2).

Analysis of the AXS data and XANES spectra is ongoing. The latter display the anisotropy expected from single-crystal analysis; to assist in interpreting these, we are employing DFT modelling using the CASTEP code. Preliminary results show good agreement for this system, as well as for reference data on metal phthalocyanates (which are standard model compounds for XANES modelling) obtained from Phil Pattison.

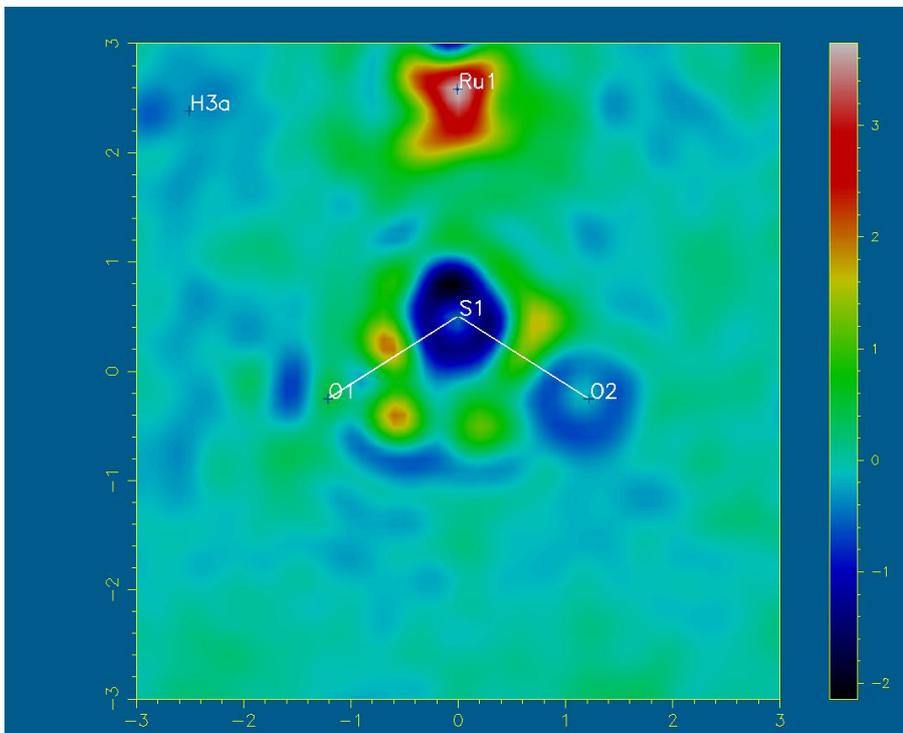


Figure 1

Photo-difference map showing a model of the ground state (in wireframe) applied to data from the photo-excited state. The deep holes around the ground-state S and O atoms indicate that these have moved in a significant fraction of the unit cells, and the small peaks nearby show two new positions for S1 and one for O2.

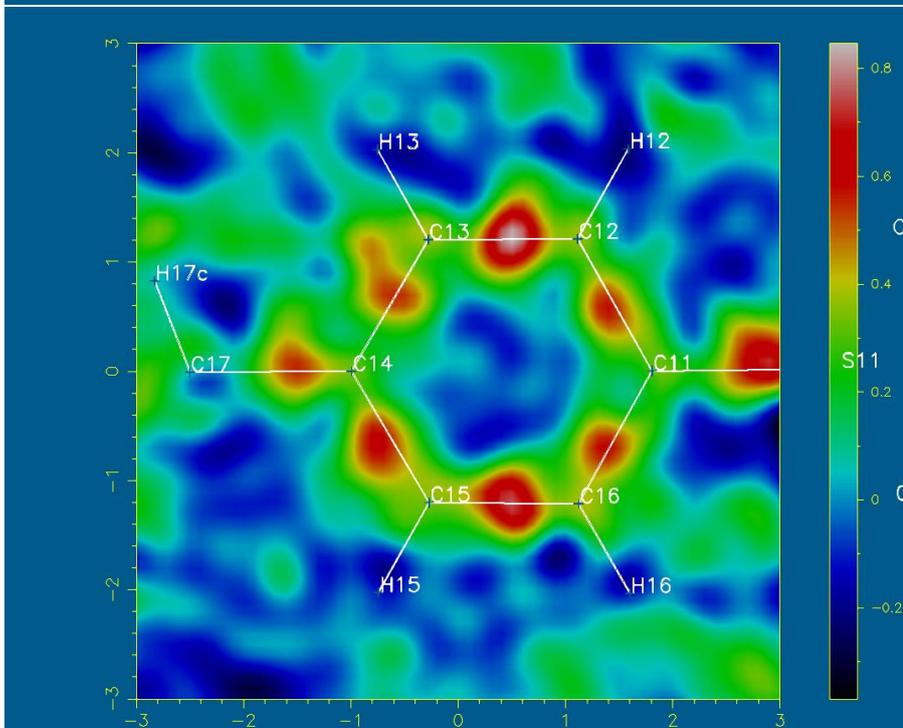


Figure 2

Difference map of the ground state, showing a spherical-atom model of the sulfonate counterion (in wireframe). This ion does not participate in the photoexcitation. The quality of the data is indicated by the peaks at the bond-critical points, demonstrating that these data can support more sophisticated charge-density models. Note the relatively small colour scale compared to Fig. 1.