

## 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.



	<b>Experiment title:</b> <b>Picosecond structural dynamics studies of the photo-dissociation &amp; geminate recombination of methylene iodide in solution</b>	<b>Experiment number:</b> CH 2168
<b>Beamline:</b> ID09B	<b>Date of experiment:</b> from: 23 <sup>rd</sup> Sept 2006 to: 26 <sup>th</sup> Sept 2006	<b>Date of report:</b>
<b>Shifts:</b> 9	<b>Local contact(s):</b> M. Wulff, Q. Kong	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants</b> (* indicates experimentalists):  <b>Jan Davidsson</b> (*) Department of Photochemistry and Molecular Science, Uppsala university, Box 523, S-7123 Uppsala, Sweden  <b>Richard Neutze</b> (*) Department of Chemistry and Bioscience, Chalmers University of Technology, Box 462, 40530 Gothenburg, Sweden.		

## Report:

Firstly we would like to note that the experiment consisted of only 9 shifts not the 12 which were allocated. This at the request of the beam line manager, and due to heavy demand of beam time.

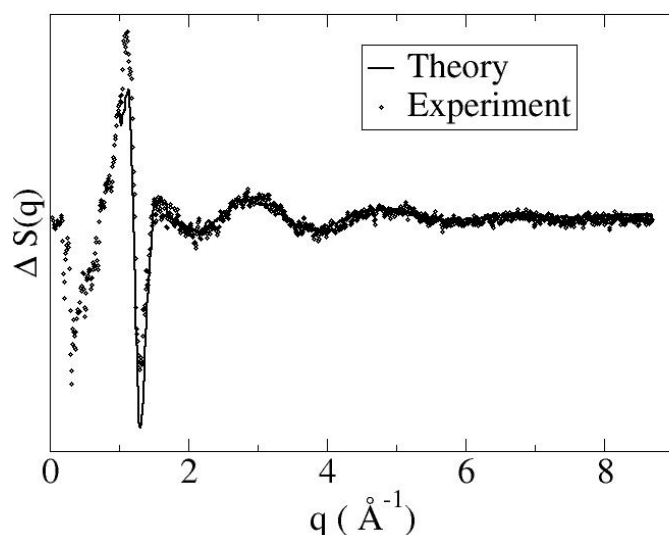
After taking approx 13 hours to set up the experiments the first measurements taken were of CH<sub>2</sub>IBr in methanol. Unfortunately we soon realised that the laser was too tightly focused resulting in hot spots being formed, and the data collected was not useful.

After the laser was defocused, and the system to be studied changed to CH<sub>2</sub>I<sub>2</sub> in cyclohexane. A large amount of data was collected. Unfortunately when further analysis of this data was performed after leaving the ESRF there was found to be significant signal in some of the -200ps control data (i.e. when the laser excitation occurs after the x-ray measurements). So approximately 1/3 of this data had to be discarded.

The next measurements taken were using CH<sub>2</sub>IBr in cyclohexane. Several pauses to allow the chopper to cool were required, and some of the data had to be rejected as the difference spectrum was clearly incorrect. The final measurements taken in the final shift were of CH<sub>2</sub>IBr in methanol, unfortunately due to frequent chopper overheating (requiring frequent and lengthy pauses in collection) and a low signal no useful data was collected.

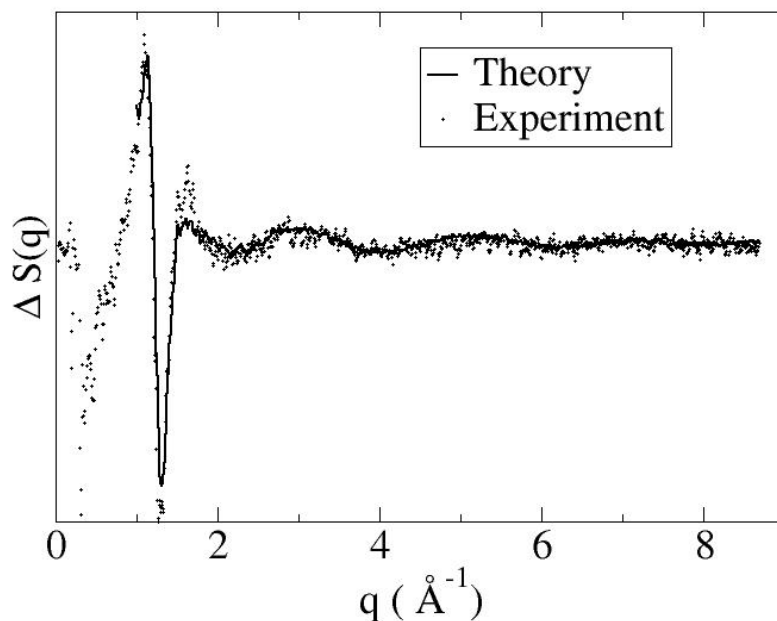
The CH<sub>2</sub>I<sub>2</sub> results look promising, and a good fit between the theory and experiment can be made (Figure 1). Unfortunately even using stretched pulses the largest signal is still from the expansion of the cyclohexane, making the analysis more difficult. The results show that the initial state consists of a mixture of CH<sub>2</sub>I<sub>2</sub>, CH<sub>2</sub>I-I and I, as would be expected from previous work [1]. In addition the long time delay data contains evidence of the formation of the predicted [2] end product C<sub>2</sub>H<sub>4</sub>I<sub>2</sub>. In addition we do not see any evidence of I<sub>3</sub><sup>-</sup>. This result is expected in a non-polar solvent. From the analysis of the data to date we feel that the rate of formation of I<sub>2</sub> from I can be seen clearly as well. The rate of the CH<sub>2</sub>I-I → CH<sub>2</sub>I<sub>2</sub> reaction is more difficult to see, and extra measurements refining the existing data with extra time points and lower noise to allow the

concentrations to be followed more closely in time would be helpful. Additional time points with longer delays would also allow the rate of formation of  $\text{C}_2\text{H}_4\text{I}_2$  to be more clearly seen.



**Figure 1 ( $\text{CH}_2\text{I}_2$  in Cyclohexane at 500 ps)**

The results for  $\text{CH}_2\text{IBr}$  in cyclohexane also look promising, and again we see a very good agreement between the theory and experiment (Figure 2). The analysis to date shows that, as expected, initially  $\text{CH}_2\text{Br-I}$  and  $\text{CH}_2\text{Br}$  are formed, and we also see evidence of the  $\text{C}_2\text{H}_4\text{Br}_2$  end product at long times, and very clearly that the I has all disappeared at long times, and again  $\text{I}_3^-$  is not formed as we have a non-polar solvent. Further measurements at the ESRF to see more clearly the rate of formation of  $\text{CH}_2\text{I-Br}$  from  $\text{CH}_2\text{Br-I}$  and the rate of formation of  $\text{C}_2\text{H}_4\text{Br}_2$ , and to improve the signal to noise ratio of the measurements would be very helpful.



**Figure 2 ( $\text{CH}_2\text{IBr}$  in Cyclohexane at 500 ps)**

## References

- [1] J. Davidsson et. al. Phys. Rev. Lett 94, 245503 (2005)
- [2] A. N. Tarnovsky et. al. J. Phys. Chem. A 108, 237 (2004)

