



	Experiment title: XSTRIP - A new detector for time-resolved EXAFS	Experiment number: MI-552
Beamline:	Date of experiment: from: 25 th Oct 2001 to: 30 th Oct 2001	Date of report: 1 st Mar 2002
Shifts: 15	Local contact(s): Sofia Diaz Moreno and Sakura Pascarelli	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Dr Andrew Dent,^a Prof John Evans,^{b*} Mr Jon Headspith^{a*} Dr Trevor Rayment,^{c*} Mr Guiseppe Salvini^{a*}		
^a CLRC Daresbury Laboratory, Warrington, UK ^b Dept of Chemistry, University of Southampton, UK ^c Dept of Physical Chemistry, University of Cambridge, UK		

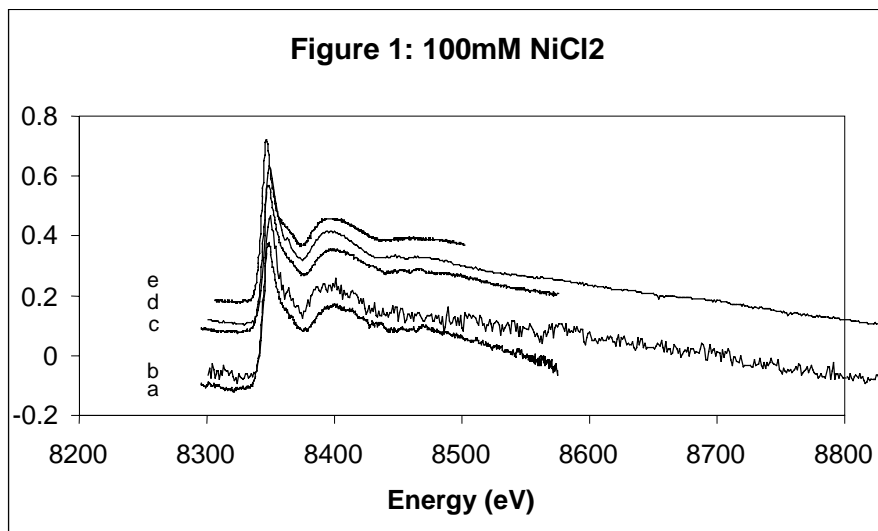
Report:

A visit was first made in August to discuss the hardware and software requirements to integrate the detector into SPEC and the ID24 data acquisition system. We are very grateful to Marie-Christine Dominguez for rewriting many of the data acquisition macros to accommodate XSTRIP. The detector was then set-up on the beamline during the October shutdown, so that it only had to be aligned at the start of the first beamtime.

The first two days were spent aligning and checking the operation of the detector and we are pleased to report that the detector performed extremely well, typically taking 35 μ s to saturate with a 60mA 16 bunch beam. In order to compare with the current ID24 CCD detector and data already collected at Daresbury using XSTRIP a 100mM NiCl₂ solution was measured and representative data are shown in Figure 1.

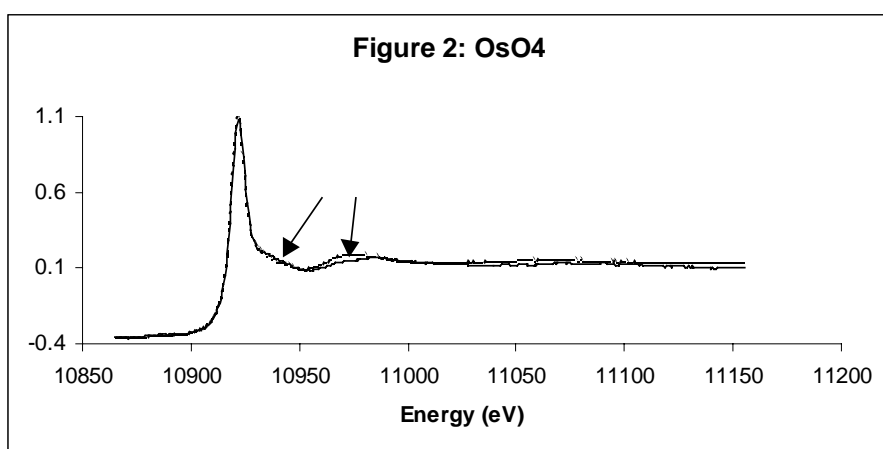
The lower two spectra (a and b) show how the increased flux at the ESRF has massively improved the data quality for a single accumulation of approximately 200 μ s. However we have been puzzled by the discovery that in summing spectra to obtain better signal to noise, non-linearities present on ID24 do not normalise out. It cannot clearly be seen with this size of diagram, but in fact spectrum c which has a total data collection time of 65ms shows significantly worse non-linearities than spectrum d collected at Daresbury in 20ms.

These features do however normalise out with the present ID24 CCD shown in spectrum e (100ms exposure), although the real time taken for these spectra is close to 3 seconds due to the delays in reading out the CCD.



We then proceeded to setup the ID24 stopped flow. It was important to choose a system that had been previously studied on ID24 to achieve a proper comparison between XSTRIP and the Princeton CCD detector. The reaction chosen was the *cis*-dihydroxylation of 2,3-dimethylbut-2-ene by OsO₄, with or without an amine promoter (pyridine or quinuclidine); this is an important reagent in synthetic organic chemistry [3], and the chemical aim was to identify the structures of the osmium species ion solution. The timescales of these reactions are between 0.01 and 2 seconds. The advantage of XSTRIP was immediately obvious, in that its flexible data acquisition system and

virtually unlimited data storage meant that the whole experiment was simpler to setup and far more efficient in the use of beam time and chemicals; the whole reaction could be studied in 1 shot, rather than having to repeat a reaction several times with different delays between the stopped-flow and CCD. Figure 2 shows the starting and end point spectra from a run of 200 data sets of 30 x 400 μ s scans (12ms per dataset); the whole run thus taking 2.4s.



Summary

The data showed that XSTRIP could operate with the full flux of an ESRF undulator. The Os data clearly showed the advantages of the efficiency of operation of XSTRIP since many time resolved data sets could be collected without having to pause for readout - a prohibitive factor with the CCD. However, there are issues over non-statistical effects which need to be investigated further. We wish to thank the ESRF for provision of beamtime.

References

[1] Criegee, R., *Justus Liebigs Ann Chem*, **1936**, 522, 75; Sharpless, K.B., *et al*, *Chem Rev*, **1994**, 94, 2483; Corey, E.J., *et al*, *J Am Chem Soc*, **1993**, 115, 12579.