The EPSRC National Crystallography Service – A Valuable Extension of your Home Laboratory

S.J. Coles[a], P.N. Horton[a], M.B. Hursthouse[a], L. Male[a], R.A. Stephenson[a], W. Clegg[b], R.W. Harrington[b], L. Russo[b].

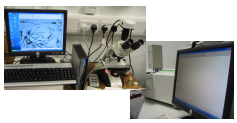
[a]School of Chemistry, University of Southampton, UK.;
[b]School of Natural Sciences, University of Newcastle upon Tyne, UK.

The UK National Crystallography Service (NCS) is an amalgamation of resources at two centres; Southampton Chemical Crystallography Group and Daresbury Synchrotron Radiation Source (Newcastle component). This service, acting as a centre of expertise, provides state-of-the-art facilities and knowledge in the technique of small-molecule crystal structure determination and is available free to all UK academics eligible to apply to the EPSRC Chemistry programme. Throughout its 25 year existence the service has pioneered development of the technique and provided innovations that have enabled structural science in the UK to flourish.
The Service offers 'data collection only' or 'full structure determination' opportunities and specialises in handling extremely demanding samples and datasets. It also offers specialist experiments, such as charge density determinations, high- and variable-temperature data collections and multiple collections to elucidate phase transitions and structure-property relationships.
The experience and expertise of the National Service staff is passed on to UK postgraduate and postdoctoral workers at our biennial Advanced Skills Workshop (held in alternate years to the Durham BCA School). This provides insights into the handling of difficult samples, both in the home laboratory and at the synchrotron.
We have just started a three year period of funding that will run until late 2009, ensuring the continued provision of these facilities for some time to come.

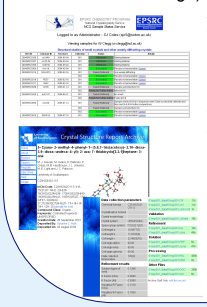
Southampton Latest News

We are proud to announce the appointment of two new members of staff. Dr. Louise Male has joined us as a PDRA and will begin the extension of our activities into the area of structure solution from powder diffraction data.
Dr. Richard Stephenson is now our Systems Administrator and will be developing the e-Science services we offer.

The renewed funding included an upgrade of our diffraction equipment and we now have an APEXII detector coupled with our high flux source. Additionally, we have replaced our liquid nitrogen cryogenic system with a pair of Oxford Cryosystems Cobra© devices.



A new component of our services, as a result of renewal funding, is the provision of a solid state characterisation facility. We can now offer Differential Scanning Calorimetry (Mettler Toledo 821e DSC) and Hot Stage Microscopy (Mettler Toledo FP90 Hot Stage).



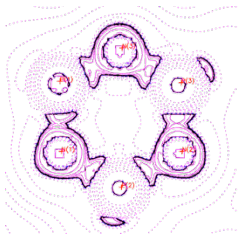
As part of our e-Science initiative we now offer a sample tracking service. All users are issued with a digital certificate that allows them to monitor the progress of their samples through our system. The sample handling process is automatically updated at various points during the experiment and this can be securely viewed at any time.

The National Crystallography Service is pioneering a new approach to the publication of crystal structure data. Our e-Crystals archive is capable of openly making available all the digital information generated during the course of a structure determination. This innovation has led us to review our publication policy, which can be viewed on our website

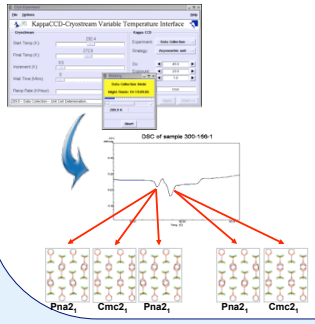
Non Routine Experiments

Our services include specialist experiments, including multiple collections to elucidate phase transitions and structure-property relationships, high- and variable-temperature data collections and charge density determinations.

Our state-of-the-art equipment allows us to rapidly collect very accurate high resolution intensity data on the most demanding samples. By special arrangement we offer data collection and advice on the determination of electron density distributions in the solid state. We have recently gained funding to investigate the process of bond formation by charge density analysis and solid state ^{17}O NMR (in collaboration with Nottingham Trent and Warwick Universities).



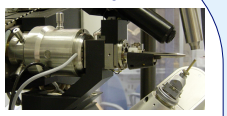
Typical Laplacian of the electron density distribution in a cyclophosphazene



Data collection in the temperature range 80-500K not only provides excellent data routinely, but also enables us to access previously uncharacterised phases. Additionally, custom built variable temperature data collection software allows us to fully characterise phase transitions by combining temperature ramping with data collection in an automated manner. The study to the left comprises 40 data collections at different temperatures and was completed in under 24 hours.

Weakly Diffracting & Hard to Handle Crystals

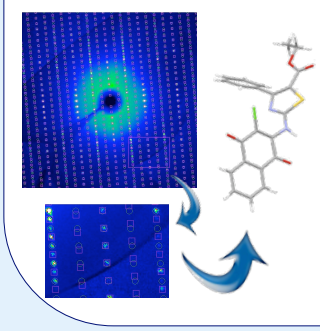
A rotating anode equipped with the world's first focusing optics for molybdenum radiation provides the brightest available laboratory source dedicated to small-unit-cell structure determinations.



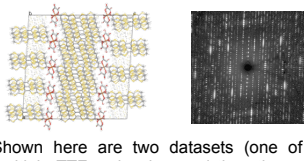
Coupled with the latest CCD technology and data processing software, this enables us to examine exceptionally small crystals (ca 10μm min dimensions) and deconvolute twinned or incommensurate datasets. This technology also enables us to derive structures originating from the most demanding areas of chemistry, such as supramolecular, nanomaterials and biological chemistry.

A typical twin refinement

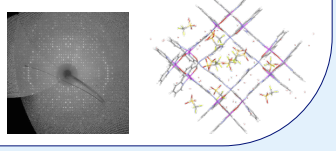
This diffraction pattern from a 1:1 twin (180° rotation about the a axis) was deconvoluted and refined to an R-factor of 8.5%



Data collection and refinement on large small molecules



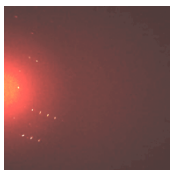
Shown here are two datasets (one of multiple TTF molecules, and the other a molecular grid) each comprising of more than 40,000 unique reflections with over 4000 parameters and refined to R-factors below 10%.

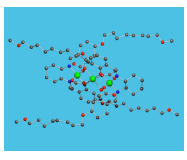


The Synchrotron Component

For those samples that are too small or weakly diffracting to provide suitable data at Southampton a linked service is provided at the Daresbury SRS. Allocations on Station 9.8 (ca. 10×3 days per annum) permit regular expert investigation of these pre-screened samples, ensuring that only the most challenging and suitable samples are run at the SRS and providing the shortest timescale for access to these oversubscribed world-leading facilities. As a result of the increased funding to 2009 Dr Luca Russo has joined the Newcastle/Daresbury team.

Can you see anything in this diffraction pattern? Weak as it is (and the diffraction was completely invisible in the home laboratory and even at Southampton), it was sufficient to generate over 13,000 reflections in a few hours for successful solution and refinement of the structure in space group *P*1. Such samples are frequently encountered in our work.





The molecular structure is shown here, with H atoms and minor disorder components (of the long chains!) omitted. This work (with Prof. Mike North at Newcastle) is part of a catalysis project. Recent SRS service publications have appeared in such high-impact journals as *Angewandte Chemie*, *Crystal Growth and Design*, *Organometallics*, *Journal of Physical Chemistry B*, and *Chemical Communications*.

It is planned that the SR service component will move to Diamond in 2008, when the SRS closes and the small-molecule beamline at Diamond becomes available.