

UK National Crystallography Service Biannual Report 2

Period covered: 01/11/2010 – 30/04/2011

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1. Preface

The contract with EPSRC was agreed and signed towards the end of the last period and we are now establishing the policies and processes that this dictates. The current period has seen the procurement and planning of the installation of the Rigaku instrument base (including the unplanned acquisition of a RAPID imaging plate system on Mo radiation, which is predominantly owned by the School of Chemistry but time can be varied between this and the rotating anodes. This instrument also provides a degree of backup and a complementary approach to the anodes). The new laboratory (instrumentation and information management system) will be fully operational in the first half of the next period and the current equipment base has performed relatively well given its age. Staffing is now up to its full strength with the appointment of Dr Claire Wilson as the PDRA based at Diamond – Claire commenced work at the start of the year.

We have now established the formal route of access to beamline I19 at Diamond, with the NCS application for a BAG (Batch Allocation Grouping), being awarded for a two-year period (Oct 2010 – Sept 2012). The process for application to access the NCS has been completely overhauled, with the introduction of a 'continuation' application, which will reduce the workload on the MAP and enable a virtual/telephone-based meeting in the future. Additionally, applications have been split into two formats – 'single research group; and 'on behalf of a consortium/department' which will enable simplified assessment and reporting procedures. The introduction of a rapid access mechanism for quick, mid-term applications has proved highly successful. The NCS publication outputs continue apace and numerous research plans are underway in the areas of systematics, charge density, data management and macromolecular crystallisation. Training and publicity events have been conducted at much the same levels as previous periods and an outreach programme has commenced.

We will present a revised (data collection only) publication policy at the MAP meeting as a discussion paper, which has been drawn up after much internal discussion and subsequent surveying of a selection of our current users.

2. Operation and Logistics

A. Overview of service use

This period has seen a healthy number of applications – both arising from the formal Call and also from the new Rapid Access route. This latter introduction has been very successful in lowering the barrier to initial access to the NCS – this type of access is awarded only once and then users are encouraged to make a full application in response to the biannual call. The number of applicants is on the rise, most probably due to ending the period of uncertainty in service provision that arose from funding issues (09-10).

Usage of the service has been commensurate with the number of applications made. We have implemented a new system of application and reporting that is aimed at streamlining these processes and better matching requests, actual demand and capacity – the 'continuation application' is concerned with assessing the use (amount and what is being done with the data) of the NCS by a particular user in a particular period and moderating accordingly for the subsequent period. We expect that this period will see a degree of acclimatisation to the new system and thereafter applications and use will be better matched and the responsible use of data considerably increased.

The proportion of samples being referred to Diamond is remaining roughly constant, but the turn around time is improving – mainly due to permanent, dedicated staffing and also to the fact that having a member of the NCS based at DLS enables us to use any unexpected, last minute spare beamtime.

B. Staff

Dr Claire Wilson started work at Diamond beamline I19 in Jan 2011. The interface between NCS Southampton and DLS has been established and Claire has settled in very well in this multi-faceted role. Frequent visits to Southampton are being made and an independent scheme of research (Charge Density) has been planned.

Dr Riccardo Montis has been appointed for 3 months to work on the macromolecular crystallisation pipeline – this has been 50% funded by the NCS and 50% by soft money and will enable the pump-priming of this support facility.

Dr Ranko Vrcelj has joined the team through a Knowledge Transfer Secondment scheme from a local company (Wallop Defence Systems Ltd) – Ranko will be looking into the assessment of crystal quality by SHG laser methods over the coming year (1 day a week).

Sarah Milsted's hours were made up to 100% (from 50%) by a related data management grant (I2S2, <http://www.ukoln.ac.uk/projects/I2S2/>) for the period Jan 2011 – March 2011 – this has allowed us to get to grips with the new application process and develop some Outreach activities.

Andrew Milsted has also been employed full time by the I2S2 project and OMII-UK (<http://www.omii.ac.uk>) for 6 months, which has enabled the design of the new sample, data and tracking system that will manage the NCS and the interface to its user-base.

There has been no alteration to the core crystallographic PDRA staffing in Southampton (Drs Horton, Tizzard & Pitak).

C. NCS Synchrotron component

The subcontracted synchrotron component at Diamond Light Source Ltd (DLS) is now in place and Claire Wilson has been appointed as the NCS team member based at and employed by DLS. She has been in post since the start of January 2011. This arrangement has allowed the NCS to make use of an extra day of standard beam time and to use some special beam condition time at the end of the last run, which cannot be scheduled as normal user time, in addition to the scheduled 5 days on I19 since the start of October 2010. As a member of both the NCS and the I19 beam line team this post provides opportunities to contribute and assist in implementing improvements to the beam line and get the best from the NCS beam time.

NCS had 5 days (each of a block 3 shifts equalling 24 hours) of scheduled time since October, with a day in November cancelled due to technical problems on the beamline which was replaced with a day in January. Since the start of the year we have been using the sample changing robot on I19 which has increased efficiency considerably, particularly reducing the time taken to thoroughly screen samples. A further 6 samples were screened during the special beam conditions.

The NCS Block allocation group (BAG) for 2-year programme mode access was successful and our next allocation of time on April 18th will be in this mode.

D. Review of user complaints/disputes and resolutions

There have been no complaints reported to either the NCS Director or Head of Service and therefore no complaint or dispute resolution has been initiated.

E. Equipment- Technical Issues

The current equipment base consists of two diffractometers (generator 13.5 years old, goniometers 13.5 and 9.5 years old, and detectors 5.5 and 9.5 years old) and nitrogen temperature cryogenic equipment (two non-liquid nitrogen cryostats 4.5 years old). As expected of equipment this age, there are likely to be numerous failures, which are not preventable by routine maintenance. The diffractometer experienced no unexpected problems and the main source of downtime was from cryogenic equipment (which was kept to a minimum due to having a spare unit). The following issues have arisen in the reporting period.

Diffractometers:

- 2 filament changes (downtime 2 days each).

Cryogenic devices:

- One Cobra unit (non-liquid N₂) had its cryodrive returned to Oxford Cryosystems for repair, requiring a new compressor. This has necessitated the temporary use of a spare Cryostream 700 (liquid N₂) while it was repaired.
- THE AD51 dry air unit was serviced by Oxford Cryosystems (this requires it to be returned to the manufacturer for about one week).
- The AD41 dry air unit had a compressor failure. As the unit is now classed as obsolete, no replacement parts could be found. The AD51 unit is able to cover for this loss, but it is therefore now running at full capacity – accordingly a

further dry air supply will be required to support the additional cryogenic systems in the new facility. We are currently investigating routes to funding a replacement.

Lab Equipment:

- New Leica microscope (M205C) arrived, providing higher magnification, clearer images, integrated image capture and image analysis.
- New X-Temp 2 'cold-mounting' device – provides a cold stream at the microscope, enabling particularly sensitive samples to be manipulated.

F. Sample Issues

Large or very long Schlenk flasks, long NMR tubes or very small, 'neck-ended' vials often contain large amount of solvent (mother liquor) and only few crystals. This makes difficult to pick the right crystal. This is especially crucial for sensitive compounds. Samples containing soft fragile crystals which grown on the bottom of vial (or on the side) where solvent has been completely evaporated. These crystals are extremely difficult to examine without causing mechanical damage. We continue to try and advise users on the best possible preparation of samples for transport and submission, and we will make this advice available on the website shortly.

The continuing problem of partially completed submission forms with sample related information missing (such as solvent/air sensitivity, possible photo reactivity, melting point of crystals) has led to incorrect storage of samples and initial crystal selections having to be aborted. In addition the empirical formula is often not provided. The new information management system will make certain fields compulsory for completion which will hopefully greatly reduce this issue.

G. Data Processing Issues

Data processing from Diamond Light Source visits has now been passed to Dr Claire Wilson, otherwise the systems and processes remain the same as before.

3. Community Activity

A. Training and Outreach

The NCS has hosted 4 visits since October. Each visit was tailored to the visiting group but each included a tour of the facilities, meeting the team and sessions with a crystallographer illustrating the technique and any special areas of interest to the group.

- Joseph Wright (Pickett Group, University of East Anglia) with 1 other member of the research group
- Lihong Li (Scott Group, Warwick University)
- Nikolaos Tsoureas (Clove Group, University of Sussex) with 2 other members of the research group
- Sergey Belyakov (Gourlay Group, Imperial College London)

The service hosted a third year undergraduate student who completed a project in outreach education in crystallography. The outcomes of the project will be worked up into materials and approaches to support workshops and competitions aimed at Key Stage 3 and above. The service also hosted a sixth form student on a work-shadowing day, and has agreed to host a 2 week work experience placement in the summer.

The NCS presented a stand at the University's Family Day – a component of Southampton's contribution to National Science and Engineering Week (around 3000 people visit this science fair every year). The event was very successful and included a

demonstration of diffraction principles (grating experiment) aimed at older children, while younger children made unit cell shapes from cardboard cut-outs. These two NCS activities intimately linked in with activities run by the School of Chemistry and colleagues from ISIS.

We have also continued to take part in University of Southampton Open Days and weekly tours of UCAS candidates.

B. Publicity

The NCS rebrand has been very successful. The website (<http://www.ncs.ac.uk>) has been developed, with additional development scheduled for 2011 as part of the revamp of the electronic infrastructure of the NCS. A second round of flyers has been distributed to heads of departments and school service managers to advertise the call. The NCS will be present on the Rigaku stand at the British Crystallographic Association Spring meeting (Keele 11th-14th April) with a banner and flyers. Further publicity activities are planned for other meetings to be held during the summer conference season.

C. User Liaison

The service has consulted users about the previous call and feedback from this has been taken into account during the production of the second call. This will continue to be the case with small changes being made to make the administration of the service easier and simpler for users.

A rapid access programme has been running allowing users to apply for access to the service between calls. The majority of these have been for one off samples, although some have been as a result of local equipment problems or failure. It is expected that these rapid access allocations will result in new users for the service both from the original rapid access users, and from increased awareness of the service. At the date of writing, the Service has received one full application from a rapid access user but a fuller picture of the success of this scheme will be available at the MAP meeting.

4. Preview of next period

A. Preview of availability over next 6 months

The next period will see the delivery and installation of the new equipment base (the RAPID is already on site). At the time of writing (early April 2011), the 007 Cu instrument is awaiting British customs clearance and it is expected that it will be delivered shortly after the Easter break. It will be possible to install this instrument in the current laboratory without any relocation of existing instrumentation and this possibility is currently being examined. We are less fortunate with the FR-E+ Mo based instrument however. Delivery of some crucial parts by Japanese suppliers to the Rigaku factory outside Tokyo is being hampered by the recent natural disaster there – events are still unfolding, but lines of communication have been established and it is currently anticipated that the instrument will be shipped to arrive in the UK around mid-late June. This date is subject to change if the situation is escalated - EPSRC and the MAP will be kept fully informed of these timescales as they unfold. Full details of the work to be carried out and the potential problems are presented in Section C below, which outlines upgrades over the coming period.

It is planned that the downtime of the facility will be less than one month. Depending on circumstances, we will call a hiatus in NCS operations – this will be no more than one month and will include diffractometer delivery, installation and commissioning at the same time as providing some time to develop new standard operating procedures and laboratory processes. Further comments on NCS availability are also included in Section C below.

B. Preview of maintenance over next 6 months

The new diffractometers will be arriving in this period. Until then there is no major service work scheduled for the current diffractometers, however the filament will need to be replaced (approximately every 2 months).

C. Preview of upgrade over next 6 months

The new equipment base will be installed during the next period. This consists of:

- Rigaku FR-E+ rotating anode Mo source with two diffractometers – one with enhanced optics and both with higher sensitivity CCD detectors.
- Rigaku 007 HomeLab rotating anode Cu source with a single diffractometer (optics and CCD detector)
- Additionally there will be a Mo sealed tube based RAPID diffractometer installed in the laboratory – whilst this is primarily a departmental diffractometer, it is complementary to the NCS equipment base and an access agreement that entails ‘swapping’ time between instruments will be put in place.

The building works required to relocate or remove existing equipment and prepare space (phase I) have been commissioned (University Estates and Facilities and Bruker) and a series of jobs have been identified thus:

- prepare services and space for relocation of powder diffractometers;
- relocate powder diffractometers;
- install new air conditioning and isolate electrics circuit for diffraction lab;
- prepare services for 007 diffractometer;
- [install and commission 007 diffractometer];
- liberate and prepare space for adjacent support lab;
- prepare services for FR-E+ diffractometer.

At this stage we may then have to wait for the delivery of the FR-E+, after which we will enter phase II of the install, which consists of:

- Decommission and remove existing FR591/KappaCCD diffractometer;
- Install and commission FR-E+ diffractometer;
- Install and commission RAPID diffractometer.

Phase I work will have little or no impact on the current operation of the NCS – in fact, if the 007 diffractometer is installed during this time, there will be an additional instrument available. This additional instrument could be crucial as we would wish to clear as much of the current backlog as possible before Phase II – to assist with this we will hire help in the form of a recently graduated PhD student from the Southampton Crystallography group. Phase II will involve some downtime and a hiatus, as described in Section A above, of no more than one month will be declared and users informed.

Appendix 1: KPI Data (All current data for 01/11/10 – 11/4/11)

	This period (01/11/10 – 11/4/11)		Last period (May-Oct 2010)	
Number of NCS Users (active)	56		39	
Number of NCS Projects	69		44	
Availability of facility for NCS use (days)	105		106	
Actual equipment uptime and use of facility by or for NCS	98		102	
Number of NCS samples processed	Total	404	Total	342
	At Southampton	342	At Southampton	273
	At Synchrotron	62	At Synchrotron	69
Number of NCS data collections performed	280		221	
Number of Full structure determinations performed	124		52	
Number of NCS samples outstanding	302		184	
Waiting for examination	17		53	
Processing	198		96	
Waiting for return to users	87 (includes a number of radioactive samples and schlenks which are awaiting special return to the user as a complete set)		35	
Number of User data sets that were completed within 1,2,3,>3 attempts	1 attempt	204	1 attempt	Unable to report against this at the time
	2 attempts	30	2 attempts	
	3 attempts	3	3 attempts	
	More than 3	6	More than 3	

	attempts		attempts	
	Unreported	107		
Number of User complaints received	0		0	
Number of NCS research outputs	73		26	
Number of NCS users of the training programme	7		8	
Number of samples classes as routine or difficult	Routine	172	Routine	Unable to report against this at the time
	Difficult	43	Difficult	
	Synchrotron	62	Synchrotron	
	Unreported	73	Unreported	

Appendix 2: Benchmark statistics

Benchmark 1

The time from arrival of a sample to logging in and informing a User of receipt will be within 2 working days for all samples

Level of samples which achieved this benchmark 98% (8 samples failed the benchmark due to a university wide network failure which meant we were unable to access our booking in system)

Benchmark 2

The time a sample is in the queue from logging in a sample to the first examination will be within 10 working days for 80% of high priority samples, within 20 working days for 80% for medium priority samples and within 30 working days for 80% of low priority samples.

Level of samples which achieved this benchmark

H-	89%
M-	93%
L-	100%

Benchmark 3

The time a sample is in the queue from examination and first communication of the outcome will be provided within 5 working days for 80% of all samples

Level of samples which achieved this benchmark 83%

Benchmark 4

The time between the first communication of the outcome of a data collection and the provision of a final result will be within 5 working days for 80% of all samples.

Level of samples which achieved this benchmark 77%

Appendix 3: Publications List

A more complete list of publications will be available at the MAP Meeting

Arising from use of the Southampton Facility

Inorganic Chemistry Vol 49 Issue 18 pages 8545 -8551 (A Fogg) (Also includes data from DLS)

MedChemLet DOI 10.1021/ML100295V (J Spencer)

Organometallics DOI 10.1021/OM2000585 (T Sheppard)

A. Platt

Source: INORGANIC CHEMISTRY Volume: 50 Issue: 6 Pages: 2553-2561 Published: MAR 21 2011

J-P. Bassin

Source: ACTA CRYSTALLOGRAPHICA SECTION E-STRUCTURE REPORTS ONLINE Volume: 67 Pages: O684-U2311 Part: Part 3 Published: MAR 2011

P. Smith

Source: INORGANICA CHIMICA ACTA Volume: 368 Issue: 1 Pages: 257-262 Published: MAR 15 2011

J. Snaith

Source: JOURNAL OF ORGANIC CHEMISTRY Volume: 75 Issue: 21 Pages: 7347-7357 Published: NOV 5 2010

M. Beckett

Source: COLLECTION OF CZECHOSLOVAK CHEMICAL COMMUNICATIONS Volume: 75 Issue: 9 Pages: 971-980 Published: 2010

Publications arising from NCS use of DLS

C. Tan, S. Yang, X. Lin, A. J. Blake, W. Lewis, N. R. Champness & M. Schröder, "High Capacity Gas Storage by a 4,8-Connected Metal-Organic Polyhedral Framework", Chem. Commun. 2011, submitted 20/1/2011, accepted 28/1/2011.