

UK National Crystallography Service Biannual Report 2

Period covered: 01/05/2011 – 30/10/2011

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1. Preface

This period has mainly been dominated by the installation of the new laboratory equipment, culminating in the formal launch of the Southampton Diffraction Centre (<http://www.southampton.ac.uk/sdc>). The Southampton Diffraction Centre is an umbrella organisation encompassing the NCS, as well as macromolecular diffraction and crystallisation and the School of Chemistry departmental and commercial services. The launch was a joint event with the University CT Imaging Centre on 16th September, where both laboratories were opened by John Denham MP and the Vice Chancellor of the University.

The transfer to new instrumentation was not without its problems – delays due to the natural disaster in Japan, the Mo rotating anode arriving broken and considerable logistics involving the removal of old equipment, refurbishment of the laboratory and installation of the new instrument base all of which was mainly performed during ‘normal’ laboratory operation. Despite these events the operation of the NCS continued, albeit under difficult conditions – operational performance predictably has dipped a little as a result of this transition and having only just completed this exercise we are likely to see a similar in the coming period.

This being said, the new equipment is now installed and tested with a few teething problems remaining, but in-hand. It remains for us to establish new operational procedures in an entirely new environment with new equipment and some downstream issues with data processing procedures have come to light and are being addressed. After initial use and testing of the new equipment we are able to collect data at the same levels (if not more) than the old laboratory and we have entered into discussions at the highest level with Rigaku as to how we address data processing. We do however have every confidence in claiming that the NCS now operates the most powerful facility of its type in the world.

We are capitalising on our world-leading position by building our scientific relationship with the equipment providers – Rigaku. During September we have had two separate visits from the Director of Small Molecule Crystallography and the VP for Software Development, both of which are significantly moving forward our capability whilst simultaneously benefitting Rigaku and its customers.

We continue to improve our customer interface by rolling out new aspects of our information management system – during this period we deployed a new application system which implements a major development in access policy.

2. Operation and Logistics

A. Upgrade of Laboratory

The transition to the new equipment was multi-faceted with many aspects requiring very careful consideration and management. The installation was delayed due to the Tsunami in Japan earlier in the year, however some components were shipped on time and hence a multi-phase install and decommissioning of old equipment was planned and proceeded as follows:

June – Cu rotating anode installed

June - Powder diffractometers relocated

July – old Bruker-Nonius equipment decommissioned and sold to University of Wurzburg

July – Mo rotating anode arrived. Discovered irretrievably broken on delivery – a new instrument required construction in the Japanese factory. The Cu anode target was temporarily replaced with a Mo one, which enabled us to bridge the gap in service provision whilst waiting for a replacement to be constructed and shipped.

July – SPIDER installed

August – Replacement Mo rotating anode arrived

September – Laboratory fully operational.



Throughout this install numerous infrastructure works were required, namely electrical, plumbing and decorative. Despite the problems with the Mo rotating anode, only 4 weeks of operation were lost – as was originally planned and the new laboratory delivered just a month behind schedule and without any noticeable disruption to NCS provision.

B. Overview of service use

This period has seen an increase in the number of user applications – both arising from the formal Call and also from the Rapid Access route. The number of users is on the rise, most probably due to ending the period of uncertainty in service provision that arose from funding issues (09-10) and an increased understanding of the benefits gained from access the NCS.

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.

C. Staff

There has been no alteration to the core crystallographic PDRA staffing in Southampton or at DLS (Drs Horton, Tizzard, Pitak and Wilson) or administrative support (Mrs Milsted). Dr Susanne Coles was employed for 3 months to assist in covering the transition period on moving to the new equipment base, where primary tasks were data collection (Soton & DLS), management of some aspects of installation, sample logging and assisting with arrangements for the launch.

D. NCS Synchrotron component

The successful NCS block allocation group (BAG) 2 year programme has been in place for the last 6 months and all the scheduled NCS beamtime has been through this access mode. NCS has had 6 days (each of 24 hours comprising a block of 3 shifts) allocated in the last 6 months (18/4/11, 4/5/11, 21/5/11, 20/7/11, 1/8/11 and 1/10/11). NCS has also made use of additional beamtime that has been made available adjacent to the scheduled 21/5/11 and 1/10/11 visits.

Typically 20-25 samples have been screened during a visit and up to approximately half of these will lead to data collections. This is highly sample dependent and varies considerably from visit to visit. The proportion of samples that result in data collection can reasonably be expected to be lower for samples referred to Diamond as they have failed at every previous stage and this is the last possible opportunity to obtain data.

The DLS based NCS team member post continues to allow the NCS to provide both complete feedback on any problems with beamtime and contribute to making improvements, allowing us to get the best out of NCS beamtime. Working closely with the I19 team has also improved the scheduling of NCS beam time to well distributed dates for visits allowing regular access and it has again allowed use to be made of some additional beam time as originally envisaged.

There are a further 5 days scheduled in the next 6 month period.

E. 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.

F. Equipment- Technical Issues

The new diffractometers have arrived and were installed during this period and are all currently up and running. The old diffractometers were shutdown, dismantled and removed. There have been a few teething problems with regards to the new set-up and getting everything running. We are now running four cryosystems (two non-liquid nitrogen cryostats (Cobras) and two liquid nitrogen cryostats (700+ cryostream and 600 cryostream)).

The main source of downtime during this period was the changeover of diffractometers (1).

Commented [SC1]: From diary??

The following issues have arisen in the reporting period:

Diffractometers:

- Chiller issues with both the Spider and Cu-007 generators resulted in an inability to generate X-rays for both instruments. An engineer from the chiller company (ATC) was called out, under the Rigaku warranty to fix the Cu-007 chiller and provide parts to fix leaks on the SPIDER.
- Problems with leaking helium regulators for the mirrors. Now resolved.

Cryogenic devices:

- One Cobra unit (non-liquid N₂) has a small helium leak. This is still unresolved and investigations continue. The Cobra unit is still operational, but requires a recharge of helium every 4 weeks. This is clearly unsustainable in the long term and resolution is being investigated.
- The Oxford Cryostream 600 and 700+ coldheads were both re-evacuated and are now running on the Spider and Cu-007 respectively.
- A new AD-51 dry air unit was acquired to allow the running of two additional cryosystems (compared to the old laboratory configuration).

G. Sample Issues

Several problems which have arisen in the past are still continuing:

- 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.
- Still a number of Schlenk flasks, long NMR tubes or very small, blind-necked vials often contain large amounts of solvent (mother liquor) and/or only a few crystals which are not easy to deal with. Also a considerable number of samples have no mother liquor resulting in crystalline material that is firmly stuck to the vial (tube) surface and requires significant manual (physical) treatment which introduces unnecessary strain to the crystals. Additionally, such samples often are not monocrystalline and sometimes partially decomposed due to solvent loss.
- A number of submitted samples have more than one type of crystalline material (different crystal type, habit, colour, etc), however users have not specified which type should be examined. Accidentally, data for unwanted side products or starting material crystals which have grown from the same solution at the same time as the expected product of the reaction might be collected instead.
- Some light sensitive samples have been submitted in colourless transparent vials. These should be wrapped in aluminium foil or put into small dark containers (or boxes). Otherwise crystals might be unnecessarily exposed to the light conditions during storage.

H. Data Processing Issues

Visits from Rigaku application scientists, as part of scheduled training on the new instrument base, have been hosted.

1. Lee Daniels (Director of Small Molecule Crystallography, Rigaku Americas) provided expert tuition in CrystalClear – dealing with twinning etc.
2. Russ Athay (Vice President, Software, Rigaku Americas) discussed incorporation of NCS requirements (data management) into CrystalClear. Also demoed alpha version of CrystalClear 3.1 due for distribution 01.04.2012.

Hardware and data architecture issues have resulted in very slow data processing, which are at present being investigated and addressed.

3. Community Activity

A. Training and Outreach

The NCS has hosted 3 visits since May. These visits have been hosted by the NCS during beamtime at Diamond Light Source

- Sofia Pascu (Bath University)
- 3 members of Kennedy group over 2 visits to Diamond Light Source (University of Strathclyde)

The service hosted a 2 week work experience placement in the summer and a sixth form student on a work-shadowing day. Four third and fourth year undergraduate students completed research projects during the last academic year, in particular one was concerned with outreach education in crystallography in collaboration with the service. 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. We have also continued to take part in University of Southampton Open Days and weekly tours of UCAS candidates.

B. Publicity

A third round of flyers has been distributed to heads of departments and school service managers to advertise the call.

The NCS was promoted heavily at the launch of the Southampton Diffraction Centre in September. This event is being used to publicise the NCS through press releases and an article highlighting the event has been submitted to the RSC periodical 'Chemistry World'.

C. User Liaison

The implementation of the online application form has suffered some teething problems. We have dealt with all user issues and have taken these on board with an eye to improving the system for the next call. We continue to act on suggestions and comments from the users to refine and improve our systems.



4. Preview of next period

A. Preview of availability over next 6 months

Asides from University closed days over the Christmas and Easter holiday periods, there is no scheduled shutdown of the facility and therefore full availability is expected.

B. Preview of maintenance over next 6 months

Apart from possible filament changes, we do not expect major service work on the diffractometers in this next period.

The Cobra helium leak is still on-going but should be resolved.

C. Preview of upgrade over next 6 months

No upgrade to any of the equipment used by the NCS is expected in the coming period.

Appendix 1: KPI Data (All current data for 01/05/11 – 11/10/11)

		This period (01/05/10 – 11/10/11)	Oct 2010 – May 2011	May – Oct 2010
Number of NCS Users (active)		68	56	39
Number of NCS Projects		68*	69	44
Availability of facility for NCS use (days)		108	105	106
Actual equipment uptime and use of facility by or for NCS		77	98	102
Number of NCS samples processed	Total	384	404	342
	At Southampton	304	342	273
	At DLS	80	62	69
Number of NCS data collections performed		238	280	221
Number of Full structure determinations performed		146	124	52
Number of NCS samples outstanding		240	302	184
Waiting for examination		20	17	53
Processing		167	198	96
Waiting for return to users		53	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	208	204	Unable to report against this at the time
	2 attempts	44	30	
	3 attempts	6	3	
	More than 3 attempts	0	6	
	Unreported	52	107	
Number of User complaints received		0	0	0
Number of NCS research outputs		55	73	26
Number of NCS users of the training programme		3	7	8
Number of samples classes as routine or difficult	Routine	166	172	Unable to report against this at the time
	Difficult	55	43	
	Synchrotron	80	62	
	Unreported	56	73	

* Projects is an old metric - we used to allow more than one project per user, now operate a one allocation per user system

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 99% (4 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-	78%
M-	88%
L-	89%

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 70%

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 75%

Commented [SC2]: Should we comment on reasons why we fall below the bar or is that dangerous?

Commented [e3]: Caused by processing and data architecture issues. Grappling with software. Improvements have been made and we are continuing to review and improve this. This will continue to affect stats into next period as some samples affected are still processing

Commented [SC4]: Alluded to in the preface – probably need to spell out here?? Maybe??

Commented [e5]: Checking this statistic update tomorrow