

UK National Crystallography Service Biannual Report 10

Period covered: 01/11/2014 – 30/04/2015

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1. Preface

Period 10 continued to consolidate NCS operations with staff, instrumentation and operating procedures well established. Claire Wilson left the Diamond position to take up a post as Staff Crystallographer at the University of Glasgow. The process to appoint a successor was undertaken with Diamond taking the lead and Dr Jeppe Christensen takes up the post effective 01/05/2015.

The number of full structure determinations continues to be at a higher level than previous periods and the number of new users continues to steadily increase. We find that the bottleneck in turning around the volume of samples necessary is shifted to the data workup stage, generally as a result of the challenging quality of the data most samples exhibit. Accordingly we have taken on a 4th Southampton-based PDRA – Dr Tony Keene had a Marie Curie Fellowship that completed its term and is working for the NCS whilst waiting to take up a new position.

The interface with Diamond and our resulting beamtime continues to run very smoothly and a steady flow of publications arising is occurring. The NCS is the most experienced user of the Helix helium temperatures cryostat and the sample changing robot and has a significant amount of beamtime. Accordingly we see a greater diversity of samples and types of chemistry than any other user group and have been conducting a study into crystal decay in the x-ray beam – an effect we are observing with more frequency than ever before. The results of this decay study will form a case study for the annual report. I19

In an interesting turn of events Rigaku purchased the single crystal diffraction arm of Agilent's business to form Rigaku Oxford Diffraction (ROD). The upshot of this is that we will move to using the CrysAlisPro software to drive diffractometers and process data. The latter we have already been doing for some time and we are very happy with its performance, whilst the former requires the software to be integrated by ROD and we will work closely with them. We foresee this 'merger' as a very positive thing for our laboratory – particularly as it fills the software void we have been trying to address for the last 4 years.

Being the International Year of Crystallography in 2014, the NCS has been significantly involved in Outreach Activities. The most notable were the Big Bang Science Fair and Gravity Fields (Grantham, Lincolnshire) events.

The NCS is looking to overlap more with others in the EPSRC Mid-Range Facilities programme. This was progressed further at a Directors meeting held in Coventry in Jan 2015. We have collaborated with the National Mass Spectrometry Service and the Chemical Database Service (CDS) through joint promotion as part of departmental roadshows and also attendance at conferences. The main conferences attended by the NCS were the BCA Autumn meeting (organised by SJC) and the RSC Dalton Southern Area Meeting, which resulted in numerous new users. The relationship with the National Service for Computational Chemistry Software was cemented at the Director's meeting.

2. Operation and Logistics

A. Overview of service use

Service use remains consistent, with the number of users, days available and samples processed all remaining roughly constant since the last allocation period. The number of samples examined at Diamond is reduced, however there were less days allocated in this period and for the entire time we did not have an on-site NCS scientist, meaning it was not possible to use spare instrument time. The trend for a significant number of Full Structure Determinations is still being upheld and the number of samples 'in the system' is reduced.

Compared to previous periods data – all Benchmarks are now met. This is due to focusing attention on time critical samples in the system and monitoring the progress of these with respect to benchmarks. Furthermore this is indicative of how

increased staffing levels are required - more difficult samples are being addressed, yet benchmarks that were previously unmet are now satisfied.

The rapid access scheme continues to attract new users between calls, the majority of whom go on to apply for regular access at the following routine call for applications – so whilst the number of users remains roughly constant, there is actually a cycling of some new users replacing those who don't currently require our services (can be up to around 10 users each 6 month period).

B. Upgrade of Laboratory

The Nitrogen generator and Cryodrive components of the ageing Cobra cryogenic system were in a critical condition and have been replaced. These form part of a multicomponent system and the vendors (Oxford Cryosystems) have agreed to sell the remaining components to us as part of an upgrade package at the same cost as if purchasing the whole product.

C. Staff

The core crystallographic PDRA staffing in Southampton has been supplemented by the appointment of Dr Tony Keene on a temporary basis. This increased level of staffing is in the main the reason for the improved benchmark statistics.

Furthermore the staffing at DLS is under change - Dr Wilson left for a permanent staff position at the University of Glasgow at the start of the period. A recruitment process has been undertaken and Dr Jeppe Christensen will commence working at DLS from the beginning of the next period.

D. NCS Synchrotron component

Access to I19, Diamond continues to be through the successful NCS block allocation group (BAG) 2 year program mode, which makes the NCS one of the main users of the beamline.

NCS had 5 days (each of 24 hours comprising a block of 3 shifts) allocated in the current 6 month period. Typically 10-20 samples have been screened during a visit and around half of these will lead to data collections. This is highly sample dependent and varies considerably from visit to visit. Our continued use of the Helix for very low temperature (30K data collection) has been very successful and we continue to typically have one day of the allocation with the Helix.

We have also continued to collect data to systematically probe the effect of radiation damage through a series of more structured experiment; a significant effect – considerably more so than when we used to use the Daresbury synchrotron. The results of this study will be available to provide information and guidance to the I19 user community and the whole chemistry community when a clear understanding has been reached. Preliminary results have been presented as a poster and the completed work will form a case study for a future annual report.

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

Routine maintenance has been performed on diffractometers. The following summarises diffractometer-based issues and resultant instrument downtime that have arisen in the reporting period:

- FRE+ filament change x 2 (1 day downtime each).
- All the above replacements and servicing have been carried out under warranty and/or service contract.

The Cobra cryogenic devices have suffered numerous problems. Various diagnoses were carried out in conjunction with support from Oxford Cryosystems, which resulted in components being upgraded (see above).

G. Sample Issues

Several persistent issues continue:

- Partially completed submission forms, particularly with sample related information missing (such as solvent/air sensitivity, possible photo reactivity, melting point of crystals). A number of users do not supply reaction schemes or expected formula.
- 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 difficult to manipulate.
- 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.
- A number of samples which degrade quickly do not have low temp storage requested.
- A number of samples which are described as solvent sensitive are sent either without solvent or in vials which are not properly sealed to prevent evaporation.

H. Data Processing Issues

In April 2015 Rigaku announced the acquisition of Oxford Diffraction from Agilent. Discussions have been on-going with Rigaku Oxford Diffraction regarding the control software for the diffractometers – namely changing from CrystalClear to CrysAlisPro (this is expected to be achieved in Q1 of 2016). We are already using CrysAlisPro to process the data for most samples.

3. Community Activity

A. Training and Outreach

We expanded our stand at the University's Family Science Day, once again took part in the Big Bang Fair and also Gravity Fields. Simon Coles has taken up the position of Education and Outreach Co-ordinator for the BCA, a Council post, and is now heavily involved in many nationwide events. NCS also took part in work shadowing days and had stands at university open days, publicising the NCS and its work to prospective students and parents.

B. Publicity

The NCS continues to publicise its services through flyers and emails at the time of call. An EPSRC mid-range facility day was attended in February, which looked at the marketing of the Mid-range Facilities and a panel focused particularly on

marketing to industry. The report from this panel is being used to guide future publicity materials. We are also starting work on the new annual report format that has been requested by EPSRC and the accompanying case studies.

NCS sponsored the RSC Southern Dalton area meeting and Simon Coles gave one of the presentations – this event directly resulted in several new users. The NCS also played a role in the BCA Autumn meeting at Burlington House, which was themed around Outreach, Education and Dissemination.

C. User Liaison

We continue to act on suggestions and comments from the users to refine and improve our systems. Building on this, we have been striving to make it clear to our user community that we can be highly responsive to urgent or unusual requests. This drive has led to good feedback from users requiring help with graphic design relating to their structures, the need for rapid turnaround times to include results in lectures and important reports/dissertations/theses/papers, suggesting additional collections to help with difficult samples and advanced experiments such as variable temperature studies and charge density.

4. Preview of next period

A. Preview of availability over next 6 months

Aside from University closed days over the bank holiday periods, the planned closures during the next period is for the annual service of diffractometers and the ECM meeting in August 2015

B. Preview of upgrade over next 6 months

None planned.

Appendix 1: KPI Data (All current data for 01/11/13 – 30/04/14)

		This Period Nov 2014 – May 2015	May– Oct 2014	Nov 2013 – May 2014	May – Oct 2013	Nov 2012 – May 2013	May – Oct 2012
Number of NCS Users (active)		64	65	68	64	67	65
Availability of facility for NCS use (days)		98	94	93	92	108	110
Actual equipment uptime and use of facility by or for NCS		92	67	86	78	94	100
Number of NCS samples processed	Total	427	403	443	376	432	417
	At Southampton	385	350	377	316	376	353
	At DLS	42	53	66	60	56	64
Number of NCS data collections performed		257	238	277	223	260	285
Number of Full structure determinations performed		170	165	166	153	172	132
Number of NCS samples outstanding		239	246	263	237	201	189
Waiting for examination		58	70	77	63	83	16
Processing		166	143	178	137	100	167
Waiting for return to users		15	33	8	37	18	6
Number of User data sets that were completed within 1,2,3,>3 attempts	1 attempt	378	365	385	284	349	289
	2 attempts	45	31	50	87	64	107
	3 attempts	4	7	8	5	15	17
	More than 3 attempts	0	0	0	0	4	4
	Unreported	0	0	0	0	0	0

Number of User complaints received		0	0	0	0	0	0
Number of NCS research outputs		*	39	40	52	44	44
Number of NCS users visiting NCS facility		3	0	2	0	0	1
Number of samples classed as routine or difficult	Routine	216	252	248	185	190	265
	Difficult	169	98	129	131	135	88
	Synchrotron	42	53	66	60	40	64
	Unreported	0	0	0	0	0	0

* extended allocation period – data assessment in progress

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.

Achievement for this benchmark = 100%

Benchmark 2

The time a sample is in the queue from logging it in 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.

Achievement for this benchmark:

High priority = 89.5%

Medium priority = 82.5%

Low priority = 81%

Benchmark 3a

The time a sample is in the queue from examination to communication of the result of the data collection to the user should be within 5 working days for 80% for Data Collection Only samples. The result of the data collection, for the purposes of this benchmark, is defined as any of the following:

1. Withdrawal of sample,
2. Failure of sample,
3. Decision to recollect (resulting in a new set of benchmark data for the recollection),
4. Decision to refer to DLS (resulting in a new set of benchmark data for the referral),
5. Provision of an .hkl file to the user.

Achievement for this benchmark = 80%

Benchmark 3b

The time a sample is in the queue from examination to communication of the result of the data collection to the user should be within 20 working days for 80% of Full Structure Analysis samples. The result of a data collection, for the purposes of this benchmark, is defined as any of the following:

1. Withdrawal of sample,
2. Failure of sample,
3. Decision to recollect (resulting in a new set of benchmark data for the recollection),
4. Decision to refer to DLS (resulting in a new set of benchmark data for the referral),
5. Provision of a publication quality .cif file to the user.

Achievement for this benchmark = 81%