

UK National Crystallography Service Biannual Report 8

Period covered: 01/11/2013 – 30/04/2014

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1. Preface

Period 8 continued to consolidate NCS operations with staff, instrumentation and operating procedures well established. 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.

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 were the subject of an ‘initial findings’ poster at the BCA Spring Meeting.

We are continuing to capitalise on our world-leading position by building our scientific relationship with the equipment providers – Rigaku and this is currently taking the form of software development and testing as well as a number of other collaborative ventures. A recent example of the latter is the installation of MiniFlex and XtaLab mini diffractometers (powder and single crystal benchtops respectively) – these are being used as training instruments and a professional video concerning our unique use of these instruments has been produced by Rigaku as a promotional tool see http://www.rigaku.com/products/smc/xtalabmini?qt-xtalab_mini_quicktabs=4#soton.

Being the International Year of Crystallography in 2014, the NCS has been significantly involved in Outreach Activities. We provided significant input into the Big Bang Science Fair (Birmingham, March 2014) and hosted an IYCr stand at the National Science and Engineering Week Family Day (Southampton, March 2014) which saw about 20,000 and 3,000 visitors respectively. A significant number of further Outreach activities are planned for the rest of the year.

The NCS is looking to overlap more with others in the EPSRC Mid-Range Facilities programme. This began afresh as a result of the Directors meeting held in Dec 2013. There is agreement between some of the ‘sister services’ in Chemistry to assist each other with promotional activities and combine forces when possible/appropriate. A strong example of this is the promotion of the NCS and the National Mass Spectrometry Service by the Chemical Database Service (CDS) – both as part of their departmental roadshows and also attendance at conferences. We have exchanged promotional material, such as powerpoint slides and fliers, in order to further enable this route to reaching out to a wider audience (with minimal overhead to all services). We have struck up a particularly strong relationship with the CDS, including working with them on the storage and representation of crystallographic/chemical information and Electronic Laboratory Notebooks.

2. Operation and Logistics

A. Overview of service use

Service use remains consistent, with the number of users remaining roughly constant since the last allocation period. The number of days the Facility is available for NCS use is slightly down due to on-going problems with ageing cryogenic equipment. The number of samples processed is significantly more than the previous period, in fact more than in any other reporting period, and most of these were examined on the Southampton facilities. The trend for a significant number of Full Structure Determinations is still being upheld and increasing slightly. The number of samples completed on the first attempt is also greater than before. The number of samples still in the system is higher than usual, reflecting not only that there have been more samples submitted this period but also that the processing of data is now the most time consuming part of the operation and involves a large amount of dedicated interpretation and analysis by crystallographic experts. This latter point is also the reason why Benchmark 3 was not met – only 71% of Full Structures were completed within 20 days of data collection (Benchmark = 80%) and this is due not only to the fact that a lot of dedicated time is required to address these

particularly hard problems (which have generally failed on users' local facilities) but also that more samples were submitted this period.

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

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).

An intern, Miss Amy Ellis, is conducting an MChem placement with the NCS and has assisted in some data collection and administrative tasks, but her main focus is an internal structural systematics research project.

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 6 days (each of 24 hours comprising a block of 3 shifts) allocated in the current 6 month period (1/11/13- 1/4/14) 30/11/13, 9/12/13, 22/1/14, 10/2/14, 13/3/14 and 13/4/14. 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.

Our attempts to use a longer wavelength than 0.6889Å were affected by problems with the beam and beam alignment. We aim to try and benefit from the increased flux and scattering efficiency at lower energies to look at particularly weakly scattering samples, especially those with larger unit cells which often do not scatter at higher resolution and gain greater spatial resolution of the closely spaced reflections with the longer wavelength radiation. We will attempt this again on future visits.

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, however preliminary results were presented as a poster at the BCA Spring Meeting (Loughborough) in April 2014.

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:

- Planned University Power outage (Weekend - but lost 2 days due to timing, just before University Easter Shutdown)
- FRE+ filament change x 2 (1 day downtime each).

- Power Spikes/Outages (random) x3 (Overall just over 1 day downtime with equipment checks and fuses replaced)
- All the above replacements and servicing have been carried out under warranty and/or service contract.

The cryogenic devices have suffered the following problems:

- One Cryodrive on the FR-E+ system failed, necessitating a return to Oxford Cryosystems for servicing/repair. Accordingly one of the diffractometers was only able to operate at room temperature for a period of 2 weeks.
- The Cobra low temperature system on one of the FR-E+ systems was unable to reach a temperature of 100K for a prolonged period. Temperatures of 120K and then subsequently 150K could be obtained. Various diagnoses were carried out in conjunction with support from Oxford Cryosystems, however the fault remains unresolved at the end of this reporting period.

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

We are continuing to work with Rigaku to develop the latest version of CrystalClear 2.1. Additionally, we have negotiated with Agilent for their CrysAlisPro software, which they have made freely available to us after some minor modifications and we are in the process of testing this with some success.

3. Community Activity

A. Training and Outreach

We have hosted 2 visits during this period and have advised a number of research groups on training options available, there appears to be growing interest in training through the NCS and we are currently exploring how best to respond to this.

As part of the International Year of Crystallography we expanded our stand at the University's Family Science Day, and once again took part in the Big Bang Fair. Next period we have been invited to take part in Primary School days and the internship scheme again.

B. Publicity

The NCS continues to publicise its services through flyers and emails at the time of call. A refresh of materials is ongoing. Collaboration with the other mid-range facilities on cross promotion has started with the Mass Spectrometry Facility and Chemical Database service with the promotion of sister services at conferences attended by any of these three facilities. Through our collaborative relationship with Rigaku, numerous publicity opportunities arise – this period we have had some visibility through the Open Lab scheme and made a promotional film on the practical crystallography education and the NCS http://www.rigaku.com/products/smc/xtalabmini?qt-xtalab_mini_quicktabs=4#soton.

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 only planned closure during the next period is for the annual service of diffractometers which has been aligned with the IUCr conference in Montreal during August 2014.

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 2013 – May 2014	May – Oct 2013	Nov 2012 – May 2013	May – Oct 2012	Oct 2011 – May 2012	May – Oct 2011
Number of NCS Users (active)		68	64	67	65	67	68
Number of NCS Projects		68*	64*	67*	65*	67*	68*
Availability of facility for NCS use (days)		93	92	108	110	106	108
Actual equipment uptime and use of facility by or for NCS		86	78	94	100	92	77
Number of NCS samples processed	Total	443	376	432	417	421	384
	At Southampton	377	316	376	353	333	304
	At DLS	66	60	56	64	88	80
Number of NCS data collections performed		277	223	260	285	301	238
Number of Full structure determinations performed		166	153	172	132	120	146
Number of NCS samples outstanding		263	237	201	189	174	240
Waiting for examination		77	63	83	16	66	20
Processing		178	137	100	167	89	167
Waiting for return to users		8	37	18	6	19	53
Number of User data sets that were complete d within 1,2,3,>3 attempts	1 attempt	385	284	349	289	330	208
	2 attempts	50	87	64	107	71	44
	3 attempts	8	5	15	17	15	6
	More than 3 attempts	0	0	4	4	5	0
	Unreported	0	0	0	0	0	52

Number of User complaints received		0	0	0	0	0	0
Number of NCS research outputs		40	52	44	44	49	55
Number of NCS users of the training programme		2	0	0	1	1	3
Number of samples classed as routine or difficult	Routine	248	185	190	265	265	166
	Difficult	129	131	135	88	57	55
	Synchrotron	66	60	40	64	88	80
	Unreported	0	0	0	0	11	56

* 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.

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 = 83%

Medium priority = 81%

Low priority = 83%

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 = 71%