

UK National Crystallography Service Biannual Report 6

Period covered: 01/11/2012 – 30/04/2013

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1. Preface

Period 6 has been a solid consolidation of the previous periods with staff, instrumentation and operating procedures well established. We are now performing a greater number of full structure determinations than in 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. The NCS is the most experienced user of the Helix helium temperatures cryostat and during this period we managed to optimise its use with the result that datasets were collected from samples that previously couldn't have been run.

We are continuing to capitalise on our world-leading position by building our scientific relationship with the equipment providers – Rigaku. The Senior Manager of Single Crystal Structure Analysis has recently visited the facility and we have established the best route forward for software development to support the NCS. A number of other collaborative ventures were also discussed.

The NCS has contributed significantly to major public engagement exercises during the period, which happens to be the Bragg Centenary and the lead up to International Year of Crystallography in 2014, with strong input and presence at the national Big Bang Science Fair (ExCeL, London) and Southampton's involvement in National Science and Engineering week – each of these events attracts several thousand members of the general public.

The NCS is looking to overlap more with others in the EPSRC Mid-Range Facilities programme. We have struck up a strong relationship with the Chemical Database Service and will be seeking to grow this, and links with other services, in the coming periods.

2. Operation and Logistics

A. Overview of service use

Service use remains approximately constant, with the number of users and samples processed remaining steady since the last allocation period. There has been another increase in the number of full structures being processed. Taking into account the number of samples which are requiring more than one attempt, the total number of datasets collected is around 540. This results in an increase in the number of samples classified as difficult.

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.

B. Upgrade of Laboratory

The laboratory plant room has undergone a much needed infrastructure upgrade to the heat extraction system (new fans, trunking, etc), which we expect to result in a higher degree of reliability for provision of chilled water for x-ray generators and operation of sample cryogenic systems. In the past this room has been unsuitable during periods of hot weather, resulting in malfunctions of chillers and therefore resulting in instrument downtime.

We continue to work with Rigaku to develop CrystalClear 3.1. Currently the service is using the latest beta version which is beta release 25. A recent visit from the Senior Manager of Single Crystal Structure Analysis at Rigaku (Masataka Maeyama, Tokyo office) outlined the future software development roadmap following the announcement that CrystalClear 3.1 will be the final version of CrystalClear to be released.

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).

D. NCS Synchrotron component

Access to I19, Diamond continues to be through the successful NCS block allocation group (BAG) 2 year program mode – during this reporting period we have been awarded our next 2 year BAG.

NCS had 6 days (each of 24 hours comprising a block of 3 shifts) allocated in the current 6 month period (30/11/12, 20/12/12, 2/2/13, 21/2/13, 18/3/13 and 20/4/13). Typically 20-25 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. 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. This is especially true with the high flux instrumentation currently in Southampton.

Two of the allocated days have utilised the Helix helium temperature sample cooling device. This has resulted in collections that would not otherwise have been possible. In fact, the NCS team has been key in developing the procedure for use of the Helix and is currently the most advanced user of this equipment – we will present this at the upcoming I19 User Workshop and thereby pass on our experience to the whole community.

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. The following summarises diffractometer-based issues and resultant instrument downtime that have arisen in the reporting period:

- Total facility closure due to a university-wide power failure (1 day downtime).
- Total facility closure due to a plant room upgrade (10 days total downtime).
- FRE+ filament change (1 day downtime).
- FRE+ drive belt tightened (1 day downtime).

All the above replacements and servicing have been carried out under warranty and/or service contract.

- The SPIDER diffractometer was restored into action in January after long downtime due to control PC failure. There continue to be issues with the control PC, particularly with the requirement to run an outdated operating system.

The cryogenic devices have suffered the following problems:

- Cobras underwent routine maintenance at the beginning of 2013.
- A new membrane was fitted to the nitrogen generator.
- There is an on-going helium leak with Cobras, however our previously reported capping has brought this down to a manageable level and this no longer interferes with the running of the service.

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.
- 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.
- Some light sensitive samples have been submitted in clear, transparent vials and a number of samples which degrade quickly do not have low temp storage requested.

H. Data Processing Issues

As mentioned previously, the service team is helping to develop CrystalClear 3.1. This software now includes the new Rigaku Integrate package, which replaces D*Trek and FS Process. In Rigaku's own tests Rigaku Integrate is consistently delivering better results than the previous programs and in our capacity as software testers we have experienced the same kind of results. We now use Rigaku Integrate for most processing and this is providing a much higher quality of dataset.

3. Community Activity

A. Training and Outreach

The service took part in both the University of Southampton Family Science Day (a component of National Science and Engineering week) and the Big Bang Fair at ExCeL in London during March. At the Big Bang Fair the NCS strongly contributed to designing and running a BCA/RAL stand to celebrate the Bragg Centenary.

We have also continued to be a key aspect of University of Southampton Open Days and weekly tours for UCAS candidates. This period has also seen the service host an intern over the Easter break and also 2 project students in their 3rd and 4th years of undergraduate study.

We will host a work experience student from a local college in the summer, as well as RSC-sponsored interns, who will work on developing new types of experiment and making large volumes of data accessible to the ChemSpider community database.

B. Publicity

The NCS is renewing its publicity materials at the current time. This is to refresh the printed materials and renew the key messages about the service as it matures and we now fully understand the capability of the facility.

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, 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 will be in August 2013. This closure will be to conduct the annual preventative maintenance programme, which we are trying to schedule at the same time as the European Crystallography Meeting in Warwick (which all NCS staff will be attending).

B. Preview of upgrade over next 6 months

The School of Chemistry is installing new bench top single crystal and powder diffractometers as part of a new instrument-based teaching facility. This will increase redundancy through having a further backup diffractometer that the NCS can use in emergencies.

As a component of the EPSRC Pioneers Equipment funding scheme we will be developing capability to perform high pressure and gas cell experiments. Once our ability to perform these experiments has matured, we intend to make these a component of the specialist capability offered by the NCS.

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

		This period (1 st Nov 2012 – 17 April 2013)	May – Oct 2012	Oct 2011 – May 2012	May – Oct 2011	Oct 2010 – May 2011	May – Oct 2010
Number of NCS Users (active)		67	65	67	68	56	39
Number of NCS Projects		67*	65*	67*	68*	69	44
Availability of facility for NCS use (days)		108	110	106	108	105	106
Actual equipment uptime and use of facility by or for NCS		94	100	92	77	98	102
Number of NCS samples processed	Total	432	417	421	384	404	342
	At Southampton	376	353	333	304	342	273
	At DLS	56	64	88	80	62	69
Number of NCS data collections performed		260	285	301	238	280	221
Number of Full structure determinations performed		172	132	120	146	124	52
Number of NCS samples outstanding		201	189	174	240	302	184
Waiting for examination		83	16	66	20	17	53
Processing		100	167	89	167	198	96
Waiting for return to users		18	6	19	53	87	35
Number of User data sets that were completed within 1,2,3,>3 attempts	1 attempt	349	289	330	208	204	Unable to report against this at the time
	2 attempts	64	107	71	44	30	
	3 attempts	15	17	15	6	3	
	More than 3 attempts	4	4	5	0	6	
	Unreported	0	0	0	52	107	
Number of User complaints received		0	0	0	0	0	0

Number of NCS research outputs		44	44	49	55	73	26
Number of NCS users of the training programme		0	1	1	3	7	8
Number of samples classed as routine or difficult	Routine	190	265	265	166	172	Unable to report against this at the time
	Difficult	135	88	57	55	43	
	Synchrotron	40	64	88	80	62	
	Unreported	0	0	11	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.

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 = 82%

Medium priority = 95%

Low priority = 72%

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 = 75%

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 = 63%