

**UK National Crystallography Service Biannual Report 9**

**Period covered: 01/05/14 – 31/10/2014**

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## **1. Preface**

Period 9 continued to consolidate NCS operations with staff, instrumentation and operating procedures well established. However, Dr Claire Wilson departs the NCS post at Diamond for a permanent position as the Staff Crystallographer in the Chemistry Department at Glasgow University – we wish her well and thank her for her tremendous hard work whilst being in post. The vacant position will take some time to fill and therefore we have temporarily appointed Dr Tony Keene in Southampton to help cover the shortfall.

The interface with Diamond and our resulting beamtime has been running very smoothly, with a steady flow of publications arising, and it will be paramount to ensure that this position is retained when recruiting Claire's replacement. The NCS remains the highest volume and most experienced user of the beamline. Accordingly we see a greater diversity of samples and types of chemistry than any other user group and have been furthering our studies into crystal decay in the x-ray beam – an effect we are observing with more frequency than ever before. The next phase of results will be presented in posters at international conferences.

The usage and performance statistics for the overall operation of the service are in a steady state at the moment – being very comparable to previous periods.

Being the International Year of Crystallography in 2014, the NCS has been significantly involved in Outreach Activities. In this period we have either conducted or provided significant input into a number of events and promotional activities ranging from Grantham to Kenya!

The NCS is overlapping more with others in the EPSRC Mid-Range Facilities programme and this has been put into practice by attending an RSC Young Scientists event in Nottingham, where the NCS, the National Mass Spectrometry Service and the Chemical Database Service had a joint stand. 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). The interaction with the CDS is particularly strong with collaborative projects on data management being scoped.

## **2. Operation and Logistics**

### **A. Overview of service use**

Service use remains consistent, with the number of users and completed samples 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. All benchmarks were met.

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**

No upgrades were planned or undertaken.

### **C. Staff**

A significant staffing change is going to be occurring. Dr Claire Wilson is leaving at the end of the period for a permanent position as the Staff Crystallographer in the Chemistry Department at Glasgow University – we wish her well and thank her for her tremendous hard work whilst being in post. She was the first employee to be employed by Diamond and required to bridge to the Southampton operation – and she defined the role and made it work. Accordingly there are no major changes planned to the structure or role of this post and discussions have been had with Diamond and a job advert is out – it is expected that interviews will be held towards the end of the year. The vacant position will take some time to fill and therefore we have temporarily appointed Dr Tony Keene in Southampton to help cover the shortfall. Extra expert analysis is now required to address the increasingly challenging samples being submitted and Tony will help in this regard as well as forming part of the beamtime team.

#### **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 (1/05/14-31/10/14), however one of these was lost due to machine issues. 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.

#### **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 annual service (2 days)
- Chiller controller and fans replaced (9 days)
- FRE+ filament change (1 day)
- Bearing replacement (1 day)

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 3 days.

#### **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**

Following successful testing in the last period and early part of this period we are using CrysAlisPro to process the data for most samples.

## **3. Community Activity**

### **A. Training and Outreach**

We took part in two primary science days with a series of activities themed around international year of crystallography. Several of the schools involved took away materials for use with other classes and to put up displays in the school about the international year. We hosted several A-level work shadowing placements and an internship.

### **B. Publicity**

The service continues to publicise its activities where appropriate. Flyers are distributed around call periods along with direct emails to users past and present. The rapid access scheme continues to bring in new users between calls.

### **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 BCA spring meeting in April 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 May – Nov 2014	Nov 2013 – May 2014	May – Oct 2013	Nov 2012 – May 2013	May – Oct 2012	Oct 2011 – May 2012
Number of NCS Users (active)		65	68	64	67	65	67
Number of NCS Projects		65*	68*	64*	67*	65*	67*
Availability of facility for NCS use (days)		94	93	92	108	110	106
Actual equipment uptime and use of facility by or for NCS		67	86	78	94	100	92
Number of NCS samples processed	Total	403	443	376	432	417	421
	At Southampton	350	377	316	376	353	333
	At DLS	53	66	60	56	64	88
Number of NCS data collections performed		238	277	223	260	285	301
Number of Full structure determinations performed		165	166	153	172	132	120
Number of NCS samples outstanding		246	263	237	201	189	174
Waiting for examination		70	77	63	83	16	66
Processing		143	178	137	100	167	89
Waiting for return to users		33	8	37	18	6	19
Number of User data sets that were completed within 1,2,3,>3 attempts	1 attempt	365	385	284	349	289	330
	2 attempts	31	50	87	64	107	71
	3 attempts	7	8	5	15	17	15
	More than 3 attempts	0	0	0	4	4	5
	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	49
Number of NCS users of the training programme		0	2	0	0	1	1
Number of samples classed as routine or difficult	Routine	252	248	185	190	265	265
	Difficult	98	129	131	135	88	57
	Synchrotron	53	66	60	40	64	88
	Unreported	0	0	0	0	0	11

\* 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 = 85%

Medium priority = 87%

Low priority = 82%

### **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 = 82%

### **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%