

**UK National Crystallography Service Biannual Report 7**

**Period covered: 01/05/2013 – 31/10/2013**

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## **1. Preface**

Period 7 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 accordingly datasets were collected from samples that previously couldn't have been run. The use of the Helix was the subject of a presentation at the I19 users workshop by Claire Wilson and the NCS contributed significantly further at this event.

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 respectively) – these are primarily being used as teaching instruments in Southampton, but will be perfectly adequate for entry level training of anyone.

The NCS has contributed to generating material for the Two Braggs exhibition at ECM28 in Warwick (August 2013) – this will subsequently be used for other events in the lead up to, and period of, the International Year of Crystallography in 2014.

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, including the RSC funding a summer intern to look into getting crystal structures into their databases. SJC will be presenting at the upcoming Mid-Range Facilities programme Director's meeting.

## **2. Operation and Logistics**

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

Service use remains strong, with the number of users remaining roughly constant since the last allocation period. The number of samples processed is slightly down on the previous period, although there are a number currently in the system. The trend for a significant number of Full Structure Determinations is still being upheld.

The benchmark data are particularly impressive this period and are considerably improved from the past. This is due to a change in operational procedure effected by the Portal electronic management system now informing NCS staff as to which samples are most pressing to investigate. Each operative is informed of the exact samples assigned to them and a priority order given, based on benchmark statistics, so that they know exactly which samples to investigate next – this replaces a paper-based approach used previously.

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**

We continue to work with Rigaku to develop the CrystalClear software, although they have now revealed the software roadmap and will not be developing CC3.1 in the immediate future. The NCS met with several Rigaku employees, including the chief software developer (Kanda-san), at the ECM28 meeting in Warwick in August 2013 – here it was established that they would continue to develop against our system (ie SIMS and Portal integration). Kanda-san will visit the NCS in Southampton before the end of 2013.

The Electronic Management system, Portal, is being developed to support the use of mobile devices to record the diffraction experiment set up and sample handling. This work has been a component of a talk at a Satellite meeting of the ECM (Data Management & CIF) and a talk on 'Tablets in the Lab' at the recent American Chemical Society meeting (Indianapolis, Sept 2013). The ability of this system to assist in the management of applications, messaging users and user input of data is continuing to be developed.

The installation of MiniFlex and XtaLab mini diffractometers (powder and single crystal respectively) has been completed. These instruments are primarily being used for teaching purposes in Southampton - already within 2 weeks of installation of the XtaLab mini 24 third year undergraduates (and additionally a number of 4<sup>th</sup> year projects and PhDs) have collected and refined their own single crystal data. These instruments won't be extensively used by the NCS, but will be perfectly adequate for entry-level training for anyone of any background and will be the basis of some NCS 'Crystallography for Chemists' workshop activities.

### **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).

Two summer interns assisted with testing and exploring our new capability to perform high pressure and gas cell experiments.

### **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 (1/5/13, 26/5/13 8/7/13, 5/8/13, 23/9/13 and our upcoming visit on 23/10/13). 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.

The use of the Helix was the subject of a presentation at the I19 users workshop by Claire Wilson and the NCS participated and contributed significantly further at this event.

We have noticed over time that radiation decay of samples is a significant effect – considerably more so than when we used to use the Daresbury synchrotron. We have set out to examine this effect and preliminary observations were presented on a poster at the ECM28 meeting (Warwick, August 2013). We are now setting out to systematically probe this effect through a series of more structured experiments – guidance and experience will be made available to the I19 user community and the whole chemistry community when a clear understanding has been reached.

### **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**

A transition to using Cryoconnect for both cobra cooling devices and the 700 Cryostream is enabling better remote monitoring of the system by the NCS team and the service engineers at Oxford Cryosystems.

Routine maintenance has been performed on diffractometers, including the annual rebuild of the rotating anode. 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 outage (1 day downtime).
- Total facility closure due to a plant room upgrade (10 days total downtime over this and the previous reporting period).
- FRE+ filament change (1 day downtime).
- Annual rotating anode rebuild (5 days downtime).
- Chiller fans replaced (4 days downtime).
- Chillers overheating due to plant room issues (4 days downtime).

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

The cryogenic devices have suffered the following problems:

- A Cobra cold head suffered a catastrophic helium seal failure, which has now been fixed (1 month without low temp on one instrument).
- Cobras underwent routine maintenance and servicing at the same time as the annual diffractometers rebuild.

## **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**

As previously mentioned, we continue to work with Rigaku to develop CrystalClear although version 3.1 that we have been beta testing is no longer being developed. We continue to use this software as it has proved to be more effective than the older 2.0 version. The new features in CC3.1 will be folded into a full stable release of the Version 2 (CrystalClear 2.1) software, which is currently being completed in Japan. Whilst we have an operational data processing arrangement, we are investigating expanding the range of software used by the NCS to improve processing of more complicated data sets eg twins and incommensurates.

## **3. Community Activity**

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

We have hosted numerous interns over the summer period: an RSC-sponsored student to investigate the automatic processing of CIFs into the ChemSpider public database; an intern to assist in developing high pressure crystallography capability; an intern to assist in developing a gas cell for in-situ environment control and a work experience student from a local college. We also hosted a visit by Emeritus Professor David Rae (ANU, Australia), which focussed around addressing particularly difficult problems and numerous publications are in preparation as a result.

The NCS contributed material towards the CCG component of the 'Two Braggs' exhibition at the ECM28 meeting (Warwick, August 2013). This exhibit will be shown elsewhere as part of the 2014 International Year of Crystallography celebrations and the NCS is involved in planning and contributing to these celebrations.

The NCS continues to be a key aspect of University of Southampton Open Days, weekly tours for UCAS candidates and department outreach events. Plans are being made for the NCS to become more fundamentally involved with outreach to a great age range, as part of the school's outreach programme.

## **B. Publicity**

The NCS has started a renewal of all its publicity materials with a redesigned flyer which highlights the ease of use of the service and quick turnaround times for data. Over 400 of these were sent to over 50 departments across the country for distribution to interested colleagues. These included materials and pharmacology departments, alongside chemistry departments and combined faculties.

The further renewal of our publicity material will be a discussion point at the upcoming management panel meeting.

## **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.

Increased focussed user engagement is planned through a) invites to practising crystallographers to come and spend a day at the NCS with their most challenging crystals in order to see what we are capable of and b) a 'crystallography for chemists' workshop where we will tutor on topics such as 'understanding a crystal structure experiment/result', 'what information you can get out of a crystal structure(s)' and 'tools for presenting crystallographic results' – this will primarily be targeted towards PDRA's and PhD students. This will be a discussion point at the next MAP meeting.

## **4. Preview of next period**

### **A. Preview of availability over next 6 months**

Aside from University closed days over the bank holiday periods, there is no planned closure.

**B. Preview of upgrade over next 6 months**

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

**Appendix 1: KPI Data (All current data for 01/05/13 – 14/10/13)**

		This Period	Nov 2012 – May 2013	May – Oct 2012	Oct 2011 – May 2012	May – Oct 2011	Oct 2010 – May 2011	May – Oct 2010
Number of NCS Users (active)		64	67	65	67	68	56	39
Number of NCS Projects		64*	67*	65*	67*	68*	69	44
Availability of facility for NCS use (days)		92	108	110	106	108	105	106
Actual equipment uptime and use of facility by or for NCS		78	94	100	92	77	98	102
Number of NCS samples processed	Total	376	432	417	421	384	404	342
	At Southampton	316	376	353	333	304	342	273
	At DLS	60	56	64	88	80	62	69
Number of NCS data collections performed		223	260	285	301	238	280	221
Number of Full structure determinations performed		153	172	132	120	146	124	52
Number of NCS samples outstanding		237	201	189	174	240	302	184
Waiting for examination		63	83	16	66	20	17	53
Processing		137	100	167	89	167	198	96
Waiting for return to users		37	18	6	19	53	87	35
Number of User data sets that were completed within 1,2,3,>3 attempts	1 attempt	284	349	289	330	208	204	Unable to report against this at the time
	2 attempts	87	64	107	71	44	30	
	3 attempts	5	15	17	15	6	3	
	More than 3 attempts	0	4	4	5	0	6	
	Unreported	0	0	0	0	52	107	
Number of User complaints received		0	0	0	0	0	0	0

Number of NCS research outputs		52?	44	44	49	55	73	26
Number of NCS users of the training programme		0	0	1	1	3	7	8
Number of samples classed as routine or difficult	Routine	185	190	265	265	166	172	Unable to report against this at the time
	Difficult	131	135	88	57	55	43	
	Synchrotron	60	40	64	88	80	62	
	Unreported	0	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 = 86%

Medium priority = 92%

Low priority = 92%

### **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 = 85%

### **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 = 85%