

**UK National Crystallography Service Biannual Report 4**

**Period covered: 01/11/2011 – 30/04/2012**

**Contents**

- 1. Preface – Simon Coles**
- 2. Operations and Logistics**
  - a. Upgrade of Laboratory**
  - b. Overview of service use**
  - c. Staff**
  - d. NCS Synchrotron component**
  - e. Review of user complaints/disputes and resolutions**
  - f. Equipment- Technical Issues**
  - g. Sample Issues**
  - h. Data Processing Issues**
- 3. Community Activity**
  - a. Training and Outreach**
  - b. Publicity**
  - c. User Liaison**
- 4. Preview of next Period**
  - a. Preview of availability over next 6 months**
  - b. Preview of upgrade over next 6 months**
- 5. Appendices**
  - a. Appendix 1: KPI Data (All data for 01/11/11 – 04/04/12)**
  - b. Appendix 2: Statistics against benchmarks**

## **1. Preface**

This period has predominantly been concerned with familiarisation with the new instrument base, which is entirely different to that previously used either by the NCS or in Southampton. Accordingly there have been a small amount of teething problems with the instrumentation, but the majority of staff time has been concerned with training on diffractometer control and software use. Additionally, as with all new instrumentation, one has to develop an understanding of maintenance schedules and requirements and we have had to evolve entirely new laboratory working procedures.

We are capitalising on our world-leading position by building our scientific relationship with the equipment providers – Rigaku. An intensive working visit from the VP for Software Development has resulted in a very close relationship where the NCS is beta testing the major upcoming release of the new CrystalClear software. Moreover we are collaborating closely on developments specifically designed for the operation of the NCS, but which will be available for the whole community and also generate a journal article.

The NCS was the driving force behind a workshop for advanced image processing using the EVAL software held at Diamond, where the developers of the software were present to tutor in hands-on exercises specifically concerned with Rigaku format data. This workshop was attended by about 20 researchers mainly originating from Southampton, Diamond, Oxford and Liverpool.

The link to the facilities at Diamond has been developing and we now have established appropriate procedures for mounting and screening highly sensitive samples that improve the efficiency of working on the beamline and transfer from Southampton. We continue to improve our customer interface by rolling out new aspects of our information management system – during this period we have developed the electronic submission, sample tracking and data download aspects.

## **2. Operation and Logistics**

### **A. Upgrade of Laboratory**

The new laboratory installations were completed in the previous reporting period, however there have been a moderate, and expected, number of teething problems. More significantly this period has been concerned with understanding and establishing a new working procedure, refining this and developing administrative procedures around it. This has involved developing a new physical workflow in the laboratory, understanding the optimal approaches for routinely using the software and designing the peripheral data management and auditing systems to interact with this new set up. Despite having to evolve this laboratory practice the general levels of throughput are very similar to those we were achieving before the new equipment install despite the type of samples we are now examining being considerably more challenging.

The only imminent upgrade remaining is that of the diffractometer control software. Rigaku have been using the 2.x version of CrystalClear for many years, however a 3.x version has been promised ever since placing the order to buy their equipment. The NCS installation was fortunately timely in that some of our requirements could be fed into this software development, so not only have we developed these ideas and requirements but also we have become their primary testers and accordingly steering the software in a mutually beneficial direction. The NCS developments are partly concerned with aspects of diffractometer control and data processing, but also significantly with data and facility management – this latter point has resulted in the development of the SIMS (Sample Information Management System) specification, which will be fully incorporated into the software and published as a standard for crystallographic data management.

### **B. Overview of service use**

This period has seen a return to operational levels similar to those we were achieving prior to the new laboratory install as evidenced by the number of samples processed and acclimatisation to the new equipment is in part demonstrated by the reduction in the number of outstanding samples. An increased number of samples processed at Diamond reflects the smoother and more efficient operating procedure at the beamline that has evolved and the increased screening power of

the new Southampton-based instrumentation. The number of user applications now appears to have levelled out after the introduction of the Rapid Access route to complement the regular call.

The remaining statistic of note is the number of data sets processed within 1,2,3,>3 attempts, which shows that there is a larger number of challenging samples that are being successfully completed and that a greater percentage of samples are being successfully examined on the first attempt. Given that there are several attempts on some samples it is worthy of note that the actual number of datasets collected was of the order of 520 (on the 421 samples processed).

### **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. NCS has had 5 days (each of 24 hours comprising a block of 3 shifts) allocated in the last 6 months (2/11/11, 14/12/11, 9/2/12, 26/2/12 and 24/3/12). As mentioned in a previous report the direct involvement of an NCS team member at Diamond has resulted in improved understanding and better scheduling of NCS beam time to give well distributed dates for visits allowing regular access wherever possible.

Typically 20-25 samples have been screened during a visit and up to approximately 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 new instrumentation in Southampton.

The DLS based NCS team member means that we continue to make the most of additional time available and give input and have involvement in developments on the beamline. We have used special beam conditions and additional time on the 25/3/12 adjacent to our allocated time on the 24/3/12 to investigate the transfer of samples which have been cold mounted ahead of the beamtime to a nitrogen filled robot dewar in an effort to continue to improve the efficiency of the use of our beamtime.

### **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**

Given the new instrumentation base, it is not possible to have developed a routine preventative maintenance schedule until fully acquainted with the machine and its operation has stabilised. However some routine maintenance, such as filament replacement, has been performed. In addition the following diffractometer-based issues have arisen in the reporting period:

- FRE+ HF/VHF. Anode water seal replaced after catastrophic failure and drive belt replaced (5 days downtime),
- FRE+VHF. Goniometer replacement (2 days downtime),
- Cu007. On-going chiller issues - during this period a plant room extractor fan has been replaced and the plant room has been cleared to improve airflow. The situation continues to be monitored (2 days downtime); Ferro-seal replacement, which involves removal of the anode and a loan anode being temporarily installed (4 days downtime),
- SPIDER. X-ray tubes running hot as a result of gradual chiller failure - new chiller installed.

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

The cryogenic devices have suffered the following problems:

- Service and compressor replacement for Cobra nitrogen generator,

- On-going helium leak with Cobras, resulting in intermittent loss of low temperature,
- Cobra for FRE+ VHF returned to Oxford Cryosystems for repairs to manifold

We have worked around the above issues as much as possible, but the loss of low temperature severely limits the type of sample that can be run under these conditions.

### **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),
- 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.

### **H. Data Processing Issues**

Previously reported processing issues have largely been resolved and we now have data processing at reasonable speeds. As outlined above, we are currently beta testing the new version (3.1) of the CrystalClear software. In collaboration with Rigaku, we have developed the SIMS format, which we intend to implement in order to link our data management system with the diffractometer software - this format will be published in an academic journal and thus be made available to the community.

## **3. Community Activity**

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

The NCS has hosted 1 visit since November. A member of Sofia Pascu's group (Bath University) visited the Southampton laboratory to learn more about the technique and in particular the advanced facilities hosted at Southampton.

The service hosted a sixth form student on a work-shadowing day. Three third and fourth year undergraduate students have embarked on research projects hosted by the service during the academic year, and the service will host a summer intern in the coming period. We have also continued to take part in University of Southampton Open Days and weekly tours of UCAS candidates.

### **B. Publicity**

The NCS continues to publicise the services it offers. This period the focus has been on promoting the advanced facilities offered by the new laboratory. The service banner was displayed at the RSC Macrocycles conference and the service was advertised during a talk at international Supramolecular Chemistry conference. A new NCS poster was submitted and accepted to the Warwick BCA meeting in April and it is hoped to display this poster at many more conferences. The poster focuses on the crystal size we are now able to run and comparison data between the new equipment and DLS.

### **C. User Liaison**

The main focus for this period has been the development of the portal service which will allow users to submit submission information electronically and to monitor the progress of their samples through the service workflow. We are hopeful this will be launched in the coming period and will be working with our users to develop this tool. We continue to act on suggestions and comments from the users to refine and improve our systems.

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

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

Asides from University closed days over the bank holiday periods, there is no scheduled shutdown of the facility and therefore full availability is expected.

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

We expect to upgrade to CrystalClear 3.1 over the next 6 months.

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

		This period (1/11/11 – 4/4/12)	May – Oct 2011	Oct 2010 – May 2011	May – Oct 2010
Number of NCS Users (active)		67	68	56	39
Number of NCS Projects		67*	68*	69	44
Availability of facility for NCS use (days)		106	108	105	106
Actual equipment uptime and use of facility by or for NCS		92	77	98	102
Number of NCS samples processed	Total	421	384	404	342
	At Southampton	333	304	342	273
	At DLS	88	80	62	69
Number of NCS data collections performed		301	238	280	221
Number of Full structure determinations performed		120	146	124	52
Number of NCS samples outstanding		174	240	302	184
Waiting for examination		66	20	17	53
Processing		89	167	198	96
Waiting for return to users		19	53	87 (includes a number of radioactive samples and schlenks which are awaiting special return to the user as a complete set)	35

Number of User data sets that were completed within 1,2,3,>3 attempts	1 attempt	330	208	204	Unable to report against this at the time
	2 attempts	71	44	30	
	3 attempts	15	6	3	
	More than 3 attempts	5	0	6	
	Unreported	0	52	107	
Number of User complaints received		0	0	0	0
Number of NCS research outputs			55	73	26
Number of NCS users of the training programme		1	3	7	8
Number of samples classed as routine or difficult	Routine	265	166	172	Unable to report against this at the time
	Difficult	57	55	43	
	Synchrotron	88	80	62	
	Unreported	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

Level of samples which achieved this benchmark = 84%.