

UK National Crystallography Service Biannual Report 5

Period covered: 01/05/2012 – 31/10/2012

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1. Preface

Period 5 is the first where we can truly consider the NCS to be fully operational, the staff have been in position for at least a couple of years in all cases and we can now say that we have a deep knowledge of the instrument base and a working procedure fully established, particularly with Diamond.

We are continuing to capitalise on our world-leading position by building our scientific relationship with the equipment providers – Rigaku. We have just had our second working visit from the VP for Software Development, which achieved the two primary goals of a) completing another round of beta testing of the new version of the diffractometer control software, CrystalClear and b) beginning the implementation of the data management protocols we have defined with them over the last year. We recently hosted a very successful visit from the President of Rigaku Americas and the President of Rigaku Life Sciences Division where our collaborative relationship was discussed heavily.

We are currently hosting a 2-month visit by Emeritus Professor David Rae from ANU, Canberra. Professor Rae's career has been devoted to developing very novel approaches to analysing particularly difficult problems and he is known for developing several new methods. The NCS hosted a visit by the software development team for the Olex2 project, with the aim of understanding these approaches and coding them into their software for the use of future generations. The meeting was highly successful and several new methods for handling difficult crystallographic problems will be incorporated into the Olex2 software in the coming months – we expect at least two publications to also arise from this collaboration.

2. Operation and Logistics

A. Overview of service use

Service use remains constant, with number of users and samples processed at a similar level to the last allocation period. There has been a slight increase in the number of full structures being processed, but it is too early to say whether this is a growing trend. Taking into account the number of samples which require more than one attempt, the number of datasets collected is around 570. Accordingly this is reflected in an increase in the number of samples classified as 'difficult'. The lower number of samples processed at DLS during this period is explained by the final day of beamtime in this Allocation Period being scheduled after the compilation of this report. This is further exacerbated by a poor alignment of the beamline resulting in a complete lack of useable data arising from another visit. The rapid access scheme continues to attract new users between calls, the majority of whom go on to apply during the next regular call.

B. Upgrade of Laboratory

Graham Tizzard has been leading the service team in the beta testing of the new diffractometer control software, CrystalClear 3.1, which the service has been using since the beta 2 release in March 2012. The NCS has been closely involved with Russ Athay, the software developer (Rigaku VP, Crystallographic Software), by feeding in bug reports and taking part in WebX sessions to directly discuss issues. Russ Athay has also visited during this period to discuss and implement this upgrade in person.

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 – we have just completed the final allocation period in this block and have submitted an application for the next 2 year BAG.

NCS has 6 days (each of 24 hours comprising a block of 3 shifts) allocated in the current 6 month period (28/05/12, 02/07/12, 29/07/12, 13/08/12, 1/10/12 and 28/10/12). One of these days (29/09/12) did however not produce any useable data due to poor alignment of the instrument and the final date is yet to arrive at the time of writing. As mentioned in previous reporting, 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 work with the robot dewar has progressed over the last 6 months with the autofill for the nitrogen having been set up and calibrated and the NCS team were closely involved in this development.

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, particularly our first annual anode service, has been performed. The following summary of diffractometer-based issues and resultant instrument downtime have arisen in the reporting period:

- FRE+ filament change (1 day downtime).
- FRE+ anode service (5 days downtime).
- FRE+HF: A framegrabber PC suffered a terminal failure. Accordingly new framegrabber PC's were installed on both diffractometers (during the anode service) as a preventative measure (5 days downtime).
- 007HF: This instrument continues to have problems with its chiller, which during warm weather meant that the diffractometer could only be run when the temperature outdoors was below 25°C. A partial solution has been implemented by relocating the chiller and new pipe work between chiller and diffractometers being installed. This instrument is currently fully operational but we are unable to determine the long-term viability of this solution until the warmer weather returns.
- SPIDER. A new chiller was installed. The control PC suffered a terminal failure through loss of its hard drive and motherboard. We are in the process of installing a new control PC with associated hardware and software (this instrument has not been operational since 4th September)

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

The cryogenic devices have suffered the following problems:

- Both cobras have had vacuum re-pumped and the helium has been refilled in one cryodrive

- There is an on-going helium leak with Cobras. Our investigation of this problem resulted in caps being fitted, which have reduced the leakage to a level where replacement of helium is the simplest option. Intermittent faulting of the instruments has been resolved as a result.
- There is an on-going issue with the nitrogen generator for the Cobra units - a membrane needs replacing and it is currently running at reduced capacity (although this does not affect the operation of the Cobra's it does place an unnecessary strain on the generator). A replacement membrane system has been ordered and this issue will be resolved in November 2012.

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

The NCS was a key exemplar and contributor to a workshop on Diffraction Data Deposition organised as a satellite of the ECM27 meeting in Bergen. The NCS continues to be at the forefront of development and practice in data management matters – spearheading in the area of crystallography and acting as an example to other fields.

The service suffered a significant hardware failure of the main data storage server. No data was lost but data processing was affected for several weeks until the replacement solution was implemented. The replacement system ensures we should not experience a repeat of this issue.

As previously mentioned this period has also seen extensive testing of Crystal Clear 3.1.

3. Community Activity

A. Training and Outreach

The NCS has hosted 1 training visit since May. Joseph Wright (UEA) came to visit the new laboratory and discuss the extended capabilities that the new instrumentation brings to the service.

The service also hosted a longer visit from Andreas Reiber (Universidad de los Andes). This month long visit concerned not only training in the technique and data work up, but also the running of a national service which Andreas is considering proposing to run in Columbia.

The service hosted a sixth form student on a work-shadowing day as well as a week of work experience for another a-level student. We have also taken part in the UoS Chemistry research experience day for undergraduates.

We have also continued to be a key aspect of University of Southampton Open Days and weekly tours for UCAS candidates.

B. Publicity

The NCS continues to publicise the services it offers. The new service poster has been displayed at several conferences and the service continues to send flyers advertising the service to departments across the country. There will be a review and redesign of publicity flyers during the next period in order to maintain their impact.

Simon Coles gave the introductory lecture at a specialist satellite meeting of the European Crystallography Meeting (Bergen) concerned with Diffraction Data Deposition. This lecture presented the NCS as a leading light in the area and the outcome of the meeting was that an IUCr working group is set up to move the agenda forward – SJC and the NCS are at the forefront of this movement.

Simon Coles will give a seminar on the NCS at Sussex Chemistry Department on 31st October 2012.

C. User Liaison

Unfortunately the time scale for the full roll out of the Portal management system to our users has suffered a setback. A review of this will be held in the new allocation period, with a scheme of work being drawn up and it is hoped to complete this work during the upcoming period. The system is however being operated very successfully to support applications, manage samples, generate usage statistics and provide information to users and this final scheme of planned work will be concerned with electronic submission of sample data and full provision of results data.

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

Aside 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 from our beta test version to the full release version of CrystalClear 3.1 in Feb 2013.

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

		This period (1/5/12 – 16/10/12)	Oct 2011 – May 2012	May – Oct 2011	Oct 2010 – May 2011	May – Oct 2010
Number of NCS Users (active)		65	67	68	56	39
Number of NCS Projects		65*	67*	68*	69	44
Availability of facility for NCS use (days)		110	106	108	105	106
Actual equipment uptime and use of facility by or for NCS		100	92	77	98	102
Number of NCS samples processed	Total	417	421	384	404	342
	At Southampton	353	333	304	342	273
	At DLS	64	88	80	62	69
Number of NCS data collections performed		285	301	238	280	221
Number of Full structure determinations performed		132	120	146	124	52
Number of NCS samples outstanding		189	174	240	302	184
Waiting for examination		16	66	20	17	53
Processing		167	89	167	198	96

Waiting for return to users		6	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	289	330	208	204	Unable to report against this at the time
	2 attempts	107	71	44	30	
	3 attempts	17	15	6	3	
	More than 3 attempts	4	5	0	6	
	Unreported	0	0	52	107	
Number of User complaints received		0	0	0	0	0
Number of NCS research outputs			49	55	73	26
Number of NCS users of the training programme		1	1	3	7	8
Number of samples classed as routine or difficult	Routine	265	265	166	172	Unable to report against this at the time
	Difficult	88	57	55	43	
	Synchrotron	64	88	80	62	
	Unreported	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 = 80%

Medium priority = 86%

Low priority = 96%

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 = 88%

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 = 82%