

Annual Report for EPSRC National Research Facilities

Facility: EPSRC National Crystallography Service

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Director: Professor Simon Coles

Facility Manager: NCS Operations Manager, Dr Graham Tizzard; NCS Service Support Manager, Sarah Milsted

Description of the Facility

The NCS facility is amongst the most powerful and highest throughput of its type in the world. Its core business is to handle and examine samples that a typical academic crystallography unit cannot. The service focusses on the technique of single crystal diffraction applied to samples submitted by UK Chemistry, and related, disciplines. This technique provides the most detailed characterisation possible for solid-state chemicals and the NCS is dedicated to investigating the smallest and weakest scattering crystals that challenge the community. In addition to structure determination the NCS also provides advanced crystallographic studies using charge density, variable temperature, high pressure and gas cell techniques. A commercial service for the industrial sector is also available. Professionalism is a trademark of the NCS – it prides itself in exemplary and rigorous processes for sample handling and tracking, data management and publishing, a bespoke user interface and an ability to deliver to a demanding set of KPIs. Sample turnaround time is exemplary, meaning that this technique is often used to progress research, as well as producing the characterisation of its final outputs. Users can select either raw/derived data being provided for their own analysis, or a full analysis service, where a sample is taken through to publication. The ability to handle the most demanding samples in the Southampton laboratory is a key feature, and provides an effective filter for a follow-on synchrotron service. The NCS has regular access to the world-leading I19 beamline at Diamond Light Source.

The track record of the NCS team is exemplary, with a proven ability to deliver a high profile and high throughput service. The facility has an exceptional publication record and is amongst the world's most prolific contributors to the Cambridge Structural Database. The NCS is well established in the research community and leads in many aspects, both nationally and globally.

Contract/Grant Period and Costs

Contract Term: 3+2 years

Start Date: 01/11/2016

End Date: 31/10/2019 (3 years) / 31/10/2021 (5 years)

Total cost: £3,900,460.96 ex VAT

Total capital cost: £166,024.01 ex VAT

Spend on track? Y

Key Performance Indicators (KPIs) and Service Level (SLs)

			SLA Level				
Type	Description	Time for Performance	Green	Amber	Red	1st Sep 2017 - 31st Aug 2018	Directors Comments
RMI	Total number of all Users	Period associated with specific report	N/A	N/A	N/A	66 (Routine service active users) / 51 (Training schemes)	
RMI	Spectrum of user types	Period associated with specific report	N/A	N/A	N/A	66 (Routine service active users) / 51 (Training schemes) / 6 (Commercial clients) / 8 (Advanced techniques)	
RMI	Number of University / Research Groups Involved	Period associated with specific report	N/A	N/A	N/A	35 (University) / 68 (Research group)	
RMI	Percentage of Access Requests Accepted	Period associated with specific report	N/A	N/A	N/A	100% (Routine service) / 100% (Advanced Technique)	
RMI	Percentage of equipment time dedicated to different access modes	Period associated with specific report	N/A	N/A	N/A	65% (Routine service, academic) / 7% (Commercial) / 20% (Host institution) / 8% (Advanced techniques)	
RMI	The number of data sets processed		N/A	N/A	N/A	881	
SL	Percentage of User enquiries responded to within Stated Window	2 working days	95% and above	>90% but <95%	90% or less	100%	
SL	Percentage of Access Requests Responded to within Stated Window	2 working days	95% and above	>90% but <95%	90% or less	100%	
SL	Percentage of Training Requests Responded to within Stated Window	2 working days	95% and above	>90% but <95%	90% or less	100%	

SL	Percentage of Training Requests Delivered within 3 months	3 months	95% and above	>90% but <95%	90% or less	100%	
SL	Number of Customer Complaints (expressed as a percentage of the Total Number of User Approvals made within the period)	Period associated with specific report	Less than 5%	5-10%	Over 10%	0%	
SL	Percentage of customer complaints resolved within Stated Window using the Dispute Resolution Plan.	Period associated with specific report	95% and above	>90% but <95%	90% or less	n/a	
SL	Percentage Uptime/Downtime per instrument of Total Available Time within Period	Period associated with specific report	95% and above	>90% but <95%	90% or less	Mo FRE+ VHF 79%	This downtime is largely a result of a collision which resulted in the detector module being sent back to Rigaku Japan for repair (2 months, but at least one other diffractometer was still operational throughout this whole period).
SL	Percentage Uptime/Downtime per instrument of Total Available Time within Period	Period associated with specific report	95% and above	>90% but <95%	90% or less	Mo FRE+ HF 64%	A persistent and ongoing goniometer issue, after numerous on-site repair attempts, required shipment back to Rigaku Japan. This is now in final factory testing and due to be returned imminently.
SL	Percentage Uptime/Downtime per instrument of Total Available Time within Period	Period associated with specific report	95% and above	>90% but <95%	90% or less	Cu 007 HF 92%	This instrument suffered a goniometer issue, which required new components and was repaired on site. At least one other diffractometer was still operational throughout this whole period.
SL	Percentage of Access Costs recovered	Period associated with specific report	10	6	2	n/a this year	
SL	Number of Publications	1 year	30	20	10	35	
SL	Number of publicity activities per year	1 year	10	8	6	10	
SL	The time from arrival of a sample to logging in and	within 2 working days	95% and above	>90% but <95%	90% or less	100%	

	informing a User of receipt						
SL	The time a sample is in the queue	From logging in a sample to examination: High Priority sample = 10 working days	95% and above	>90% but <95%	90% or less	95.10%	
SL	The time a sample is in the queue	From logging in a sample to examination: Medium Priority samples = 20 working days	95% and above	>90% but <95%	90% or less	95.60%	
SL	The time a sample is in the queue	From logging in a sample to examination Low Priority samples = 30 working days	95% and above	>90% but <95%	90% or less	96.20%	
SL	Time from examination to end result	Data collection = 5 working days	95% and above	>90% but <95%	90% or less	95.10%	
SL	Time from examination to end result	Full Structure Analysis= 20 working days	95% and above	>90% but <95%	90% or less	95.10%	

Users

User statistics.

NCS users fall into six categories in this reporting period:

- Research Group Principal Investigators. 57 NCS PI's, of which 8 were new users. We do not record the number of students for each PI accessing the NCS, however an accepted estimation an average user would have 2-3 students (some groups are into double figures).
- UK service crystallographers. 9 users fall into this category, which is an increase on the 2017 report. This usage mode supports large numbers of researchers as departmental crystallographers access the NCS to analyse difficult samples from across *their* entire user base.
- Collaborators using Advanced Techniques. 5 users performed pilot studies in this period.
- Industrial Users. There are 6 companies regularly accessing the commercial service. All of these are from the health and pharmaceutical sector.
- International Collaborations. These are detailed in the International Interactions section.
- People accessing the training programme. These are detailed in the Societal Impacts section.

The NCS users originate from 35 different institutions. 82% of our users are based in chemistry departments, while the remaining 18% originate from materials science, defence materials, pharmacy and biochemistry departments. The geographic spread of users is even and touches all regions of the UK. User groups cover 17 Research Areas (an increase of 4 on the 2017 reporting period) and these are detailed in the Strategic Fit section of this report. Some

time on the facility equipment is allocated to the host institution and the facility is regularly used in local training, demonstration and outreach events.

User satisfaction survey

Over 60% of active users in each call (50 respondents in total) replied to a user satisfaction survey as follows:

- The NCS is easy to use, e.g. ease of application, clear sample forms and guidance [4.85/5]
- The NCS is responsive to user requests, e.g. rapid turnaround, additional data collections [4.90/5]
- The NCS is helpful with regard to publication, e.g. all data necessary provided, assistance with crystallography sections etc. as per type of service requested [4.85/5]
- I would recommend the NCS to a colleague [4.96/5]

The response rate for the survey is up this year and all the scores have either remained in line with previous years or increased ('ease of use' and 'would recommend' have increased).

Some representative comments illustrating the value of our services to our users include:

- "Many of our papers would not have been publishable without the help of your researchers and they cover all bases including helping with CIF, CheckCIF, discussions around bond lengths and angles for we x-ray illiterate!"
- "NCS provide an excellent and invaluable service to the UK academic community. They have been hugely helpful in advancing our science and we are very grateful."
- "Excellent service, communication with the personnel at the service is always positive and very helpful."

Scientific Excellence

Publishing Highlights

The record for journal publications in this period is very similar to that of last year. A 2012 perspective article on the future direction of crystallography (Coles & Gale, *Chem Sci*, 2012, 3, 683-689) outlined the NCS operation. As of May/June 2018, this highly cited paper continues to be in the top 1% based on Web of Science's highly cited threshold for the publication year and field. In this reporting period 34 co-authored papers were published and this is supplemented by at least the same number of papers also arising from our data collection only service. The top 5 papers of the period during this period are summarised as:

- 1) Noncovalent Interactions of π Systems with Sulfur: The Atomic Chameleon of Molecular Recognition (Motherwell, UCL) **ANGEWANDTE CHEMIE** <https://doi.org/10.1002/anie.201708485>. This paper links structural data with solution state NMR to understand the interaction between a thioether sulfur atom and a pi system to provide the key component to a "top pan molecular balance".
- 2) Trimerisation of Carbon Suboxide at a Di-titanium Centre to form a Pyrone Ring System (Cloke, Sussex) **CHEMICAL SCIENCE** <https://doi.org/10.1039/c8sc01127c>. This paper reports an unprecedented reaction and uses structural data to underpin the DFT calculations which reveal its mechanism.
- 3) Modular $[\text{Fe}^{\text{III}}8\text{M}^{\text{II}}6]^{n+}$ ($\text{M}^{\text{II}} = \text{Pd}, \text{Co}, \text{Ni}, \text{Cu}$) Coordination Cages (Breachin, Edinburgh) **INORGANIC CHEMISTRY** <https://doi.org/10.1021/acs.inorgchem.7b02674>. This work reveals enhanced antiferromagnetic exchange when moving from a monomer to heterometallic cage compound and involved a considerable amount of very challenging crystallography.
- 4) Ligand tuneable, red-emitting iridium(III) complexes for efficient triplet-triplet annihilation upconversion performance (Pope, Cardiff) **CHEMISTRY A EUROPEAN JOURNAL** <https://doi.org/10.1002/chem.201801007>. This paper reports a study where a ligand was systematically altered in a family of complexes and crystallography was key in underpinning the DFT calculations that were able to predict their triplet emitting wavelengths.
- 5) Stereoselective and Stereospecific Reactions of Cobalt Sandwich Complexes: Synthesis of a New Class of Single Enantiomer Bulky Planar Chiral P-N and P-P Ligands (Richards, UEA) **CHEMISTRY A EUROPEAN JOURNAL** <https://doi.org/10.1002/chem.201705113>. The complexes reported in this paper contain a new class of ligand for application in asymmetric catalysis and crystallography contributed vitally to understanding their exact steric and conformational arrangements.

Advanced Techniques Highlights

The advanced techniques mode of access to the NCS was introduced in the last reporting period and of these were due to be phased in over a two year period. They are therefore in differing states of maturity and embedding, with variable temperature being the most ready, through to high pressure where we have just finished establishing the support laboratory required to begin experiments. We will therefore pick highlights accordingly – and it is important to bear in mind that there is considerably more work-up and analysis required for these methods and so there is a longer lead in time to publication.

Variable temperature experiments in this reporting period have yielded a number of exciting results.

- We performed an extensive dehydrolysis study on a MOF material for Drs Claire Wilson and Mark Murrie (University of Glasgow) where we were able to obtain data of a sufficient resolution that it was possible to observe the sequential loss of two bound water molecules while the sample retained its crystallinity and the structure of the complex altered accordingly. A collaborative paper is now in preparation.
- With Dr Ian Gass (University of Brighton) we structurally tracked a phase change where it was possible to monitor a high spin to low spin transition through changes in bond lengths and angles. A manuscript is in early stages of preparation.
- A broad ranging set of studies has been planned, and embarked on, with Davide Bonifazi (Cardiff University). The work is based on the concept of freely rotating molecules in the solid state and chemically tailoring these to reduce this motion and thereby understand the driving force behind it. Some promising, reproducible results have been generated and now inform the design of the next stage of work. We are planning work on a whole range of chemically similar systems which do/may exhibit similar behaviour.

Working on studies where gases interact with crystalline solids is now established at the NCS and is rapidly generating results and interest. The highlight of this work is a strong and well established collaboration with Prof Andrew Weller (Oxford), where we have drawn up an ambitious programme of work. By reacting a gas with a crystalline coordination complex, due to the constraints of the lattice, we can produce ‘impossible complexes’ i.e. those that simply wouldn’t be favourable to form in solution. The Weller group regularly visit the NCS and integrate with our staff. The first results for publication of work arising from this collaboration have been submitted to JACS. In this period we have initiated two Charge Density projects – one with Sally Freeman (University of Manchester) and Carl Schwalbe (University of Aston) aimed at understanding biological activity in pharmaceuticals and another with Annie Powell (KIT, Germany) attempting to relate coordination mode to magnetic properties in an exceptionally challenging system (for the Charge Density approach).

The support laboratory for high pressure studies (gasket drill and fluorescence spectrometer) is now commissioned and we will be conducting studies of this nature in the forthcoming period.

Methods Development:

We have an ongoing project to increase understanding of radiation induced decay in small molecule samples and therefore develop mitigating data collection strategies. The first phase of this work has been completed and is about understanding the effect and is in the final stages of preparation for publication. In this period we have begun an international collaboration concerned with using our methodology at other synchrotron radiation sources and to this end we have hosted a visit from our international collaborators where we shared knowledge about the effect and trained them how to measure it. We have now designed an international standardisation project – for which we have recently been awarded beamtime at the Advanced Photon Source (Chicago, USA) to begin work.

Community Leadership:

The Director is a member of the International Union of Crystallography’s Committee on Data (CommDat) and is heading up a global community consultation on the curation, use and reuse of raw data in chemical crystallography. This work will inform policy at IUCr which will be cascaded out to all national bodies and journal publishers – a survey has been drawn up and the results of this will be synthesised in the forthcoming reporting period.

During this period Simon Coles has also become the Chemistry lead for GO FAIR, an EU initiative aimed at making research data open. This work has led to a significant involvement in driving this agenda forward with the International Union of Pure and Applied Chemistry.

The NCS was at the heart of a CDT application with Southampton colleagues from Physics, Engineering and Biological Sciences – Diamond were primary partners and extremely supportive of the bid. The bid was led by the Director and

had the theme of uniting and cross-fertilising X-ray based diffraction and imaging technologies. Unfortunately the application didn't get past the outline stage, but the process did highlight new methodologies and engage a range of different commercial companies (from Nikon to Rolls Royce). Arising from this bid is an exploratory collaboration with Nikon, which is looking to bring the Diffraction Computed Tomography technique to the home laboratory – and be applicable to molecular materials. The Director has had several planning meetings with the company and has prepared samples for measurement and method development which are currently being assessed in Denmark.

Case Study

We include a case study on the work of Dr George Kostakis, University of Sussex, with this report. Dr Kostakis has used the NCS for several years and is a prolific submitter of samples and uses all modes of the service i.e. data collection only for non challenging samples, full structure solution (for the majority of submissions as his chemistry often produces challenging crystals) and he also regularly sends students to our training activities. George works on functional coordination compounds, predominantly for magnetism and memory storage applications and the NCS has collaboratively published 10 papers with him since 2015. The main aspect of this research is using the topology of the structure of a coordination compounds as a descriptor that can be linked to physical properties. This work therefore crucially relies on crystallography and really gains worth as more structures are determined – and so for both aspects the NCS plays a fundamental role.

Impact: Training, Outreach and Societal Impacts

Training courses and workshops.

Three major events were conducted during the reporting period:

- An 'Approaches to twinned crystals' workshop was held in January and had 10 attendees. This was designed for those with considerable crystallographic experience, but requiring an ability to address more complex problems.
- A 'Using OLEX² software' workshop for intermediate crystallographers in May had 22 attendees.
- A 'Hirshfeld Atom Refinement (HART) with OLEX²' workshop in June was designed for experienced crystallographers and had 19 attendees, including three from overseas.

Feedback gathered from all course attendees has been excellent and we continue to respond to this and community feedback when planning events, which has influenced the scheduling of workshops going forward.

Activities to promote the facility beyond its core user base.

The Director has promoted the NCS to a number of different audiences outside of the existing/core user base e.g. seminar in Oxford Physics; GCRF planning workshop in Africa. The NCS sponsored an RSC early career researchers Macrocycles & Supramolecular Chemistry symposium in December 2017 where the director gave a talk. Simon also gave a talk at the UK-MOF event (Southampton) in April which highlighted the advanced techniques and their application in this research area and which factored into an application to become an official RSC section.

Partnership with the Knowledge Centre for Materials Chemistry (KCMC) allowed us to promote the NCS to commercial and academic users at the Materials Research Exchange 2018. Collaboration with the KCMC on a CDT outline bid (led by the Director) meant working with the Centre for Process Innovation (host of the KCMC), amongst other innovation and commercial organisations and this led to significantly enhanced visibility for the NCS in this sector.

Public engagement.

In June the NCS took on a BCA-funded project to develop a series of exemplar resources for Ada Lovelace Day. Centred on female crystallographers of our time, these resources include posters, postcards, screen advertising and a talk and all of these will be made available and promoted through the BCA so that anyone can readily run an outreach event of this nature.

The NCS was involved in many schools outreach events during the period. Events included 'Hands-on diffraction' with sixth form students (July); 'King Edwards School Science Festival' workshops (June) and work shadowing experiences for 6th formers from across the region. The NCS has developed a portfolio of outreach activities which can be delivered to audiences from primary school through to a-level students and also to the adult audiences through events such as Pint of Science – these are being promoted and made available to all through a BCA project spearheaded by the Director. Simon Coles is the lead for the BCA, and now the global crystallographic community, on a project to coincide with the International Year of the Periodic Table – the periodic table will be illustrated through crystal structures and built up

through news items and releases for each element as the year proceeds. Support has been secured from CCDC and applications for funding have gone into the RSC and STFC to support the development of learning materials for schools and the general public.

Facility staff training and career development.

Dr James Orton was appointed as a research technician primarily responsible for commercial work and progressed through probation and a range of training activities, including our own workshops. Dr Wim Klooster progressed through probation and completed a one week residential charge density training at Laboratoire de Cristallographie & Résonance Magnétique & Modélisations (CRM2) as well as participating in the international Quantum Crystallography summer school (Erice, June). All members of the team attended the BCA conference in Warwick and Sarah Milsted attended the BIG Network STEM communicators' conference. The HART workshop hosted by the NCS was a training opportunity for all our staff and was complemented by an in house session.

Impact: Economic Impact

Commercial Service

The commercial service commenced on 1st Sept 2017 and work billed for to date is around £50,000. This means the technician's post which was funded for 18 months by the University of Southampton is self-sufficient and we can continue to develop this aspect of the service. We have retained the custom of users of this service through the transfer from the departmental service. We are putting together a more standardised offering for commercial clients which we hope will simplify discussions with interested parties and minimise the time from enquiry to work being undertaken.

We are actively exploring new, additional models for commercialisation. The potential for the NCS to form strategic business partnerships, as opposed to the "pay per structure" model, is strong – particularly if as part of a package with other University of Southampton capabilities. We have co-promoted ourselves with the Knowledge Centre for Materials Chemistry (KCMC, <http://materialschemistry.org.uk/>) at the Materials Research Exchange this year. We are also consulting on a proposal to establish a diffraction service in the United Arab Emirates. The proposed UAE service would offer powder and materials diffraction services, with a possibility that single crystal diffraction would be offered through sub contract to the NCS. This proposal is at the very early stages and our input is being sought on how to set up, structure and run a service of this nature.

We are also in the early stages of setting up a collaborative project with Professor Chris-Kriton Skylaris of the University of Southampton and Merck Chemicals Ltd which will include co-authorship of papers and will provide training for a sponsored PhD student.

Additional funding received

A modest amount of funding has been awarded by the BCA for development of Education and Outreach infrastructure and resources (see Impact – Training section).

Proposals seeking funding to support academic projects with existing collaborators/users are being, or have been, written with Graeme Day and Andrew Cooper (ERC Synergy), John Wallis (Leverhulme), Andrew Weller (Diamond PhD studentship & EPSRC), Robert Kingsford-Adaboh (GCRF), Sedat Ture (TUBITAK).

Leveraging the NCS and using it as a way to experimentally validate a computational modelling approach, an EPSRC proposal with Profs Day and Woods was submitted to the Feasibility Study call associated with Automating Science Discovery. The proposal was funded (EP/S015418/1) and will start at the end of 2018. Due to the limited amount of funding available the NCS contributes facilities in-kind and will supervise a PhD student and PDRA conducting this aspect of the work. The strategy is that the NCS will be core to future follow-on applications in the area and will ultimately receive additional funding through this route.

International Interactions

International Collaborations making use of the facility this period include: William Zuercher (USA), Annie Powell (KIT, Germany), Tony Keene (Ireland), Hugo Meekes (Netherlands), Mohammed Nur-e-Alam (Saudi Arabia),

Srinivasulu Aitipamula (Singapore), Waro Nakanishi (Japan), Mohamed Gaye (Tunisia), Ibrahima Thiam (Senegal), Abdurrahman Sengul (Turkey) and several groups from the Chemistry Department in Cagliari, Sardinia. All these collaborations have resulted in preparation or publication of 9 manuscripts. Work arising from our collaboration with the UNC Eshelman School of Pharmacy (USA) was presented at the ACS August 2018 National Meeting.

Additionally to these, a selection of highlights include: An extended visit from, Sedat Ture (Turkey) and the continuing international collaboration led by the NCS working on radiation decay at synchrotrons (Oxford, Aarhus, Bremen, San Francisco & Melbourne.) Beamtime for this at Diamond has led to a successful application to the Advanced Photon Source (Chicago), where the NCS will combine with these international collaborators to explore different aspects of the phenomenon that cannot be addressed at Diamond. A high impact publication raising community awareness of this hitherto ignored/unnoticed phenomenon has been prepared during the reporting period and will be submitted imminently.

Simon Coles is on both the steering and scientific committees of the Pan African Conference on Crystallography (Feb 2019) in Accra (Ghana). This has now become a dual conference since combining with the AFLS (African Light Source) – an initiative to secure funds and build a synchrotron on the African continent. As a result of close connection/collaboration with the Local Organising Committee, Simon Coles has assumed a leading role not only in organising the scientific program but also at higher political levels (it is expected that due to the importance of establishing an African synchrotron several government ministers from a number of nations will be present). Simon is personally responsible for securing funding (ca £8000 from the RSC, CCDC & Diamond) to support african student attendance through bursaries. He is also collaborating with Diamond to deliver a live, on-line remote beamline control session at the conference.

In May Simon Coles led a Royal Society funded workshop in Ghana to establish a network and develop proposals to submit to the Global Challenges Research Fund. Around 40 participants ranging from chemists, engineers and computer scientists to politicians and policy makers met and developed proposals around Organic Photovoltaics and Biomass Conversion – NCS is a key feature of the former.

Improvement

Equipment Upgrade

The ancillary equipment associated with the advanced techniques has largely been commissioned across all 4 techniques and initial experiments have been undertaken with NCS users as detailed in the Scientific Excellence section. The high pressure equipment has required the most significant input with the sourcing of a gasket drill and the in-house construction of a fluorescence spectrometer to measure the pressure inside the cell. Continued training of staff members (as detailed in the Training section) has also improved the service we are able to give in all the Advanced Techniques areas.

No further equipment upgrades are currently planned in the current funding period.

Access and User experience

The method of access and peer review for the Advanced Techniques was established by our Strategy and Allocations Panel at their April 2018 meeting. This process has been designed to have a low barrier to entry, whilst clearly identifying what results would be expected and the follow-on steps (e.g. funding applications) that they would enable.

Work on the replacement electronic sample management system, Portal2, has progressed well. The system development is complete and we have now reached the testing phase. All that then remains is the timing of the full launch, which will have been achieved before the end of the next reporting period.

A standard operating procedure is being drawn up for commercial clients. This not only formalises our working procedure, a significant matter for some clients, but it has also allowed greater standardisation across the academic service.

The training programme continues to develop with new activities being undertaken and planned in this period. The steer for these developments is strongly in accordance with feedback from our users and previous course participants.

Strategic Fit

The NCS fundamentally supports the EPSRC 2015 Strategic Plan and current Delivery Plan at the highest of levels by providing underpinning knowledge across the whole of Chemistry that informs other fields. As a world leading facility, it enables a broad portfolio of work that helps place the UK as a leading global research nation and feeds in to most aspects of the outcomes framework. The technique of crystallography and thereby the facility, transcends disciplinary boundaries and accordingly supports most elements of interdisciplinary research that touch on the chemical sciences. The NCS forms part of the 'National Capability' landscape by providing "support for excellent, long-term disciplinary and multidisciplinary research in engineering and the physical sciences." Applicants are asked to self-identify which Balancing Capability Research Area(s) they work in. This year users have identified 4 more areas than last year.

Users have identified the work the NCS supports as being in the following areas:

Prosperity Outcome	Balancing Capability Research Area
Resilient Nation	Carbon capture and storage, Energy storage, Hydrogen and alternative energy vectors, Materials for energy applications, Solar technology
Healthy Nation	Chemical biology and biological chemistry
Productive Nation	Catalysis, Computational and theoretical chemistry, Synthetic coordination chemistry, Synthetic organic chemistry, Synthetic supramolecular chemistry, Analytical science, Chemical structure, Light matter interaction and optical phenomena, Polymer materials, Chemical reaction dynamics and mechanisms, Condensed matter: magnetism and magnetic materials

A review of NCS marketing and user engagement strategy has recently been instigated. Future marketing and engagement will have an emphasis on new users in Balancing Capability Research Areas where the Strategic Focus Highlights are 'Access to research infrastructure' and/or 'Partnering with business'.

Sustainability

The commercial service has been successfully introduced and invoiced for a significant amount of money in its first year – this pays its own way and makes a modest profit. However, in the context of sustainability of the service it is equally important to consider driving towards more efficient operation of the service. Embedding the NCS in bigger community initiatives and being seen as a global leader also makes for a more sustainable operating basis.

Efficiency Developments

Work on Portal2, the newly written version of the NCS's management software, has entered the testing phase. This is designed to give users and staff a more streamlined application and allocation management tool. Additional information, including automated updates from the diffractometers on data collected, will be stored by the new system. This will allow our users to monitor their allocation and the progress of samples through the facility process much more closely and will reduce the administrative burden on the crystallographers in the laboratory. It is anticipated that this will also reduce the time taken to monitor metrics required by EPSRC. The system has been written with a new modular architecture and on well established development platforms, which will improve its ability to be upgraded and maintained long into the future. Development of a Standard Operating Procedure has enabled us to understand the process better and become more efficient at delivering a consistent service.

Sustainability Plans

The continued development of both the commercial and advanced techniques services opens up important potential funding streams and development of both of these continues to be a priority for the NCS for the

upcoming year. A range of funding applications have been made and these are anticipated to grow as the advanced techniques become more embedded in the NCS provision. The commercial service has grown steadily, however with more time investment in attracting new customers the revenue will increase at a faster rate. Mechanisms for achieving this are outlined in the Economic Impact section of this report. There are also other elements of service provision that will be considered as routes to generate income. The potential to charge a modest amount for training in academia e.g. to CDTs will be investigated, but this has even greater possibilities with the proposed UAE service (see Economic Impact section), where we have quoted for significant training events. We have also factored in a charge for consultancy on service provision as part of the UAE proposal.

Summary

Year one of operation in the current funding period involved setting up a range of new elements of the NCS. This second year (second reporting period), has largely been one of embedding these new elements into the regular operation of the service.

Our most significant achievement has been the launch of the Commercial Service. This has been a very successful endeavour, with several industry customers forming an entirely new group of users for the NCS. There has been a technical member of staff appointed, who has delivered a considerable amount of contract work – we have billed for around £50k this year, which exceeds our original set target.

The second new element of service provision is that of the advanced techniques, which are in fact four different types of science and experimental approach. These are fully embedded and growing, however at different rates as we have introduced them sequentially in a phased manner in order to manage change in workloads. Variable temperature work is the most mature with several studies producing results that are being worked up for publication. The gas environment work has perhaps provided the most exciting results, with a range of work being undertaken and a high profile article submitted. Charge density studies are now beginning to happen and the high pressure support labs are established, with studies scheduled.

Finally, we have an established training programme delivering to several audiences at different levels of expertise. We have delivered the number of training events that we proposed and believe this to be a sustainable level.

There have been several fundamental problems with each of the diffractometers during this period. The support from the manufacturer has been extensive, including factory recall and on site rebuilds. Despite these problems, which are essentially beyond our control, the NCS was able to study all samples within acceptable KPI levels and was not once at a standstill.

We continue to operate at a sustained level of scientific excellence and provide a balanced service for the UK community. We also operate internationally, with a range of collaborations in structural characterisation, advancing/understanding the technique and in advanced techniques. In the global crystallography community we demonstrate a sustained level of leadership and continue to innovate and deliver a varied outreach programme.

1. Title of Case Study: Topologically directed discovery of functional co-ordination materials
2. Grant Reference Number or Facility Name: University of Sussex, Dr George Kostakis, accessing National Crystallography Service, University of Southampton.
3. One sentence summary: Data generated by the National Crystallography Service is helping researchers address major challenges in synthetic chemistry
4. One paragraph Summary: Data from the University of Southampton's National Crystallography Service (NCS) is helping chemists at the University of Sussex address major challenges in synthetic chemistry to produce functional and transformative materials.
5. Key outputs in bullet points: NCS data is enabling: <ul style="list-style-type: none"> transformative multidisciplinary science leading to new knowledge new collaborations with chemists to study the properties of compounds the training of chemical topological scientists to design and develop new materials that will address the needs of the future industry the training of students who will understand the potential application of their research and develop skills to enable them to progress in emerging sectors and traditional industries
6. Main body text Data from the University of Southampton's National Crystallography Service (NCS) is helping chemists at the University of Sussex address major challenges in synthetic chemistry to produce functional and transformative materials. Dr George Kostakis, Senior Lecturer In Physical/Inorganic Chemistry at the University of Sussex, said: "Our research combines techniques and expertise from inorganic, co-ordination, topology, theoretical and organic chemistry. This TOPological Co-ordinatIon Chemistry (TOPIC) approach will broadly systemise the discovery of new functional co-ordination compounds, complementing state-of-the-art techniques to harvest new materials. Our comprehensive programme aims to: <ul style="list-style-type: none"> introduce a conceptually different synthetic methodology exploiting compositional parameters systemise and topologically rational assemble co-ordination compounds correlate topology and property relationships with applications in interdisciplinary fields <p>"This research will create the necessary background to develop new methodologies for materials with specific properties in areas as disparate as catalysis, biochemistry and molecular magnetism. It has the potential to increase the storage capacity/memory of devices, and improve and lower the costs of drugs.</p> <p>"We work with compounds whose synthesis is complicated and attempts at the systemisation of these compounds has been very sparse. If we could elucidate the mechanisms and processes to make these compounds more generally available, it would be very beneficial. We could overcome the challenges of a reproducible synthesis, controlling the shape and size, as well as the fine-tuning of chemical and physical properties.</p> <p>"X-ray crystallography produces a lot of detailed structural information, in order to maximise the potential of this information we have set up a specific database – the Polynuclear Inorganic Clusters Database.</p> <p>"Working with the NCS at Southampton is integral to our research. The materials we are investigating can be weakly diffracting, and the NCS has all of the relevant equipment and expertise to provide a full characterisation of the material which is essential for our project.</p>

"We have a really good relationship with the NCS. We have been using their services for the past four years and they know what we are trying to achieve and what they can do to help facilitate this. They are in constant contact as they collect and solve datasets."

The collaboration between the Kostakis Group and the NCS is already having an impact, with nine co-authored papers already having been published.

7. Names of key academics and any collaborators:

Manchester (EPR)

Akien (Lancaster 89Y-NMR)

Spivey (Imperial College)

Arseniyadis (Queen Mary's)

8. Sources of significant sponsorship (if applicable):

£92,400 EPSRC EP/MO23834/1, Feb 2015

9. Who should we contact for more information?

Dr George Kostakis, University of Sussex (G.Kostakis@sussex.ac.uk)

01273877339

Assessment Criteria:

The assessment panel will assess the Annual Report against the criteria outlined below, scoring each with a score from 1-6. At the end, an overall score will be given. The score and feedback should enable the facility, together with their steering committee and EPSRC contact to ensure the best possible service is provided to the user community.

- 1) **Key Performance Indicators (KPIs) and Service Levels (SLs):** Over the reporting period, did the facility meet all KPIs and SLs laid out in the contract (score 6 = all KPIs and SLs met, very high standard / score 5 = all KPIs and SLs met, high standard). If not, did the facility do their best in mitigating negative impact for the users and did the facility take steps to improve performance in the future? To which degree were these steps successful? (not very successful: score 2, partially successful: score 3, predominantly successful: score 4). If the facility has not met more than 2 of their KPIs and SLs AND has not taken any steps to mitigate effects and improve performance, score 1).
- 2) **Scientific Excellence and Users:** Does the facility support scientific excellence in the UK to its user community? Does the facility actively engage with a variety of user communities and provide support for special needs (students, business collaborations etc.)? (1: Does not meet the criteria in any way; 2: partially meets the criteria, but with major weaknesses; 3: partially meets the criteria, but there is room for improvement; 4: meets the criteria; 5: meets the criteria and sometimes exceeds expectations; 6: exceeds expectations)
- 3) **Impact: Training, Outreach, Societal and Economic Impact:** In what way does the facility support the generation of broader impact, such as training of skilled people, enabling broader societal and/or economic benefits of scientific work e.g. through collaborations, and promote the Engineering and Physical Sciences among the wider public. (1: Does not meet the criteria in any way; 2: partially meets the criteria, but with major weaknesses; 3: partially meets the criteria, but there is room for improvement; 4: meets the criteria; 5: meets the criteria and sometimes exceeds expectations; 6: exceeds expectations)
- 4) **Improvement:** Has the facility thought of ways to improve the user experience and ensure its long term attractiveness and sustainability? (1: This aspect has not yet been thought of at all; 2: Some aspects of continuous improvement have been considered, but are not yet implemented; 3: some aspects of continuous improvement are being implemented; 4: some successes of continuous improvements can be demonstrated; 5: continuous improvement is integral to how the facility operates; 6: work on continuous improvement of the facility exceeds expectations)
- 5) **Strategic Fit:** Can the facility show that it supports work in areas of strategic priority to EPSRC? Does the facility take steps to align itself with EPSRC's balancing strategies? (1: Does not meet the criteria in any way; 2: partially meets the criteria, but with major weaknesses; 3: partially meets the criteria, but there is room for improvement; 4: meets the criteria; 5: meets the criteria and sometimes exceeds expectations; 6: exceeds expectations)

Overall assessment and score: Does the facility meet the assessment criteria? Which areas could be improved? Are any actions recommended to improve the benefits for users and EPSRC from this facility? (1: Does not meet the criteria in any way; 2: partially meets the criteria, but with major weaknesses; 3: partially meets the criteria, but with minor weaknesses; 4: meets the criteria; 5: meets the criteria and sometimes exceeds expectations; 6: exceeds expectations)

Annual Report for EPSRC Mid-Range Facilities – Contract Manager’s Commentary

Facility: _____

Address: _____

Director: _____

Facility Manager: _____

EPSRC Contract Manager responsible: _____

Contract Manager’s comment on the performance of the facility, including a longer term view.

Change log

Name	Date	Version	Change
Michele Erat	18/07/2014	1.0	N/A
Michele Erat	06/08/2014	1.1	Version after consultation with portfolio managers
Michele Erat	21/01/2015	2.0	Adapted with recommendations from the Capital Equipment SAT
Michele Erat	11/05/2015	2.1	New timelines
Louise Tillman	01/07/2016	3.0	Updated for 2016 with amendments based on feedback from Mid-range Facilities Statements of Need Panel
Simon Crook	17/7/2017	4.0	Updated for 2017
Simon Crook	16/7/2017	5.0	Updated for 2018 to include Sustainability and some ammendments to other questions.