

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, Sue Boundford

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 most-weakly 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 rapid, 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 - it is critical in underpinning structural science in the UK that impacts across many areas including energy, catalysis and next-generation materials .

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: £198,221.09 ex VAT

Spend on track? Y

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

			SLA Level				
Type	Description	Time for Performance	Green	Amber	Red	1st Sep 2018 - 31st Aug 2019	Directors Comments
RMI	Total number of all Users	Period associated with specific report	N/A	N/A	N/A	65 (Routine service active users) / 49 (Training schemes)	
RMI	Spectrum of user types	Period associated with specific report	N/A	N/A	N/A	65 (Routine service active users) / 49 (Training schemes) / 8 (Commercial clients) / 5 (Advanced techniques)	
RMI	Number of University / Research Groups Involved	Period associated with specific report	N/A	N/A	N/A	60 (University) / 66 (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	988	
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 35.6%	See Note 1 below.
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 62.4%	See Note 2 below.
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 97.52%	A water leak caused a one week loss of operation.
SL	Percentage of Access Costs recovered	Period associated with specific report	10	6	2	8.75%	See Note 3 below.
SL	Number of Publications	1 year	30	20	10	47	
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 informing a User of receipt	within 2 working days	95% and above	>90% but <95%	90% or less	100%	See Note 4 below.

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	94.35	See Note 4 below.
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	93.15	See Note 4 below.
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	96.6	See Note 4 below.
SL	Time from examination to end result	Data collection = 5 working days	95% and above	>90% but <95%	90% or less	94.3	See Note 4 below.
SL	Time from examination to end result	Full Structure Analysis= 20 working days	95% and above	>90% but <95%	90% or less	94.25	See Note 4 below.

Note 1

A catastrophic collision resulted in total lack of operation from early December until May. Several components had to be repaired, however the root cause of the collision took a considerable time to resolve - components were returned to the factory and numerous on-site repairs attempted. The situation was eventually resolved after several site visits by a Rigaku factory engineer (from Tokyo). Since repair the instrument has been 100% reliable and is aligned to a greater degree of accuracy than ever before.

Note 2

In the previous period a persistent and ongoing goniometer issue required numerous on-site repair attempts and ultimately shipment back to Rigaku Japan for factory repair. During the start of this period approximately 3 months were taken up with factory testing, shipping, installation and commissioning of the goniometer. Since that time the goniometer has been completely reliable and the instrument has only been down due to expected x-ray generator maintenance/repair.

Note 3

NCS is free at the point of access for academics and therefore commercial income is the only possible route for cost recovery. 76 commercial samples were studied, generating an income of £64k. Access costs (staffing and maintenance) for this period were £731742 and therefore the recovery rate is 8.75%.

Note 4

An entirely new electronic user and sample management system was implemented in this period. It was launched at the start of the May-Oct allocation period. Numerous, generally minor, issues were uncovered in the first 3 weeks of use. Whilst these were rapidly addressed, the system was operating live. Therefore we were still recording "test" samples introduced in order to fix the problems. This had unintentional and unforeseen influences on the way SL data are recorded. There was therefore a period of 3 weeks where statistics are unreliable and accordingly negatively effects metrics for all the following categories.

Users

User statistics.

NCS users fall into six categories in this reporting period:

- Research Group Principal Investigators. 65 NCS PI's, of which 4 were new users. We do not record the number of students for each PI accessing the NCS, however an accepted estimation is that an average user would have 2-3 students (some groups are however into double figures).
- UK service crystallographers. 9 users fall into this category. This usage mode in-turn supports large numbers of researchers as departmental crystallographers access the NCS to analyse difficult samples, screened on their own facilities, from across *their* entire user base.
- Advanced Techniques collaborators. 5 Collaborators in this category worked with the NCS in this period.
- Industrial Users. There are 10 companies registered with the NCS and 8 of them 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 38 different institutions. 78% of our users are based in chemistry departments, while the remaining 22% 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 21 Research Areas (an increase of 4 on the 2018 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 (46 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 [4.7/5]
- The NCS is responsive to user requests, e.g. rapid turnaround, additional data collections [4.8/5]
- 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 [4.89/5]
- I would recommend the NCS to a colleague [5/5]

The response rate for the survey is on par with previous years and the scores essentially remain in line with them.

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

- "This is a world leading national service of huge strategic and scientific importance. The quality of infrastructure and analytical expertise should be hugely valued by the UK."
- "From a personal perspective I simply would not be able to publish the quality of work I do without the service and the expertise of its staff."
- "All my dealings with the NCS have been excellent. They have been very helpful and supporting our research. The quality of our research outputs has been enhanced by this service. It is an asset to the research community."

However, we are anxious that this survey is useful in terms of getting and acting on feedback from users in order to improve the service. Some comments we can readily react to and will address in the coming reporting period, the most worthy of note being:

"The new online portal is great. In time it would be helpful to have "Help" sections available for each of the data entry points to help new users who are unfamiliar with the system." [In progress, predominantly completed]

"Would be useful to receive the DotHyPix, RosHyPix cif files with each data set." [Completed]

There are also comments that are harder to address and we will consider these over time, examples include:

"Samples that need to be sent to Diamond have taken a long time and so some have gone off, or we no longer have fresh crystals"

"Have less frequent application periods."

"Make my crystals grow better. Possibly more synchrotron time as samples we send tend to be either small or weakly diffracting."

Publishing Highlights.

The level of 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. At the time of reporting, this paper has received 339 citations and continues to be in the top 1% based on Web of Science's highly cited threshold for the publication year and field. Simon Coles has been commissioned by Structure and Bonding, a Springer journal-book hybrid, to write a review on 21st Century Service Crystallography, which details the current state-of-the-art in the field and will also form an update to replace the outdated 2012 description of the NCS operation. This review will be significant in scope and is due to be published at the end of 2019, when it will replace the 2012 Chemical Science perspective article as the method of citing the NCS.

In this reporting period 47 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. Some selected highlights of these include:

- Two papers in collaboration with Skabara (Glasgow): "Non-covalent close contacts in fluorinated thiophene-phenylene-thiophene conjugated units: understanding the nature and dominance of O...H versus S...F and O...F interactions towards the control of polymer conformation" **Chemistry of Materials**, <https://doi.org/10.1021/acs.chemmater.9b01886> and "Intermolecular Interactions in Molecular Crystals and Their Effect on Thermally Activated Delayed Fluorescence of Helicene-Based Emitters" **Journal of Materials Chemistry C**, <https://doi.org/10.1039/c8tc03390k>. Skabara has worked with the NCS for ~25 years and these high impact articles in the field of developing organic semiconductor materials illustrates the vital contribution of crystallography to developing this important field.
- Collaboration with a Materials Science team from Oxford (Bogani, Anderson) led to "Tailored Homo- and Hetero- Lanthanide Porphyrin Dimers: a Synthetic Strategy for Integrating Multiple Spintronic Functionalities into a single molecule" **Chemical Science**, <https://doi.org/10.1039/c8sc03762k>. NCS work characterised very large covalently linked porphyrin oligomers for use as molecular spintronic devices.
- "The asymmetric aza-silyl-Prins reaction: synthesis of enantiopure piperidines" **Organic Letters**, <https://doi.org/10.1021/acs.orglett.8b03283> by Dobbs et al (Greenwich) describes the design and development of the first asymmetric aza-silyl-Prins reaction which gives rise to valuable and diverse piperidines and pipecolic acid derivatives in both high yields and as single enantiomers.

Advanced Techniques Highlights.

The advanced techniques modes of access to the NCS are relatively new and now becoming established at the NCS. Given they have had to develop from nothing and are 'long-duration', both experimentally and in data analysis / write-up, they are in differing states of maturity and embedding. In fact, a new mode of access was established in this period, that of supported access for non-specialists to neutron diffraction facilities – here a collaborative application with Manfred Bochmann (UEA) to access the ISIS neutron facility enabled characterisation of a unique Au-hydride and this work is currently being written up for publication. This has led to establishing a mechanism whereby the NCS can work with ISIS to promote more facilitated use of its single crystal diffraction facility (SXD).

Variable temperature experiments in this reporting period have yielded a number of exciting results. The most notable outcome is with Dr Ian Gass (University of Brighton) which 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 has been uploaded to the ChemRxiv repository [Hysteretic Thermal Spin-Crossover and Symmetry Breaking in Heteroleptic Fe(II) Complexes Using Alkyl Chain Substituted 2,2'-Dipyridylamine Ligands **ChemRxiv**, <https://doi.org/10.26434/chemrxiv.9641990.v1>] – a first for the NCS on this preprint server. A MOF dehydrolysis study with Wilson and Murrie (Glasgow) where we were able to obtain sufficient resolution data to observe the sequential loss of two bound water molecules is now in an advanced state of preparation.

In this period work on our primary Charge Density project with Sally Freeman (University of Manchester) and Carl Schwalbe (University of Aston) was hampered by the unexpected death of Carl, who was the main coordinator and intermediary. Data has been collected and results worked up and now discussions are planned with Manchester colleagues to understand how to analyse in the light of their research. A technically very demanding project with Annie Powell (KIT, Germany) attempting to relate coordination mode to magnetic properties has moved forward with collected data currently being analysed. A variable temperature study has been initiated with John Wallis (Nottingham Trent University), where very unusual intramolecular interactions have been observed at normal resolution – data have been collected and a complementary solid-state NMR study begun in Southampton.

The highlight of the gas environment work is a 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 from this collaboration have been published in a premier journal [Modulation of σ -Alkane Interactions in $[\text{Rh}(\text{L}2)(\text{alkane})]^+$ Solid-State Molecular Organometallic (SMOM) Systems by Variation of the Chelating Phosphine and Alkane: Access to η^2, η^2 - σ -Alkane $\text{Rh}(\text{I})$, η^1 - σ -Alkane $\text{Rh}(\text{III})$ Complexes, and Alkane Encapsulation **Journal of the American Chemical Society**, <https://doi.org/10.1021/jacs.8b09364>] with further publications in preparation. In collaboration with Weller and Prof Matthew Rosseinsky (Liverpool) we were successful in gaining funding (50% Diamond Light Source, 50% Southampton DTP) for a PhD studentship to extend this approach well beyond current capability. The studentship will work closely with all 4 centres to develop a new type of gas flow cell, combined with GC-MS, that will enable the monitoring of the kinetics of reactions and adsorption processes. This represents a new capability and will be made available to UK academia through Diamond Light Source and the NCS on completion.

Advancement of high pressure studies has required modified instrumentation (X-ray collimator and beamstop). These modifications were initially attempted in-house, but we had to refer to the instrument manufacturer, which has a 6 month lead time for this work. However, this work is now being picked up through the appointment of a Southampton PhD student associated with the NCS who is connecting variable temperature and pressure studies.

Methods Development & Community Leadership.

The NCS is a key partner in a 3-way venture with Merck and Rigaku to develop the "crystal sponge" method to be adopted by the chemistry and pharmaceutical communities as a way to obtain structures of uncrystallisable compounds. It is expected that this will lead to a significant commercialisation opportunities (see Economic Impact).

An exploratory collaboration with Nikon, looking at the application of Diffraction Computed Tomography to molecular materials in the home laboratory, has been undertaken. There have been several planning meetings and samples have been measured and are being assessed in Denmark in order to develop the method.

The NCS has a pioneering project to increase understanding of radiation induced decay in small molecule samples and develop mitigating data collection strategies. In collaboration with macromolecular crystallographers (Garman, Oxford) the first phase of this work has been completed and our publication [Radiation damage in small molecule crystallography: fact not fiction **IUCrJ**, <https://doi.org/10.1107/S2052252519006948>] is the first report of this effect in small molecule crystallography and illustrates its seriousness, breadth and impact to the entire community. This has triggered an international collaboration concerned with using our methodology at other synchrotron radiation sources and to this end we hosted a visit from our international collaborators (from Berne and Aarhus). This initiated an international standardisation project – which has begun with beamtime at the Advanced Photon Source (Chicago, USA) and will be extended to the Advanced Light Source (Berkeley, USA).

Simon Coles was a co-organiser of the 2nd Pan-African Conference on Crystallography, which was attended by high ranking African politicians as well as academics, due to the ongoing debate about investment in an African synchrotron facility - Simon is now an advisor on this significant infrastructure project.

In July 2019 the Editor of Crystallography News died unexpectedly – at very short notice Simon Coles became the interim editor of this magazine that goes out to BCA members around the world and has produced one issue so far.

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 community survey has been conducted and forms the basis of recommendations which will be formalised at an international workshop (Prague, August 2020) which is being organised by Simon Coles. Simon is the Chemistry lead for GO FAIR (an EU initiative aimed at making research data open) and lead the development of the Chemistry Implementation Network (<https://www.go-fair.org/implementation-networks/overview/chemistryin/>). This has led to a significant involvement in driving this agenda forward with the International Union of Pure and Applied Chemistry and a paper describing this is about to be published in **Data Intelligence**.

Case Study (Carl Redshaw).

We include a case study on the work of Prof Carl Redshaw, University of Hull. Prof Redshaw has been an NCS user for over two decades, predominantly using the Data Collection Only service, although has used the occasional Full Structure Analysis for particularly demanding samples. Carl has provided samples across a range of inorganic

coordination chemistry areas, but is now particularly active in the highly topical field of designing environmentally-friendly plastics. In this respect the NCS supports work on a large UKRI/EPSRC grant – ‘Evolving a circular plastics economy’, which also feeds into travel grants with China and Japan (EPSRC, Royal Society and British Council).

Impact: Training, Outreach and Societal Impacts

Training courses and workshops

Three major training events were conducted during the reporting period:

An ‘Introduction to Crystallography’ workshop was held in January 2019 and had 7 attendees;

‘Approaches to Modelling Disorder’ workshop was held in June 2019 and had 12 attendees. This was designed for those with considerable crystallographic experience, but requiring an ability to address more complex problems;

‘Collecting and Processing Data’ (6 attendees) was held in May 2019 as a focussed session to train Early Career Researchers in groups that we collaborate with on advanced techniques.

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.

A number of materials were developed during the reporting period to promote the NCS provision of Advanced Techniques – these have been well received and so will be used for increased activity in this area. Fliers have been included in conference packs (BCA Spring Meeting, RSC Macrocycles and Supramolecular Chemistry). A poster was presented at the BCA Spring meeting and has also at several University of Southampton events with external attendees. A seminar entitled “Smaller, Faster, Better, New research at the frontiers of single crystal diffraction” was given at the University of Kent (Simon Coles invited speaker) and will be recycled at other locations. This formed the basis of a presentation at the Rigaku Oxford Diffraction European User Meeting (Peter Horton, University of Regensburg) which illustrated how a service crystallography laboratory can expand its capability.

Simon Coles organised an RSC Local Section event (June 2019) aimed at illustrating techniques and applications of Thermal Methods – it was sponsored by the NCS and had around 40 attendees from industry and academia and the NCS was promoted in numerous ways. A range of other sponsorship opportunities have been identified and approaches for this method of marketing developed – these will take place in the next reporting period.

One of our papers this year [The Curious Case of Acetaldehyde Phenylhydrazone: Resolution of a 120 Year Old Puzzle where Forms with Vastly Different Melting Points Have the Same Structure, *Crystal Growth and Design* <https://doi.org/10.1021/acs.cgd.8b01459>] had a compelling background story of a problem that defied a Nobel Prize winner over a century ago and was solved recently by collaboration with Terry Threlfall (an 84 year old emeritus scientist at Southampton closely associated with the NCS since retiring in the 1990’s). We used this as a significant publicity vehicle – the paper has had over 2300 views and became a significant thread on Twitter. We ensured the story was picked up by the popular press, with articles in *Physics Today*

(<https://physicstoday.scitation.org/doi/10.1063/PT.6.1.20190606a/full/>) and *Chemistry World*

(<https://www.chemistryworld.com/news/solution-to-120-year-old-puzzle-reveals-new-chemical-phenomenon/3010955.article>). The story was also picked up by numerous news aggregators e.g.

<https://soylentnews.org/article.pl?sid=19/06/08/2219212> and newsletters (University, Faculty & Department).

Public engagement.

The University of Southampton hosts the largest outreach event of its kind in southern England - SOTSEF (<https://www.sotsef.co.uk/>). NCS has been involved in this since its beginnings nearly 20 years ago. This year saw over 5000 visitors and the NCS stand had its usual activities, as well as Peter Horton trialling some pilots as part of an outreach qualification. We also ran an International Year of the Periodic table stand which was spun out of the NCS activities. Simon Coles was part of a team (with his local scout group) awarded funds from The Science Council for a community event in National Science and Engineering Week - NCS activities were core to this event, which attracted 300 people. NCS was involved in many schools outreach events during the period, including ‘Hands-on diffraction’ with sixth form students (July), outreach activities at many Open Days and work shadowing experiences for 6th formers from across the region. In collaboration with the Cambridge Crystallographic Data Centre, Simon Coles leads an global community project for the International Year of the Periodic Table, which is illustrated through crystal structures and built up through news items for each element as the year proceeds (<https://www.ccdc.cam.ac.uk/Community/educationalresources/PeriodicTable/>). Some RSC funding has been secured to support the development of schools and the general public learning materials based on this resource.

Facility staff training and career development.

All NCS staff undertook training and development activities. The BCA Spring Meeting (Nottingham, April 2019) was attended by all staff; Graham Tizzard and Peter Horton were involved in internal EPSRC fund applications (ECR & Institutional Equipment); Wim Klooster presented at the International Charge Density Meeting (Gottingen, July 2019); Sue Boundford attended numerous administrator training activities; James Orton attended several personal development courses (Thinkwrite: Writing Books workshop; Writing Quality Papers workshop); Chris Holes attended the Annual Spectroscopy in a Suitcase meeting (RSC, August 2019); Goulielmina Anyfanti was a student at the BCA/CCG Intensive Teaching School in X-ray Structure Analysis (March 2019); the NCS hosted an undergraduate summer internship (Matilda Rhodes).

Impact: Economic Impact

Commercial Service.

The commercial service generated £64,000 during this reporting period. The technician post which provides the data collection, analysis and reporting aspect of the commercial service was initially funded for 18 months by the University of Southampton. The remaining legal, financial and administrative work on this service is provided by the Service Support Coordinator position. At this level of turnover the technician post is self-sufficient. This level of operation gives a reliable platform from which we can operate and continue to develop this aspect of the NCS. We have reviewed our operation and adapted to a more standardised offering for commercial clients. This includes a Standard Operating Procedure for performing the experimental and reporting work and a defined workflow from point of enquiry all the way through to invoicing. This workflow integrates as smoothly as possible with other departments at the University of Southampton, such as Legal and Finance. On the whole this approach has simplified discussions with interested parties and minimised the time from enquiry to work being undertaken whilst also easing the invoicing process.

There were 4 new users of the commercial service this period. This indicates that there is still more of an achievable market to engage with, however the traditional 'Big Pharma' area is relatively saturated. We are therefore beginning to target our marketing efforts in different areas and to different types of organisation eg Contract Research Organisations. This presents some challenges as such companies are less visible than large business and we are developing a strategy to address this issue. 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. This is particularly if the NCS offering can be made as part of a package with other University of Southampton capabilities. During this reporting period the University of Southampton has restructured and the School of Chemistry is now within the Faculty of Physical and Engineering Sciences and this presents an opportunity as far as collective enterprise is concerned. The NCS has now worked with nC² – one of the Faculty's business units providing engineering consultancy. This work has initially generated ca £4000 to date via Differential Scanning Calorimetry measurements (a supporting experiment for our variable temperature advanced techniques work), however this is with the view to also providing diffraction expertise. Being part of this consultancy at the university level provides much greater visibility and the ability to be a component of a package.

A significant opportunity has arisen in collaboration with Rigaku (our instruments manufacturer/provider) and Merck Performance Materials. Details of this are the subject of a Non-Disclosure Agreement currently being drawn up with these two companies. The NCS will provide a significant amount of quality assurance work which should lead to a commercialisation opportunity. Rigaku will provide training and any necessary equipment and in return the NCS will conduct the work. While this is currently only generating in-kind training and equipment to the NCS, it is expected that the commercialisation opportunity will then be run by the NCS at which point there will be a greater return. A significant amount of work towards this goal will be conducted in the coming reporting period.

Additional funding received

Proposals to fund academic projects with existing collaborators/users are being, or have been, written with: Graeme Day and Andrew Cooper (ERC Synergy) – Failed at first attempt but at the time of writing we learnt of the successful second attempt, some crystallographic analysis funded and academic input into the project as a whole; Andrew Weller (Diamond PhD studentship & EPSRC DTP) – Both halves of the studentship were funded and work commences October 2019 and this will develop new gas cell methodology for the whole community; European colleagues in Charge Density/Quantum Crystallography field (COST Network) – second attempt failed, despite scoring over the threshold; ECR Small equipment proposal (EPSRC Resounding fund) – led by Peter Horton, not funded; EPSRC Core equipment (UoS internal) – request for a new goniometer was deemed too costly for a single item.

Simon Coles successfully led a bid with STFC to operate the successor to the National Chemical Database Service. The Physical Sciences Data-science Service came into operation during this period and while no direct funding comes to the NCS there is considerable synergy and efficiency gains in operating these two NRFs. On a related note, Simon was strongly involved in advising and writing a bid to operate an NRF in Computed Tomography in which the University of Southampton are key partners. Working across NRF's will uncover synergies and possibilities for efficiency savings as well as being a basis for sharing best practice.

International Interactions

International Collaborations making use of the facility: William Zuercher (USA), Annie Powell (KIT, Germany), Tony Keene (Ireland), Hugo Meekes (Netherlands), Minas da Piedade (Portugal), Mohammed Nur-e-Alam (Saudi Arabia), Robert Kingsford-Adaboh (Ghana), Srinivasulu Aitipamula (Singapore), Mohamed Gaye (Tunisia), Ibrahima Thiam (Senegal), Abdurrahman Sengul (Turkey) and groups from the Chemistry Department in Cagliari, Sardinia. These collaborations have resulted in preparation or publication of 12 manuscripts.

Simon Coles sat on the steering and scientific committees of the Pan African Conference on Crystallography (Feb 2019, Ghana), a dual conference combining with the AFLS (African Light Source) which began the process to fund and build a synchrotron in Africa. This included conversations and agreements between high ranking academics and politicians from many developing African nations – several Ghana government ministers attended. Simon secured UK funding (£8000: RSC, CCDC & Diamond) to support african student attendance, coordinated a live, on-line remote beamline control session with Diamond, chaired sessions, judged posters & ran workshops. The culmination was formation of an independent African Crystallographic Association. Simon's experience with working in this region led to a pivotal role in a partnership agreement between the University of Southampton and the University of Ghana (the VC and Deans of UoG visited Southampton twice in this period) and he is now key in exploring joint funding opportunities, support from the World Universities Network and larger GCRF bids.

Based on our recently published, high impact, radiation damage work at Diamond, a collaboration has been formed to explore this effect at synchrotrons around the world. The NCS joins the universities of Oxford, Aarhus & Bern with scientists at the ALS (Berkeley) and Australian synchrotrons in a study to begin to benchmark this effect. This led to a successful application to the Advanced Photon Source (Chicago), where 6 days beamtime was conducted by the NCS and Bern teams. Data is currently being worked up. This has led to Simon being on the scientific advisory board for the APS and leading the formation of a team of European academics who will meet in 2020 to formulate a large network proposal based around 'data quality'.

Visits from international collaborators: Prof David Rae (ANU, Canberra) continued a series of visits to look at extremely complex structure refinement problems arising from the NCS. In collaborative applications two international visits have been funded for the next period: Dr Ibrahima Thiam (Dakar, Senegal) funded by the Senegalese government application and Enrico Podda (Cagliari, Italy) will visit on an Erasmus scheme.

Improvement

Equipment Upgrade.

During the reporting period there were significant problems with equipment. One goniometer was returned from the factory in Japan after significant downtime and attention from engineers and has performed without returning problems for 10 months. A second goniometer had a very similar problem and was not in action for most of this period – fortunately a repair was possible on site (after much engineer input) and an entirely new kappa block was installed. This installation means the instrument is showing a greater level of alignment precision than ever before. It was fortunate that these goniometer issues didn't overlap – and as a whole the facility remained available for use and managed to achieve most KPIs at the highest level.

A new water chiller was acquired and replaced one that was ca 20 years old and becoming unreliable. Some minor equipment purchase is necessary to support the advanced techniques and a new collimator and beamstop has been ordered (these need to be custom made) to enable high pressure experiments. A collaboration with Diamond Light Source through a newly funded PhD studentship will produce a new capability for gas cell experiments - a new cell is being made for Southampton (enabling 5 bar pressure as opposed to the current capability of ca 1 bar) which will enable efficient screening for Diamond experiments. The studentship will develop the technique at Diamond and in collaboration with the Research Complex at Harwell to couple gas cells to mass spectrometry – thus providing entirely new capability through insight into the kinetics of solid-gas reactions.

Access and User experience

We have now launched our electronic sample management system (Portal2) and its uptake is progressing well. Some initial implementation issues affected performance in May, however the system was developed by the Research Software Group (part of the Software Sustainability Group) as part of an EPSRC Impact Acceleration project and the team fixed issues rapidly. The system is now fully adopted by the service and its users and is functioning well – as testified by strong feedback eg “The application process is really smooth and user-friendly, so it didn't take long at all” (Prof P.J. Skabara, Glasgow). This new system modernised much of the web-based electronic infrastructure supporting the service, in particular the user interface and administrative aspects. The system has a range of benefits, including:

- Ease of use
- User access to and retrieval of data
- User telemetry for all aspects of sample handling, data acquisition and processing
- Ability for users to submit samples electronically
- Enhanced security
- Better collaboration between the service and its users
- Better back up and disaster recovery
- Improved workflow
- Enables a greater degree of regulatory compliance
- Facilitates quick retrieval of documents
- Access rights model much improved and flexible

A Standard Operating Procedure has been derived and is in use for commercial clients, providing much more rigour and compliance with their processes. 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 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 2019 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, while helping drive goals in most aspects of the Priority 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.” NCS is a strong, key part of UK research infrastructure (with a current entry in the Research and Innovation Infrastructure Portal). An analysis of the current UK Research Infrastructure Roadmap shows it contributes to the Physical Sciences & Engineering infrastructures directly at the Capability and Discovery level (where X-ray diffraction is a named technique) and also it in turn supports the Application (eg Electronics) and Challenge levels (eg Advanced Materials).

Applicants to access the NCS are asked to self-identify which of EPSRC's Research Area(s) they work in. This year, in a total of 289 responses (>1 Research Area can be identified per user), users have identified 1 new area when compared to previous years (Condensed matter: electronic structure).

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

Prosperity Outcome	Priority Framework 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, Condensed matter: electronic structure

Further to this response, users were able to provide 'other information', which resulted in around 60 responses

and identified further areas of interest such as 'Energetic Materials', 'Small Molecule Activation', 'Surface Science', 'Medicinal and Bioinorganic Chemistry', 'Photonic Materials', 'Electrochemistry' and 'Liquid Crystals'.

Sustainability

The commercial service is now in a steady state of operation. It has invoiced for a significant amount of money in its first year – this pays its own way and makes a modest profit. We now have a basis on which to develop and are making efforts to increase the customer base. We realise that the conventional approach has a finite market – which we are not too far from reaching. Therefore we are evolving approaches to engage with SMEs and different markets and also different modes of operation e.g. through consultancy consortia (see Economic Impact section). 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, diversifying our capabilities and being seen as a global leader also makes for a more sustainable operating basis (see previous sections).

Efficiency Developments

Portal2 is live and providing an excellent user experience - an important component of maintaining a satisfied and engaged user community. Additional functionality and benefits include a streamlined application and allocation management process for users and staff; automated updates from the diffractometers on data collected; better telemetry for users; reduction in administrative burden on the NCS team; reduction in time taken to monitor/collect KPIs & SLAs required for reporting; modular architecture on well established development platforms, which improves its ability to be upgraded and maintained long into the future. Development of a Standard Operating Procedure has enabled us to understand the commercial workflow better and become more efficient at delivering a consistent service. Opportunities for small equipment funding to update components or support advanced techniques are more commonplace and we will continue to pursue these.

Sustainability Plans

Growth of the commercial service: We expect gradual growth as we reach out to smaller companies and diversify our approach. However there is potential for a step change through the collaboration with Merck and Rigaku where we not only conduct a high profile contract to take a product to market, but then go on to exploit that as part of NCS service provision – more detail on this project will be presented in future reports.

Engagement with larger community efforts: We have long term strategies including: Involvement in large consortia grants (see ERC Synergy above); international initiatives both in crystallography and much broader aiming projects (see International section); the potential to charge a modest amount for training in academia e.g. to CDTs; acting as consultants for crystallographic endeavours (see last annual report re proposed UAE service).

Development of advanced techniques: Provision of these capabilities is challenging with the resources available, but is growing and gaining traction. Continued effort will open up new funding streams through collaborative funding applications, which we expect to see as these techniques become more embedded in NCS provision.

Synergy with other NRFs: A close alignment to the new PSDS service, which has a basis in crystallographic data, means that in the long term the NCS will be at the heart of driving data science for structural chemistry.

Summary

Year one of operation in the current funding period involved setting up a range of new elements of the NCS. Year two has largely been one of embedding these new elements into the regular operation of the service. Year 3 (this reporting period) has been about consolidating these and learning how the NCS can deliver them alongside core activities, how to balance restricted resources and where we need to focus efforts to grow and move forward.

It has been a solid year for core activities, with performance and outputs not dropping despite many other, new commitments. 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 and we now believe to be resolved, the NCS was able to study all samples within acceptable KPI levels and was not once at a standstill. Our most significant achievement has been the full establishment of the commercial service, which has been a very successful endeavour, with several industry customers and now forming an entirely new group of users for the NCS. The technical member of staff is now fully trained and has delivered a considerable amount of contract work – we have billed for around £64k this year, which exceeds our original set target and is sustainable.

The second new element of service provision is that of the advanced techniques, which are five different types of science/experimental approach. These are embedded and growing, but at different rates due to being introduced in a phased manner in order to manage change in workloads and ability to ramp up. Variable temperature work is mature with publications forthcoming. The gas environment work has perhaps provided the most exciting results, with a range of work being undertaken and high profile article published and in preparation. Charge density studies are now beginning to happen and the high pressure support labs are established with studies scheduled. A publication based on facilitating access to the neutron facility is being prepared and we have a high profile article that has opened up understanding of the phenomenon of sample radiation damage. The rolling training programme is fully established and delivering to a range of audiences at different levels of expertise.

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: Evolving a Circular Plastics Economy
2. Grant Reference Number or Facility Name: EP/S025537/1 and EP/R023816/1
3. One sentence summary: National Crystallography Service data is providing crucial information in the global challenge to find an alternative to plastic.
4. One paragraph Summary Data from the University of Southampton's National Crystallography Service (NCS) is providing key information to researchers at the University of Hull in their work to discover new environmentally-friendly plastics.
5. Key outputs in bullet points: <ul style="list-style-type: none"> • Strengthening ties with China and Japan through overseas travel grants from the EPSRC, The Royal Society and the British Council. • Exchange of personnel and sharing of equipment. • Broadened collaborations across the University of Hull research landscape and local industry with the award of an UKRI Creative Circular Plastic grant.
6. Main body text Plastic pollution is one of the biggest challenges facing our planet today. Historically, plastics are petrochemical-based and resistant to decay, and some plastics such as polyethylene can take centuries (on average 400 years) to decompose in landfill sites. This means that much of the plastic that has been produced during our lifetime still remains in its near original form. These plastics have started to leach into our rivers and oceans - often in microscopic form – and, as a result, they are starting to enter the food chain. Widespread media coverage has increased the pressure to move away from single use plastics and either recycle much more of what we currently use or to invest in research and development to discover new environmentally-friendly plastics that retain the desirable properties required for widespread application. Researchers at the University of Hull are exploring ways that renewable natural resources could be used to develop alternative materials with greener, biodegradable features. Professor Carl Redshaw, Professor of Inorganic Materials Chemistry at the University of Hull said: "One route to such materials is to polymerize monomers accessible from natural resources. To achieve this, a catalyst is generally required, which typically comprises a metal centre surrounded by bound organic compounds (ligands). By changing the nature of the ligands in terms of their size, shape and charge, it is possible to change both the catalytic activity of the system and the resulting polymer properties. "X-ray crystallography plays a central role in defining the structure-activity relationships of these catalysts, which in turn allows the optimum catalyst structure to be identified." The Redshaw group has a long-standing collaboration with the NCS at Southampton stretching back 20 years. Carl said: "Data from the NCS is critical to moving our research forward and has led to us achieving results that have made it possible to get many successful grants. "We can do a range of research in our labs at Hull but we have one of the oldest working diffractometers in the country that just can't handle many of the samples that we are producing. The only definitive way of knowing exactly what we have got is to get an X-ray diffraction study done on a single crystal, so to be able to send our

samples to Southampton to get this data is essential to our work. We need to know exactly what the catalyst structure is, as that helps us determine the ratio we use with the monomer that we are going to polymerize to make the polymer. Alongside this we are also carrying out investigations to ensure that the resulting polymer does not contain toxins.

"If we can identify the right catalyst, the ultimate aim would be to replace plastics such as polythene with a polymer with the same properties that would biodegrade in a couple of years. That would be a big step forward.

"Data from the NCS has helped us obtain a grant from the Plastics Research Innovation Fund (PRIF) which has facilitated us actively engaging with departments across the University, and talking to end-users such as supermarkets, local councils and industry to find out more about what they require."

7. Names of key academics and any collaborators:

Professor Carl Redshaw, Professor of Inorganic Materials Chemistry at the University of Hull.

The Evolving a Circular Plastics Economy grant is an institutional award and involves many collaborators across the University of Hull research landscape, including Dr Pauline Deutz (Geography), Prof. Dan Parsons and Dr Will Mayes (Energy & Environment Institute), Dr Vicky Skoulou and Dr Sharif Zein (both from Chemical Engineering), Prof. Rudi Wurzel (Politics), Prof Richard Barnes (Law), Prof. Nishikant Mishra and Prof. Gerald Midgley (Management Systems), Dr Felix Why (Psychology), Dr Chandra Kambhampati (Computer Science) and Dr Michael Farrelly (English).

Crystallographic work (dataset workup) is either conducted in-house (Dr Tim Prior) or externally (Dr Mark Elsegood at Loughborough).

8. Sources of significant sponsorship (if applicable):

£914,631, UKRI via the EPSRC 01 Jan 2019 to 30 June 2020 – 'Evolving a circular plastics economy'.

£79,539, EPSRC, 18 March 2018 to 17 April 2021 – 'Coordination chemistry approaches to societal issues: environment and health'.

9. Who should we contact for more information?

Carl Redshaw: c.redshaw@hull.ac.uk; 01482 465219.

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.