Annual Report for EPSRC National Research Facilities

Facility: EPSRC National Crystallography Service

Address: NCS, Chemistry, University of Southampton, Highfield Campus, University Road, Southampton, SO17 1BJ

Director: Professor Simon Coles

Facility Manager: NCS Operations Manager, Dr Graham Tizzard; NCS Coordinator, Dr Robert Bannister

Value Proposition
Crystal structure analysis is the dominant and most informative characterisation technique available and is a cornerstone underpinning cutting edge research in chemistry and related disciplines. The National Crystallography Service (NCS) provides structural characterisation support which crucially drives research across the range of disciplines performing chemical synthesis and materials characterisation. The NCS facility is amongst the most powerful and highest throughput of its type in the world. Building on this platform, its core service provides state-of-the-art structure characterisation, significantly beyond that found in home departments, for chemists, materials scientists and crystallographers in academia and industry. Crystallisation or subsequent diffraction quality of a new compound is not predictable and so synthesis chemists need timely recourse to higher-powered facilities, but not for all their samples – thus making a centralised facility the only scalable and financially viable option. The ability to handle the most demanding samples in a University-based facility provides an effective screen for a follow-on synchrotron service. The NCS has regular access to the world leading I19 beamline at Diamond Light Source, making the use of this facility as efficient, cost effective and timely as is possible. 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. The NCS greatly enhances the reach of the technique by the in-situ study of dynamic solid-state systems at high temperature, pressure and in different gaseous environments. This underpins developments in areas of strength e.g., energy, carbon capture, functional and catalyst materials, but requires specialised equipment and significant experience to perform such experiments, often considered the preserve of the expert crystallographer. The NCS broadens the impact of these techniques by making them available to the wider chemistry community. The NCS is a world leading facility and spreads its expertise by educating, facilitating, and empowering non-experts across the community, particularly in providing advanced training opportunities. It increases the levels at which structure characterisation can be carried out in the UK and enables a step change in accessibility of the technique to entirely new communities.
Scientific Excellence
Core Service Highlights The NCS provides a state-of-the-art service for structure characterisation, significantly beyond that found in home departments, for chemists, materials scientists and crystallographers in academia and industry. It enables the UK to remain a world-leader in structural science, providing structural characterisation support which is crucial to drive research across the range of disciplines performing chemical synthesis. The NCS underpins cutting edge and impactful research, as demonstrated by the publications list and the particular highlights given below. A recent review by Simon Coles in the 2020 issue of 'Structure and Bonding' (21st Century

Challenges in Chemical Crystallography) entitled “Leading Edge Chemical Crystallography Service Provision and Its Impact on Crystallographic Data Science in the Twenty-First Century” extensively details the scientific excellence delivered by the NCS core service and is now the standard citation for the use of the NCS.

The exceptional level of publications outputs (see Publications section below) is testament to the scientific excellence both supported and driven by the NCS. In this reporting period 24 co-authored papers were published and this is supplemented by at least the same number of papers also arising from the data collection only service. Some publication highlights, selected from very high Impact Factor journals, during this period include:

- A paper by the crystallography group in Southampton, where a highly complicated crystallographic analysis was only possible due to the facilities and expertise of the NCS, was published in Nature (https://doi.org/10.1038/s41586-021-03194-y). Despite the system(s) being based on a very simple organic salt, we discovered a highly complex system, which reproducibly and consistently produced the largest crystal structure ever characterised (in terms of Z’). Coles and Hursthouse, the two NCS directors over the years, have around 80 years experience between them and are two of the most published crystallographers ever – both consider this to be their most significant and impressive crystallographic study.
- A whole new family of differentially substituted phthalocyanine-porphyrin hybrids was recently accessed due to some elegant synthesis work by collaborators at the University of East Anglia. The products resulted in highly challenging crystallographic work, which was necessary to unequivocally characterise them, that was overcome by the NCS. The results were published in Angewandte Chemie (https://doi.org/10.1002/anie.202016596).
- In close collaboration with colleagues from Sussex University, a new approach to C-H activation was discovered and characterised. A range of techniques were required to fully understand and confirm the chemistry where a Cu(II) complex forms a radical species which then initiates an alkyne C-H bond activation. The product formed post-catalysis was confirmed by some intricate crystallographic analysis and the work was published in the journal ‘JACS Au’ (https://doi.org/10.1021/jacsau.1c00310).
- ‘Rotaxanating Metallo-supramolecular Nano-cylinder Helicates to Switch DNA Junction Binding’ is an example of a high impact publication where recourse to high-powered X-ray facilities was a crucial contribution to completing a very complex study. The NCS performed data collection for colleagues in Birmingham, who were collaborating with large groups in France and China and the results were published in JACS (https://doi.org/10.1021/jacs.0c07750).

Advanced Techniques Highlights

The advanced techniques provided by the NCS perform solid-state chemistry studies and drive a range of transformative research areas. They centre around the use of in-situ diffraction experiments to monitor in-operando dynamic processes in complex systems, which addresses major global issues as well as providing fundamental understanding e.g. providing atomic resolution detail as to how MOFs can be utilised for carbon capture and hydrogen storage while variable temperature work sheds light on the functioning of organic electronics and catalysts. The techniques provided are variable temperature, high pressure, gas cell, charge density/quantum crystallography and access to neutron single crystal diffraction. Besides the technical advances in these areas described elsewhere in this report, there are two main outputs of note in this reporting period.

A paper in JACS resulting from NCS collaboration with Weller (York) is a particular highlight from the solid-state – gas reactivity work. It reports a series of exceptionally novel compounds that could not be synthesised by any other route than reaction of a single crystal with a gas. A series of linear and branched alkanes were reacted with a rhodium complex, generating σ-bound products. The gases ranged from propane to 3-methylpentane, which enabled a systematic study into the steric impact of these novel ligands on the nature of both the complex and the overall crystal structure.

The second highlight was in the area of charge density / quantum crystallography. Here the NCS collected ultra-high resolution data on a series of compounds where systematically varying functional groups were placed in the peri- positions of a napthalene core. These functional groups were chosen and designed to be 'reactive' with one another to different extents. The quality of the data enabled accurate modelling of the charge densities and thereby calculation of quantum mechanically derived properties. The results were combined with solid-state NMR and revealed various stages of bond formation never before characterised to this resolution. The results were published in Angewandte Chemie and had associated press releases. This work is presented as a case study – see the final section of this report.

Methods Development

The Crystal Sponge (CS) technique is a new way of characterising the molecular structure of samples only producing nanograms of analyte, where routine crystallisation is unfeasible, or with a sample that refuses to crystallise. It is based on soaking a porous crystal in a dilute solution of the analyte in question and performing X-ray diffraction on the host-guest complex. This technique requires a particular, technical approach and so has not been widely adopted. The NCS teamed up with Rigaku (Japan) and Merck (Germany) companies to explore the reproducibility and remit of the technique with a view to establishing a commercial operation and applying it to academic research areas. Rigaku initiated a 'Joint Evaluation Project' whereby Merck provide commercial grade CS materials and through Knowledge Transfer train the NCS to conduct the technique. Rigaku provided the NCS with ca £400k of state-of-the-art diffraction equipment to conduct the work. The NCS role is to embed the technique and explore routes to making it available as a service – this aspect was funded by an EPSRC Impact Acceleration Account grant which bought out the time of Dr Orton, established a well-founded crystal soaking laboratory and paid for consumables. During the reporting period the project was initiated, the soaking laboratory established, the diffraction equipment installed and a significant body of work conducted to establish the scope of a commercial service. The project has been highly successful, producing many new results on pharmaceutical-like materials and expanding the envelope of compounds studied. The project was publicised to the community via a talk at the BCA 2021 Spring Meeting, generating considerable interest. Accordingly, a commercial and academic CS service has been proposed as part of the NCS renewal. In the forthcoming reporting period the remit will be expanded to academic research samples through a second Impact Acceleration Account grant, a business model and commercial service will be established in collaboration with Merck and the CS operation will become a core part of the NCS operation. This activity is globally unique – the technique is the preserve of a handful of academic research laboratories and no other facility in the world can deliver a CS service at scale, either to industry or academia. Our results indicate it will be a transformational technique that opens crystallographic characterisation to entirely new areas of the research community.

Through NCS work with Weller et al (York) and a collaboration with Diamond Light Source, significant advances are being made in the development of gas cells for single crystal diffraction. The current NCS work involves the use of an advanced 'static' cell and is the first facility outside of a synchrotron to use such technology. However, through a PhD studentship jointly supported by Diamond Light Source and the University of Southampton, we are now significantly advanced in the development and prototyping of a 'flow' gas cell for single crystal diffraction. This will not only enable the gas environment to be varied in a controlled way, but also the sensing of the content of the gas stream coming out which will enable monitoring and characterisation of reactions. To the best of our knowledge, this is the first of its type in the world and when complete will enable entirely new information about solid-gas reactions to be gathered.

Community Leadership

During this period a significant review in the journal/book hybrid 'Structure & Bonding' was published by Simon Coles. The review is entitled 'Leading Edge Chemical Crystallography Service Provision and Its Impact on Crystallographic Data Science in the Twenty- First Century'. It describes the equipment, infrastructure and processes required to operate such a cutting edge and large facility, bringing in synchrotron radiation developments. It then describes how such facilities feed into databases, in turn fuelling a range of different data mining and other research activities, and is beginning to enable data science in the chemistry-based disciplines. This review also contains extensive supplementary material comparing facilities data and is now the formal citation for the use of the NCS.

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 workshop organised by him at the IUCr Congress and General Assembly in August 2021 (https://www.iucr.org/resources/data/commdat/prague-workshop-cx) was designed to gather global input into the requirement for an organised and supported approach to managing raw data. The NCS activity in this area was presented as an exemplar. The workshop was a resounding success with the result that Simon is the global community lead to write a White Paper to be taken to the IUCr executive providing recommendations as to how the Union should proceed in this area. The workshop outcomes have been published widely as full articles in the crystallographic popular press (American Crystallographic Association and British Crystallographic Association with others to follow).
Simon Coles is a leading advocate for FAIR data in the chemistry community. Recent work has driven this agenda forward, particularly by using the approaches developed by the crystallography community as an example to others. Simon is a member of the International Union of Pure and Applied Chemistry (IUPAC) Task Force on FAIR data which encompasses all this work and will report its recommendations for implementation in late 2021.

Publications

The number of publications that the NCS is associated with is very considerable a three-year list is too long to incorporate into the body of this report and so is appended at the end. The NCS generates different types of outputs that are dictated by the type of operation – these are crystal structure characterisations where NCS collects data and passes it to a user to work up, analyse and report; crystal structure determinations that NCS performs and reports in collaboration with users; larger scale projects where NCS drives the research and its publication.

Crystal structures invariably form a key component of a paper if it is possible to acquire them – for many they are an invaluable, definitive characterisation of a new chemical compound and therefore are always considered critical to a paper. We therefore choose to categorise the papers in the following way (indicating how they might be equated to H, M, L as requested, however we stress that this is inappropriate for NCS operation).

The NCS publication categories are thus:

- DCO (Data Collection Only – approximately equivalent to L). NCS collects data on samples which cannot be examined using local facilities or where there are no local facilities - the user solves, analyses and publishes the data themselves. The user is required to at least acknowledge the service in the publication using a particular form of words and preferably also cite a paper outlining the NCS operation (Coles & Gale, Chem Sci, 2012, 3, 683-689). Publications are therefore tracked in several ways – searching for the acknowledgement, sifting citations of the NCS paper and requesting returning users to provide publication outputs. Tracking of these papers can at times be difficult to enforce and accurately perform – we believe there are likely to be some additional papers however it is not a sensible use of staff time to take the additional measures required to trace these.
- FSA (Full Structure Analysis - approximately equivalent to M). NCS collects, solves and analyses data on samples which cannot be examined using local facilities or where there are no local facilities – NCS collaboratively publishes the crystallography results in a publication driven by the user. NCS staff are authors on the publication, which facilitates tracking (supplemented with searching for the acknowledgement, sifting citations of the NCS paper and requesting returning users to provide publication outputs).
- C (Crystallography led study - approximately equivalent to H). A study led by the NCS or where the facility has played a core role. These are solid-state structural chemistry studies as opposed to those performing crystallography for the purpose of characterisation of a new compound. These papers are often based on the NCS advanced techniques and methods development and would not be possible without NCS staff expertise and state-of-the-art instrumentation. NCS staff are always co-authors, enabling accurate tracking.

NCS Publications from the period 1st September 2020 – 31st August 2021

1 (DCO) M. R. Ward and I. D. H. Oswald, *Crystals*, 2020, **10**, 1–13.

2 (DCO) J. J. Jones, A. P. M. Robertson, G. M. Rosair and A. J. Welch, *Russ. Chem. Bulletin*, 2020, **69**, 1594–1597.

3	(FSA) P. J. Holliman, C. P. Kershaw, E. W. Jones, D. Meza-Rojas, A. Lewis, J. McGettrick, D. Geatches, K. Sen, S. Metz, G. J. Tizzard and S. J. Coles, *J. Mater. Chem. A*, 2020, **8**, 22191–22205.
4	(FSA) Z. S. Al-Taie, Z. S. Al-Taie, S. R. Anetts, J. Christensen, J. Christensen, S. J. Coles, P. N. Horton, D. M. Evans, L. F. Jones, F. F. J. De Kleijne, S. M. Ledbetter, Y. T. H. Mehdar, P. J. Murphy and J. A. Wilson, *RSC Adv.*, 2020, **10**, 22397–22416.
5	(FSA) Z. S. Al-Taie, J. M. Anderson, L. Bischoff, J. Christensen, S. J. Coles, R. Froom, M. E. Gibbard, L. F. Jones, F. F. J. de Kleijne, P. J. Murphy and E. C. Thompson, *Tetrahedron*, 2021, **89**, 132093.
6	(FSA) C. M. N. Choubeu, B. N. Ndosiri, H. Vezin, C. Minaud, J. B. Orton, S. J. Coles and J. Nenwa, *J. Coord. Chem.*, 2021, **0**, 1–13.
7	(FSA) C. M. N. Choubeu, B. N. Ndosiri, H. Vezin, C. Minaud, J. B. Orton, S. J. Coles and J. Nenwa, *Polyhedron*, 2021, **193**, 114885.
8	(FSA) R. R. Mittapalli, S. J. Coles, W. T. Klooster and A. P. Dobbs, *J. Org. Chem.*, 2021, **86**, 2076–2089.
9	(DCO) P. De'Ath, M. R. J. Elsegood, C. A. G. Halliwell and M. B. Smith, *J. Organomet. Chem.*, 2021, **937**, 121704.
10	(FSA) N. Abdullah, L. N. Ozair, H. Samsudin, G. J. Tizzard, S. J. Coles and M. I. Mohamadin, *J. Coord. Chem.*, 2021, **74**, 1947–1964.
11	(DCO) T. Xing, C. Jiang, M. R. J. Elsegood and C. Redshaw, *Inorg. Chem.*, 2021, **60**, 15543–15556.
12	(FSA) M. A. Altahan, M. A. Beckett, S. J. Coles and P. N. Horton, *Inorganics*, 2021, **9**, 1–17.
13	(DCO) X. Zhang, K. Chen, M. Chicoma, K. Goins, T. J. Prior, T. A. Nile and C. Redshaw, *Catalysts*, 2021, **11**, 1–19.
14	(FSA) J. Devonport, L. Sully, A. K. Boudalis, S. Hassell-Hart, M. C. Leech, K. Lam, A. Abdul-Sada, G. J. Tizzard, S. J. Coles, J. Spencer, A. Vargas and G. E. Kostakis, *JACS Au*, 2021, **1**, 1937–1948.
15	(DCO) A. A. Y. Guilbert, Z. S. Parr, T. Kreouzis, D. J. Woods, R. S. Sprick, I. Abrahams, C. B. Nielsen and M. Zbiri, *Phys. Chem. Chem. Phys.*, 2021, **23**, 7462–7471.
16	(C) E. Podda, M. Carla Aragoni, M. Arca, G. Atzeni, S. J. Coles, G. Ennas, F. Isaia, V. Lippolis, G. Orru, A. Scano, J. B. Orton, A. Pintus and A. Scano, *J. Nanosci. Nanotechnol.*, 2021, **21**, 2879–2891.
17	(DCO) O. Santoro, M. R. J. Elsegood, S. J. Teat, T. Yamato and C. Redshaw, *RSC Adv.*, 2021, **11**, 11304–11317.

18 (C) A. J. Bukvic, A. L. Burnage, G. J. Tizzard, A. J. Martínez-Martínez, A. I. Mckay, N. H. Rees, B. E. Tegner, T. Krämer, H. Fish, M. R. Warren, S. J. Coles, S. A. Macgregor and A. S. Weller, *J. Am. Chem. Soc.*, 2021, **143**, 5106–5120.

19 (C) G. J. Rees, M. B. Pitak, A. Lari, S. P. Day, J. R. Yates, P. Gierth, K. Barnsley, M. E. Smith, S. J. Coles, J. V. Hanna and J. D. Wallis, *Angew. Chemie - Int. Ed.*, 2021, **60**, 23878–23884.

20 (DCO) M. G. Albuquerque, R. S. B. Gonçalves, C. H. da S. Lima, F. L. de A. Maia, S. de P. Machado, L. do N. Oliveira, T. U. da Silva, J. L. Wardell and S. M. S. V. Wardell, *J. Mol. Struct.*, 2021, **1246**, 1–14.

21 (FSA) K. M. Fortune, C. Castel, C. M. Robertson, P. N. Horton, M. E. Light, S. J. Coles, M. Waugh, W. Clegg, R. W. Harrington and I. R. Butler, *Inorganics*, 2021, **9**, 1–20.

22 (FSA) A. N. Cammidge, F. Alkorbi, A. Díaz-Moscoso, J. Gretton, I. Chambrier, G. J. Tizzard, S. J. Coles and D. L. Hughes, *Angew. Chemie Int. Ed.*, 2021, **60**, 7632–7636.

23 (DCO) K. Wang, K. Chen, T. Bian, Y. Chao, T. Yamato, F. Xing, T. J. Prior and C. Redshaw, *Dye. Pigment.*, 2021, **190**, 109300.

24 (DCO) D. Mandal and G. M. Rosair, *Crystals*, 2021, **11**, 1–12.

25 (C) E. Podda, S. J. Coles, P. N. Horton, P. D. Lickiss, O. S. Bull, J. B. Orton, A. Pintus, D. Pugh, M. C. Aragoni and R. P. Davies, *Dalt. Trans.*, 2021, **60**, 3782–3785.

26 (DCO) L. S. De Moraes, J. Liu, E. Gopi, R. Oketani, A. R. Kennedy and Y. H. Geerts, *Crystals*, 2021, **11**, 1004.

27 (C) İ. Yılmaz, N. Acar-Selçuki, S. J. Coles, F. Pekdemir and A. Şengül, *J. Mol. Struct.*, 2021, **1223**, 129271.

28 (FSA) H. M. O. Connor, S. Sanz, A. J. Scott, M. B. Pitak, W. T. Klooster, S. J. Coles, F. Chilton, E. J. L. Mcinnes, P. J. Lusby, H. Weihe, S. Piligkos and E. K. Brechin, *Molecules*, 2021, **26**, 1–9.

29 (DCO) T. M. Seck, F. D. Faye, A. A. Gaye, I. E. Thiam, O. Diouf, M. Gaye and P. Retailleau, *Eur. J. Chem.*, 2020, **11**, 285–290.

30 (FSA) M. A. Altahan, M. A. Beckett, S. J. Coles and P. N. Horton, *Phosphorus, Sulfur and Silicon and the Related Elements*, 2020, **195**, 952–956.

31 (FSA) S. M. Meier-Menches, B. Aikman, D. Döllerer, W. T. Klooster, S. J. Coles, N. Santi, L. Luk, A. Casini and R. Bonsignore, *J. Inorg. Biochem.*, 2020, **202**, 110844.

32 (FSA) A. Garau, L. Lvova, E. MacEdi, G. Ambrosi, M. C. Aragoni, M. Arca, C. Caltagirone, S. J. Coles, M. Formica, V. Fusi, L. Giorgi, F. Isaia, V. Lippolis, J. B. Orton and R. Paolesse, *New J. Chem.*, 2020, **44**, 20834–20852.

33 (DCO) M. Kieffer, R. A. Bilbeisi, J. D. Thoburn, J. K. Clegg and J. R. Nitschke, *Angew. Chem. Int. Ed.*, 2020, **59**, 11369–11373.

34 (DCO) H. C. Gardner, A. R. Kennedy, K. M. McCarney, E. Staunton, H. Stewart and S. J. Teat, *Acta Cryst.*, 2020, **C76**, 972–981.

35 (DCO) M. Molloy, A. F. R. Kilpatrick, N. Tsoureas and F. G. N. Cloke, *Polyhedron*, 2021, **212**, 115574.

36 (FSA) G. Picci, J. Milia, M. C. Aragoni, M. Arca, S. J. Coles, A. Garau, V. Lippolis, R. Montis, J. B. Orton and C. Caltagirone, *Molecules*, 2021, **28**, 1–16.

37 (FSA) I. R. Butler, D. M. Evans, P. N. Horton, S. J. Coles and P. J. Murphy, *Organometallics*, 2020, **40,** 600-605.

38 (FSA) I. R. Butler, D. M. Beaumont, A. M. I. Bruce, N. N. Zaitseva, J. A. Iggo, C. Robertson, P. N. Horton and S. J. Coles, *Aust. J. Chem.*, 2020, **74**, 204–210.

39 (C) R. Montis, L. Fusaro, A. Falqui, M. B. Hursthouse, N. Tumanov, S. J. Coles, T. L. Threlfall, P. N. Horton, R. Sougrat, A. Lafontaine, G. Coquerel and A. D. Rae, *Nature*, 2021, **590**, 275–278.

40 (DCO) C. A. J. Hooper, L. Cardo, J. S. Craig, L. Melidis, A. Garai, R. T. Egan, V. Sadovnikova, F. Burkert, L. Male, N. J. Hodges, D. F. Browning, R. Rosas, F. Liu, F. V. Rocha, M. A. Lima, S. Liu, D. Bardelang and M. J. Hannon, *J. Am. Chem. Soc.*, 2020, **142**, 20651–20660.

Impact

Training Courses and workshops hosted by NCS

The training highlight of the year was an online webinar series “NCS Crystallography for beginners” which was conducted in collaboration with the CCDC. The webinars were aimed at undergraduate students as a way for them to be able to improve their research projects, given the reduction in available lab time due to COVID. The workshops were specifically on Olex2, Mercury and using the CSD, with a particular focus on the basics of the various software offerings, how to use them and what they could be applied to. The partnership with CCDC was particularly useful, given their experience of running events on this scale and so their support on delivery was invaluable.

The webinars were fully booked in advance (within 24 hours of going live), which ultimately resulted in between 75-100 people actually attending each session. Many more people asked to be able to attend, watch the events afterwards or if we would be running future sessions. There was only minimal promotion on social media, yet still the posting reached over 50,000 people, with 3,000 engagements i.e ‘Likes’, etc. The event page itself was viewed over 5,000 times. The response was quite unexpected and high demand for such training was evident. We also had many requests from post-graduates, PDRAs and academics for a similar course pitched at a slightly more advanced level. The recorded webinars have now been made available on the NCS YouTube channel and website training page (see section 13). There are plans to run more of these in the future and build a library of resources.

The NCS provided training for the AI3SD-Directed Assembly joint EPSRC Networks (virtual) summer school (https://www.ai3sd.org/2021/05/20/ml4mc-summer-school/). Simon Coles was a tutor and lead mentor for a project group that successfully ran over 6 weeks and explored the application of Machine Learning approaches to finding and categorising interesting features in raw diffraction data.

Activities to promote the facility beyond its core user base

Simon Coles presented the NCS and its pharma-related capabilities, particularly the developing novel Crystal Sponge capability at the Rigaku Pharmalytical Summit https://www.rigaku.com/Summit-2021/Pharma. This was a global event attended by over 300 members of the pharmaceutical industry and which generated leads for the NCS commercial service.
The Crystal Sponge work, which will provide an entirely new capability for the NCS, was presented at the BCA 2021 Spring meeting as a talk by James Orton, which generated considerable discussion. His associated contribution to the poster session won the Industrial Group poster prize.

As noted in the previous section, Simon Coles was the lead organiser of a global community workshop at the International Union of Crystallography 2021 Congress. This workshop gathered global input into the requirements for managing raw data and the NCS activity in this area was presented as an exemplar. Simon is the global community lead on writing a White Paper to be taken to the IUCr executive providing recommendations as to how the Union should proceed in this area. The workshop outcomes have been published widely as full articles in the crystallographic popular press (American Crystallographic Association and British Crystallographic Association with others to follow).
Peter Horton presented a talk to the Education group the 2021 American Crystallographic Association conference. It described how we have adapted teaching methods to support a hybrid online/physical environment for hands-on training with instruments augmented by online methods to guide students.

Public engagement

Due to pandemic restrictions, most of our established methods and routes for public engagement were not possible. National level engagement activities essentially stopped, while some local ones were taken online. The director was involved in designing and conducting online sessions for Hampshire Scouts, enabling 100's of young people to gain their science badge while conducting experiments from home. He was also involved in an online outreach event for the Royal Society of Chemistry Mid-Southern Counties Section and conducted a crystal growing competition.

For the second year running the Southampton Science and Engineering Festival, the largest of its type in the south of the country, took its activities online. The usual crystallography stand converted its activities into instructions for experimenting from home and the director was present to meet members of the public online at specific times to explain what we do. The resources made available by the NCS for SOTSEF 2021 online are available at http://sotsef2020.geodata.soton.ac.uk/all_activities/?zone=chemistry

Perhaps the most significant activity of the period was a collaboration with the Cambridge Crystallographic Data Centre. Based on the International Year of the Periodic Table project led by Simon Coles (https://www.ccdc.cam.ac.uk/Community/educationalresources/PeriodicTable/) we set out to develop some outreach resources. The result was "Battlecards" - a game for any age and background, based on the well-known Top Trumps, https://www.ccdc.cam.ac.uk/Community/educationalresources/PeriodicTable/activities/. A series of cards were designed and made available for print-out and followed up with a launch online, particularly on Twitter and Instagram. Subsequently we developed an approach whereby the game itself could be played on Instagram. The Battlecards are dual branded but hosted by the CCDC, a world-wide organisation which maximises the reach of the game.

Facility staff training and career development

Dr James Orton is an NCS Research Technical Professional, who moved from providing the commercial NCS structure analysis service to being the lead for the Rigaku-Merck JEP project on Crystal Sponges. This significant step up in responsibility, not only for introducing a new technique but also management of a team, wholly merited a promotion, a process which James readily achieved. Dr Eleanor Dodd joined the team as a Research Technical Professional, replacing Dr Wim Klooster who held a PDRA post, and taking over responsibility for providing the commercial service. Including Mr Christopher Holes (part-time with NCS) there are now three RTP's in the NCS team and so we have embarked on developing a programme that supports and recognises them specifically and ensures a career development path. They are now integrated with the University technician community, while James and Christopher have begun (Eleanor to follow) the process of professional registration, specifically with the Registered Science Technician scheme (RSciTech) program.

Four team members (Laura McCormick, Eleanor Dodd, Robert Bannister and James Orton) attended a professional personal coaching course available to Post-Doctoral Researchers in the department. Entitled 'Develop from within' this course developed a personalised reading of an individual's strengths and development areas and all attendees strongly felt it helped them identify areas to concentrate on a seek to develop (mostly in professional practice but also an element of personal).

Conferences and workshops continue to be a key way for staff to develop. Due to pandemic restrictions these were limited to on-line attendance only, however several valuable events were attended. The British Crystallographic Association Spring Meeting continues to be a priority event each year and at the 2021 meeting James Orton presented the Crystal Sponge project to the Industrial Group and received very strong feedback indeed. The team also attended (online) the hybrid International Union of Crystallography Congress in Prague, which was a 6-day event with 7 parallel sessions and provided many opportunities to stay abreast of current developments in the field. Finally, all members of the team attended a range of online workshops during the period – the most notable being the Rigaku Advanced Crystallography Summer School and the 14th High Pressure Diffraction workshop. Both these events were fundamentally training exercises and certainly resulted in increasing the skills of participants and are significant in the field, providing recognised training certificates.

Cost Recovery

The NCS is free at the point of access, which is the only viable financial model. This is because a) researchers cannot predict in advance whether they will produce samples that could only be investigated using advanced facilities/expertise and b) only some samples for a particular study will require advanced facilities whilst the remainder can be studied locally. For such a mixed model it is impractical to implement and anticipate anything other than a 'free-at-point-of-access' mechanism. Also access to synchrotron facilities is already free at the point of access and therefore charging to access via a NCS is incompatible with this model. Access to the NCS is subject to peer review on scientific merit and moderated to ensure the volume request is appropriate for the project being supported, which prevents access purely to avoid paying local charges.

However, some cost recovery is obtainable through a very successful commercial service. The commercial arm of the NCS serves the pharmaceutical industry with a professional and efficient service – it is used by several different companies, including big pharma. Additionally, it is possible to recover some costs from external grant applications – up until now this route has been somewhat limited and has tended to cover capital more than running costs.

Year	Running Costs	Grants	Other Academic	Students	Industry	Other*	%
2018	£576,000				£35,000		6.1
2019	£514,000				£49,000		9.5
2020	£465,000				£36,000	£12,000	10.3
2021	£518,000				£33,000	£48K (IAA)	15.6

Figures rounded to the nearest £1000

The reporting period has been a mix of busy periods of activity and some tougher times due to periods of lockdown or other pandemic related restrictions on facility activity. This has impacted our commercial clients as well as ourselves. With that said, £33,000 industry income represents a 6.4% cost recovery. Whilst this is short of the 10% KPI, it does show that even during a time where many in industry are cutting back costs, the NCS is seen as critical to their operations.

The NCS was successful in obtaining an EPSRC Impact Acceleration Account grant to cover the costs of setting up a facility for crystalline sponge work and to prepare for a commercial partnership with Merck. This, with the commercial service, resulted in a cost recovery of 15.6% which far exceeds the KPI requirement.

The figures above also do not account for a £400k in-kind contribution from Rigaku, in the form of a new instrument necessary for work on the crystal sponge project. This is further discussed in Section 12.

There is potential for increasing cost recovery levels if the NCS can embark on new activities and the Statement of Need exercise identified a number that would achieve this. These form the basis of our future plans in the next period of the NCS.

- A significant increase in advanced techniques work was clearly considered worthwhile and would be in demand. We anticipate this initially being free at the point of access in order to perform proof of concept work. Proof of concept would be expected to lead to incorporation (and therefore funding) into larger, funded project proposals e.g. to research councils and charities.
- Additionally, a crystallisation service was considered a very worthwhile addition. This would lend itself both to external grant income and to a highly attractive commercial service. We expect to incorporate our Crystal Sponge service into this endeavour, as well as other innovative crystallisation technology.

Users

NCS users fall into six categories: a. Research Group Principal Investigators. 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 (although some groups are well into double figures). b. UK service crystallographers. As 'gateway' users, this access mode supports large numbers of researchers, as departmental crystallographers access the NCS to analyse difficult samples from across their entire user base. c. Collaborators using Advanced Techniques. d. Commercial Users. All of these are from the health and pharmaceutical sector. e. International Collaborations. More details are provided in the Links section of this report. f. People accessing the training programme. There were no face-to-face training activities held in this period (see Impact section of this report). Prospective academic users (categories a-c above) must make an application in response to a six-monthly call to access the NCS. This application must estimate an allocation of expected samples that will require the NCS facilities i.e., there is great uncertainty as to how many new compounds will be made, how many of these can be crystallised and how many of these will be of lesser quality and therefore not examinable with local facilities. Furthermore, departmental service crystallographers (category b above) apply on behalf of several academics in their department, further increasing the uncertainty around the likely number of samples that will be produced. On application these users are asked to identify academics they are submitting on behalf of, however no information on numbers of students, PhD candidates or PDRAs is requested from category a-c users due to the significant additional requirements that would be placed on users to gather this information for an application relating to speculative samples. We cannot therefore report on the level of granularity of users as requested. It should also be noted that commercial users are counted as companies, not individuals, and within each company there are generally many research scientists who use our services but through a gateway user - again we do not request this level of information of our clients. Furthermore, the NCS has not previously kept records of new and repeat users, but this information is now included here for the first time. The user information presented in the table below is therefore that which is most appropriate for the NCS and which we are currently able to gather.

Year	Academic	Industry	Other	No. Repeat	No. unique
2018	65	8	49		
2019	66	6	51		
2020	67 (+48)*	7	0**	6 repeat industrial, 56 repeat academic	1 new industry contact, 11 new academic
2021	74 (+63)*	20 Users from 6 companies	0**	6 repeat companies 62 repeat academic	0 new companies, 12 new academic***

*Some of the academic users are departmental crystallographers who submit samples on behalf of other users in their department, totalling an additional 63 users of the service.
** No in person training events took place during this period due to COVID restrictions within the department.
**Of those with an account new users represent 16% of our total users this year.

Research Areas

NCS users originate from 58 different institutions. Approximately 85% of our users are based in chemistry departments, while the remaining 15% originate from chemical engineering, natural science, materials science, defence materials, pharmacy and biochemistry departments. The geographic spread of users is even and touches all regions of the UK. The most common research areas include catalysis, chemical structure, synthetic chemistry (organic, supramolecular, organometallic, inorganic etc.), carbon capture, energy storage, small molecule activation, materials for energy applications.

User Surveys / Satisfaction

Due to the requirement for a community driven Statement of Need exercise (SoN) to be conducted during this period, there had been significant engagement and various mechanisms for feedback and input – both from current and potential users. Concerned that we would cause a level of 'survey fatigue' which would not provide meaningful feedback, it was decided not to conduct the usual satisfaction survey at the same time. Further, due to the pandemic restrictions, it was decided that rather than forcing users to reapply every 6 month period that allocations are simply rolled over on a pro-rata basis. The user satisfaction survey is an integral part of the application process for returning users – and with this exercise not being conducted the user survey was therefore not conducted either. As operations now move towards a post-pandemic level, we will be resuming the normal application rounds and therefore also the user satisfaction survey.

We continue to receive feedback through more informal routes such as within email correspondence. However, the following comment is of particular note as it was received as part of a journal article review:

"There are problems with the X-ray data, and these have been rightly highlighted by the authors. The fact that data were collected by the NCS means that it is unlikely that anyone could collect a better dataset, and it would be a shame if this chemistry were hidden away because of this. The authors do not give too much emphasis on metric parameters which is sensible, but I would accept the X-ray data as presented - I for one defy anyone to achieve better!"

However, what is of exceptional note is that during the reporting period a full Community Statement of Need exercise was conducted. This is a considerable consultation exercise and while it does not replace direct feedback on usage of the service it does in some respects imply satisfaction – and many positive comments were received from satisfied users during the exercise. The community engagement and consultation exercise was multi-facetted with numerous activities, with the most relevant to this reporting being a widely distributed (including by the British Crystallographic Association, indicating the key community role it sees the NCS playing) survey fronted by an independently convened group. The results of this survey are presented at https://ncs.ac.uk/ncs/wp-content/uploads/sites/426/2022/02/Statement-of-Need-Questionnaire-Results.pdf. 193 respondents provided user community demographics from academic and commercial sectors, feeding into further targeted discussions conducted by running focus groups and interviews. 91% said that the use of facilities offering collection of data on weakly diffracting samples would be useful or essential to their research. 83% said advanced structure determination (e.g. from datasets of twinned crystals) would be useful or essential to their research. Between 67 and 78% of community members indicated their research would greatly benefit from new insights enabled by advanced techniques, while a further 20% would now consider the potential benefits. Additionally, 85% indicated that an advanced crystallisation service would be highly valuable. Some indicative comments contributed during this exercise that indicate satisfaction with the NCS include:

"This is a fantastic service that is essential for many researchers in universities where there is no single crystal diffraction facility. Without it we could not carry out our research. The costs of housing and staffing these facilities in our own department are too high and shared central facilities support by experienced staff are essential."

"NCS's ability to deal with weak diffractors and twinned crystals is really essential for our work."

The survey was a primary source of information in defining the key community requirements of the NCS and these are expressed in the Statement of Need response which can be found at https://www.ncs.ac.uk/ncs/wp-content/uploads/sites/426/2022/02/Statement_of_Need_2020.pdf.

Service Demand

The service provided can be separated into two types based on the extent of the study (see also Publications Section) - Full Structure Analysis (FSA) and Data Collection Only (DCO). The DCO service is for experienced users requiring access to more advanced instrumentation at the NCS to collect data on challenging crystals. The FSA service is accessed by users who are not familiar with solving crystal structures or require the help of expert crystallographers for particularly difficult structures. The fundamental difference is that the DCO service generally only requires instrument time and is relatively quick to turn around, although crystal manipulation and selection can be challenging, and data may often need reprocessing. The FSA service requires the same amount of resourcing to collect the data but a considerable amount of expert crystallographer input to generate a result. The Service Level for processing the data resulting from the DCO service is 5 days, whereas the target for processing FSA samples is 20 days.

On application, an allocation is awarded which is prioritised into high, medium and low priority samples. On receipt a sample is logged into our Portal system with time until data collection Service Levels of 10, 20 and 30 days for High, Medium and Low priority samples respectively (this prioritisation system is regardless of whether a sample is FSA or DCO as prioritisation is based on scientific merit).

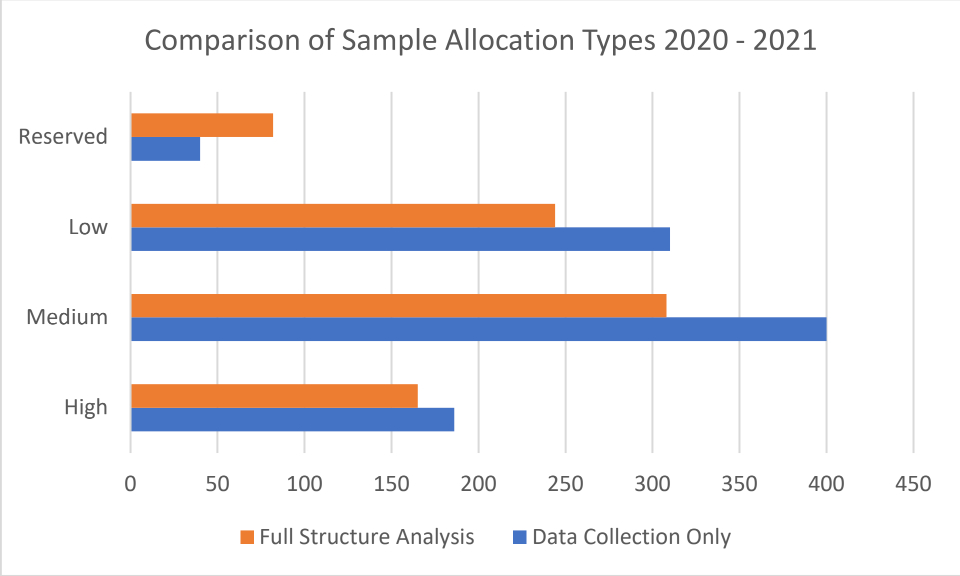
All commercial samples are FSA, but instead of L/M/H priority these are treated as bands 1/2/3. This banding refers to the difficulty of the sample and therefore how much resource is likely to be required to generate a result. The banding is directly linked to a pricing structure. Band 1 requires less than 4 hours on an instrument and is a relatively simple structure to solve; Band 2 is between 4 and 24 hours on the instrument and is reasonably difficult to process; Band 3 includes any sample requiring > than 24 hours data collection or a particularly challenging data analysis. Whilst these figures don't specifically align to the high, medium and low brackets for academics, they provide an indication of instrument time required for routine, difficult and extremely challenging samples. It is noted that processing times can vary drastically depending on sample difficulty. The table below summarises the Service Level criteria for DCO and FSA analyses.

Service				Commercial	
	DCO*	**FSA***		**Band**	**Time****
H	15	30		**3**	>24
M	25	40		**2**	4-24
L	35	50		**1**	<4

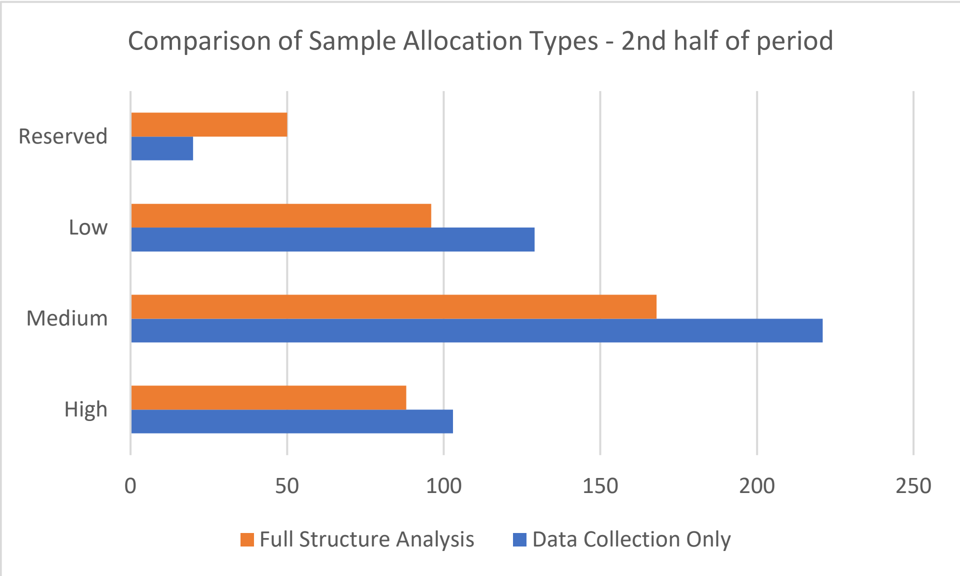
* Total number of working days to provide result
** Time on instrument (hours)

Theoretical Demand

The NCS operates a biannual call for applications for access to generate allocations. Academics apply for the number of samples they expect their group will generate in a 6-month period. The NCS Strategy and Allocations Panel (SAP) review this request on scientific merit and to ensure a reasonable workload for the service. An additional category, 'Reserved', is introduced at this point: if a large request is made then some samples can be allocated here – these will not be included in Service Level reporting and are examined only if there is spare capacity and no turnaround time guarantee is given. The Reserved mechanism allows the service capacity to be fine-tuned at the stage when the demand is essentially unknown. This is the basis of *theoretical* demand and capacity levels. The number of samples allocated during this reporting period are shown below.



The ratio of H:M:L is allocated to provide an appropriate workload. While there is a higher proportion of DCO samples, over 40% of the capacity relates to FSA samples, which are more demanding of time and expertise.
The number and spread of samples allocated in the 2020-2021 period is identical to the 2019-2020 period. This is to be expected, as in the first half of this period user allocations were rolled over to relieve unnecessary pressure on all during a demanding pandemic restricted time. The second half of the period, when users had to apply for their allocations again, shows a typical spread with most samples being in low and medium data collection only and medium full structure analysis categories.



Actual Demand Vs. Capacity

Service capacity has been affected somewhat by reduced capacity in the chemistry department. Often during this period we were only allowed one person from the group in the building. Datasets could be processed at home but a reduced occupancy in the department did reduce the amount of data that could be collected. Demand for the service has also been lower in the last two years than in previous years, due almost exclusively to restrictions on people being able to access their laboratories. Previously, to measure the actual demand vs capacity, the number of samples submitted in a period was compared to turnaround time for respective KPIs. Due to instrument upgrades, as discussed in section 9, it has not been possible to properly monitor the rate at which samples go through the system. Therefore, to report actual demand for this period it is only possible to report the number of samples completed in this period compared to the previous year. Whilst this does not accurately provide a sample turnaround time, it does show a trend in demand for the service. Since the ability to process samples has not been affected, and the NCS has been prioritised by the University to allow us to continue working, turnaround time has not been the biggest factor in our demand vs. capacity.

	2019-2020	2020-2021
Low	130	164
Medium	198	277
High	171	174
Reserved	34	69
Total	533	684
DCO	339	470
FSA	194	214

We report an increase of 22% in completed samples over the previous year. The first half of the year complete 278 samples, while in the second half the remaining 406 were completed, suggesting a return to more normal demand for the service.
Following upgrades to the Portal2 system to account for the instrument upgrades, in the next reporting period it will be possible to return to tracking turnaround time to ensure that demand is met.

Risks

A risk register is maintained for all capital equipment, either whole entities or components where appropriate, that the facility crucially relies on and has a value >£10k. The register is regularly reviewed by the Operations and Management Team (OMT), which passes concerns or strategic requirements to the Strategy and Allocations Panel (SAP).

In this period Core Equipment identified through the register was installed (goniometer, cryostat, microscope & optics). Further, the aims of the NCS for 2022-2027 were defined by a Statement of Need exercise. In combination with these needs and the risk register, the equipment base was extensively reviewed and the requirement for another replacement goniometer and detector incorporated into the renewal proposal.

The register also identified a persistent water chiller problem causing failure of the most important X-ray generator in very hot weather – a replacement has now been installed and a further chiller has been identified for replacement due to age and unreliability.
Other facility related risks are assessed by the OMT and/or SAP as appropriate. NCS is embedded in School of Chemistry strategy, with the Head of Department attending OMT once a quarter and SAP twice a year, which ensures senior management are aware of major issues. The risks considered include:

- Those associated with staffing absence or resignation - reasonable number of PDRAs so some redundancy, Director covered by department,

- Financial - regular review to ensure overspend, contingency plans if funding renewal doesn't occur,
- Disaster recovery e.g. fire, pandemic – covered by Departmental policy and mitigation/recovery measures put in place,
- Host institution issues e.g. infrastructure failure – reporting channels clear, close two-way interaction with School Facilities Manager.

Management of the current Covid-19 pandemic situation illustrates the strong risk prevention measures in place. The department cautiously opened soon after the first lockdown ended in June 2020, with the NCS amongst the first to return due to the low level of laboratory occupancy required and high potential for remote control and monitoring of experiments. Prior to return, extensive and rigorous Risk Assessments and Method Statements were devised at several different levels – research groups leaders were engaged and trained from the outset and templates cascaded down through the levels to ensure risk levels minimised. Controlled access and occupation levels were devised and coupled with rigorous hygiene and PPE measures to ensure infection control. Weekly reviews were performed and reported up the chain of command. With time the department moved to different tiers of control which involved gradual increase of occupancy with associated review and rewrite of RAMS processes and documentation. Extensive Covid-19 safety training has been provided to all staff reoccupying the buildings with documentation, meetings and support provided via online systems. A rigorous system of notification, risk assessment and sign off is required when further staff, servicing engineers, etc are admitted into the department. Throughout this time a dedicated member of senior staff has overseen the Covid-19 response and risk assessment. The NCS has been deemed business critical, with suitable procedures in place, meaning that it is amongst the last operations to be shut down in the case of lockdown. With all these risk prevention measures in place it has been possible for the NCS to continue to operate under pandemic conditions throughout the entire reporting period.

KPIs and SLs

			SLA Level				
Type	**Description**	**Time for Performance**	**Green**	**Amber**	**Red**	**1st Sep 2020 - 31st Aug 2021**	**Directors Comments**
RMI	Total number of all Users	Period associated with specific report	N/A	N/A	N/A	74 Routine Service users (+63 from departmental crystallographers)	
RMI	Spectrum of user types	Period associated with specific report	N/A	N/A	N/A	63 individual service users, 11 departmental crystallographers, 6 commercial clients	
RMI	Number of University / Research Groups Involved	Period associated with specific report	N/A	N/A	N/A	58 Institutions / 106 Groups	
RMI	Percentage of Access	Period associated	N/A	N/A	N/A	100% (Routine service) / 100%	

	Requests Accepted	with specific report				(Advanced Technique)	
RMI	Percentage of equipment time dedicated to different access modes	Period associated with specific report	N/A	N/A	N/A	60% (Routine service, academic) / 10% Commercial / 20% Host institution / 10% (Advanced techniques)	
RMI	The number of completed samples		N/A	N/A	N/A	684	This is an increase of 149 samples on last year indicating a gradual return to our normal figures. 278 between Sept and Feb and then 406 between March and August. The latter half of the year is much closer to our normal statistics pre COVID. **
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 of Total Available Time within Period	Period associated with specific report	95% and above	>90% but <95%	90% or less	97.6%	Several equipment upgrade installations contribute to the facility availability. Particularly with Covid restrictions in place it was not possible for >1 person to be in the laboratory at any one time. The installation of a whole new diffractometer on the spare Cu port for Crystal Sponge work and the much needed upgrade to the ultra-high intensity Mo goniometer were the two main events that contributed to facility downtime.

SL	Percentage of Access Costs recovered	Period associated with specific report	10	6	2	15.6	
SL	Number of Publications	1 year	30	20	10	40	
SL	Number of publicity activities per year	1 year	10	8	6	13	
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%	
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	79.6%***	Due to pandemic restrictions regarding access to the Chemistry department and multiple occupation of spaces, the laboratory was only able to operate at 25% occupancy. So effectively only 1 member of staff could be in the facility at a time and there are 4 instruments to support. Instrument upgrades also compounded this problem. Despite this obstacle and largely due to judicious scheduling and exceptional flexibility of staff, statistics of 67-90+% efficiency is an impressive achievement.
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	90.7%***	
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	88.6%***	
SL	Time from examination to end result	Data collection = 5 working days	95% and above	>90% but <95%	90% or less	72.3***	
SL	Time from examination to end result	Full Structure Analysis= 20 working days	95% and above	>90% but <95%	90% or less	67.8***	

** This number does not take into account any advanced technique experiments such as high pressure, gas cell or crystal sponge experiments. The latter has had a single diffractometer dedicated to it for a year and the group have undertaken 350 experiments throughout the year. The experiments involve soaking 3 crystal sponges with an analyte and often all 3 sponges are run on the diffractometer leading to a vast number of datasets which are quite difficult to process and solve.

*** Collection and processing statistics for the period are skewed by an unfortunate situation with the Portal2 system. The recent upgrade of a number of the instruments has resulted in a change in the technical data architecture of the facility. Whilst this upgrade has greatly increased collection times and capacity, data is now acquired too rapidly to be saved directly onto the data server as per the old instrumentation. We are currently in the situation where we have a mixture of old and new instrumentation which is saving data in different ways. This has led to problems with Portal2 accurately detecting when a sample has been run as the bespoke system is hard-wired into the old instrumentation approach. This has resulted in apparent delays that are mostly artificial. At the time of writing, we have begun an extensive rework of the facility data infrastructure so that it can scan for the generation of new datasets – we expect this work to be complete by Easter 2022 and by mid 2022 for the final upgrade to be completed. Thereafter we expect Service Level reporting to return to normal.

Links

NCS has input and collaboration with numerous **Organisations and Facilities:**

- Diamond, through beamline I19 (EH1) are formally key partners in delivering the NCS. This has led to access and use of I19 EH2 and more currently also to I15. Optimising access to Diamond facilities and opening up to more advanced techniques was the subject of a significant amount of activity around the Statement of Need, resulting in new proposed access routes. Furthermore, Diamond and Southampton co-fund and supervise a PhD student aligned to the NCS and split over the two sites, whose goal is to develop and apply a new type of gas cell for in-situ/in-operando studies.
- A collaboration with the SXD beamline at the ISIS neutron source has seen the facilitation of access and data analysis for NCS users that wouldn't otherwise have been aware / capable of using such a facility. This has resulted in publications and a formal arrangement would be proposed for future delivery of the NCS.
- SJC is also the Director of the Physical Sciences Data-science Service and is driving convergence of the two NRFs on crystallographic data management, aggregation and publication.
- SJC advised on the models and approaches for establishing the Computed Tomography NRF and sits on management advisory panel of the Southampton site.
- SJC is a consultant feeding into the direction and setup for the ChemMatCARS beamline at the Advanced Photon Source (Chicago).
- SJC led community discussions/workshops around embedding Quantum Crystallography into regular crystallographic analysis. Firstly with a consortium involving 20 parties across Europe and then more broadly at a global community event. SJC is the lead proposer looking at routes to fund developments in the area.
- Radiation Decay project through a collaboration with Dr Grabowsky (Bern) that is informing synchrotrons (specifically Diamond, APS and ESRF) as to how to deal with this phenomenon.
- BCA Education & Outreach Officer and CCDC – SJC coordinates public engagement initiatives and projects at a national level.
- SJC sits on the IUCr Committee on Data and IUPAC FAIR data taskforce – and through these Unions is driving global approaches to managing, curating and sharing raw data in Chemical Crystallography.

The following **International** institutions have formal links with the NCS, generally relating to student exchange and training and mostly resulting in collaborative papers (these links have been largely dormant during the global pandemic as research activity in most of these regions has been significantly reduced, however papers were produced during this period with Cagliari and UCAD):

- Karlsruhe Institute of Technology - PhD student placement and training, Nature paper published
- Radboud University (Nijmegen) – combined techniques to solve phase transition problems, several papers and student exchange
- King Saud University – collaboration leading to publications
- Wakayama University – student visits and several papers using the charge density technique
- University of Malaya - student training visit and papers published
- University of Mauritius - SJC conducted a training workshop, leading to several papers
- University of Ghana and KNUST - SJC held several training workshops under Royal Society and Leverhume projects, resulting in being a Local and Scientific Organiser of the PCCr2 Pan-african Conference on Crystallography in Accra. Application for World University Network funding for researcher mobility between Southampton and UoG in progress
- University of Cagliari – long collaboration (SJC visiting Prof providing training many visitors to NCS – 1 student in this reporting period, Enrico Podda via Eramus scheme).
- Turkey (Zonguldak & Gebze – SJC visiting academic) continual stream of papers and visits (Gebze student Erasmus visit during reporting period).
- SJC is responsible for setting up and coordinating Cagliari and Gebze as Erasmus exchange partners with Southampton
- UCAD Senegal – Ibrahima Thiam training visit in the reporting period, 3 papers published
- Yaounde University (Cameroon) – SJC provided a training course, resulting in a stream of papers.

Improvements and Future Plans

The methods development section of this report describes the implementation of the novel Crystal Sponge approach. This was facilitated by a project in collaboration with the companies Merck and Rigaku. The Rigaku contribution to the project was the provision and installation of a new state-of-the-art diffractometer worth £400k specifically tailored for the technique. This enabled work to begin immediately without having to make protracted and uncertain funding applications. This proof-of-concept project was primarily a Knowledge Transfer exercise where NCS staff were skilled up and the potential to deliver the technique as a service was explored. The project was highly successful and the provision of a service deemed viable – a contract is being drawn up with Merck to deliver a service to industry, which also has the potential for delivery to academia.

An EPSRC Impact Acceleration Account award (£60k) enabled the establishment of a fully provisioned laboratory for the soaking of crystal sponges prior to structural characterisation. This included numerous incubators, a fridge, pipettes and a wide range of smaller items and consumables. The award also bought out the time of Dr James Orton, enabling him to focus on establishing the laboratory and refining the technique.

A successful EPSRC Core Equipment proposal submitted in the summer of 2020 led to much needed and significant instrument upgrades. The primary advance was the replacement of the goniometer for the ultra-high intensity Mo source, which had become unreliable and the cause of numerous breakdowns, including having to be returned to the factory in Japan. This replacement has provided a 4x faster data collection time, bringing the instrument up to the same technical level as the rest of the components and thereby maximising its potential – this also cements the sustainability of the most important facility instrument. A high-powered microscope increases the capability and throughput of the facility. A new cryogenic system and a very substantial optics upgrade enabled the full operation of the new crystal sponge instrument – at 3x the X-ray flux levels previously achievable.

The Statement of Need exercise to understand and establish the requirement for a national crystallography service was conducted during this period. This is a significant opportunity to widely engage with the community and establish requirements – from which logical improvements can be planned. The Statement of Need was successful and provided a blueprint for the next generation of the service. All the above improvements and some further planned ones fed into the development of a proposal for the service renewal with significant enhancements and new capabilities – this proposal was submitted at the beginning of August 2021.

Website

The NCS website is split into two resources: the home site (http://www.ncs.ac.uk) and the Portal site. The home site is mainly a resource to provide details about services, equipment and contact information. It also hosts reports and case studies.

The old NCS webpage, constructed in 2010 had very much begun to show it's age and was also a bespoke construction that had become almost impossible to administer. A complete overhaul of the whole site was therefore planned and conducted during the reporting period. As a result, the NCS home website has had a complete refresh and been moved to a new WordPress platform. This has given us much greater control over the content and layout of the site and will enable us to edit and migrate content long into the future. A student intern from the university was hired, as a website developer, to build the framework of the site, design a modern and intuitive layout and provide a ready to publish version. She also provided some training so NCS staff can maintain and edit the site in-house in the future.

The site contains all the information content that was in the original pages, such as beam time dates, routes for access, services provided etc. It also now hosts some of our training materials, such as YouTube tutorials and associated resources. We will develop these materials further and building up this area of the site to provide a library of resources and tutorials. This is especially helpful timing, given the recent interest in webinars and online workshops. This new website also allows us to track information that was previously unavailable to us. Using the Exact Metrics program, we can see the number of sessions on our website in a duration, pageviews, the average session duration and the total number of users. We can track how many new and returning visitors there have been to a page and which pages are most popular. The new site has not yet been advertised but these are statistics we can report as of the next annual report.

Our Portal2 site, where users can track their samples through the process of being analysed, is much more current and up to date. Discussions have begun, however, to understand how to adapt the system to allow users to apply for advanced technique experiments. Further developments on this will be provided in the next annual report.

Case Study
We provide a Case Study with Professor John Wallis from Nottingham Trent University (and John Hanna from Warwick University) where the NCS contribution was core, and which is the culmination of over a decade of very close collaboration. The result was a 'Hot Article' publication in the topflight journal Angewandte Chemie. A summary presentation of this Case Study and the full PDF download can be accessed via the NCS website at https://www.ncs.ac.uk/case-studies/.

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