Annual Report for EPSRC National Research Facilities

Facility: EPSRC National Crystallography Service

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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. In its globally leading role, the NCS drives innovation and adoption of novel instrumentation, software, methods, analysis, standards and dissemination.

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

2020 review by Simon Coles 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 the 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 16 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 highly novel Cu(II) complex was shown by to cause Alkyne C–H Activation via a radical-triggered mechanism – this was only possible to understand once the post-catalysis crystal structure was solved and shown to comprise two 3-coordinate copper centres doubly bridged by chloride anions (JACS Au; https://doi.org/10.1021/jacsau.1c00310).
Reversible P–P bond cleavage is relatively rare and notable and this behaviour in a highly unusual Ir(III) / Au (I) complex was only observable and understandable after a comprehensive X-ray crystallographic analysis of the free ligand and both the bound/cleaved complexes (Chem. Comm. https://doi.org/10.1039/D2CC00706A).
The NCS was able to fully and accurately characterise, for the first time, the structure of a well-known compound, 'Noria', enabling Prof James and his team to understand its novel behaviour as a host for the synthesis of an exciting and relatively new class of materials known as porous liquids (Chem. Sci. https://doi.org/10.1039/D1SC03367K).
A serendipitous discovery of a highly unusual and deeply interesting phenomenon was made when Dr Butler observed peculiar and particular crystallisation behaviour of a relatively innocuous compound. The spontaneous self-assembly of a novel arrangement of water molecules, about which the compound crystallised, a so-called molecular water pipe, was elucidated by a combined X-ray and neutron study (IUCrJ; https://doi.org/10.1107/S2052252522003396).
Prof Pope and his team made a series of luminescent Iridium(III) complexes and analysis of their crystal structures enabled establishing an understanding of the link between the systematic variation of ligand substituents and very precise tuning of their phosphorescence (Inorg. Chem.; https://doi.org/10.1021/acs.inorgchem.1c02121)
Working with Prof Goldup, crystallographic analysis was able to unambiguously support confirmation of a novel synthetic strategy to induce specific chirality between two interlocking molecules in a rotaxane (Nat. Chem.; https://doi.org/10.1038/s41557-021-00825-9).

The NCS operates a structure analysis service for industry partners, which is usually a commercial-in-confidence operation. However, in this period there was a publishing highlight arising from Astra Zeneca's use of this service. A crystal structure was able to opportunistically highlight an unnoticed incorrect tautomer assignment and was reported in "Tautomerism troubles: proton transfer modifies the stereochemical assignments in diastereoisomeric structures of spiro-cyclic 5-methyl-2H-imidazol-4-amine dimers" (Acta Cryst. Sect. E; https://doi.org/10.1107/S205698902100668X).

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 and new additions to the service described elsewhere in this report, there are two main outputs of note in this reporting period. The proposal for the NCS renewal was developed and awarded in this period, with a key difference to previous provision being the inclusion / expansion of advanced techniques in collaboration with Newcastle University and we will describe these developments in the next report.

The highest profile piece of advanced techniques work was the publication in Angewandte Chemie of a collaborative study with Profs Wallis (Nottingham Trent University) and Hanna (University of Warwick). Entitled "Mapping of N-C bond formation from a series of crystalline peri-substituted naphthalenes by charge density and solid-state NMR methodologies" this study used a combination of high-resolution electron density studies with a novel solid-state NMR approach to observe and characterise the formation of an (intramolecular) bond. The underpinning work was the subject of a Case Study in our previous report (https://ncs.ac.uk/ncs/wp-content/uploads/sites/426/2022/02/Case-study-Bond-Formation.pdf). The publication of this paper, which was the 'Editors Choice' and therefore in the top 10% of papers in this journal, was the subject of press releases both by Southampton (https://www.southampton.ac.uk/chemistry/news/2021/10/06-new-research-allows-to-see-chemical-bond-formed.page) and Nottingham Trent (https://www.ntu.ac.uk/about-us/news/news-articles/2021/11/scientists-monitor-the-formation-of-a-chemical-bond-for-the-first-time2). There is the additional novelty that the Vice Chancellor of the University of Southampton is a co-author on this paper, which arises from a previous position as a Professor of Chemistry (and user of the NCS) at the University of Warwick.

Methods Development

The Crystal Sponge 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. The adoption of this technique by the NCS team, via a funded knowledge transfer collaboration with Merck and Rigaku was detailed in the previous annual report. During the current reporting period there was considerable consolidation of the technique in the team – it is a fickle process and depends on a range of conditions as well as the behaviour of the analyte in question. Following the establishment of appropriate laboratory facilities and initial training, the NCS has been able to embed the technique and also begin some independent research into understanding and controlling the process - until now it has been shown to work and optimised, but not actually properly and fundamentally understood. Three PhD students associated with this NCS activity are involved in systematic experimental and computational studies to uncover more detailed insight into the process.

A second round of EPSRC Impact Acceleration Account funding bought out the time of Dr Orton to lead this embedding work. 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 2022 Spring Meeting, generating considerable interest. Accordingly, a commercial and academic Crystal Sponge service has been proposed as part of the NCS renewal explore routes to making it available as a service – we are currently conducting a 'blind validation study', run by Merck to establish the NCS credentials in providing the technique and enable us to become a service delivery partner. 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 Crystal Sponge 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 (both for use in our facility, but also collaborating with Diamond in the development and prototyping of a 'flow' gas cell for single crystal diffraction). Recently we procured a bespoke glovebox equipped with a microscope which has been installed in a laboratory adjacent to the main NCS facility. The only similar capability in the UK is at Diamond Light Source.

This is primarily for supporting gas cell experiments, as it will be possible to select sensitive crystals and conduct the difficult and precise process of loading them into the gas cell under an inert atmosphere.

The most notable new experiments performed in this area were on CO2 uptake and absorption in ZIFs (a class of MOFs), where we show it is possible to accurately determine the absorption sites within the pores and the extent to which the framework deforms on gas absorption, which is a whole new level of information in the field and will inform further development of these systems. This is a collaboration with chemical engineers, and we expect it to overlap heavily with the proposed technique development associated with the new electron diffraction facility (see below).

Community Leadership

During this period a significant technological advance came to light - dedicated Electron Diffraction. SJC is a member of Rigaku's Customer Advisory Board and input into development of a new instrument that will transform the field – based on an electron microscope, a dedicated electron diffractometer will be able to perform structural studies on nanocrystals (as opposed to microcrystals with X-rays). This advance opens up whole new areas of chemistry, physics and materials science that couldn't previously access single crystal diffraction. Following up on this development, Simon Coles was PI on a series of proposals that led to success with an EPSRC Strategic Equipment bid. A £3.2M partnership with University of Warwick will establish an instrument at each site to provide a unified national facility for all UK researchers. The NCS are therefore now established community champions and will be the lead for a UK landscape analysis that will gather demand (geographic and scientific) to inform future investments and developments. Further details also provided in the 'Future Developments' section below.

Simon Coles led a successful EPSRC Software for Research Communities proposal (ranked first out of 232 submissions) which is a collaboration with the Olex2 software team at Durham University. The lead up to this involved extensive engagement with the Quantum Crystallography community at a global level to buy-in and scope requirements and will go on to engage with the UK Research Software Engineer community as the project progresses. The endeavour will develop a system that will seamlessly bridge from conventional crystallography to Quantum Crystallography and enable better quality structures and quantum properties to readily be calculated every single crystal structure that is ever produced. This system will curate software coming out of research groups and make it available to all, thereby maximising the longevity, use and efficiency of global research software outputs in this field. The project was launched with a dedicated session to gather views and requirements from the whole community at the International Charge Density Meeting (Aarhus, May 2022).

The Director is the UK 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 is to inform policy at IUCr which will be cascaded out to all national bodies and journal publishers. He is the lead author of a White Paper providing recommendations as to how the Union should proceed in this area and which is informed by his previous engagement activities with the global community. This was written during this reporting period and is currently being considered by the IUCr Commissions before being taken to the IUCr executive. It will be presented and discussed at a workshop (IUCr Congress 2023, Melbourne) being co-organised by Simon. Simon is a founding Editor of IUCr Raw Data Letters, a new concept in journal publishing which addresses some key data management problems and was launched September 2022.

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 communicated its recommendations (https://doi.org/10.1515/ci-2021-0304) during the reporting period. This work fed directly into Simon being one of the chemistry community leads in the WorldFAIR, EU-funded project covering 13 disciplines in this area.

Simon Coles is the founding PI of the Physical Sciences Data Infrastructure, PSDI www.psdi.ac.uk, a national initiative around data management and reuse which conducted a pilot study (£1M funded by EPSRC Digital Research Infrastructure) during this reporting period and is now making the case to become a key new piece of the UK research infrastructure.

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:

- DCO (Data Collection Only). 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). 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). 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 2022

1. (DCO) M. Molloy, Alexander F.R. Kilpatrick, Nikolaos Tsoureas and G. N. Cloke, *Polyhedron*, 2022, **212**, 115574.
2. (FSA) A. A. A. Al-Riyahee, P. N. Horton, S. J. Coles, A. J. Amoroso, S. J. A. Pope, *Polyhedron*, 2022, **225**, 116079.
3. (FSA) L. A. Panther, D. P. Guest, A. McGown, H. Emerit, R. K. Tareque, A. Jose, C. M. Dadswell, S. J. Coles, G. J. Tizzard, R. González-Méndez, C. A. I. Goodall, M. C. Bagley, J. Spencer and B. W. Greenland, *Chemistry - A European Journal*.
4. (DCO) R. Huckvale, A. C. Harnden, M. J. Cheung, O. A. Pierrat, R. Talbot, G. M. Box, A. T. Henley, A. K. De, A. E. Hallsworth, M. D. Bright, H. A. Akpinar, D. S. J. Miller, D. Tarantino, S. Gowan, A. Hayes, E. A. Gunnell, A. Brennan, O. A. Davis, L. D. Johnson and S. Klerk, *Journal of Medicinal Chemistry*, 2022, **65**, 8191–8207.
5. (DCO) T. Xing, M. Derbyshire, M. R. J. Elsegood and C. Redshaw, *Chemical Communications*, 2022, **58**, 7427–7430.
6. (FSA) M. A. Altahan, M. A. Beckett, S. J. Coles, P. N. Horton and C. L. Jones, *Inorganica Chimica Acta*, 2022, **539**, 120998.
7. (FSA) N. Sawicka, C. J. Craze, P. N. Horton, S. J. Coles, E. Richards and Simon J.A. Pope,

Chemical Communications, 2022, **58,** 5733–5736.
8. (DCO) X. Zhang, T. J. Prior and C. Redshaw, *New Journal of Chemistry*, 2022, **46**, 14146–14154.
9. (FSA) S. J. Coles, P. N. Horton, P. Kimber, W. T. Klooster, P. Liu, F. Plasser, M. B. Smith and G. J. Tizzard, *Chemical Communications*, 2022, **2042**, 1–4.
10. (FSA) D. J. Cutler, M. Coletta, M. K. Singh, A. B. Canaj, L. J. McCormick, S. J. Coles, J. Schnack and E. K. Brechin, *Dalton Transactions*, 2022, **51**, 8945–8948.
11. (C) S. J. Coles, D. I. Robinson, A. Davesne and T. L. Threlfall, *Crystal Growth and Design*, 2022, **22**, 3613–3624.
12. (FSA) T. J. Blundell, A. L. Morritt, E. K. Rusbridge, L. Quibell, J. Oakes, H. Akutsu, Y. Nakazawa, S. Imajo, T. Kadoya, J.-I. Yamada, S. J. Coles, J. Christensen and L. Martin, *Materials Advances*, 2022, **3**, 4724–4735.
13. (FSA) A. Jose, D. Guest, R. LeGay, G. J. Tizzard, S. J. Coles, M. Derveni, E. Wright, L. Marrison, A. A. Lee, A. Morris, M. Robinson, F. von Delft, D. Fearon, L. Koekemoer, T. Matviuk, A. Aimon, C. J. Schofield, T. R. Malla, N. London and B. W. Greenland, *ChemMedChem*, 2022, **17**, 1–12.
14. (DCO) J. L. Garbutt, C. F. da Costa, M. V. N. deSouza, S. M. S. V. Wardell, J. L. Wardell and W. T. A. Harrison, *Acta Crystallographica Section E: Crystallographic Communications*, 2022, **78**, 619–624.
15. (FSA) A. S. Diallo, I. E. Thiam, M. Gueye-Ndiaye, M. Dieng, J. Orton, S. J. Coles and M. Gaye, *Acta Crystallographica Section E: Crystallographic Communications*, 2022, **78**, 349–353.
16. (DCO) J. S. Sweet, R. Wang, P. Manesiotis, P. Dingwall and P. C. Knipe, *Organic and Biomolecular Chemistry*, 2022, **20**, 2392–2396.
17. (FSA) T. J. Blundell, J. R. Lopez, K. Sneade, J. D. Wallis, H. Akutsu, Y. Nakazawa, S. J. Coles, C. Wilson and L. Martin, *Dalton Transactions*, 2022, **51**, 4843–4852.
18. (DCO) P. Shaw, S. J. Hassell-Hart, G. E. Douglas, A. G. Malcolm, A. R. Kennedy, G. V. White, L. C. Paterson and W. J. Kerr, *Organic Letters*, 2022, **24**, 2750–2755.
19. (FSA) I. R. Butler, D. M. Evans, P. N. Horton, S. J. Coles, S. F. Parker and S. C. Capelli, *IUCrJ*, 2022, **9**, 364–369.
20. (DCO) A. Ignaszak, N. Patterson, C. O'Brien, A. True, M. R.J. Elsegood, T. J. Prior and C. Redshaw, *RSC Advances*, 2022, **12**, 11672–11685.
21. (DCO) A. Ojeda-Porras, R. Aouzal, C. Wilson and J. Prunet, *Tetrahedron*, 2022, **106-107**, 132630.
22. (DCO) I. A. Kühne, A. Ozarowski, A. Sultan, Kane Esien, A. B. Carter, P. Wix, A. Casey, M. Heerah-Booluck, T. D. Keene, H. Müller-Bunz, S. Felton, S. Hill and G. G. Morgan, *Inorganic Chemistry*, 2022, **61**, 3458–3471.
23. (DCO) M. Edgar, M. R. J. Elsegood, P. Liu, C. R. Miles, M. B. Smith and S. Wu, *European Journal of Inorganic Chemistry*, 2022, **2022**, e202200017.
24. (FSA) A. K. Edmonds, C. S. Oakes, Storm Hassell-Hart, Didier Bruyère, G. J. Tizzard, S. J. Coles, R. Felix, H. J. Maple, G. P. Marsh and J. Spencer, *Organic and Biomolecular Chemistry*, 2022, **20**, 4021–4029.
25. (DCO) J. R. J. Maynard, B. Galmés, A. D. Stergiou, M. D. Symes, A. Frontera and S. M. Goldup, *Angewandte Chemie - International Edition*, 2022, **61**, e202115961.
26. (FSA) M. Carla Aragoni, E. Podda, M. Arca, A. Pintus, V. Lippolis, C. Caltagirone, R. H. Bartz, E. J. Lenardão, G. Perin, R. F. Schumacher, S. J. Coles and J. B. Orton, *New Journal of Chemistry*, 2022, **46**, 21921–21929.
27. (DCO) D. Dimitrova, C. McMahon, A. R. Kennedy, J. A. Parkinson, S. G. Leach, L. T. Boulton, D. D. Pascoe and J. A. Murphy, *Tetrahedron*, 2022, **128**, 133120.
28. (FSA) R. E. Powell, M. R. Lees, G. J. Tizzard and P. J. Van, *Acta Crystallographica Section C: Structural Chemistry*, 2022, **78**, 63–69.
29. (FSA) D. J. Cutler, M. Coletta, M. K. Singh, A. B. Canaj, L. J. McCormick, S. J. Coles, J.

Schnack and E. K. Brechin, *Dalton Transactions*, 2022, **51**, 8945–8948.
30. (DCO) X. Zhang, T. J. Prior, K. Chen, O. Santoro and C. Redshaw, *Catalysts*, 2022, **12**, 1–23.
31. (DCO) L. Barluzzi, S. R. Giblin, A. Mansikkamäki and R. A. Layfield, *Journal of the American Chemical Society*, 2022, **144**, 18229–18233.
32. (DCO) K. Maciel de Santiago-Silva, B. Taciane, L. do N. Oliveira, F. L. de A. Maia, J. C. Castro, I. C. Costa, D. B. Lazarin, J. L. Wardell, S. M. S. V. Wardell, M. G. Albuquerque, C. H. da S. Lima, W. R. Pavanelli, M. de L. F. B. Bispo and R. S. B. Gonçalves, *Antibiotics*, 2022, **11**, 1402.
33. (DCO) T. M. Horsley, M. F. Mahon, J. P. Lowe, R. M. Bailey and D. J. Liptrot, *ACS Catalysis*, 2022, **12**, 8214–8219.
34. (DCO) W. T.A. Harrison, *Acta Crystallographica Section E: Crystallographic Communications*, 2022, **78**, 737–741.
35. (DCO) G. M. d Lima, C. H. da S. Lima, S. de P. Machado, E. T. da Silva, T. U. da Silva, J. L. Wardell and S. M. S. V. Wardell, *Journal of Molecular Structure*, 2022, **1269**, 133783.
36. (DCO) T. W. Price, I. Renard, T. J. Prior, V. Kubíček, D. M. Benoit, S. J. Archibald, A. M. Seymour, P. Hermann and G. J. Stasiuk, *Inorganic Chemistry*, 2022, **61**, 17059–17067.
37. (FSA) A. de Juan, D. Lozano, A. W. Heard, M. A. Jinks, J. M. Suarez, G. J. Tizzard and S. M. Goldup, *Nature Chemistry*, 2022, **14**, 179–187.

Impact

Training Courses and workshops hosted by NCS

We adapted well to the lockdown period, running a series of online workshops that were well attended. However, our transition back to normal/pre-pandemic activities was admittedly slower. In the reporting period we trained approximately 10 PhD students to run the ‘Spectroscopy in a Suitcase’ activity in preparation to take it out to schools as an NCS coordinated activity.

The NCS facilities attained a Laboratory Efficiency Assessment Framework, LEAF, bronze award (amongst the first 3 laboratories in the University to do so). This is a self-assessment process which is then externally examined once a particular level has been achieved. Taking a lead, the NCS then ran a training workshop in collaboration with Peter Morgan (a Geography Research Technical Professional and UoS LEAF ambassador) to guide other chemistry users through the process, which resulted in five other groups embark on the scheme that day.

Whilst we have had a slightly slow restart to training activities following lockdown, we have several plans for the start of our next period. The new and highly novel NCS apprenticeship scheme will begin as part of the NRF renewal, where the NCS will host a number of residential students to spend time with NCS staff and facilities to learn about our processes and capabilities as well as increase their key crystallographic skills. These apprentices will then become NCS ambassadors back in their home institutions and become part of a growing cohort through networking and workshop activities long into the future.

We have planned training visits from international Research Technical Professional colleagues – from Germany (Regensberg) via the ERASMUS scheme and the Manager of the X-Tech Labs initiative in Benin (Cotonou) for three months. We will also be restarting the advanced training series of workshops, with a disorder workshop in Southampton and a second session planned as a focussed activity for the Midlands in Birmingham, which will enable us to reach a wider audience without excessive travel. Finally, we are in discussions with CCDC planning a follow up to our successful ‘Crystallography for beginners’ course that we ran collaboratively online during lockdown.

Activities to promote the facility beyond its core user base

The main areas of activity which will broaden the NCS remit by a very considerable amount have been driving the uptake and implementation of the exciting new areas of Electron Diffraction and Quantum Crystallography. Both have involved a considerable amount of community awareness raising and educating regarding the potential of these techniques. This has been through a range of national and international community engagement activities – online events, in-person events at international conferences and surveys. There is no doubt that these two techniques represent the future directions of the field, opening up crystallography to entirely new subject areas and providing new levels of information from crystallographic results. These are the two primary 'hot topics' in structural science right now and the NCS is leading the way in both. Further details are provided elsewhere in this report – two major grants in these areas were awarded during the reporting period and there are many promotional activities planned for the next period.

Also, Simon Coles is a world-recognised leader and champion for FAIR research data – and is leading the way through working with two international scientific unions – the International Union of Pure and Applied Chemistry and the International Union of Crystallography. In this work the approaches adopted by the NCS are constantly used as an exemplar and in the field of chemical crystallography Simon is the lead within IUCr for championing publication and access to raw data to support scientific studies. Following an international workshop in Prague (summer 2021), built on considerable community engagement through surveys and popular press articles, Simon has been the lead author on a white paper on the matter, which is currently being considered by various Commissions of the Union. There will be a workshop at the IUCr Congress 2023 in Melbourne, co-organised by Simon, on the adoption and implementation of his recommendations.

Public engagement

This period saw the return of an in-person Southampton Science and Engineering Festival (SOTSEF). The event was a huge success with thousands of members of the public showing up on the day. The activities NCS ran included crystal gardens, salt names, unit-cell cut and stick activities alongside some others.

We primed the team for this activity with a visit to Droxford Primary School as part of their Science week. Two NCS staff members went to the school and ran a day of unit cell building and salt names which was exceptionally well received, receiving the following feedback:

"On behalf of all of us at Droxford Junior School, we wanted to say thank you so much for coming in this week and working with the children. We really appreciate you giving up your own time and planning and resourcing such a brilliant session for the children.

We have had such positive feedback from the children, staff and parents."

Finally, following the training of a number of PhD students to run the Spectroscopy in a Suitcase activity, we ran our first session at Itchen College.

Facility staff training and career development

One of our technicians, Dr James Orton, was successful in becoming a 'Registered Scientist'. The application process was supported by the UoS, as part of the Technicians Commitment initiative. Several of the NCS staff contributed to a 1-day conference showcasing the broad and varied roles which technical staff play within the university. This included various seminars and workshops on ways that technical staff can engage with, and best utilise, professional services for grant applications, publications and raising awareness of support networks and resources available.

There was a dedicated poster and networking session throughout the day, with a formal presentation session (chaired by the RSC).

The NCS attended the BCA Spring meeting with James Orton, Eleanor Soper, Robert Bannister and Robert Carroll presenting posters and Robert Bannister, Simon Coles & Robert Carroll giving talks on the crystal sponge method. Eleanor Dodd presented a poster at ICDM9 in Aarhus Denmark and Robert Carroll and Eleanor Soper presented posters at the 25th International Conference on the Chemistry of the Organic Solid State (ICCOSS) in Ohrid, Macedonia.

James Orton & Robert Bannister attended a conference and workshop, Opportunities and Landscape of Funding for Research Technical Professionals, held at the University of Warwick by the Warwick Analytical Science Centre (WASC). This was a meeting for Research Technical Professionals to learn how to apply for funding and how facilities can be included on grants.

Finally, the NCS was awarded the LEAF bronze award to show our commitment to sustainable laboratory operations. This was an effort made by the whole group to improve our general operation and strive for a more sustainable operation. We will continue to renew our LEAF award moving forward and will strive to achieve a silver award in the near future.

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 recur 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	£12,000			£36,000		10.3
2021	£518,000	£48,000			£33,000		15.6
2022	£426,000	£31,000			£24,000		12.9

**Figures rounded to the nearest £1000*

This reporting period has seen quite a significant drop off in use from our most regular client. This has been softened by an increase in use from two other smaller clients but is not sufficient to cover the difference. It is not yet clear what the cause for this reduction in use is (pandemic related, no longer require crystallography, etc).

A regular NCS user, Prof Andrew Weller (York), was successful in obtaining an EPSRC grant to utilise NCS advanced techniques access to conduct gas cell experiments. The NCS is in the process of increasing this kind of activity and we hope it will yield more successful grant opportunities in the future. This, with the commercial service, resulted in a cost recovery of 12.9% which exceeds the KPI requirement.

Looking forwards, the crystallisation service is likely to attract further external grant income and provide a valuable commercial service. In the medium term we expect to attract intense industry interest in the new electron diffraction capability and as this becomes more embedded in the NCS it will provide further custom.

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. New
2020	67 (+48)**	7	0**	1 new industry contact, 11 new academic
2021	74 (+63)*	6 companies	0**	12 new academic
2022	67 (+70)*	6 companies	0**	10 new academic

*Number of applicants to the service

**Some of the academic users are departmental crystallographers who apply and submit samples on behalf of other users in their department
*** No in person training events took place during this period due to COVID restrictions within the department.

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

A User Satisfaction Survey was conducted in this period and was sent to 64 recent users of the service, from which we received 27 responses. Respondents were given 4 statements and asked to rate them 1 (Strongly disagree) - 5 (Strongly agree). The statements and average responses were as follows:

- The NCS is easy to use, e.g. ease of application, clear sample forms and guidance – 4.81
- The NCS is responsive to user requests, e.g. rapid turnaround, additional data collections etc – 4.81
- 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. – 4.81
- I would recommend the NCS to a colleague – 4.96

Respondents were also asked to provide any additional comments (over 20 were received) the overwhelming majority of which were about quality of service and were extremely positive. Some examples include:

> *“Without this service it would be impossible for us to gain crystallographic data. It is really key for us to continue to perform research. The service is easy to use and communication has been great. Please keep it up!”*
>
> *“We have been using the NCS service for a number of years now. The team has been extremely helpful and reliable in terms of data collection and assisting in preparation of manuscripts for publication. We do not have any in-house facilities available to us and so this service is invaluable for our research activities.”*
>
> *“The NCS is an outstanding service without which I wouldn't have been able to publish a significant number of papers over many years.”*
>
> *“All my interactions with people at the NCS have been of the highest standard and they are a fantastic group of researchers. Super helpful and absolutely essential to my research throughout my career.”*

Two key themes / terms that cropped up throughout these comments are ‘reliable’ and ‘essential’.

Some feedback was also received which indicated areas where the service could improve (and approaches to do so are already underway):

> *"The 6-monthly applications are quite time consuming and arduous, but understand that these might be necessary"*
>
> *"Turnaround time is not always great, but I guess that's down to weight of demand."*

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).

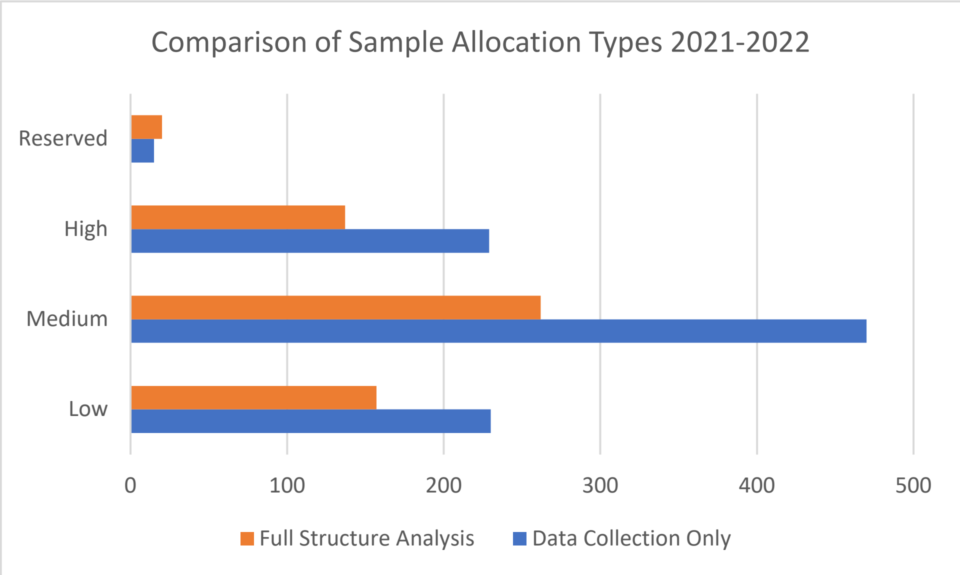
All commercial samples are FSA, but instead of L/M/H priority these are treated as bands 1/2/3. This banding refers to the difficulty of the sample and therefore how much resource is likely to be required to generate a result. The banding is directly linked to a pricing structure. Band 1 requires less 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, approximately 40% of the capacity relates to FSA samples, which are more demanding of time and expertise. Due to an increase in instrument speed and because the majority of time is spent on FSA samples we have allowed more data collection only samples through and relied less heavily on the reserved category.

Actual Demand Vs. Capacity

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 mentioned in the last report, 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. 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	2021-2022
Low	130	164	125
Medium	198	277	290
High	171	174	149
Reserved	34	69	8
Total	533	684	572
DCO	339	470	351
FSA	194	214	221

Taking a 3-year view, it is seen that there is an upturn in samples run in the 2020-2021 period, which we have attributed to a ramp up in synthetic chemistry activities following the pandemic lockdown period (many groups returned to the lab to find several samples had crystallised due to a long period of being undisturbed). The 2021-2022 period represents something of a return to regular (pre-pandemic) levels. In the next reporting period we will be able to more accurately compare our turnaround times to KPIs and better reflect our capacity.

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).

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.

The service is to employ an apprentice technician, who's principal job will be the maintenance of the equipment and minimising the risk of instrument failure. This will allow us to undertake a higher level of active monitoring and management of all safety and operations.

Comprehensive and effective management of the Covid-19 pandemic situation illustrates the strong risk prevention measures in place. This includes active management, responsive planning, development of risk assessments and change in working practice.

KPIs and SLs

			SLA Level				
Type	**Description**	**Time for Performance**	**Green**	**Amber**	**Red**	**1st Sep 2021 - 31st Aug 2022**	**Directors Comments**
RMI	Total number of all Users	Period associated with specific report	N/A	N/A	N/A	67 Academic service users (+70)* users	
RMI	Spectrum of user types	Period associated with specific report	N/A	N/A	N/A	57 individual users and 10 service crystallographers (+6 commercial companies)	
RMI	Number of University / Research Groups Involved	Period associated with specific report	N/A	N/A	N/A	58 Institutions / 127 Research Groups	
RMI	Percentage of Access Requests Accepted	Period associated with specific report	N/A	N/A	N/A	100% Routine Service	
RMI	Percentage of equipment time dedicated to different access modes	Period associated with specific report	N/A	N/A	N/A	60 % Standard NCS time, 10% Commercial clients, 20% Host institution, 10% advanced techniques	
RMI	The number of completed samples		N/A	N/A	N/A	572*	
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	2 working days	95% and above	>90% but <95%	90% or less	100%	

	to within Stated Window						
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	99.6%	
SL	Percentage of Access Costs recovered	Period associated with specific report	10	6	2	12.9%	
SL	Number of Publications	1 year	30	20	10	37	
SL	Number of publicity activities per year	1 year	10	8	6	16	
SL	The time from arrival of a sample to logging in and	within 2 working days	95% and above	>90% but <95%	90% or less	100%	

	informing a User of receipt						
SL	The time a sample is in the queue	From logging in a sample to examination: High Priority sample = 10 working days	95% and above	>90% but <95%	90% or less	** 8 days	An on-going issue in the last reporting period, extending through this and into the next is the gathering of accurate reporting data via Portal. The upgrade caused unanticipated effects, which we are working hard to resolve. Reporting data has been difficult to generate and is unreliable – this and pandemic recovery are the reasons for not meeting the highest sample turn-around targets.
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	** 20 days	
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	** 33 days	
SL	Time from examination to end result	Data collection = 5 working days	95% and above	>90% but <95%	90% or less	** 3 days	
SL	Time from examination to end result	Full Structure Analysis= 20 working days	95% and above	>90% but <95%	90% or less	** 12 days	

* This number does not take into account any advanced technique experiments such as high pressure, gas cell or crystal sponge experiments. The latter has a diffractometer dedicated to it and the group have undertaken approximately 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.

** We are currently unable to access benchmark statistics from our Portal2 site due to maintenance and upgrades. As such we are only able to access accurate information on the average turn around time for H/M/L and DCO/FSA samples. This is what has been reported here. We have taken the values from after the point our system polling was switched on.

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 also on occasion to I15. Optimising access to Diamond facilities and opening up to more advanced techniques was the subject of community engagement in the 2021 Statement of Need, resulting in new proposed access routes to be made available as part of the service renewal (see below). 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 partnership with Newcastle University (Dr. Michael Probert) to deliver components of the NCS service offering for advanced techniques was a major component of the 2022 renewal and commenced on 1st August 2022. Actual delivery of the experimental capability will begin in 2023 as new equipment in Newcastle is commissioned and made available for use. Newcastle will deliver the ENaCt crystallisation service as well as some dedicated high pressure and ultra-low temperature facilities.
- 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. This culminated in a proposal to the EPSRC Software for Research Communities programme, which was successful. This involves a partnership with the development team of the leading Olex2 crystal structure analysis software based at Durham University. The project launched in the summer of 2022 at an international conference and will develop a platform to integrate software from all over the world into a seamless system.
- A long involvement with the instrument manufacturer Rigaku led to participation in the development of a new type of diffractometer based on electron radiation rather than X-rays. The NCS team led a proposal to the EPSRC Strategic Equipment programme to partner with the University of Warwick to provide a national facility to make this ground-breaking technique widely available. The proposal was successful in August 2022 and a formal partnership between Southampton, Warwick and Rigaku will be setting up the facility, and national access to it, during 2023.
- SJC is also the Director of the Physical Sciences Data-science Service (PSDS) and is driving convergence of the two NRFs on crystallographic data management, aggregation and publication. A parallel activity, the Physical Sciences Data Infrastructure (PSDI) is also led by Simon and will align strongly to PSDS, and therefore NCS.
- 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 advised on the models and approaches for establishing NXCT, the Computed Tomography NRF, and sits on management advisory panel of the Southampton site.
- 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 which are currently active and generally relate to student exchange and training and resulting in collaborative papers:

- SJC is a consultant feeding into the direction and setup for the ChemMatCARS beamline at the Advanced Photon Source (Chicago).
- X-TechLab Benin – the first facility in Africa to develop widespread multinational access, in-part built on the NCS model and SJC acted as an advisor. The service manager will visit Southampton for a 3-month residential training period in 2023.
- Karlsruhe Institute of Technology - PhD student placement and training, Nature paper published
- Wakayama University – student visits and several papers using the charge density technique

- University of Cagliari – long collaboration (SJC visiting Prof providing training both in Cagliari and to many visitors to NCS over 2 decades).
- King Saud University – collaboration leading to 2 publications
- Turkey (Zonguldak & Gebze – SJC visiting academic) continual stream of papers and visits.
- 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.
- SJC has been responsible for setting up and coordinating Cagliari and Gebze as Erasmus exchange partners with Southampton
- University of Salahaddin (Kurdistan, Iraq) – two papers in preparation for publication
- UCAD Senegal – Ibrahima Thiam training visit in 2021, 6 papers published
- Yaounde University (Cameroon) – SJC provided a training course, resulting in a stream of papers.

Improvements and Future Plans

The previous annual report detailed a project with Merck and Rigaku, funded by EPSRC Impact Acceleration Account, concerning knowledge transfer and establishment of the Crystal Sponge technique at NCS. This has concluded and the current aim is to complete a validation study which will demonstrate that NCS can deliver robust, industry-standard results. Our aim in 2023 is then to develop a business partnership with these companies around the technique where NCS is a service delivery partner. In the meantime, a service and projects will be rolled out by the NCS to the UK academic community as part of our Advanced Techniques offering.

The August 2022 renewal of the NCS (EP/W02098X/1) includes a new partnership with Newcastle University that has been established to grow the advanced techniques aspect of the service. Through our colleague, Dr Michael Probert, in the Chemistry department at Newcastle a range of new services will be made available. The principle new addition is the inclusion of their ENaCt crystallisation technology – this ideally complements the Crystal Sponge activity in Southampton and together they form the advanced crystallisation capability that was envisioned in the Community Statement of Need. Further, Newcastle has unique capabilities for ultra-low temperature data collection and especially tailored equipment for high pressure studies that they will make available through the NCS. This component of the service was funded via the EPSRC call to provide the NCS and some funds also contributed by Newcastle University have enabled new diffractometry and crystallisation automation instrumentation to be installed.

Simon Coles was the PI on a recently awarded proposal (EP/W029588/1) to the EPSRC 'Software for Research Communities' call. This activity is partnering with the Olex2 team at the University of Durham that produce the de-facto standard software used by many crystallographers (80,000 users worldwide). The proposal (ranked 1st out of 232 applications) is to gather Quantum Crystallography software developed in research groups around the world into a single, maintained platform where the different codes interoperate seamlessly with each other. So this work will ensure the products of research technique development are not lost and are optimally and efficiently utilised. The project, named Quantum Box, commenced 1st September 2022 and the NCS team acts as a champion both as the vehicle for community engagement (software developers and end users) and the alpha test environment for developing the platform. The Southampton developer on this project is a Research Software Engineer and recently performed the upgrades to our Portal system, so is fully familiar with the field and the NCS operations.

During the course of this reporting period the NCS partnered with colleagues at the University of Warwick to develop a case and then a full proposal to the EPSRC Strategic Equipment fund to launch the technique of dedicated Electron diffraction and turn it into a service. The proposal (EP/X014444/1) was ranked 1st in its panel round and was awarded in August 2022. The project has NCS at the core and will use its existing structures to deliver a national facility split over two sites (Warwick and Southampton) and partnering with the equipment manufacturer Rigaku. A Press Announcement can be viewed at: https://www.southampton.ac.uk/news/2023/01/crystal-electron.page. This technique has much promise and ideally complements existing X-ray diffraction facilities in departments across the country and the advanced capabilities of the NCS – it enables study of nanocrystals, while even the most powerful X-ray facilities (including a synchrotron) can only examine micro-scale crystals. From 1st January 2023 the dual-site facility will be set up (refurbishment, staff recruitment, equipment installation, community promotion, access mechanisms) in order to be made available for national access from July 2023. These two instruments will be amongst only 5 available worldwide to come on stream in 2023. NCS is therefore taking a primary leadership role in promoting the capabilities of this nascent technique to the global user community while rolling out the largest capacity facility in the world. Based on applications and work conducted at this facility, the NCS will lead a landscape assessment for EPSRC in order to inform wider funding and adoption in the UK.

EPSRC Core Equipment funds in 2020 supported upgrade of the 2010 vintage equipment which supports the NCS. This upgrade is currently 2/3 complete and a follow-on 2022 Core Equipment proposal requests funds to complete this upgrade path and at the same time establish recently available detector technology (a curved, as opposed to flat detector, meaning greater coverage, faster and enabling rapid and/or dynamic experiments). As part of this upgrade, we will replace the final goniometer for the high intensity Mo source, which has become unreliable and the cause of numerous breakdowns, including having to be returned to the factory in Japan. This replacement will provide 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. Additionally, this Core Equipment proposal will also request accessories for Crystal Sponge and the Electron Diffraction facility that will make the NCS the only place in the world capable of conducting particular advanced crystallisation and in-situ diffraction studies on nanocrystals.

Website

The NCS website is comprised of two separate 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.

In the last period our home website was given a complete refresh and moved to the Wordpress platform. This has continued to be maintained and updated and NCS staff have been developing Wordpress skills to do so. As we are entering a new period the website will need further additions to better advertise the advanced techniques that we have on offer and help users more readily access the services.

We had significant developments to our instruments that meant that our Portal2 site was unable to accurately record when samples had been completed. This was an issue mentioned in the last report and so in this period we began work on upgrading Portal2 in order to be able to accurately track sample status again. This work is still ongoing and has temporarily affected our ability to obtain accurate statistics on our samples. We are working to ensure we have this available again as soon as possible.

In addition to the repairs to Portal2 we also started work, in this period, to allow users to better request advanced techniques via our usual applications process. The works are due to be completed in the next reporting period and will allow us to ramp up advanced technique work and better integrate with our future partner site at Newcastle.
Case Study
We provide a Case Study focussing on some recent in-house work on the Crystal Sponge technique. The Crystal Sponge method can enable crystallographic analysis and structural conformation for chemical samples that stubbornly refuse to crystallise, or for those only available in very limited (nanogram) quantities. Here we focus on a novel structure of Whisky Lactone, an oil with no previous structural characterisation. The case study can be found here - https://www.ncs.ac.uk/ncs/wp-content/uploads/sites/426/2023/02/Crystal-Sponge-Case-Study.pdf

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