

INTERNATIONAL UNION OF CRYSTALLOGRAPHY

IUCr Newsletter

Volume 28 · Issue 1

Collected archival edition

Original article text and available illustrations

Contents

Extraction incomplete: missing article positions 2.

Letter from the President

The whence and the where to of crystallography	4
--	---

Editorial

Editorial	6
Visualizing an unseen enemy; mobilizing structural biology to counter COVID-19	17

Letter to the Editor

First molecular structure of coronavirus spike protein mapped by cryo-EM – a perfect example of how our work impacts society	9
--	---

IUCr journals news

IUCr Journals introduces its three Commissioning Editors	11
Acta Cryst. E welcomes Section Editors Sean Parkin and Graciela Díaz de Delgado	13
Open-access transformative agreements: is your institution included?	15

Commentary

Improving the solubility of berberine	20
There's many a good tune played on an old fiddle – a new colour for Alfred Werner's isomer counting	22
Mirolysin structures open a window on gum disease	25

Feature article

Using the Cambridge Structural Database in research and teaching: a personal view	28
Raw data availability: the small-molecule crystallography perspective	35
Not so elementary, my dear Wyckoff	42

Special report

IAEA activities on synchrotron radiation applications	47
---	----

Meeting report (IUCr supported)

17th International Summer School on Crystal Growth	54
19th International Conference on Crystal Growth and Epitaxy	56
11th School of the Argentinean Association of Crystallography	62

Meeting report

77th Annual Pittsburgh Diffraction Conference	60
2019 Meeting of the Crystallographic Society of Japan	64
Sixth joint Diamond-CCP4 MX workshop	67
Joint Polish-German Crystallographic Meeting 2020	68

Outreach

Winners of the 2019 IUCr Crystal Growing Competition	72
--	----

History of crystallography

Royal Society Biographical Memoirs – now free to read online!	74
---	----

Awards and prizes

Mitchell Guss honoured in Australia Day 2020 Honours List	80
---	----

Notice

On the 80th birthday of Professor (retired) Dr Dr h.c. Peter Paufler	82
--	----

Obituary

Leonard Derwent Hamilton (1921–2019)	84
Michael Woolfson (1927–2019)	87
Mark A. Beno (1951–2019)	90

IUCr supported events

IUCr supported meetings	92
-------------------------	----

Meetings calendar

Meetings calendar	94
-------------------	----

LETTER FROM THE PRESIDENT

The whence and the where to of crystallography

Sven Lidin



Students at the IUCr-supported 2019 CIFIESTA (Naples, Italy), an intensive course covering the extraction, dissemination and use of scientific knowledge from the structure determination experiment to database-driven discovery.

Our field celebrated a 100-year anniversary in 2014, the centenary of the Nobel Prize awarded to Max von Laue for the experiment that provided vital insight into the nature of X-rays and opening the door to the most powerful method in the toolbox of structural chemistry. At that time, there was no method available for probing the nature of solids. In 1913, H. C. Jones wrote in *A New Era of Chemistry*: "We do not know what is the formula of rock salt and we have no reliable means of finding out these simplest matters about solids. Our ignorance of solids is very nearly complete."

Since then, the diffraction of X-rays by crystals has been complemented by other diffraction methods, by a large number of spectroscopic techniques, by microscopies and not least by theoretical methods. These methods, together with the development of X-ray diffraction from a slow and laborious method that required long measurements and manual data processing (even during the time of my PhD work in the 1980s a relatively simple structure could require a week of measurement time), have turned many structural problems into fast and straightforward projects that may even allow for the direct study of solid-state reactions *in situ*. The IUCr has always been very inclusive in its view of complementary techniques, and this is why we have [commissions](#) for NMR crystallography, XAFS, small-angle scattering and quantum crystallography. We are there to find solutions to problems, not to define a home turf.

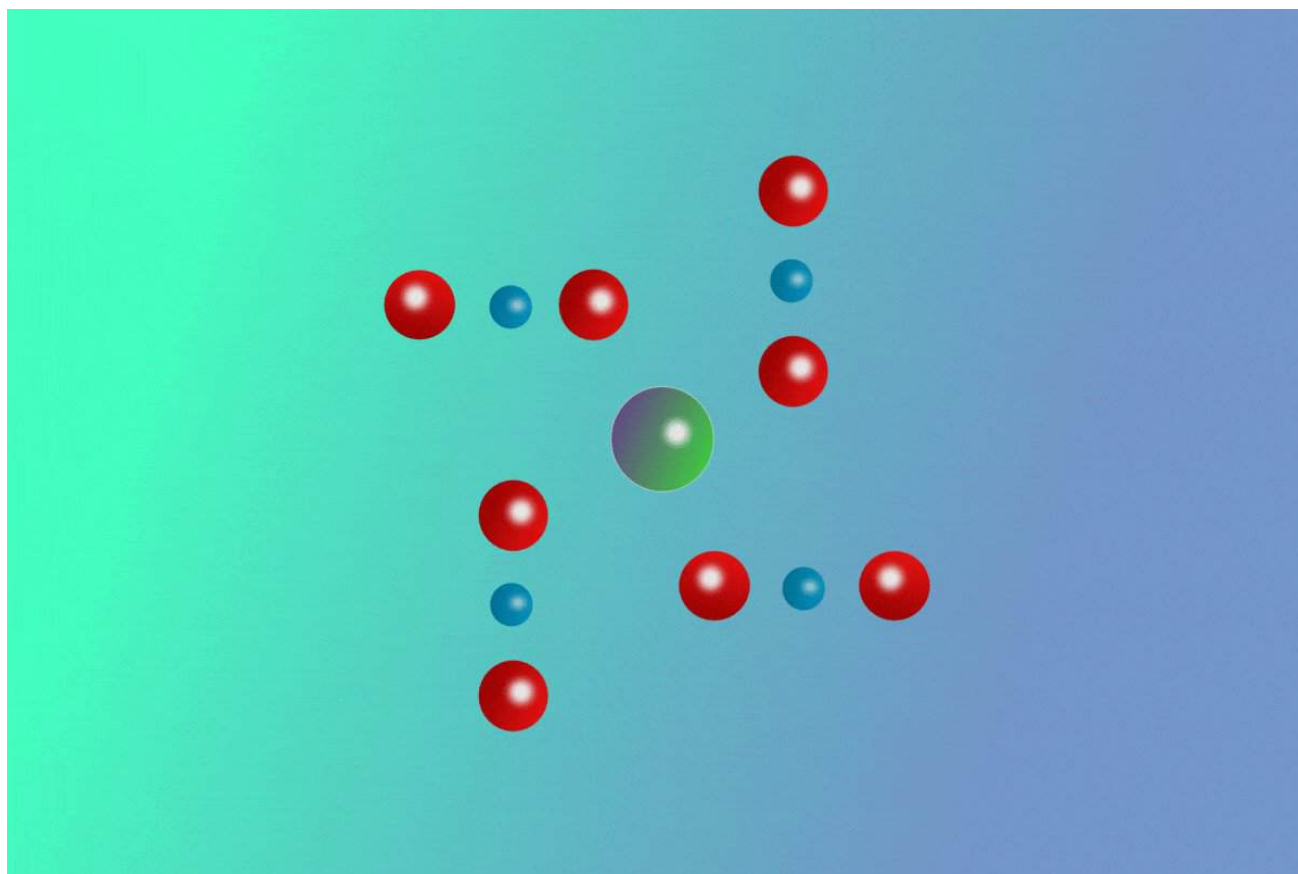
Other commissions highlight the multi-faceted use of crystallography: Aperiodic Crystals, Biological Macromolecules, Crystallography in Art and Cultural Heritage, Crystallography of Materials, High Pressure, Inorganic and Mineral Structures, Magnetic Structures, Structural Chemistry. Crystallography has become an integral part of many of the sciences. Although the time when you could get a Nobel Prize by solving a crystal structure may have passed, it may still be difficult to get it if you don't. Careful studies of biochemical systems and materials require detailed structural knowledge. From a content perspective, the *where to* of crystallography is clear: although we team up with all the other techniques of structural science, we still have a long way to go before we can reverse the statement of H. C. Jones into "Our knowledge of solids is very nearly complete."

Crystallographic data have the advantage of ease of standardisation. This is why crystallography has been at the forefront of managing large amounts of data in prescribed formats. Of course, much of this is owed to the foresight of great crystallographers and the excellence of our publishing and development team at the [IUCr journals](#), preparing us all for the age of open data. It also allows quality assurance of crystallographic work, making crystallography an ideal training ground for scientists. The [outreach programme](#) from the IUCr has been very successful, and we owe this not only to dedicated individuals all over the world but also to the very nature of crystallography. It is a subject where *rigor* can, and should, be applied without any adhering *mortis*. Teaching and learning science are about that balance. This is why our outreach programmes are so important. We should be involved because we are, frankly and objectively, very good at it.

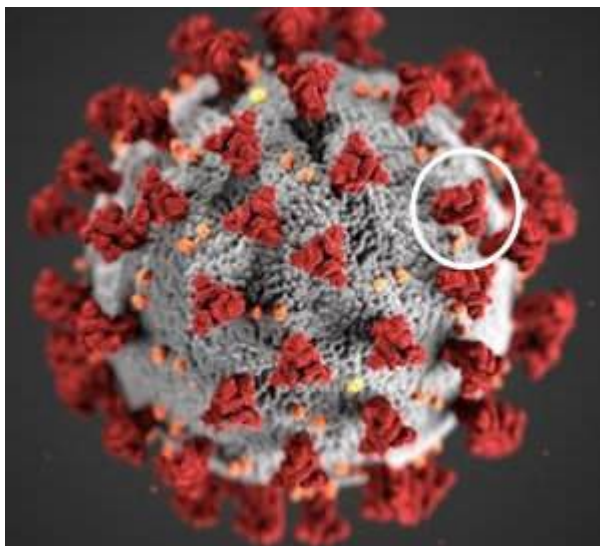
EDITORIAL

Editorial

Mike Glazer



We are living in strange times, what with weeks of floods here in the UK, huge fires in Australia, fears of climate change, and, as if that is not enough, now we have the spread of the so-called coronavirus (COVID-19). Talking of which, I received a letter from Marv Hackert (Immediate Past President of the IUCr) informing us of some most exciting and timely work carried out on the structure of the spike protein in COVID-19 (see [Letter to the Editor](#)) by Jason McLellan and his team at the University of Texas in Austin, USA. The spike in question refers to the circled unit in this figure, and this is the nasty bit that helps the virus to gain entry into the cell. Don't you love viruses?



Coronavirus.

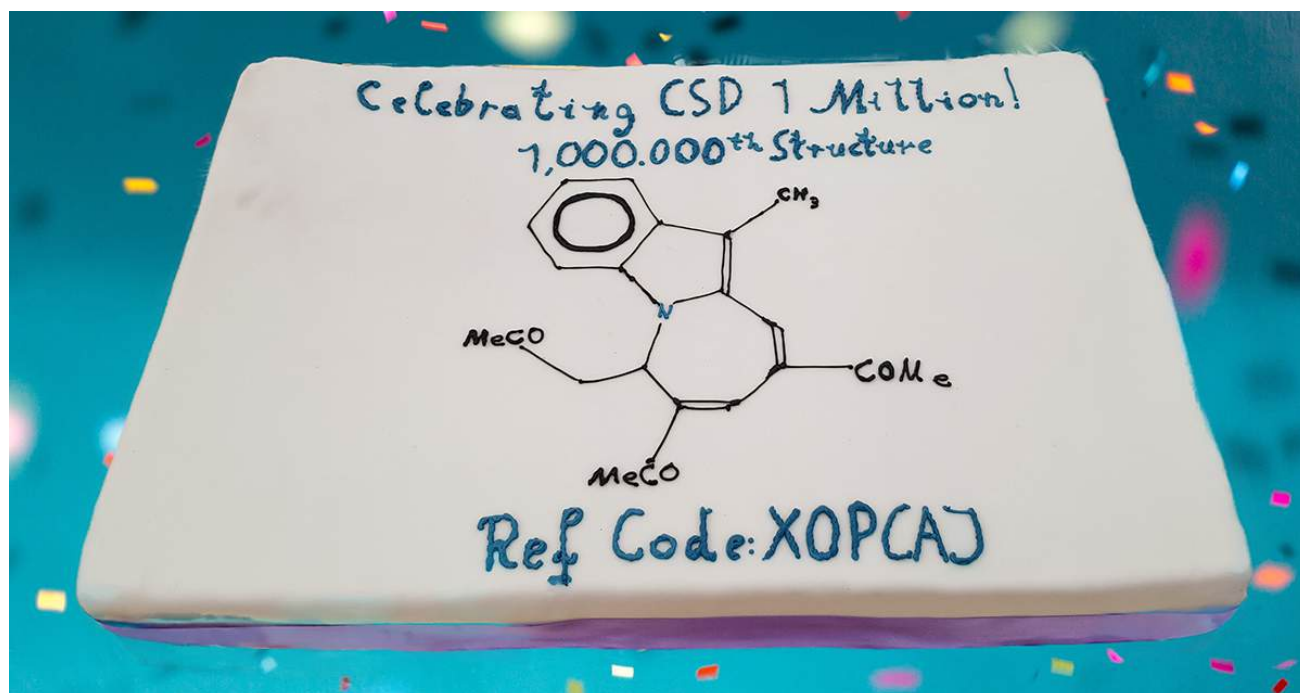
You can read up more about this important work at

<https://science.sciencemag.org/content/early/2020/02/19/science.abb2507> and

<https://thetexan.news/ut-austin-researchers-announce-breakthrough-in-developing-a-vaccine-for-the-coronavirus/>

Work is proceeding on the structure of this virus around the world, for example at the [Diamond Light Source](#) (UK), [Argonne](#) (USA) and the University of Lübeck/BESSY II (Germany) – see the [report](#) of the Joint Polish-German Crystallographic Meeting 2020 in this issue of the *IUCr Newsletter*. **If you ever needed to explain the importance of crystallography, now is the time!**

Also in this issue, we have an [article](#) from Bill Clegg on the Cambridge Structural Database (CSD), an essential source of structural information. Bill provides a personal discussion of how the CSD has helped in both teaching and research. As you will surely know by now, last year the CSD reached its millionth structure. At the 32nd European Crystallographic Meeting in Vienna, Austria, there was even a cake specially made for the occasion.



Once more, Massimo Nespolo gives us [insight](#) into a topic in symmetry that is often overlooked even by trained crystallographers, namely the question of orbits. We are all familiar, at least we think we are, with the concept of Wyckoff sites, but the associated orbits are also worth understanding.

I was pleased to hear recently from the Royal Society (UK) that they were finally releasing their biographical memoirs free of charge. I was able to get them to let me publish [here](#) with Helen Eaton from the Royal Society a listing of all the scientists who have had links with crystallography (53 in all) and who, at the same time, were Fellows of the Royal Society. These memoirs are fascinating thorough accounts of the lives and achievements of deceased Fellows. You will find in this list names well known to many of you already, but also several others. No doubt in due course, there will be a memoir for [Michael Woolfson](#) who passed away recently.

The question of making available raw data is of current interest internationally. Here, we crystallographers and especially the IUCr have been well ahead of the game because, through much of the last century, crystal structure publications have often included tables of structure factors. I well remember as a graduate student many hours of delight having to copy them out by hand in order to put them into our Ferranti Pegasus Mark II computer! Here, Simon Coles and Amy Serjeant describe their [raw data survey](#) with an emphasis on small-molecule crystallography.

Finally, we tend to associate the International Atomic Energy Agency (IAEA) with nuclear energy. However, it also has been involved in accelerators, including those used in synchrotron radiation. You can read a compelling account [here](#) on this aspect of the work of the IAEA and how it is providing expertise and aid to developing countries. For instance, the construction of the SESAME synchrotron in Jordan is of significance as is the programme for African countries.

One more thing. At present, many countries are in lockdown, and here in the UK people over the age of 70 (e.g. me!) are likely to be confined to our homes for months. I think the same is, or soon will be, true elsewhere. Let us hope that the news will be better soon, but we have to be prepared for the long haul and serious changes to our way of living. No-one knows how all this will turn out. In the meantime, I hope you will all remain healthy.

LETTER TO THE EDITOR

First molecular structure of coronavirus spike protein mapped by cryo-EM – a perfect example of how our work impacts society



I suspect that we are all hearing a lot about the coronavirus (SARS-CoV-2) on the news these days. I am not sure if the work of University of Texas (UT) at Austin's Jason McLellan structure determination of the spike protein of the coronavirus by cryo-EM has made it into your local and national news, but I wanted to share this story with you as an example of how our work impacts society and how the world of structure determination has changed so dramatically over the past few years – from years to days to get a large, complicated protein structure.

Jason uses both X-ray crystallography and cryo-EM to study viruses that affect the respiratory system, and has been recognized for his work as a past recipient of the ACA Margaret Etter Award for promising young investigators. After the Chinese released the sequence of the new coronavirus, Jason and his team engineered a variant of the spike protein to increase its stability and expression for study. They ordered the nucleic acid to be synthesized for the modified spike protein. Within 25 days of receiving the engineered nucleic acid, they cloned the gene, expressed the protein, isolated it, used our cryo-EM facility to determine the structure AND submitted [their paper](#) to *Science*, which came out mid-February!

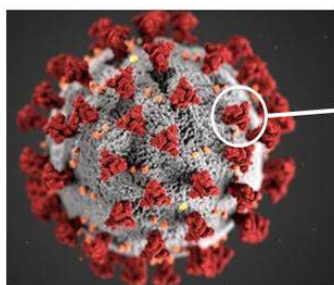
Jason has been interviewed on the news a lot since then. Knowing that I have been doing 3D printing of biomolecules, Jason sent me the coordinates of the coronavirus spike protein and asked if I could make a 3D printed model for him before he was going to be interviewed by Bill Hemmer on Fox National News. I

did so over the weekend (it took more than 2 days to print the model) and I presented the model to Jason on the following Monday morning. The photo below of Jason holding the 3D printed model was taken from our recording of the TV news interview. I added the sketch of the whole coronavirus particle posted by the Centers for Disease Control and Prevention to illustrate the location of the surface spike protein that Jason and his group determined. I am very proud of our whole group of young structural biologists at UT, and delighted to have been able to assist in this small way. I wanted to share this with friends who could appreciate the moment.

Breakthrough in Coronavirus Research Results in New Map to Support Vaccine Design -

Feb 19, 2020 (Austin Am. Statesman)

Dr Jason McLellan
interviewed on Bill
Hemmer Reports -
Fox National News –
Feb. 24, 2020.

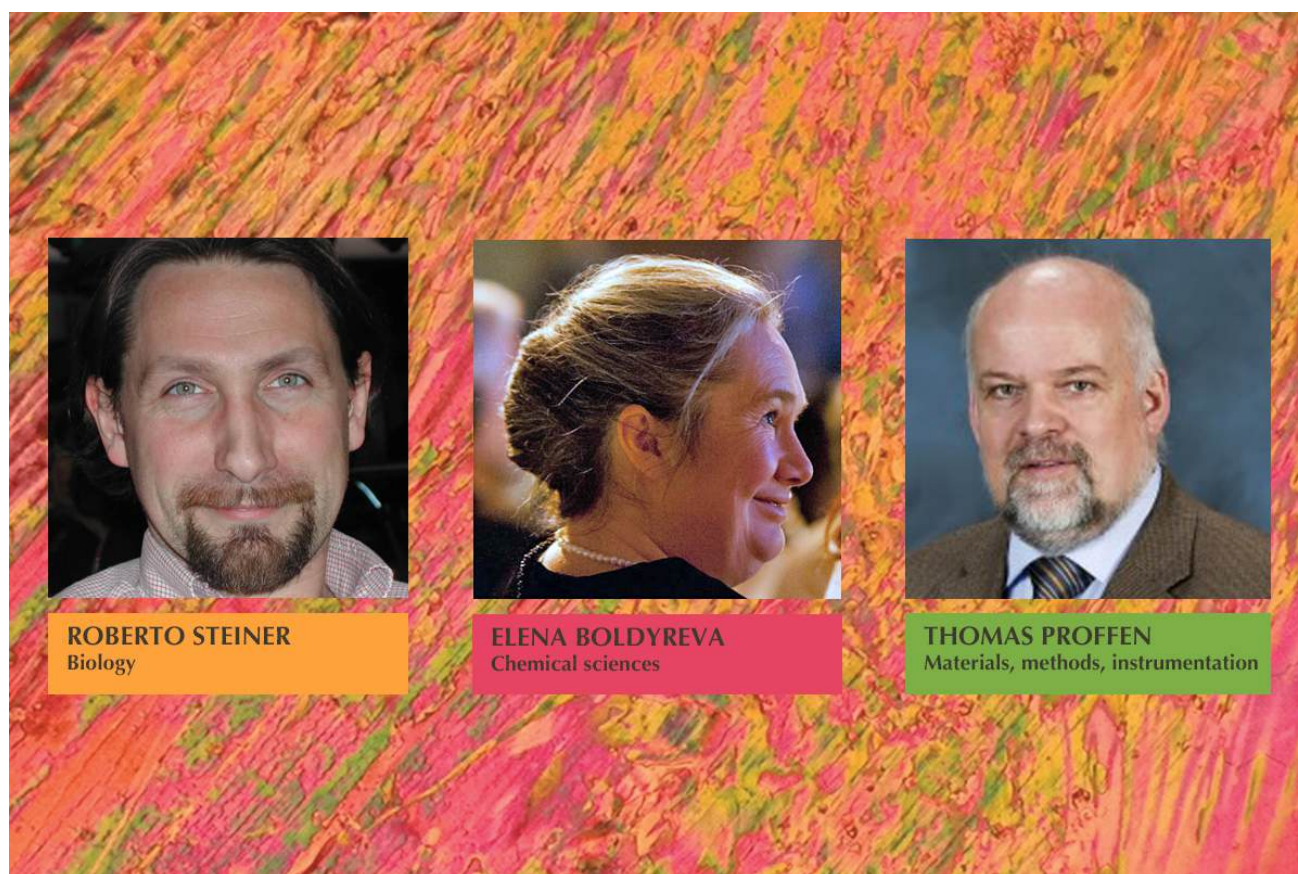


Marv Hackert, Immediate Past President of the IUCr

Department of Molecular Biosciences, University of Texas at Austin, TX, USA

IUCR JOURNALS NEWS

IUCr Journals introduces its three Commissioning Editors



The IUCr is pleased to announce the appointment of three Commissioning Editors for [IUCr journals](#). "The appointment of Commissioning Editors – to work across all relevant IUCr journals in commissioning high-quality individual papers and special issues, and hopefully attract authors, reviewers and editors who have not worked with IUCr journals previously – is new to IUCr journals," said Editor-in-Chief, Andrew Allen. "The Commissioning Editor role is envisaged to be comparable to that of a Section Editor and will involve extensive interaction with the journal Section Editors and Co-editors."

The three Commissioning Editors and their subject areas are as follows:

Biology: Roberto Steiner

Professor Steiner (Randall Centre for Cell and Molecular Biophysics, King's College London, UK; roberto.steiner@kcl.ac.uk) has over 20 years of experience in the use of X-ray crystallography applied to understanding biological macromolecules and their mechanisms of action. He also has a strong passion for disseminating best practices in the field having been a tutor and contributor at many international schools and workshops. Roberto has published in a number of IUCr Journals and has served as a Guest Editor for a [CCP4 issue of Acta Cryst. D](#). He is in the process of commissioning a special issue on serial crystallography and has selected a number of top articles that appear in a [virtual special issue](#). While initially focusing on **IUCrj**, *Acta Cryst. D* and *Acta Cryst. F*, Roberto intends to expand his work to the other journals with remit relevant to structural biology – especially *Acta Cryst. A*, *J. Appl. Cryst.* and *J. Synchrotron Rad.*

Chemical sciences (chemistry and chemical crystallography): Elena Boldyreva

Professor Boldyreva (Boreskov Institute of Catalysis, Russian Academy of Sciences and Novosibirsk State University, Novosibirsk, Russia; eboldyreva@catalysis.ru) was already well known to IUCr journals having served as a Co-editor for *Acta Cryst. B* for nine years, and more recently as a Main Editor of *Acta Cryst. E*. She has published in **IUCrJ**, *Acta Cryst. A*, *Acta Cryst. B*, *Acta Cryst. C*, *Acta Cryst. E* and *J. Appl. Cryst.*; however, her publications mainly focus on chemistry and chemical crystallography. Elena is using her extensive experience in dealing with the chemical crystallography community at the international level to forge a stronger interaction between this community and IUCr journals, and has a number of ideas for special issues, including Chemical crystallography and new materials, Chemical crystallography and drug research, Chemical crystallography and solid-state transformations, and virtual Editors' selection issues.

Materials, methods and instrumentation: Thomas Proffen

Dr Proffen (Neutron Sciences Directorate, Oak Ridge National Laboratory, Oak Ridge, TN, USA; tproffen@ornl.gov) has served as an active Co-editor of *J. Appl. Cryst.* since 2014. He has a distinguished publication record with many citations across much of the applied crystallography field, and has published in *Acta Cryst. A*, *Acta Cryst. B*, *J. Appl. Cryst.* and *J. Synchrotron Rad.* His main emphasis in publications is centred on materials, neutron scattering and applied crystallography, including advanced methods, software and instrumentation development. Thomas is conversant with the user communities of large-scale national and international facilities, and conducts various mentoring and outreach activities in support of young researchers, together with gender and ethnic diversity. He is developing plans for a special issue on machine learning in crystallography.

This inaugural team spans a subject, geographical and gender distribution that should serve well in developing the Commissioning Editor role for the IUCr journals. Once a successful operation is established, the appointment of a further Commissioning Editor from the developing world will be considered.

IUCR JOURNALS NEWS

Acta Cryst. E welcomes Section Editors Sean Parkin and Graciela Díaz de Delgado



Sean Parkin (left) and Graciela Díaz de Delgado, *Acta Cryst. E*'s new Section Editors.

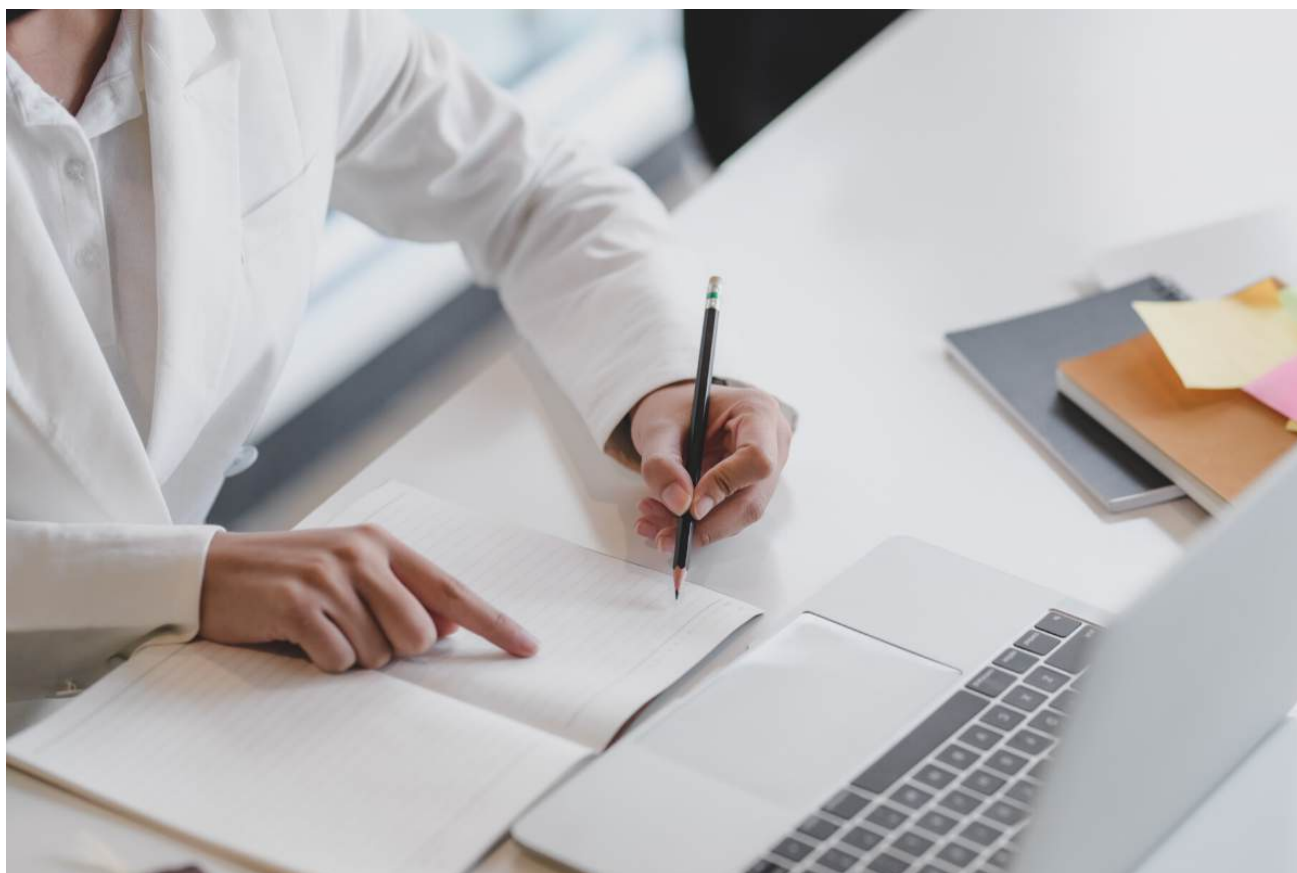
Acta Crystallographica E: Crystallographic Communications has recently welcomed two new Section Editors: Sean Parkin (University of Kentucky, Lexington, KY, USA; s.parkin@uky.edu) and Graciela Díaz de Delgado (Universidad de Los Andes, Mérida, Venezuela; gdiazedelgado@gmail.com). They have joined existing Section Editors Chiara Massera and Luc Van Meervelt in further developing *Acta Cryst. E* in its transformation to the IUCr's open-access structural communications journal for the rapid high-quality publication of fully validated structures.

Professor Parkin, already an experienced Co-editor of both *Acta Cryst. E* and *IUCrData*, was appointed in summer 2019 to succeed former Section Editor Elena Boldyreva, who had been [selected](#) as the IUCr Journals Commissioning Editor for Chemical sciences. Sean has published widely in the chemical and crystallographic literature, including nearly 150 papers in IUCr journals (*Acta Cryst. A*, *Acta Cryst. B*, *Acta Cryst. C*, *Acta Cryst. D*, *Acta Cryst. E* and *J. Appl. Cryst.*), and has contributed to *International Tables for Crystallography* (Volume F). Starting with his graduate studies, Sean's career has been devoted to various aspects of structural chemistry and chemical crystallography, including their application in biology and biotechnology. He is an expert in the design, procurement and state-of-the-art application of X-ray diffractometers and crystallographic facilities, and is an experienced teacher of chemical crystallography and crystallographic methods. He is keen to bring new Co-editors on board who will increase the subject, geographical and gender diversity of the Editorial Board, and make it more reflective of *Acta Cryst. E*'s developing authorship and readership.

The retirement at the end of 2019 of long-standing Section Editor Helen Stoeckli-Evans, to whom we extend our sincere thanks, prompted Professor Díaz de Delgado's appointment. Graciela is already well known to the IUCr as an author (in *Acta Cryst. A*, *Acta Cryst. C* and *Acta Cryst. E*), reviewer, member of the IUCr Executive Committee and Chair of the IUCr Calendar Committee. Her career has been devoted to a wide range of interests in structural chemistry and chemical crystallography, and in the use of single-crystal and powder diffraction for the characterisation of materials. Of particular note are her interests in active pharmaceutical compounds and their application, as well as in natural products isolated from medicinal plants of the Venezuelan Andes. She is determined to encourage submissions from Latin America and the southern hemisphere in general, and to help potential authors, especially graduate students and postdocs, overcome the obstacles (such as structure validation issues) that may prevent them from submitting an interesting manuscript to *Acta Cryst. E*.

IUCR JOURNALS NEWS

Open-access transformative agreements: is your institution included?



In 2019, the IUCr's publication partner, Wiley, signed a transformative agreement with Projekt DEAL, a representative of nearly 700 academic institutions in Germany. This meant that researchers at these institutions are able to publish research or review articles open access in the following IUCr Journals:

Acta Crystallographica Sections A-D and F
Journal of Applied Crystallography
Journal of Synchrotron Radiation

with no direct charge. Similar deals with Austria, Finland, Hungary, the Netherlands, Norway and Sweden followed, and we are now pleased to announce that a new [agreement](#) with [Jisc](#) means that UK-based researchers at participating institutions will also be able to publish open access in the above IUCr journals at no direct cost. The agreement aims to support and accelerate open-access publishing in the UK and is the most extensive UK-based transitional agreement, covering 138 Jisc member institutions. Researchers based at participating UK institutions can now also browse and read all Wiley and IUCr journals. Liam Earney, Executive director for digital resources at Jisc, says: "This new agreement is a step-change in the transition to open access to UK research. This agreement offers all universities within the consortium, regardless of how much or little they publish, an opportunity to rapidly transition toward full and immediate open access in a financially sustainable way."

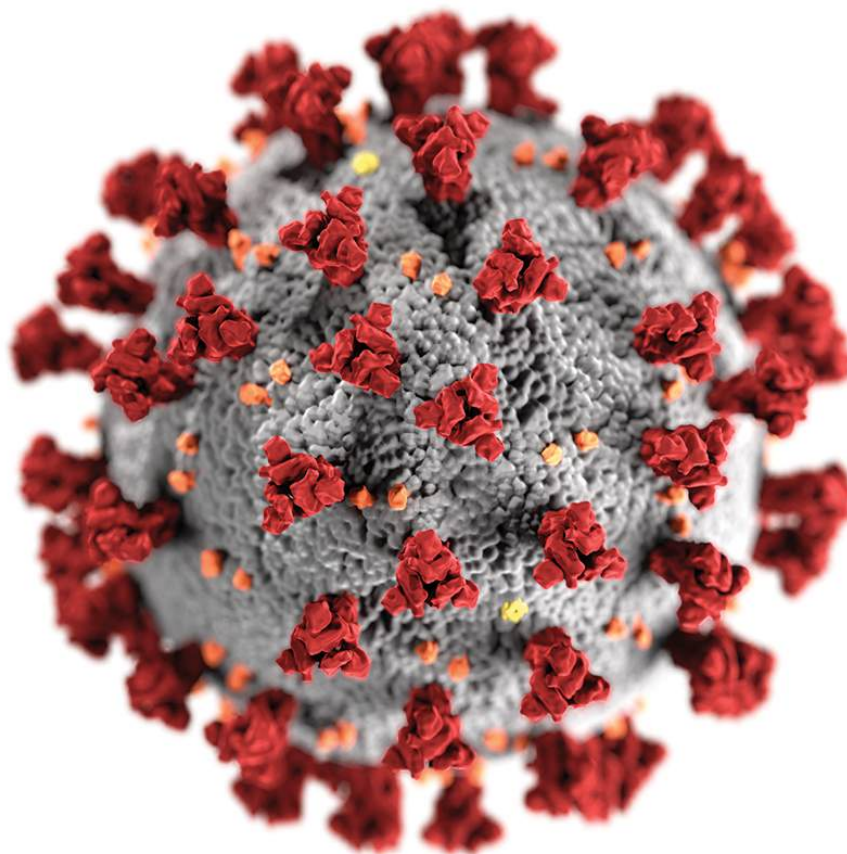
If, after [submission](#), your article is accepted for publication, the IUCr will pass on details of your article to Wiley so that open access can be arranged. Open-access articles in IUCr Journals are generally more

highly cited and downloaded than non-open-access articles; for more information on open access and to check that you qualify for this arrangement, see [here](#).

EDITORIAL

Visualizing an unseen enemy; mobilizing structural biology to counter COVID-19

Edward N. Baker



The COVID-19 pandemic currently sweeping round the world reminds us all of our vulnerability to ‘enemies’ we cannot see. In New Zealand we are all in lockdown for the next month, as are many populations, and none of us can see how long this will have to continue. Science of course holds the key to countering the pandemic, but our inability to ‘see’ the agent responsible generates fear, and, in some individuals, disbelief, while it also limits the strategies that can be applied.

Crystallography has a special role to play in this crisis. In its broadest sense, the term crystallography has come to embrace a range of approaches, from traditional single-crystal X-ray analysis through electron and neutron diffraction to cryo-electron microscopy (cryo-EM) and a variety of scattering technologies. Some are relatively mature, while others such as cryo-EM are undergoing revolutionary change. Collectively, they provide a uniquely powerful atomic scale view into the natural world, and when used judiciously, often in combination, can enable the three-dimensional structures of extremely complex molecules and molecular assemblies to be determined with sometimes breathtaking speed.

In many ways the current situation parallels that at the onset of the HIV-AIDS epidemic in the 1980s. In the absence of a straightforward route to a vaccine, crystal structures of key viral proteins, the HIV protease and reverse transcriptase, provided the crucial templates for structure-based drug design, and in a remarkably short space of time effective drugs became available for clinical use. This was arguably the first great triumph for the use of crystallography in drug development. The virus responsible for the

present COVID-19 pandemic, known as SARS-CoV-2, is very different from HIV. It belongs to the wider family of coronaviruses that includes those responsible for the SARS (Severe Acute Respiratory Syndrome) and MERS (Middle East Respiratory Syndrome) outbreaks of 2003 and 2012, respectively. These viruses have trimeric protein ‘spikes’ on their surface that have attained an ability to attach to a receptor protein that is widely prevalent on cells of the human respiratory system: the ACE2 receptor. The virus also encodes two long overlapping polyproteins that are processed into smaller, functional, polypeptides that are essential for viral replication. The processing is carried out by two viral proteases, which are now of enormous interest as potential drug design candidates (as was the case for the HIV protease).

What is truly exciting, considering that at time of writing only three months have passed since COVID-19 was first identified, is the speed with which some of these key structures have been determined. Already we have three-dimensional structures for three of them; the Main protease, M^{pro}, resolved by crystallography at 2.1 Å resolution by a Chinese group (Jin *et al.*, 2020); the Spike protein, characterized by cryo-EM by two groups in the USA (Walls *et al.*, 2020; Wrapp *et al.*, 2020); and the viral RNA polymerase, characterized by cryo-EM in China (Gao *et al.*, 2020). The structure of the human ACE2 receptor has also been solved by cryo-EM in China, in complex with the receptor-binding domain of the viral Spike protein (Yan *et al.*, 2020). There is undoubtedly much more to come as researchers analyse the conformational changes that occur as receptor binding is followed by entry into cells, and target other key viral proteins.

Since its earliest days in the mid-1900s structural biology has been notable for a high degree of collegiality and willingness to share resources, methods and results. This, too, deserves to be celebrated as it is crucial that as many researchers as possible should bring their skills together at a time like this. One manifestation of this collegiality is the role of the worldwide Protein Data Bank (wwPDB), which makes the three-dimensional structural information for macromolecular structures freely available throughout the world. All of the SARS-CoV-2 structures so far determined have been deposited in the wwPDB, checked, annotated and released, most of them in advance of formal publication. This gives researchers anywhere the opportunity to participate in the search for therapies. Already, the M^{pro} structure has been the target of extensive screening in the search for potential drug leads, both by the Chinese researchers themselves and in the United Kingdom at the Diamond synchrotron facility. At the latter site a massive fragment screening exercise is in progress and a [web-based call](#) has been made for chemists, drug designers and others to contribute to the initiative.

At this point, research is accelerating and by the time this editorial is published and read, many more advances will have been made. One cannot know what the outcomes of these efforts will be, when a vaccine might become available or whether structure-based drug design will indeed produce effective drugs. My purpose in writing this editorial, however, is to applaud the energy and skill of the researchers, and their public spirit in sharing their results. And I take a vicarious pride in the fact that the discipline to which I have given my professional life – crystallography – has such an important role at a time of international crisis.

References

- Gao, Y., Yan, L., Huang, Y., Liu, F., Zhao, Y., Cao, L., Wang, T., Sun, Q., Ming, Z., Zhang, L., Ge, J., Zheng, L., Zhang, Y., Wang, H., Zhu, Y., Zhu, C., Hu, T., Hua, T., Zhang, B., Yang, X., Li, J., Yang, H., Liu, Z., Xu, W., Guddat, L. W., Wang, Q., Lou, Z. & Rao, Z. (2020). *bioRxiv*, <https://doi.org/10.1101/2020.03.16.993386>.
- Jin, Z., Du, X., Xu, Y., Deng, Y., Liu, M., Zhao, Y., Zhang, B., Li, X., Zhang, L., Peng, C., Duan, Y., Yu, J., Wang, L., Yang, K., Liu, F., Jiang, R., Yang, X., You, T., Liu, X., Yang, X., Bai, F., Liu, H., Liu, X., Guddat, L. W., Xu, W., Xiao, G., Qin, C., Shi, Z., Jiang, H., Rao, Z. & Yang, H. (2020). *bioRxiv*, <https://doi.org/10.1101/2020.02.26.964882>.
- Walls, A. C., Park, Y.-J., Tortorici, M. A., Wall, A., McGuire, A. T. & Veesler, D. (2020). *Cell*, **180**, 1–12.

Wrapp, D., Wang, N., Corbett, K. S., Goldsmith, J. A., Hsieh, C.-L., Abiona, O., Graham, B. S. & McLellan, J. S. (2020). *Science*, **367**, 1260–1263.

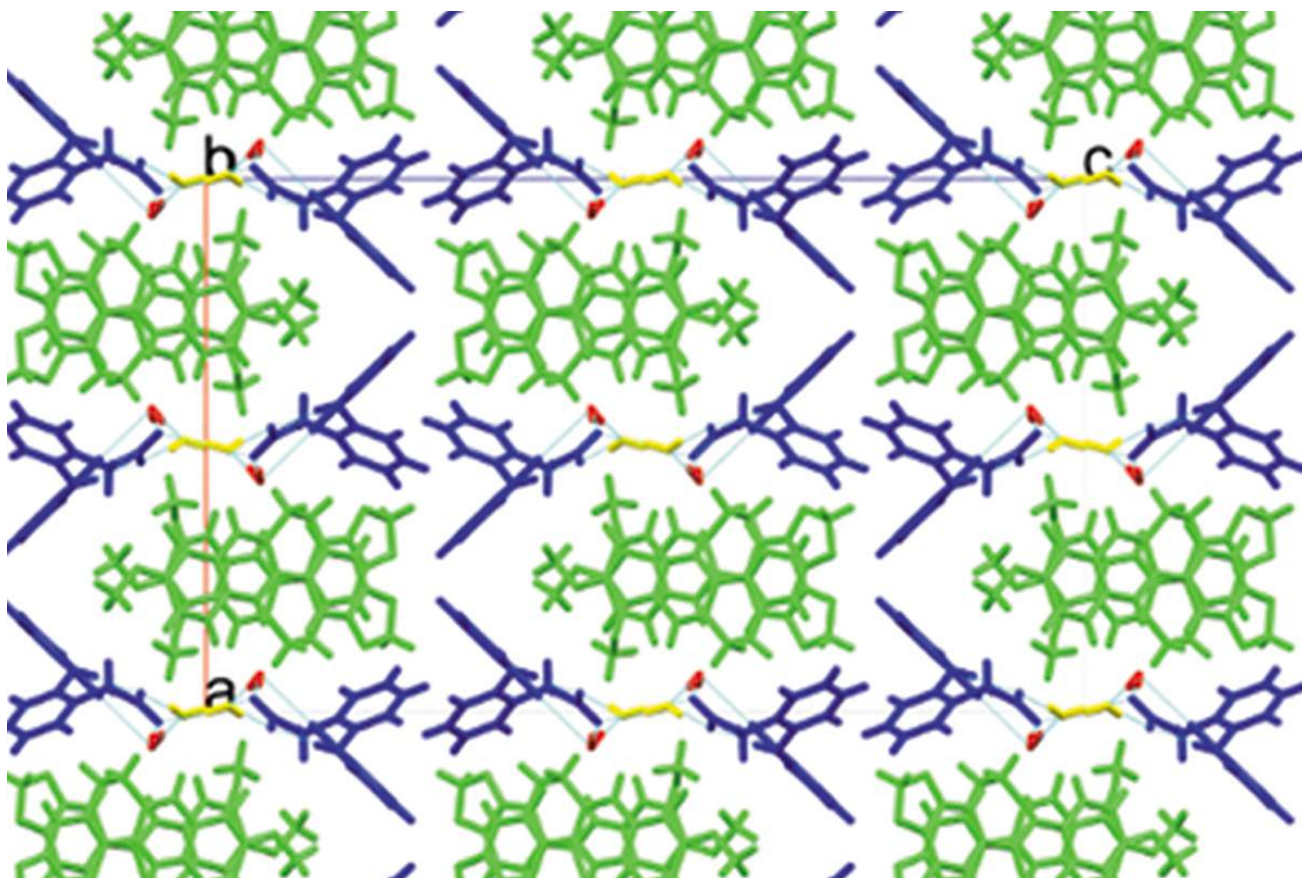
Yan, R., Zhang, Y., Li, Y., Xia, L., Guo, Y. & Zhou, Q. (2020). *Science*, **367**, 1444–1448.

This article was originally published in *IUCrj* (2020). **7**, 366–367; *Acta Cryst.* (2020). **D76**, 311–312 and *Acta Cryst.* (2020). **F76**, 158–159. It has also been included in a [special virtual issue](#) presenting articles and abstracts on coronaviruses that have been published in IUCr journals.

COMMENTARY

Improving the solubility of berberine

José A. Fernandes



While a certain compound can be considered as an active pharmaceutical ingredient (API) in controlled situations, it may fail as a medicine due to problems related to its physical, chemical or biological properties. Moisture, heat or oxygen may jeopardize the chemical or physical stability of the compound, compromising its long-term storage. On the other hand, the form in which the compound is available may present poor solubility in water, preventing its absorption by the human body. The preparation of cocrystals is among the solutions that have been used to circumvent these kinds of problems. If one of the components of a cocrystal is an API, the crystal is classified as a pharmaceutical cocrystal. Conversely, the other component is called a coformer. It is the screening of the vast number of available coformers that allows the optimization of the target parameter (e.g. improving stability toward moisture).

In spite of berberine (BBR) being a water-soluble drug with a wide spectrum of medical applications, it presents poor intestine absorption *via* oral administration (Fratter & De Servi, 2014). Additionally, this API is highly hygroscopic and presents an extremely bitter taste. Cocrystals have been prepared in attempts to solve these problems (Lu *et al.*, 2019; Wang *et al.*, 2016).

Recently, in *Acta Crystallographica Section C*, Sun *et al.* (2019) proposed a possible solution for improving the water solubility of BBR by the formation of multicomponent pharmaceutical salts (or ionic cocrystals in a wider definition; Kumar & Nanda, 2018) with diclofenac (DIC), another known API, as coformer. Thus, they managed to prepare DIC salts of BBR as a dihydrate (denoted I) and four other solvates [hemidichloromethane dihydrate (II), ethanol (III) and methanol (IV and V, mono- and disolvates,

respectively)]. These salts were readily prepared by slow evaporation of mixtures containing the corresponding solvents.

All crystals show an N—H...O hydrogen-bond interaction between the amino group of DIC and the carboxylate group of BBR comprising an $S(7)$ graph-set motif. Additionally, O—H...O hydrogen-bond interactions are also present in I, II and V, comprising rings with $R_4^4(12)$ and $R_6^4(12)$ graph-set motifs in I, a one-dimensional chain in II and an isolated hydrogen bond in V. Additionally, π - π interactions are also present in all the compounds, except for II.

Sun *et al.* (2019) have contributed to the library extension of berberine presentations. Further studies may result in the use of the prepared compounds for new formulations of berberine with improved properties.

References

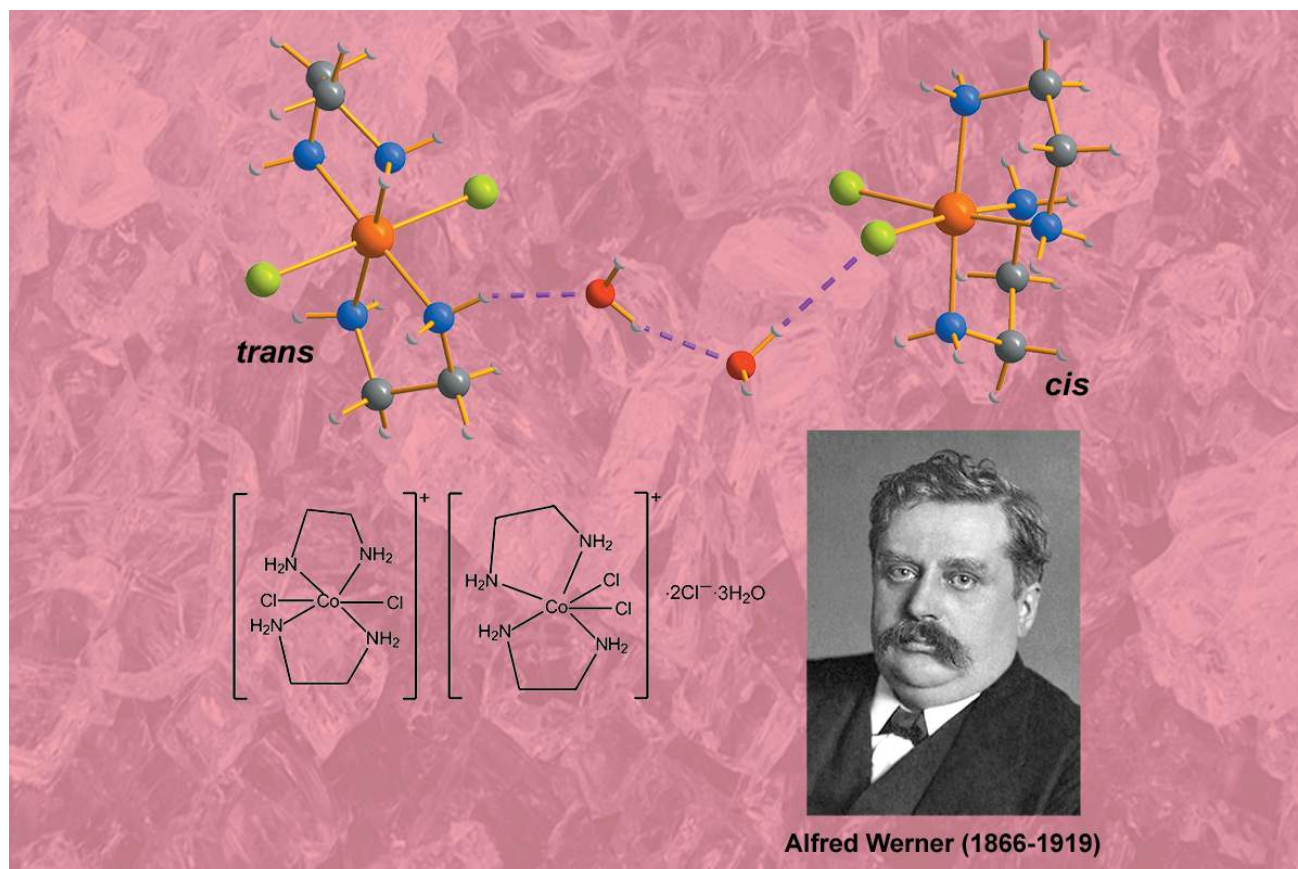
- Fratter, A. & De Servi, B. (2014). *Int. J. Drug Deliv. Technol.* **5**, 33–42.
- Kumar, S. & Nanda, A. (2018). *Mol. Cryst. Liq. Cryst.* **667**, 54–77.
- Lu, Q., Dun, J., Chen, J., Liu, S. & Sun, C. C. (2019). *Int. J. Pharm.* **554**, 14–20.
- Sun, W., Zuo, L., Zhao, T., Zhu, Z. & Shan, G. (2019). *Acta Cryst.* **C75**, 1644–1651.
- Wang, C., Perumalla, S. R., Lu, R., Fang, J. & Sun, C. C. (2016). *Cryst. Growth Des.* **16**, 933–939.

This article was originally published in *Acta Cryst.* (2020). **C76**, 117.

COMMENTARY

There's many a good tune played on an old fiddle - a new colour for Alfred Werner's isomer counting

Edwin C. Constable



Chemical history often teaches that Alfred Werner (1866–1919) was the 'Founder of Coordination Chemistry' (Kauffman, 1966). In the sense that he was responsible for the modern vision of coordination chemistry and the clear distinction between oxidation number, *Hauptvalenz*, and coordination number, *Nebenvalenz* (Constable & Housecroft, 2013; Werner, 1893, 1911, 1920), this accolade is justified. However, as Bernal & Lalancette (2020) state in their publication in *Acta Crystallographica Section C: Structural Chemistry*, the reality is a little more complex.

Although Werner and his co-workers in Zürich were prolific experimentalists, most of these studies were performed after the publication of his seminal work of 1893 (Werner, 1893). In writing his *Beitrag zur Konstitution anorganischer Verbindungen*, Werner drew extensively on the large body of experimental work published by other chemists. Bernal & Lalancette (2020) particularly identify the compounds prepared by Sophus Mads Jørgensen (Kauffman, 1959) and Otto Linné Erdmann as being key elements in the development of his coordination model. This is not the place to revamp the controversy between Werner and Jørgensen, which is adequately documented elsewhere (Kauffman, 1960; Constable, 2019), although the interpretation is currently subject to some degree of revisionism (Kragh, 1997). It is certainly true that a vast number of the compounds used by Werner in developing his model were first prepared or characterized by Jørgensen or Erdmann, but this is also not the entire story.

The coordination chemistry of cobalt started at the end of the 18th Century CE with the description of what subsequently proved to be $[\text{Co}(\text{NH}_3)_6]^{3+}$ by Tassaert (1798). This led to an exponential expansion of interest in cobalt chemistry in the following half century or so. Although the contributions of Jørgensen or Erdmann are exceptional, the contributions of Leopold Gmelin, Emil Dingler, Karl Georg Winkelblech (Karl Marlo), Wilhelm von Beetz, Georg Vortmann, Frederic Just Claudet, Edmond Fremy and Karl Friedrich August Rammelsberg and many others are of equal importance (Constable, 2019). Among the unsung heroes of 19th Century coordination chemistry are Friedrich August Ludwig Karl Wilhelm Genth and Oliver Wolcott Gibbs who described a vast number of cobalt compounds in a series of publications in the 1850s and 1870s (Kauffman, 1977; Genth, 1851; Gibbs, 1873, 1875*a,b*; Gibbs & Genth, 1857*a,b,c*). They were also responsible for the popularization of the names for coordination compounds based on prefixes such as *luteo-*, *croceo-* and *purpureo-*, based on the colour of the prototype cobalt(III) complexes.

One of Werner's arguments for the octahedral coordination of complexes such as those of cobalt(III) relied on the number of isomers that could be isolated. Compounds of the type $[\text{CoA}_4\text{X}_2]^+$ (A_4 = four amine N-atom donors and X = halido ligand) were a critical part of the establishment of the octahedral coordination geometry as opposed to a hexagonal planar one. Only two isomers, *cis*- $[\text{CoA}_4\text{X}_2]^+$ and *trans*- $[\text{CoA}_4\text{X}_2]^+$, were to be expected for octahedral complexes, whereas three would be found for a hexagonal planar one (Fig. 1). First described by Jørgensen (1889), the isolation of only two forms of $[\text{CoCl}_2(\text{en})_2]\text{Cl}$, viz. violet *cis*- $[\text{CoCl}_2(\text{en})_2]\text{Cl}$ and green *trans*- $[\text{CoCl}_2(\text{en})_2]\text{Cl}$, was one of the observations used to support the proposed octahedral geometry. The argument was that in no case had more than two isomers been found for $[\text{CoA}_4\text{X}_2]^+$ complexes, although this is not an unambiguous establishment of the geometry, in particular because it relied on the *non*-observation of a third isomer.

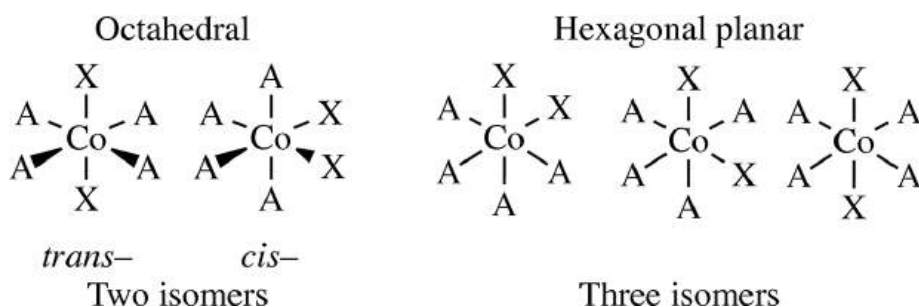


Figure 1. The number of isomers expected for octahedral and hexagonal planar six-coordinate complexes.

Now, 131 years after the original publication by Jørgensen, the crystal structure of a new form of $[\text{CoCl}_2(\text{en})_2]\text{Cl}$ is reported by Bernal & Lalancette (2020). This compound is a purple form that contains equal amounts of the *cis*- $[\text{CoCl}_2(\text{en})_2]\text{Cl}$ and *trans*- $[\text{CoCl}_2(\text{en})_2]\text{Cl}$ isomers. It is really quite remarkable that this purple form has not been noted before, considering that the preparation of the *cis*- $[\text{CoCl}_2(\text{en})_2]\text{Cl}$ and *trans*- $[\text{CoCl}_2(\text{en})_2]\text{Cl}$ isomers is incorporated in so many undergraduate practical courses at universities around the world. The authors speculate what might have happened if Werner had been aware of, or if his co-workers had isolated, this form. It would probably not have demolished the case for the octahedral geometry, as this was built on evidence from more compounds than $[\text{CoCl}_2(\text{en})_2]\text{Cl}$ alone. Nevertheless, it would likely have caused him some sleepless nights!

This report is both interesting and important as it allows us to reassess the early work of Werner and brings an emphasis to the occurrence of cocrystals of complexes. Although the phenomenon of cocrystallization is well-established and investigated for organic compounds and pharmaceuticals, it is less well-documented or recognized in coordination chemistry. A search in *Scifinder* (<https://www.cas.org/products/scifinder>) for the terms 'cocrystal' and 'cobalt' combined yielded 234 hits, suggesting that there is a rich area for data-mining here.

References

Bernal, I. & Lalancette, R. A. (2020). *Acta Cryst.* **C76**, 298–301.

- Constable, E. C. (2019). *Chemistry*, **1**, 126–163.
- Constable, E. C. & Housecroft, C. E. (2013). *Chem. Soc. Rev.* **42**, 1429–1439.
- Genth, F. A. (1851). *Keller-Tiedem's Nordamerikanischer Monatsbericht für Natur- und Heilkunde*, **2**, 8–12.
- Gibbs, W. (1873). *Am. J. Sci.*, Ser. 3, Vol. 6, pp. 116–125.
- Gibbs, W. (1875a). *Proc. Am. Acad. Arts Sci.* **10**, 1–38.
- Gibbs, W. (1875b). *Proc. Am. Acad. Arts Sci.* **11**, 1–51.
- Gibbs, W. & Genth, F. A. (1857a). *Am. J. Sci.* **23**, 234–265.
- Gibbs, W. & Genth, F. A. (1857b). *Am. J. Sci.* **23**, 319–341.
- Gibbs, W. & Genth, F. A. (1857c). *Am. J. Sci.* **24**, 86–107.
- Jørgensen, S. M. (1889). *J. Prakt. Chem.* **39**, 1–26.
- Kauffman, G. B. (1959). *J. Chem. Educ.* **36**, 521–527.
- Kauffman, G. B. (1960). *Chymia*, **6**, 180–204.
- Kauffman, G. B. (1966). In *Father of Coordination Chemistry*. Berlin, Heidelberg: Springer-Verlag.
- Kauffman, G. B. (1977). *Isis*, **68**, 392–403.
- Kragh, H. (1997). *Br. J. Hist. Sci.* **30**, 203–219.
- Tassaert, B. M. (1798). *Ann. Chim. Phys.* **28**, 92–107.
- Werner, A. (1893). *Z. Anorg. Chem.* **3**, 267–330.
- Werner, A. (1911). In *New Ideas on Inorganic Chemistry*. Translated, with the author's sanction, from the second German edition. New York, Bombay, Calcutta: Longmans, Green and Co.
- Werner, A. (1920). In *Neuere Anschauungen auf dem Gebiete der anorganischen Chemie*, 4th ed. Braunschweig: Friedrich Vieweg & Sohn.

This article was originally published in *Acta Cryst.* (2020). **C76**, 312–313.

COMMENTARY

Mirolysin structures open a window on gum disease

Evette S. Radisky

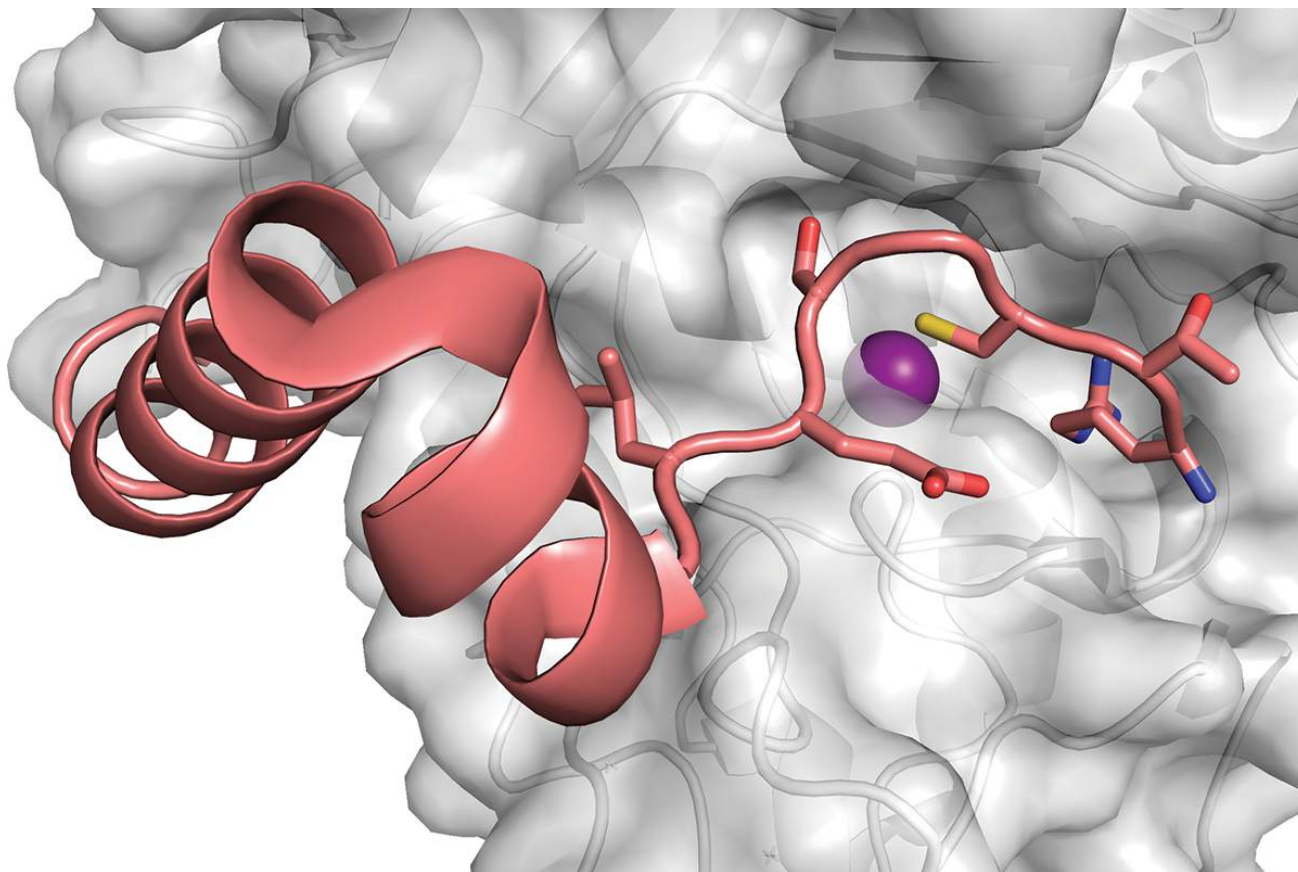


Figure 1. Structure of promirolysin reveals how the pro-segment inhibits enzyme activity prior to proteolytic activation. The N-terminal residues of the pro-segment (salmon) occupy the active site cleft of the catalytic domain (gray), with a key cysteine residue coordinating the catalytic zinc ion (purple). This figure was generated using *Pymol* from the coordinates of PDB structure [6r7v](#), reported in this issue by Guevara, Rodriguez-Banqueri *et al.*

Periodontitis, commonly known as gum disease, is a chronic inflammatory disease characterized by dysregulation of the oral microbiota in the subgingival space below the gum line (Hajishengallis, 2015). Healthy gums are compromised by a proliferation of pathogenic anaerobic bacteria, most commonly the triad of *Porphyromonas gingivalis*, *Treponema denticola* and *Tannerella forsythia*. These pathogens release 'virulence factors', molecules that aid their colonization and survival in the subgingival niche and their evasion of the host immune response. Synergistic interactions of the pathogenic microbes with normal oral microbiota lead to a sustained host inflammatory response, degrading periodontal tissues, eroding bone, and ultimately leading to tooth loss. Severe periodontitis affects around 10% of the human population, and health implications are not limited to oral health; the chronic inflammatory conditions precipitated by the disease can extend to systemic manifestations, contributing to risk of cardiovascular and respiratory diseases, diabetes, rheumatoid arthritis, and cancer (Hajishengallis, 2015; Peters *et al.*, 2017).

Proteases are amongst the most abundant virulence factors produced by periodontal pathogens. These enzymes degrade host proteins to feed bacterial growth, and also serve as key mediators of immune-subversive mechanisms, for example by degrading or inactivating endogenous antimicrobial peptides and components of the host complement system (Hajishengallis, 2015). One of the proteases implicated in these activities is mirolysin, a metalloprotease secreted by *T. forsythia*. Mirolysin cleaves multiple components of the complement pathway to protect the pathogen against complement-mediated killing, and degrades the antimicrobial peptide LL-37, a human host defense peptide secreted by epithelial and immune cells at sites of infection (Jusko *et al.*, 2015; Koneru *et al.*, 2017). As a recently discovered mediator of *T. forsythia* survival and immune evasion, mirolysin offers a potential therapeutic target, yet until now the structural basis for its latency, activation and substrate specificity has not been defined.

In this issue of **IUCrJ**, Guevara, Rodriguez-Banqueri *et al.* report high-resolution crystal structures of promirolysin, defining the mechanism of latency of the precursor protein (Guevara *et al.*, 2020). Like nearly all proteases, mirolysin is first produced as a zymogen, maintained in an inactive state by the presence of an N-terminal extension known as a pro-peptide, pro-segment or pro-domain (Fig. 1). Upon proteolytic cleavage of the pro-segment, the active site of a protease becomes exposed and accessible to substrates. While unified by this common theme, different clans and families of metalloproteases show myriad variations in the details of how latency is maintained and activation subsequently achieved. These variations have been carefully cataloged in over 60 crystal structures of metalloprotease zymogens, including many from the Gomis-Rüth research group (Arolas *et al.*, 2018). Here, the group finds that the mechanism of latency of promirolysin closely resembles the cysteine switch mechanism of matrix metalloproteinases (MMPs) and a number of other zinc metallopeptidases (Morgunova *et al.*, 1999; Arolas *et al.*, 2018). A stretch of the pro-segment occupies the enzyme active site cleft in an orientation opposite to that of a productively bound substrate, positioning a conserved cysteine residue to bind the catalytic zinc. The cysteine side chain displaces the normally bound water molecule that plays the essential hydrolytic role in proteolysis, thus rendering the zymogen inactive.

While this basic cysteine switch mechanism of latency is well preceded, the promirolysin structure reveals a surprisingly extensive interface between the pro-segment and the catalytic domain. The zymogen structure reveals a buried surface area of 2302 Å² between these structural elements, greater than the average buried surface area for protein-protein complexes, which is remarkable given the small size of the 34-residue pro-segment. The pro-segment and catalytic domain interact through a balance of favorable hydrophobic interactions complemented by an array of hydrogen bonds and salt bridges, the same forces that shape affinity and selectivity of intermolecular protein-protein interactions (Xu *et al.*, 1997). The extensive nature of the broad interface between pro-segment and catalytic domain suggests the possibility that the pro-segment may be capable of inhibiting the enzyme *in trans*, and serving as a template for engineering protein-based mirolysin inhibitors of therapeutic utility. Similar approaches have shown promise for engineering therapeutic inhibitors of ADAM family metallopeptidases, based upon their much larger pro-domains (Moss *et al.*, 2007; Wong *et al.*, 2016).

Guevara, Rodriguez-Banqueri *et al.* further report a high-resolution structure of active mirolysin bound to a peptide product, providing insight into determinants of substrate specificity of the protease (Guevara *et al.*, 2020). A preference of mirolysin for cleavage on the N-terminal side of basic residues (Koneru *et al.*, 2017) is explained by the presence of an aspartate residue positioned to form a salt bridge with the substrate. This aspartate residue is conserved among the pappalysin family of metalloproteases to which mirolysin belongs, suggesting that similar specificity may be typical of the entire family, and that the mirolysin structure can offer a model to understand substrate recognition for the larger family. Additionally, this serendipitously trapped product complex may provide yet another starting point from which active site-targeted mirolysin inhibitors might be developed. A similar serendipitous discovery of a product fragment captured in a crystal structure of MMP-13 recently led to the design of a potent peptidomimetic inhibitor of this metalloprotease (Gall *et al.*, 2019). Together the new structures of

mirolysin reported here offer a window from which to understand the function of this protease in gum disease, with a glimpse of how one might inhibit this intriguing target.

References

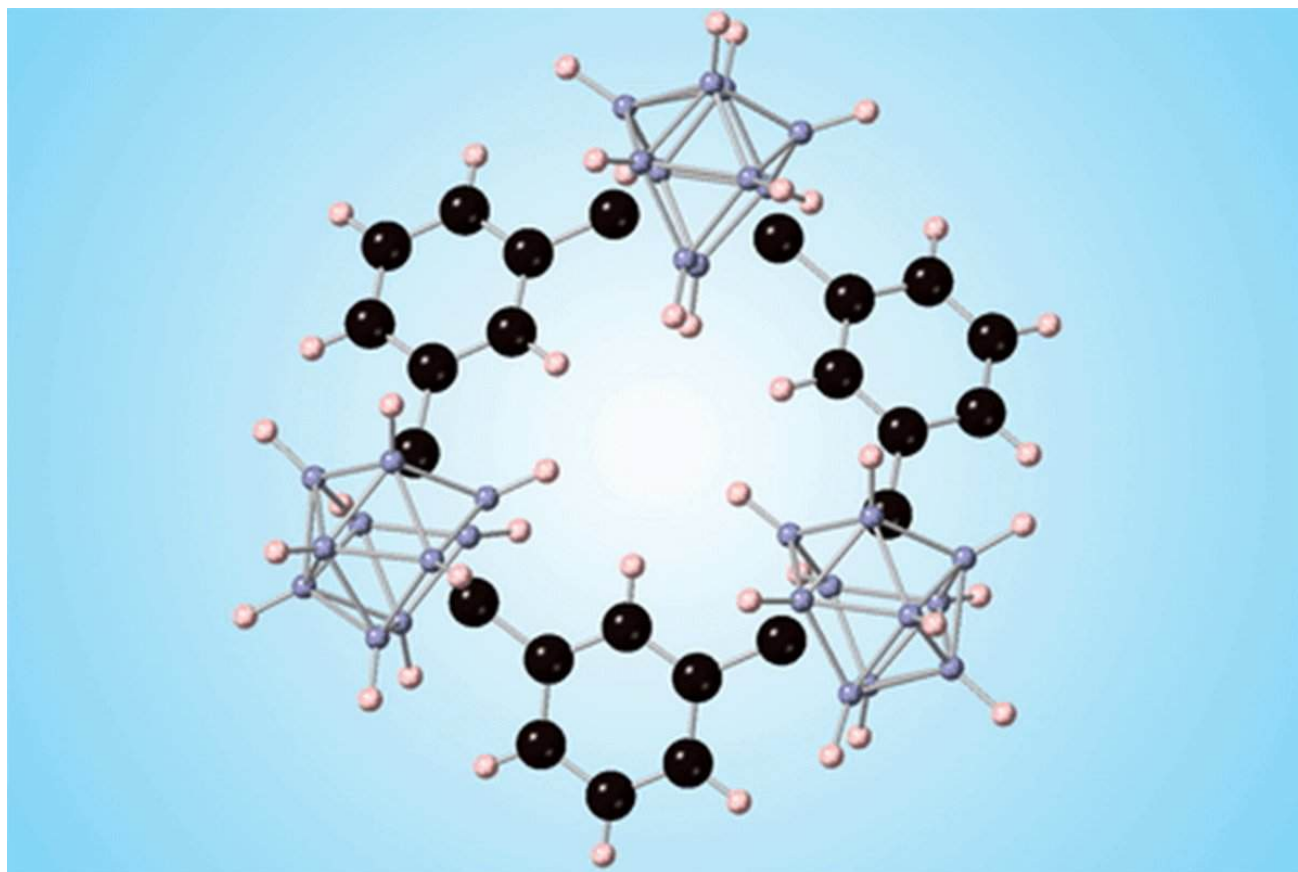
- Arolas, J. L., Goulas, T., Cuppari, A. & Gomis-Rüth, F. X. (2018). *Chem. Rev.* **118**, 5581–5597.
- Gall, F. M., Hohl, D., Frasson, D., Wermelinger, T., Mittl, P. R. E., Sievers, M. & Riedl, R. (2019). *Angew. Chem. Int. Ed.* **58**, 4051–4055.
- Guevara, T., Rodriguez-Banqueri, A., Ksiazek, M., Potempa, J. & Gomis-Rüth, F. X. (2020). *IUCrJ*, **7**, 18–29.
- Hajishengallis, G. (2015). *Nat. Rev. Immunol.* **15**, 30–44.
- Jusko, M., Potempa, J., Mizgalska, D., Bielecka, E., Ksiazek, M., Riesbeck, K., Garred, P., Eick, S. & Blom, A. M. (2015). *J. Immunol.* **195**, 2231–2240.
- Koneru, L., Ksiazek, M., Waligorska, I., Straczek, A., Lukasik, M., Madej, M., Thøgersen, I. B., Enghild, J. J. & Potempa, J. (2017). *Biol. Chem.* **398**, 395–409.
- Morgunova, E., Tuuttila, A., Bergmann, U., Isupov, M., Lindqvist, Y., Schneider, G. & Tryggvason, K. (1999). *Science* **284**, 1667–1670.
- Moss, M. L., Bomar, M., Liu, Q., Sage, H., Dempsey, P., Lenhart, P. M., Gillispie, P. A., Stoeck, A., Wildeboer, D., Bartsch, J. W., Palmisano, R. & Zhou, P. (2007). *J. Biol. Chem.* **282**, 35712–35721.
- Peters, B. A., Wu, J., Pei, Z., Yang, L., Purdue, M. P., Freedman, N. D., Jacobs, E. J., Gapstur, S. M., Hayes, R. B. & Ahn, J. (2017). *Cancer Res.* **77**, 6777–6787.
- Wong, E., Cohen, T., Romi, E., Levin, M., Peleg, Y., Arad, U., Yaron, A., Milla, M. E. & Sagi, I. (2016). *Sci. Rep.* **6**, 35598.
- Xu, D., Tsai, C. J. & Nussinov, R. (1997). *Protein Eng.* **10**, 999–1012.

This article was originally published in *IUCrJ* (2020). **7**, 3–4.

FEATURE ARTICLE

Using the Cambridge Structural Database in research and teaching: a personal view

William Clegg



A favourite structure. W. Clegg, W. R. Gill, J. A. H. MacBride and K. Wade (1993). *Angew. Chem. Intl Ed.* **32**, 1328, doi: 10.1002/ani3.199313281; CSD REFCODE PETZOD.

In 2019 the Cambridge Structural Database (CSD; Groom *et al.*, 2016) reached the significant milestone of a million entries (Fig. 1; Taylor & Wood, 2019): crystal structures of organic and metal-organic compounds reported in formal publications, patents, some conference proceedings and personal depositions of otherwise unpublished results as *CSD Communications*. These 1,000,000+ structures, along with those in other structural databases for inorganic, metallic and biological materials, provide a convenient and comprehensive resource for scientists involved in crystallographic and related research, training, education and scholarship (Bruno *et al.*, 2017).

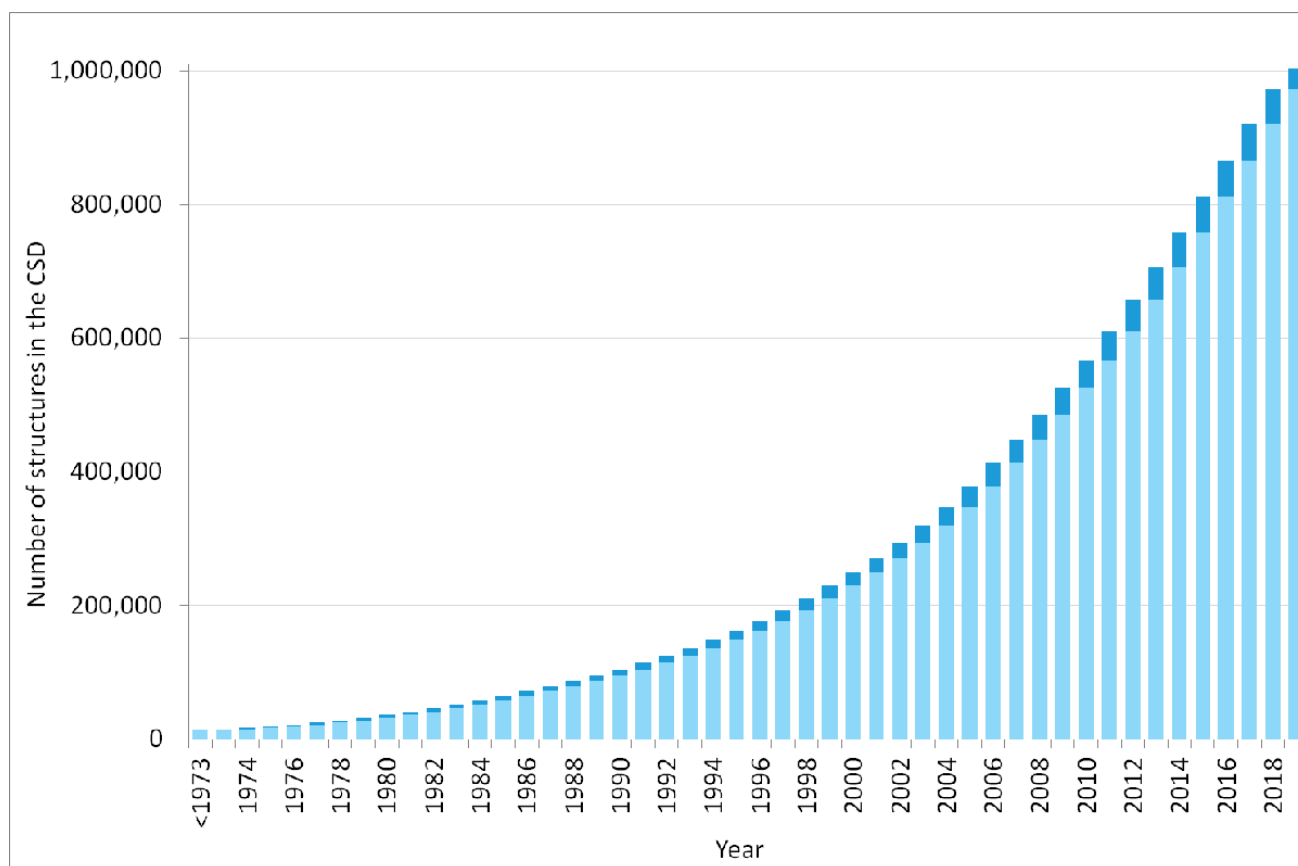


Figure 1. Growth of the CSD since 1972. The darker blue bars show the number of structures added annually. Date: 6 June 2019.

The ‘CSD one million’ achievement was celebrated at various events, including major national and international conferences. An evening session at the 32nd European Crystallographic Meeting (ECM32) in Vienna, Austria, in August 2019, organised and hosted by the Cambridge Crystallographic Data Centre (CCDC, the curators and developers of the CSD), included presentations from some users representing a range of applications of the CSD. I was invited, as an author of over 3000 CSD entries, to speak about the impact of the CSD on my life in crystallography. In view of the interest shown in this presentation, a version of it is provided here for a wider audience. The topics covered will relate to the research and teaching activities of many readers.

My career in crystallography, which spans 50 years, reaches back to the relatively new and modest-scale operation of the CCDC. This was started by Olga Kennard in 1965, and occupied rooms adjacent to my own PhD research laboratory in the Lensfield Road chemistry building in Cambridge University, UK, in the early 1970s (Groom & Allen, 2014). The impact of the CSD over this period can conveniently be summarised under three headings, each illustrated by a photograph taken in my shared Newcastle University, UK, office, where I have a part-time continuing engagement following formal retirement in 2009.

1. Research and publications

I first began to make significant use of the CSD in connection with crystallographic research in the 1980s. This was in a version maintained at Daresbury Laboratory as part of a broader provision of chemical database resources, the UK Chemical Database Service (CDS; Fletcher *et al.*, 1996). I later chaired the CDS management panel for a few years before taking up a part-time secondment to a Joint Appointment at Daresbury, at which point this might have been seen as a conflict of interest. This version, supplied with a convenient independently developed software search system, was known as Crystal Structure Search and Retrieval (CSSR) and provided faster interactive search protocols than the initial *Quest*

software available from CCDC. We later adopted CCDC's much-improved *ConQuest* software (Bruno *et al.*, 2002) as part of a locally installed CSD annual major release and periodic updates, an approach that continues now alongside the internet *WebCSD* access (Thomas *et al.*, 2010).

The CSD is an indispensable tool at all stages of a research project having any significant crystallographic component, whether personal or collaborative, and whether this is in specific chemical topics or a more general structure determination service operation. It has enormous value before, during and after results are submitted and published.

In the formulation and development of a new project, one key question is the extent to which the research area has already been explored and reported by others. Coverage of the extant literature is greatly assisted and simplified by appropriately constructed CSD substructure and text searches to reveal relevant crystal structure results. Access to an exhaustive compendium of published structures helps avoid unnecessary duplication of effort, particularly in cases where a supposedly new compound proves to be a starting material or unexpected by-product with an already established crystal structure. Some diffractometer control systems link to databases of known unit cells derived from the CSD.

For structure solution or refinement that proves problematic, perhaps because of disorder or challenging data quality, CSD entries can provide useful and reliable structural geometries for techniques such as fragment searches, rigid group refinement and appropriate constraints and restraints. During and after refinement, the CSD also serves as a library of results for validation, especially if unusual geometrical features are found; the CSD program *Mogul* finds particular application here (Bruno *et al.*, 2004; Cottrell *et al.*, 2012). Following the completion of a structure determination, at the stage of writing up results for publication, the CSD is a comprehensive compilation of relevant known structures that can be used for comparison and discussion, highlighting similarities, trends and possibly genuine novelties. Much time and effort can be saved in research literature surveys, particularly for crystallographers working across many diverse fields of chemistry. This gives confidence that relevant published structures have not been overlooked, and contributes to the high volume of publications that crystallographers can often achieve (Fig. 2).



Figure 2. A personal collection of reprints from almost 50 years of publications.

After the publication of any results that include new organic or metal-organic crystal structures, these are added to the CSD in due course. Usually, this occurs relatively quickly, the structures having already

been deposited with the CCDC as part of the manuscript submission process in fulfilment of journal policy requirements. A deposition number supplied by CCDC appears in the paper and is part of the new CSD entry, which is subject to an embargo by CCDC until the journal formally publishes it. In this way, the CSD serves as a permanent curated public record of the published structures.

2. Teaching and training

The CSD has contributed significantly to undergraduate chemistry teaching and postgraduate/postdoctoral research training programmes and events in which I have been involved over many years, including associated published material (Fig. 3).

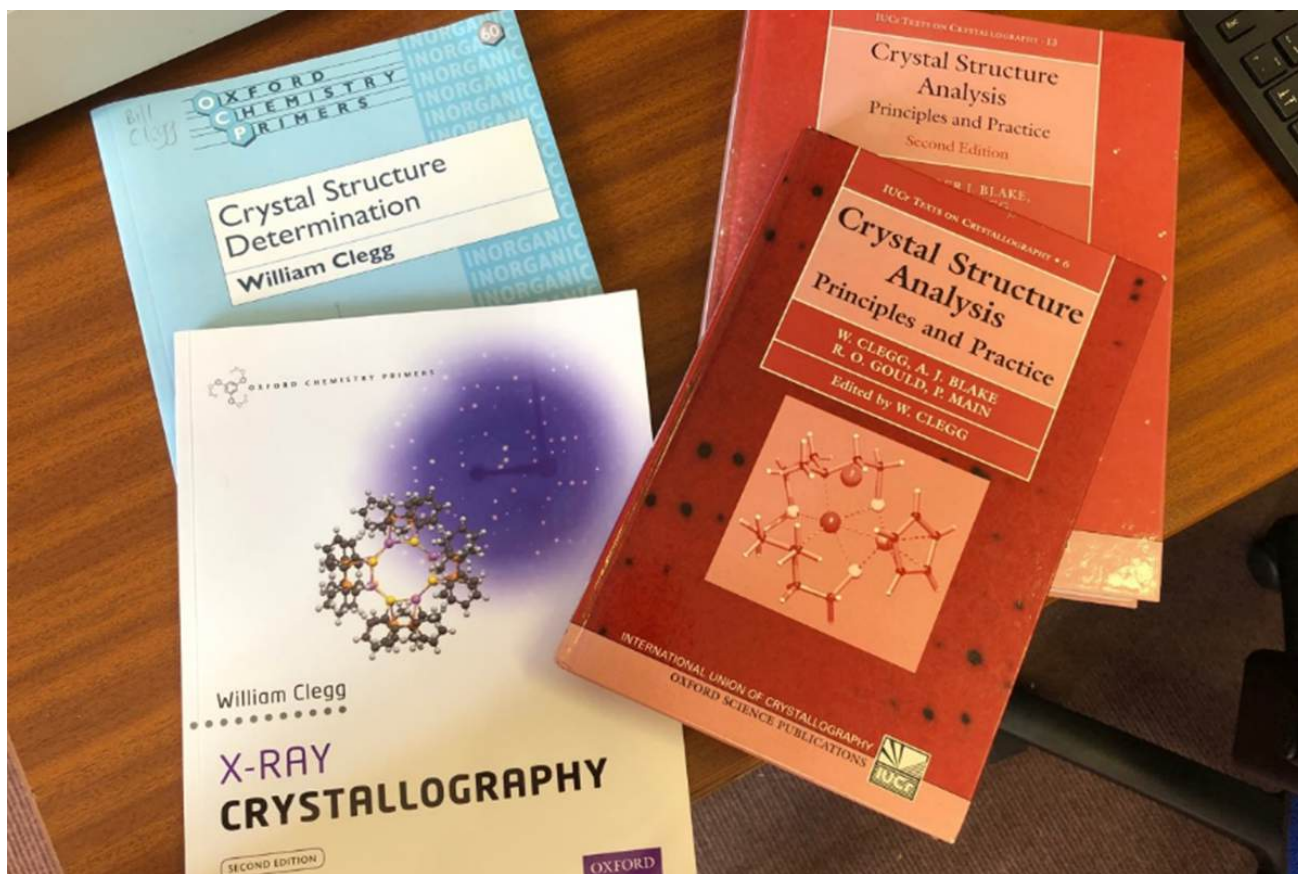


Figure 3. Crystallography textbooks written/edited by the author.

In research training, the CSD serves the same purposes as outlined above in general research terms. It provides a catalogue of known structures and their geometry for information, comparison and validation. It is a valuable resource, not only for those who are mainly engaged in crystallographic research, but also for those in synthetic chemistry research groups who make use of crystallography as one of their characterisation techniques, either by direct hands-on involvement or through a provided structure determination service. It aids their interpretation of their own results and the assessment of their significance in the context of previously reported research.

At the chemistry undergraduate level, in Newcastle, we have used CSD searches and the CSD molecular graphics program *Mercury* (Macrae *et al.*, 2020) in lectures, demonstrations and practical classes. We use it as a tool for teaching various aspects of molecular geometry, including topics such as torsion angles, ring conformation, isomers, chirality and coordination geometry. It is important to make this a hands-on experience and not just a demonstration ‘from the front’, and to develop small-scale investigative projects rather than just a prepared script to follow so that students explore structural features for themselves. As the contents of the CSD are crystal structures and not just molecular

structures, topics covered also include intermolecular aspects such as hydrogen bonding and other interactions, together with polymorphism and solvates. Lectures also briefly cover the topic of structural databases, including the CSD. One of my colleagues at Newcastle, Peter Hoare, has also developed a subset of CSD structures for use in secondary school teaching of chemistry (Hoare & Henderson, 2014; Hoare, 2016).

The Oxford Chemistry Primer text *X-Ray Crystallography* (second edition), shown in Fig. 3 (Clegg, 2015), includes several detailed case studies and numerous other illustrative examples of structures taken from the CSD. In each case, the CSD REFCODE is given, and there are online resources, including the measured diffraction data and the complete crystal structure in CIF format, so that students can follow the crystallographic processes and analyses themselves if they have access to appropriate software.

3. Unpublished results

It would be inaccurate to describe the CSD as a collection of all known organic and metal-organic crystal structures; it encompasses only those that are known in the public domain, and this is a major distinction! Estimates vary widely for the number of unpublished structures, but there is general agreement that they would inflate the CSD to more than double its current size – how much more than double is debated.

There are many reasons, good and bad, why solved and refined crystal structures do not get published; perhaps this is a topic for another *IUCr Newsletter* article. Much of the research has been carried out using public funding, and there are increasing pressures for open access to public-funded research results and data. There is growing awareness, at least in some parts of the world, that the failure to publish such results represents a problem having ethical dimensions that may be regarded as anything from a pity to a scandal.

The availability of such unpublished results would add hugely to the value of what is already in the public domain, giving better coverage and more reliable data for analyses of structural trends and patterns in what is sometimes known as ‘data-mining’ research. Even so-called ‘negative results’ are worth making known, for completeness, if only to help others avoid wasting time and resources in unnecessarily repeating essentially the same work; this includes results such as unexpected structures, by-products and unreacted starting materials.

My own experience here is probably typical of many other prolific crystallographers working in a wide range of chemical areas. Fig. 4 shows just a selection of unpublished structures (each in its own filing cabinet entry) from several decades of collaborative research projects. It is an under-representation because more recent results have been stored only electronically with no paper record for retention in this way.



Figure 4. Filing cabinets of unpublished crystal structures.

Some of these will yet be published (a few are already submitted), but most, realistically, will not. The practical approach, now being actively pursued, and far preferable to consigning everything to eternal non-publication as a total waste of time and resources, is to refresh each structure with a further refinement using current software to generate a CIF output in accordance with modern standards, resolve any issues that now become apparent and deposit it as a *CSD Communication* (Rogers, 2019). This approach generates a CSD entry without a journal publication, with an associated DOI and on a par, in CSD terms, with published entries. Although the work is not subject to formal peer review for publication, it is validated through standard *checkCIF* procedures (International Union of Crystallography, 2020; Spek, 2020), with a facility for authors to respond to significant alerts. These responses are retained as part of the complete entry in the CSD. *CSD Communications* are also manually curated by a team of CCDC editors in the same way as structures published in journals. A comparative survey has shown that, in general, structures contributed to the CSD from these two sources are of similar overall quality and reliability (Tovee, 2018).

The *CSD Communication* approach is strongly supported and encouraged by CCDC as a facility to help crystallographers make their unpublished results available to others. Its use is growing, so *CSD Communications* are likely to represent an increasing proportion of the CSD contents in future years. At the time of writing, *CSD Communications* comprise close to 3% of the total database entries. Adding to this is one of my main activities now during part-time research activities.

Conclusion

The CSD is well established as a critical resource in modern chemical crystallography. Its uses and applications are broader than initially envisaged. These include a comprehensive compilation of crystal structures in the public domain, data for structural comparisons and validation, a library of geometrical fragments for a range of crystallographic and theoretical calculations, a rich vein of treasures for data mining and attractive material and software for training and teaching purposes. Recent developments have made it more easily and widely accessible, simple to use and attractive for the public deposition of structures having no formal publication.

This is not the place for a discussion on the relative merits of, and access to, the CSD and the Crystallography Open Database, COD (Gražulis *et al.*, 2012). The CSD has approximately double the

number of entries than the COD, which is not restricted to organic and metal-organic structures. In this article, I have exclusively addressed the use of the CSD as it currently exists, comprising the compiled database itself, its facilities for deposition, validation, curation, maintenance and access, and its powerful associated software. The question of free open access can be addressed elsewhere; perhaps someone will do so in response to this article.

References

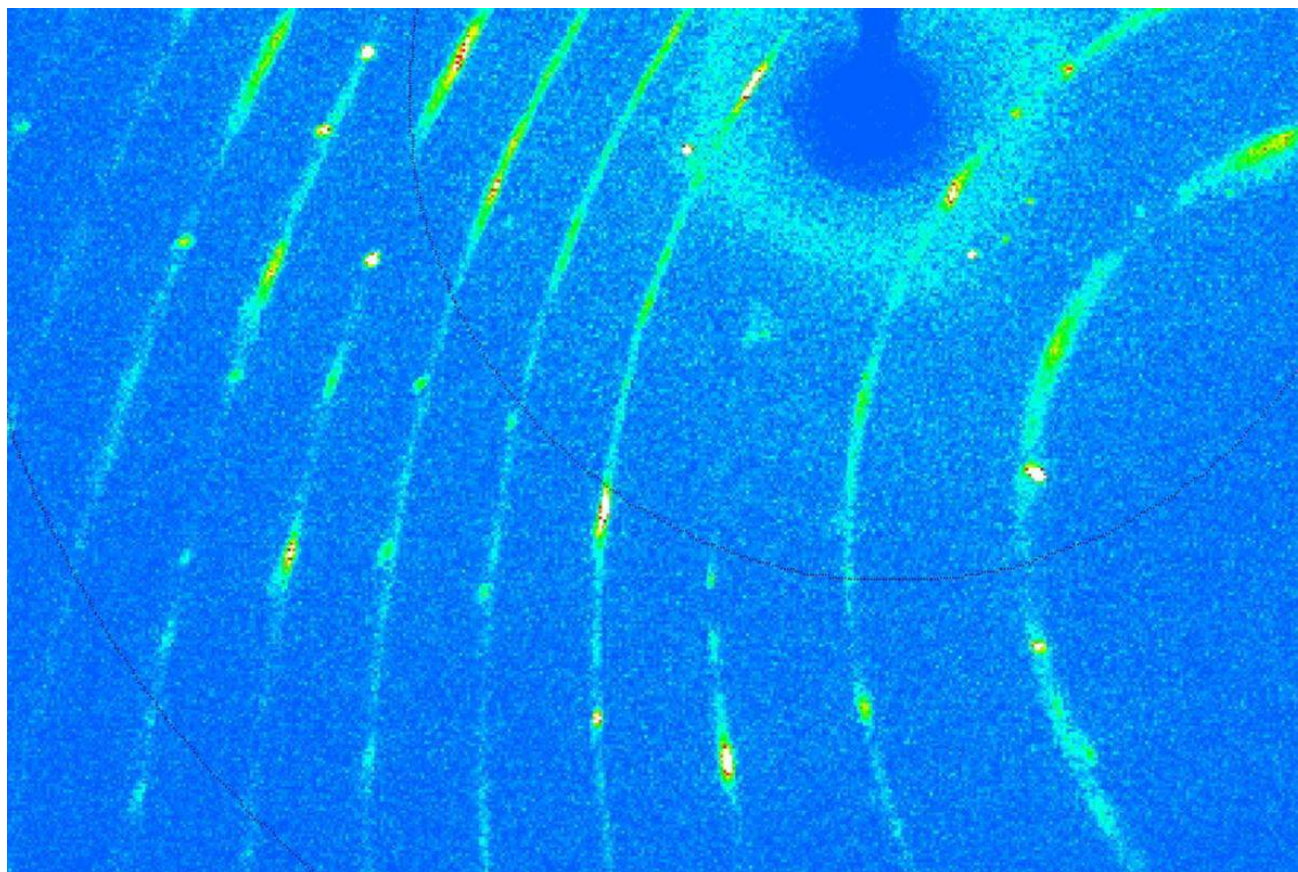
- Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* **B58**, 389–397.
- Bruno, I. J., Cole, J. C., Kessler, M., Luo, J., Motherwell, W. D. S., Purkis, L. H., Smith, B. R., Taylor, R., Cooper, R. I., Harris, S. E. & Orpen, A. G. (2004). *J. Chem. Inf. Comput. Sci.* **44**, 2133–2144.
- Bruno, I. J., Gražulis, S., Helliwell, J. R., Kabekkodu, S. N., McMahon, B. & Westbrook, J. (2017). *Data Science J.* **16**, 1–17.
- Clegg, W. (2015). *X-Ray Crystallography*, 2nd edition. Oxford: Oxford University Press.
- Cottrell, S. J., Olsson, T. S. G., Taylor, R., Cole, J. C. & Liebeschuetz, J. W. (2012). *J. Chem. Inf. Model.* **52**, 956–962.
- Fletcher, D. A., McMeeking, R. F. & Parkin, D. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.
- Gražulis, S., Daškevič, A., Merkys, A., Chateigner, D., Lutterotti, L., Quirós, M., Serebryanaya, N. R., Moeck, P., Downs, R. T. & Le Bail, A. (2012). *Nucleic Acids Res.* **40**, D420–D427.
- Groom, C. R. & Allen, F. H. (2014). *Angew. Chem. Intl Ed.* **53**, 662–671.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Hoare, P. & Henderson, S. (2014). *Educ. Chem.* **51**, 14–17.
- Hoare, P. (2016). Abstracts of the 66th Annual Meeting of the American Crystallographic Association, Denver, CO. [Abstract 01.10.07](#).
- International Union of Crystallography (2020). <https://checkcif.iucr.org/>
- Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Pidcock, E., Platings, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* **53**, 226–235.
- Rogers, E. (2019). <https://www.ccdc.cam.ac.uk/Community/blog/share-data-as-a-csd-communication/>
- Spek, A. L. (2020). *Acta Cryst.* **E76**, 1–11.
- Taylor, R. & Wood, P. A. (2019). *Chem. Rev.* **119**, 9427–9477.
- Thomas, I. R., Bruno, I. J., Cole, J. C., Macrae, C. F., Pidcock, E. & Wood, P. A. (2010). *J. Appl. Cryst.* **43**, 362–366.
- Tovee, C. (2018). <https://www.ccdc.cam.ac.uk/Community/blog/2018-11-12-structure-quality/>

William Clegg is one of the founding Editors of *Acta Cryst. E*; please see his latest article in that journal, "Some reflections on symmetry: pitfalls of automation and some illustrative examples" [here](#).

FEATURE ARTICLE

Raw data availability: the small-molecule crystallography perspective

Simon ColesAmy Sarjeant



A diffraction image from a challenging sample that surprisingly yielded a structure sufficient to inform the chemists of their reaction outcome. However, the refinement was unstable and produced unreliable derived geometric parameters.

In a research climate that encourages the application of 'FAIR' data principles (that scientific data be Findable, Accessible, Interoperable and Reusable), crystallography has been able to hold its head high. Development of the Crystallographic Information Framework has led to the standardisation of datafile formats (and, more importantly, the precise codification of machine-readable terms for describing all useful attributes of data and associated metadata). Journals have required deposition of derived data in the form of atomic positional coordinates and displacement parameters, and, in many cases, the structure factors or Rietveld profiles from which models have been derived. The value of collecting these results as aggregated collections of searchable structural models in databases and journals has been well demonstrated over the last half-century.

However, in recent years, in line with concerns about research reproducibility (a [2018 Special Collection of Nature articles](#) illustrates concerns in a variety of disciplines), focus in crystallography has shifted towards the desirability – or need – to retain and make available the primary experimental data ('raw' data) coming from the instruments. The IUCr commissioned a Diffraction Data Deposition Working Group (DDDWG) to consider the motivation and value of routinely storing and making available raw data sets.

The [DDDWG's final report \(2017\)](#) spanned all the IUCr Commissions and the opportunities for these communities to harness the massive increases in archiving capabilities, even for raw data. The first of 14 Recommendations was:

Authors should provide a permanent and prominent link from their article to the raw data sets which underpin their journal publication and associated database deposition of processed diffraction data (e.g. structure factor amplitudes and intensities) and coordinates, and which should obey the 'FAIR' principles.

Several case studies across biological and chemical crystallography and powder diffraction were published to demonstrate the value of preserving raw data (Helliwell *et al.*, 2017).

At the inaugural meeting of the [IUCr Committee on Data](#) (CommDat) during the August 2017 IUCr Congress in Hyderabad, India, a start was made on discussing the Commissions' reactions to the DDDWG's final report. Subsequent detailed discussion in the Commission on Biological Macromolecules led to an article published in IUCr Journals with a specific implementation plan encouraging the systematic archival of experimental data with trusted repositories, such as the PDB, and linking to raw data from publications where possible (such linking is available from IUCr Journals, for example) (Helliwell *et al.*, 2019). The powder diffraction community has also begun to react to the DDDWG's final report, see for example, Aranda (2018).

However, for structural chemistry, a general hypothesis was aired during the CommDat meeting that it is not necessary for small-molecule crystallographers to make *all* raw data available, although there are many clear cases where this is desirable. In most cases, small-molecule data are clean and simple. If, in an ideal situation, it is possible to demonstrate that all Bragg diffraction has been accounted for, that there is nothing of interest remaining in the images and that they have been processed into structure factors appropriately, then why would it be necessary to retain the raw data?

Surveying the field

Here follows a summary of our exploration of this hypothesis, the first step of which was a survey ([announced](#) in the *IUCr Newsletter* and sent to a number of individuals) questioning current raw data management practices, as these underpin the ability to make the data available in the first place. Following our summary of the survey results presented here, we will hold a [chemical crystallographers' workshop](#) at the 2020 IUCr Congress and General Assembly from which we intend to develop initial guidance on best practices for archiving small-molecule crystallographic raw data and making it publicly available.

While our personal experiences led us to believe that the small-molecule community lagged behind other disciplines in their readiness to store and share raw data, we were curious to know if this was truly the case and, more importantly, why. The methodology used was the design of a short online survey in consultation with CommDat colleagues, which was then disseminated through various crystallographic and social networks. A total of 193 responses were received from around the world, representing a good cross-section of academia, industry, government laboratories, researchers, professors and staff crystallographers. The questions, responses and raw survey data have been deposited in the Chemical Crystallography Community grouping of the Zenodo repository [here](#).

Current raw data archiving practice

While a majority of survey respondents, nearly 80%, claim they do archive their raw data in response to a binary yes/no question, more probing questions reveal that our initial suspicions were largely correct. Few people archive their raw diffraction data in a systematic, searchable, robust and secure way. In the main, data archiving is predominately performed 'in-house' – that is, on laboratory or office computers and with little backup, either off-site or to a secondary hard drive. Only a very few respondents store

their raw data on facility/institutional archives, and almost nobody uses an independent or commercial cloud-based approach.

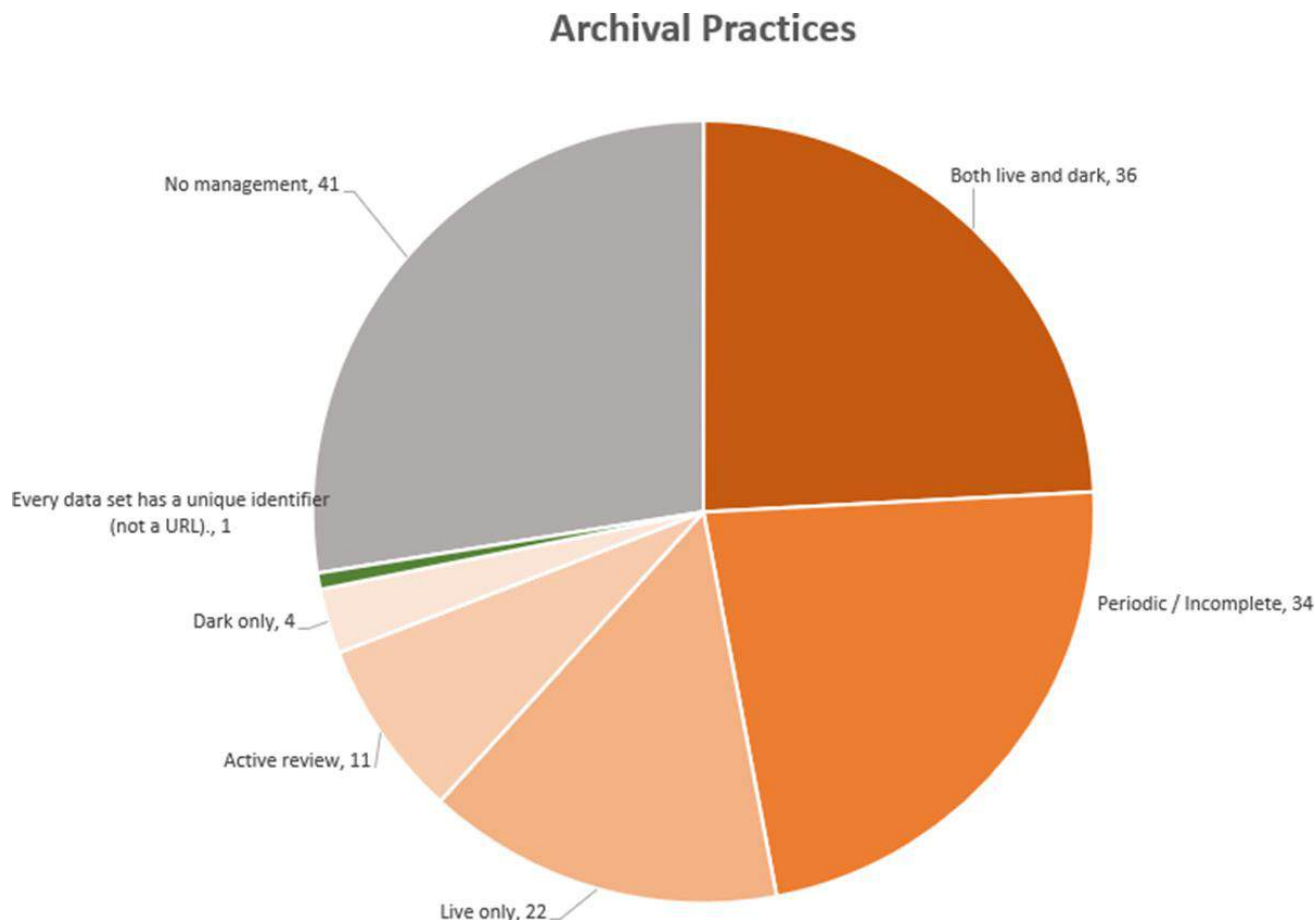


Figure 1. Data management strategies amongst chemical crystallographers.

For those who do archive their raw data, more than a quarter have no management of their archives. The rest claim some management practices, though these are often 'dark', *i.e.* not accessible by others and to be used only for disaster recovery, or they are intermittent and incomplete (Fig. 1). The majority of archives are not only inaccessible to all but the facility manager but also unsearchable. By unsearchable, we mean that there is no structuring of the information or indexing based on a number of key identifiers/fields. Any search capability that does exist relies on operating-system-based tools and pivots on a single identifier, such as folder name (which invariably relates to a sample identifier). Sometimes, there is additional grouping, but this is in arbitrary categories such as year of data collection or which diffractometer was used (although such categorisation may suggest suitable metadata for structuring an efficient distributed search/indexing system). This behaviour leads to a situation where only those 'in the know' are able to find data for a particular experiment. Though it is admirable that the majority of respondents to the survey do currently take some steps to back up their raw data, clearly more can be done and there are likely to be a number of 'quick wins' that can be achieved at little or no cost (a considerable contributing factor) simply by raising awareness.

The availability of raw data

The predominant culture in small-molecule crystallography appears to be somewhat protective: 152 out of 186 respondents declare that data are not made *readily* available to collaborators. This is likely to reflect the fact that many of these facilities are relatively small-scale enterprises run by, at most, a handful of people, many of whom have to charge service fees in order to continue operating. This is generally in sharp contrast to macromolecular crystallography facilities. Macromolecular crystallography has taken the lead in making experimental data freely available, but while many great initiatives from

this community have been followed by other crystallography subdisciplines in the past, this may be an approach that is less attractive in small-molecule crystallography. Another aspect of raw data availability is compliance with mandates imposed by funding institutions to make all experimental data openly available. Of our survey respondents, more than half claimed to be unaware of funding mandates, yet suspected that they must exist. Most respondents would also be willing to follow policies to make raw data sets for funded research accessible at some time after measurement, roughly evenly split between those who would do so for all their raw data sets and those who would do so only where mandatory. There were 17 respondents who claimed that they would not comply with such a policy, even if such compliance were mandatory. Generally, the aspirations of funders in adopting these approaches are well founded, in that they want data (generally funded by taxpayers) to be more widely exploited. However, the state of policies varies significantly around the world, particularly in terms of implementation and policing. These mandates can, therefore, be very polarising in the research community and often grudgingly followed at minimum compliance levels only – they are rarely embraced for their aspirations or intentions.

To understand why some may be resistant to or unable to comply with data archiving, it is revealing to look at the reasons why facilities might not currently archive or make their data available. Forty percent of those who do not archive their raw data (14 out of 35) believe this is an unnecessary measure. However, the most common reason people don't back up their data appears to be a lack of appropriate infrastructure. Some respondents declare a lack of ability as a reason, and we suspect, therefore, that 'infrastructure' refers not only to supplying space for storage but also to the tools required to manipulate, transform, annotate, search and validate raw data. A lack of finances is also a clearly stated factor, with more than half of respondents stating that they are unwilling to absorb the costs associated with archiving and managing raw data. The current funding and operating models of small-molecule crystallography facilities are quite simply unable to cater to these aspects – this is unlikely to change and points to the requirement for community-level and centralised solutions and approaches.

The value of making raw data available properly

A key factor often overlooked in data management activities is incentivisation. Initially, there is often resistance to changes in routine practice, and when these changes are imposed by external institutions or funding agencies, they are all too often viewed as punitive. It is, therefore, important to understand what our community sees as the value of good data management and to articulate this clearly. Responses to the survey in this regard were generally positive, with many of the opinion that making raw data available would enable new scientific insights (Fig. 2). Validation of results is a clear driver for making data available, while training and methods development were also considered worthy incentives.

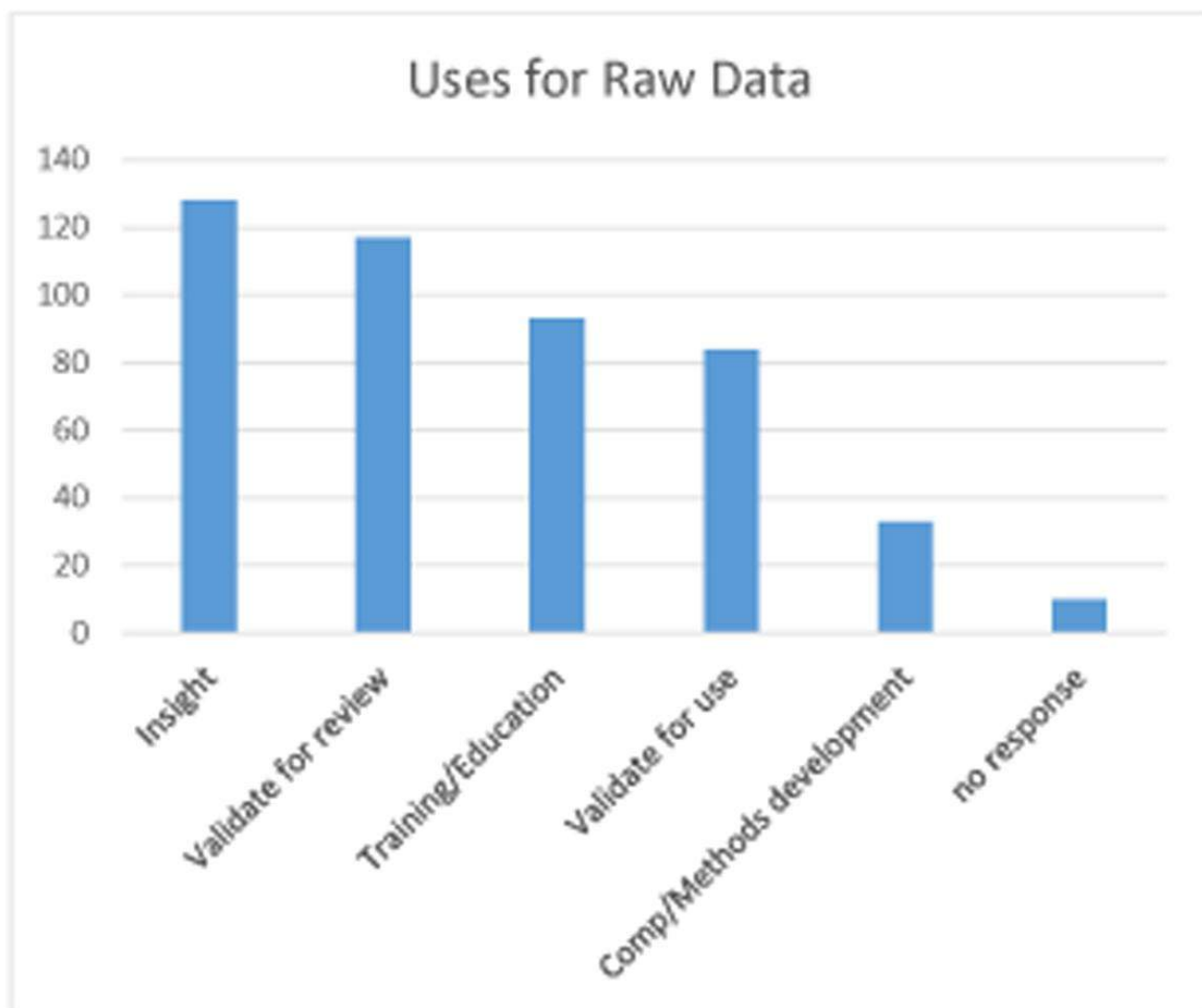


Figure 2. The benefits of having access to raw data, as identified by the survey respondents.

It is also worth noting that this discussion has centred around service crystallography – the predominant mode of operation for most facilities. However, there is a large and growing element of crystallography that we will group here as 'advanced techniques', including, but by no means limited to, quantum crystallography, dynamic crystallography (covering numerous methods), neutron scattering, electron diffraction, nuclear magnetic resonance crystallography and diffuse-scattering analysis. These advanced techniques are becoming foundational methods for many areas of research – and generally produce diffraction data of a lower standard (or requiring a deeper level of analysis) than that commonly accepted in service crystallography. Arguably, it is these advanced techniques that will have the strongest need for robust raw data management, validation and sharing – although there will of course be commonalities with some of the tougher service crystallography examples such as disordered and incommensurate structures. In all of these cases, the drivers for sharing will be to generate further insight – or that future methods might be able to extract more information without the need to repeat the experiment.

Centralised neutron facilities have had an established approach to raw data preservation for some time and this has more recently led to a greater degree of availability as the ability to 'publish' has developed, *i.e.* repository capability of assigning DOIs and opening up to the internet – see, for example, [ILL](#) and [ISIS](#) data management and DOI policies. In the macromolecular community, raw data archiving is becoming just another part of the process of publication – largely in the face of fraud and out of a sense of being able to do a better job of modeling the structure oneself. Most small-molecule crystallographers

would agree that routine well-behaved structures don't need re-refining just to measure bond distances or angles, let alone re-integrating the entire dataset. However, the respondents of this survey do seem to agree that in cases of difficult refinements, disorder, twinning and modulation, having access to the raw diffraction images would benefit the community. Additionally, more than half of all respondents felt access to raw data was essential in examining pathological samples or for validating scientific claims and quality (Fig. 3).



Figure 3. Motivation for archiving raw data sets.

Clearly, while the community does seem to agree that raw data archiving is a worthwhile practice, most lack the funds, capability, infrastructure and motivation to do so in a structured way (particularly one that would then readily enable wider sharing). Yet, as already proven by the macromolecular community, there is a real benefit to having raw data available. When we look back over the evolution of crystallographic data sharing, more crystallographers made more of their experimental data available once a standard format for data became available – the CIF (Crystallographic Information File). Though standard formats for sharing experimental data, e.g. FCF, existed around the same time as the CIF, it did not become routine for crystallographers to share their structure factors until relatively recently when this became an automated output of the refinement and embedded in the CIF. In the same vein, we should now consider how sharing of raw data can be made an automatic part of publishing crystallographic data. What steps can we, as a community, take to ensure this valuable practice becomes part of everyday operations? Who should bear the costs involved and the responsibility for maintaining such an archive?

In conclusion, we focus on two factors in this article – data management practice and the sharing of data to support scientific assertions or findings. While we use the term archiving to refer to data management practices in general, we understand that this includes the aspect of locking data away to keep it safe, as opposed to sharing it for the greater good. With a modest amount of culture change and relatively little extra money or effort, it should be possible for small-molecule crystallography facilities to manage their raw data so that it is easy to make it more widely available. However, without clear guidance on when to make data available and without centralized tools and infrastructure to support the process, widespread data sharing will continue to be elusive in small-molecule crystallography. Where is that guidance to come from, and who will create and maintain the necessary tools and infrastructure?

Should you wish to be part of the conversation, please join us in Prague on 22 August for a community-driven workshop addressing issues of raw data management and availability – see [here](#) for the schedule.

The authors would like to acknowledge [John Helliwell](#) and [Brian McMahon](#) for their significant input during the preparation of this article.

References

Aranda, M. A. G. (2018). *J. Appl. Cryst.* **51**, 1739–1744.

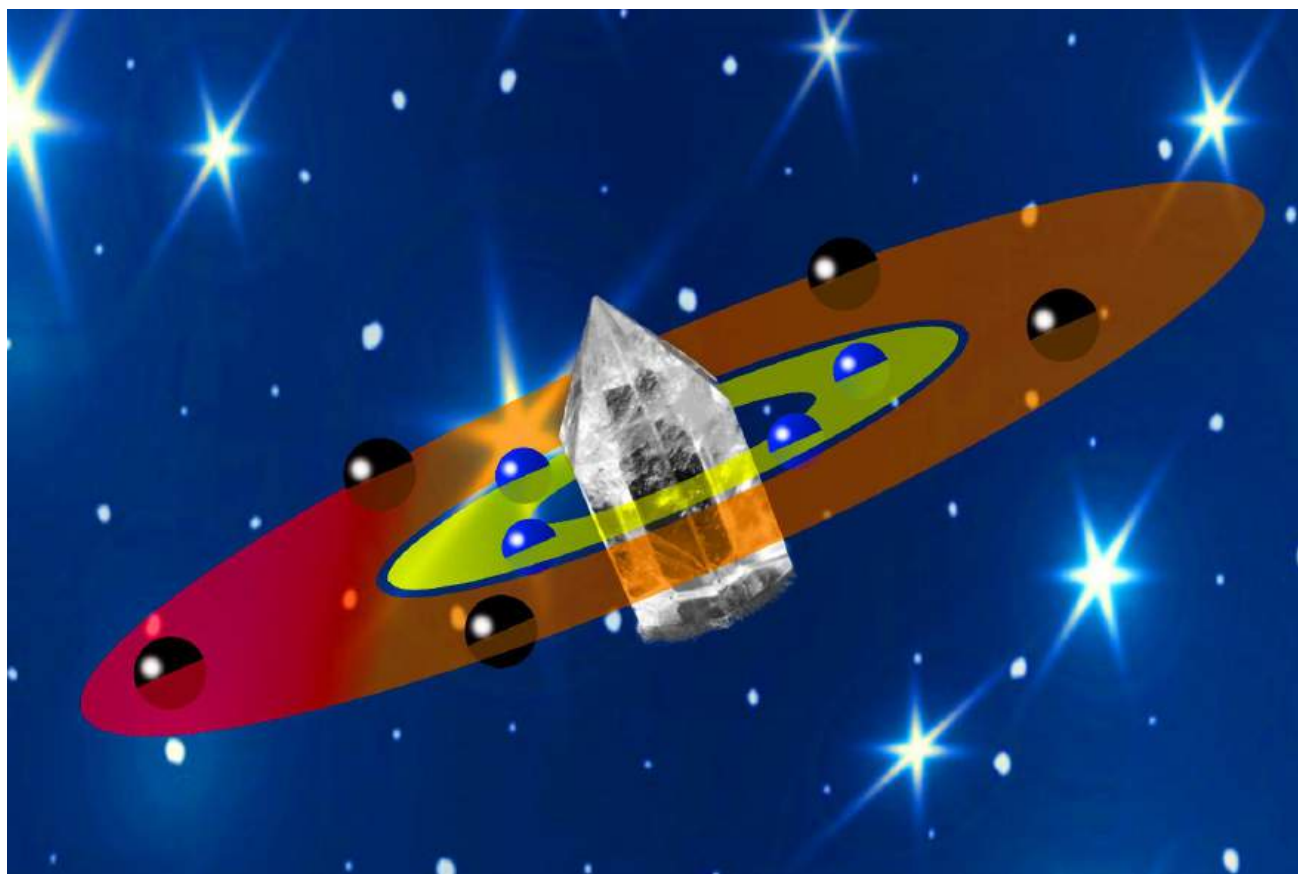
Helliwell, J. R., McMahon, B., Guss, M. & Kroon-Batenburg, L. M. J. (2017). *IUCrj*, **4**, 714–722.

Helliwell, J. R., Minor, W., Weiss, M. S., Garman, E. F., Read, R. J., Newman, J., van Raaij, M. J., Hajdu, J. & Baker, E. N. (2019). *Acta Cryst.* **D75**, 455–457.

FEATURE ARTICLE

Not so elementary, my dear Wyckoff

Massimo Nespolo



Among the terms that every crystallographer uses on a daily basis, “Wyckoff position” is undoubtedly one of the most common. Anyone who has ever solved and published a crystal structure will be familiar with the “Wyckoff letter”, a handy shortcut to identify “something” in a given (type of) space group. In a world where it has become imperative to save space and time, it is indeed an obvious choice to say that a certain atom “is in position 2e of $P2_1/c$ with $y = 0.4125$ ”. This is a shortcut for “an infinite set of equivalent atoms with coordinates $0, 0.4125, \frac{1}{2}$ and the equivalent coordinates obtained through the operations of $P2_1/c$ ”. But what is a Wyckoff position? Few readers may actually have ever considered such a question, and they would most likely not be able to provide a rigorous answer.

Classifying crystals

In our daily work as crystallographers, we constantly classify crystals according to different criteria. Choosing a feature shared by different crystals allows us to put those crystals in a *class*. We actually use quite a number of classes, and to avoid complicated and longish titles, we label some of the classes in different ways: *systems*, *families*, etc. We say that all crystals with the same type of point group belong to the same *geometric crystal class*, all crystals with the same type of point group *and* the same type of lattice (P , I , F , C , etc.) belong to the same *arithmetic crystal class*, all crystals with the same morphological symmetry are in the same *crystal system*, all crystals with the same lattice symmetry are in the same *lattice system*, all crystals with the same type of conventional unit cell stay in the same *crystal family*, and so on.¹ All these categories are indeed *classes* in mathematical terms, *i.e.* a collection of objects (concrete or abstract) that can be unambiguously defined by a property shared by all its

members. Wyckoff positions are classes of crystallographic orbits. The latter term is much less known, yet it is much simpler to define and more fundamental.

You use them but you do not name them: crystallographic orbits

Consider a crystal and forget all its defects, including the surface; in other words, describe your crystal as ideal. Next, consider an atom somewhere in the unit cell of that ideal crystal. It has an infinite number of equivalent atoms that are obtained by applying the symmetry operations of the space group (they are infinite in number: don't forget the translations!). This infinite set of equivalent atoms is called a *crystallographic orbit*. It is really that simple. Then why is the name so unfamiliar? Most likely because it was introduced quite late.² Crystallographic orbits are classified according to two criteria:

1. The *eigensymmetry* (inherent symmetry) of the orbit, *i.e.* the group of operations that leave invariant the orbit as a whole (exchanging, in general, atoms belonging to the same orbit). This eigensymmetry can never be lower than a subgroup of the space group (otherwise that orbit could not be realized in that space group), but can be higher (a supergroup). The class of crystallographic orbits whose eigensymmetry coincides with the space group leads to the name *characteristic orbits*. The class of crystallographic orbits whose eigensymmetry is higher than the space group is referred to as *non-characteristic orbits*. For instance, in our example of $P2/c$ (Figure 1a), the crystallographic orbit generated by an atom at $0, y, \frac{1}{4}$ with y different from 0 or $\frac{1}{4}$ or $\frac{1}{2}$ or multiples, coincides with the space group (Figure 1b) and is therefore a characteristic orbit. However, for $y = 0$ or $\frac{1}{2}$ the eigensymmetry is $P2/m$ with a half translation along c (Figure 1c), whereas for $y = \frac{1}{4}$ or $\frac{3}{4}$ it is $A2/m$ (Figure 1d); these orbits are therefore non-characteristic orbits.

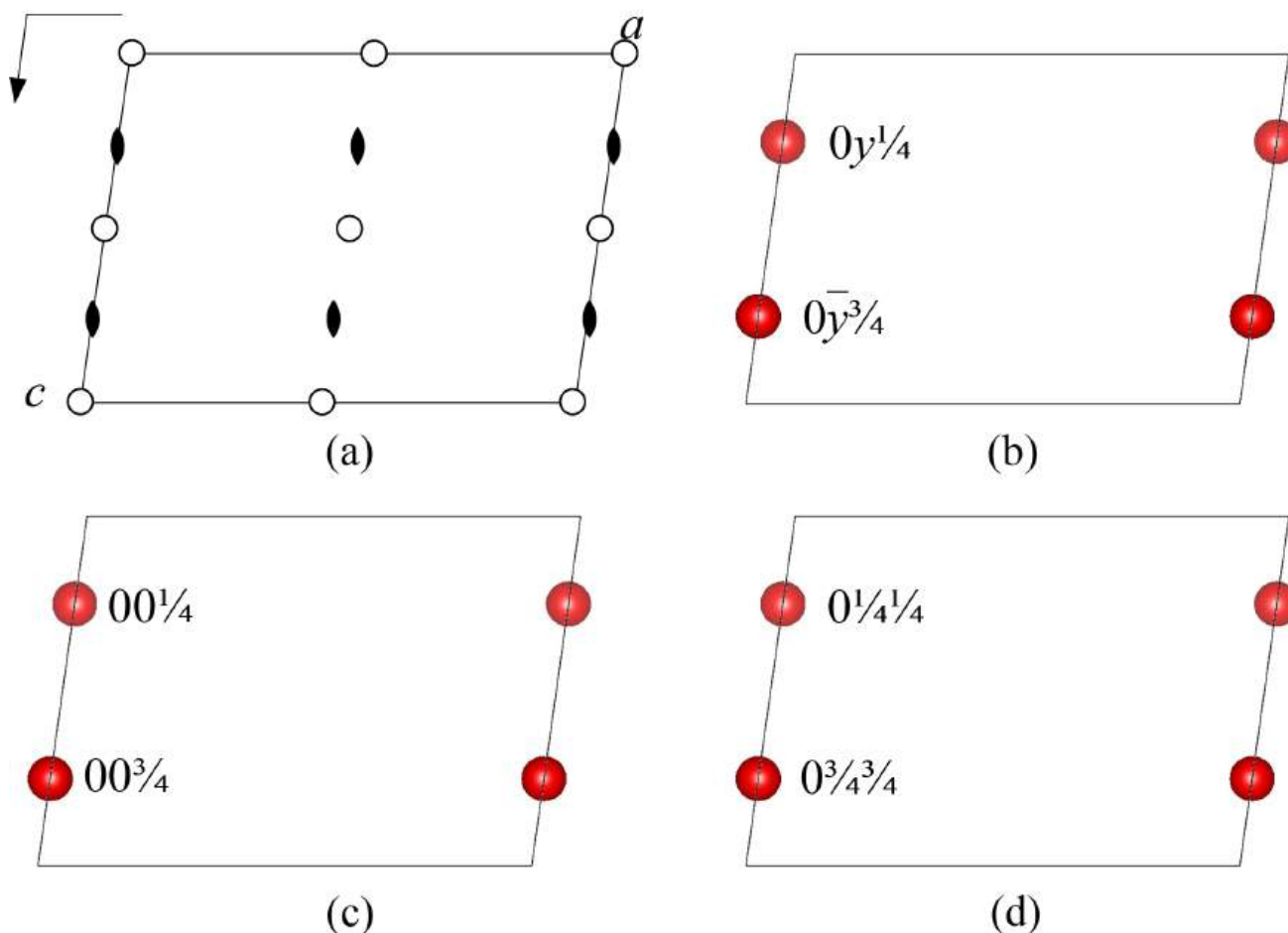


Figure 1. (a) Symmetry elements in $P2/c$. (b) An atom placed at $0, y, \frac{1}{4}$ with $y \neq 0, \frac{1}{4}, \frac{1}{2}$ or multiples generates a characteristic orbit. (c) An atom placed at $0, 0, \frac{1}{4}$ generates a non-characteristic orbit with eigensymmetry $P2/m$. (d) An atom placed at $0, \frac{1}{4}, \frac{1}{4}$ generates a non-characteristic orbit with eigensymmetry $A2/m$.

2. The *site-symmetry groups* of the positions belonging to the crystallographic orbit. The operations of the space group that leave a position fixed in space form a group that is called the site-symmetry group of that position. Wyckoff positions are simply classes of crystallographic orbits based on site-symmetry groups.

Site-symmetry groups and Wyckoff positions

Let us consider a position in the crystal, identified by its coordinates x_1, y_1, z_1 . Among the operations of the space group, some leave this position fixed: at least the identity operation, but often some other operations as well. If we gather all the operations that leave x_1, y_1, z_1 fixed, we obtain a group that is called the site-symmetry group of the position x_1, y_1, z_1 . Let us call it S_1 ; it is obviously a subgroup of the space group. If we now fix our attention on another different position with coordinates x_2, y_2, z_2 , its site-symmetry group will be indicated by S_2 .

The relationship between S_1 and S_2 provides the criterion to classify crystallographic orbits; the result is classes of crystallographic orbits known as Wyckoff positions. The relationship between S_1 and S_2 is the basis for the classification of crystallographic orbits:

1. $S_1 = S_2 = 1$, i.e. the site-symmetry group contains only the identity. The two positions x_1, y_1, z_1 and x_2, y_2, z_2 , as well as all the other symmetry-related positions (x_j, y_j, z_j) , belong to a class of crystallographic orbits referred to as *general* because none of the operations of the space group, apart from the identity, fixes any of these positions. A different set of coordinates (x'_j, y'_j, z'_j) defining another different and independent crystallographic orbit again has the site-symmetry group $S' = 1$ and so belongs to the same class. This class is known as the *general Wyckoff position*.
2. $S_1 = gS_2g^{-1} > 1$, where g is a symmetry operation of the space group G . The relationship between S_1 and S_2 is known as *conjugation* and is a *similarity* relation. S_1 and S_2 are known as conjugate subgroups of G . Conjugation can be pictorially described as “do the same somewhere else”. Two site-symmetry groups that are conjugate subgroups of the same space group contain operations of the same kind but at least some of the symmetry elements are located or oriented differently in space. Remember that a crystallographic orbit contains an infinite number of positions (x_j, y_j, z_j) that are located differently in space. They cannot have the same site-symmetry group (with the obvious but trivial exception of the general position) but because they are symmetry-related, their site-symmetry groups are also tightly related: they are indeed *conjugate*. Now, all the crystallographic orbits whose site-symmetry groups are conjugate subgroups of the space group belong to the same class of crystallographic orbits, and are called *special Wyckoff positions*.
3. S_1 and S_2 are not conjugate subgroups of the space group G . The corresponding crystallographic orbits belong to different classes, i.e. to different Wyckoff positions.

Let us consider the example of space group $P4mm$. A position with coordinates $x, 0, z$ is left invariant by the identity operation and by a reflection through a mirror plane perpendicular to $[010]$ and passing through $y = 0$, so that its site-symmetry group has order 2 and is denoted symbolically by $.m$. By applying the infinitely many symmetry operations of $P4mm$ to $x, 0, z$ one gets infinite positions, with coordinates $x, 1, z$; $-x, 0, z$; $-x, 1, z$; $0, x, z$; $0, -x, z$; $1, x, z$; $-1, -x, z$ and so on. Let us consider the position with coordinates $0, x, z$. It is left invariant by the identity and by a reflection through a mirror plane perpendicular to $[100]$ and passing through $x = 0$, so that its site-symmetry group has order 2 and is again symbolically indicated as $.m$. Now, the site-symmetry groups of the two positions are not identical:

the reflection is performed through two mirror planes oriented differently. However, they are conjugate subgroups of $P4mm$. Indeed, by applying, for example, a fourfold rotation about $[001]$ through the origin to $x,0,z$, one obtains $0,x,z$. Formally, the site-symmetry group $\{1, m_{x0z}\}$ becomes $\{1, m_{0xz}\}$ under conjugation:

$$4[001]_{00z}\{1, m_{x0z}\}4^{-1}[001]_{00z} = \{1, m_{0xz}\}.$$

If we choose another operation of $P4mm$ we get a different position with a different site-symmetry group, which is again conjugate to $\{1, m_{x0z}\}$. In the following table a few examples are explicitly shown.

Operation	Position	Site-symmetry group
$4[001]_{00z}$	$0xz$	$\{1, m_{0xz}\}$
$t(010)$	$x1z$	$\{1, m_{x1z}\}$
$4[001]_{\frac{1}{2}\frac{1}{2}z}$	$1xz$	$\{1, m_{1xz}\}$

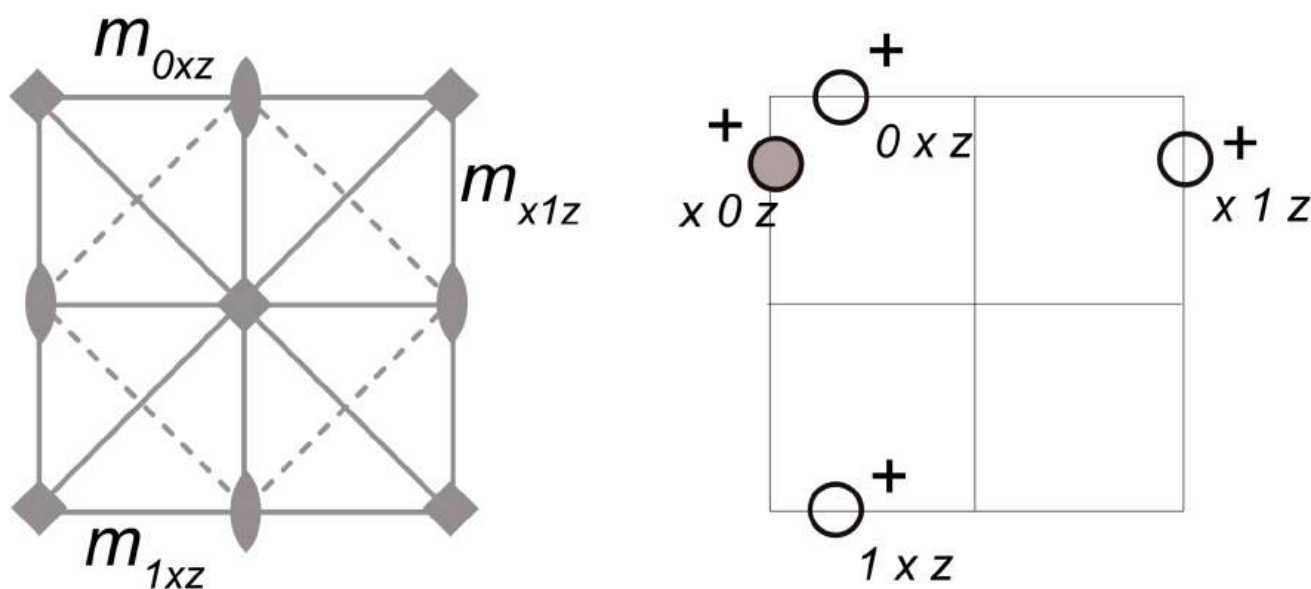


Figure 2. Diagram of the space group $P4mm$. Left: symmetry elements; right: four of the atomic positions belonging to the Wyckoff position 4e.

Figure 2 illustrates this table. On the left are the positions of the relevant symmetry elements in $P4mm$ with the corresponding mirror planes and a glide plane marked. On the right are the positions of atoms, with the shaded one at the starting point $x,0,z$.

These atomic positions, and all the infinitely many positions that are obtained by applying the infinitely many operations of the space group, are located differently in space and, quite obviously, have different site-symmetry groups. They are, however, all conjugate subgroups of $P4mm$ and therefore belong to the same class of crystallographic orbits in $P4mm$. This class is called the 4e Wyckoff position.

A somewhat richer example is $I4mm$, where the I -centring vector leads to an additional population of the orbits. In particular, the translation $t(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ applied to $x,0,z$ leads to $\frac{1}{2}+x, \frac{1}{2}, \frac{1}{2}+z$, the site-symmetry group of which is $\{1, m_{x\frac{1}{2}z}\}$. The position $x, \frac{1}{2}, z$, which in $P4mm$ belongs to a different class of crystallographic orbits with respect to $x,0,z$, is now in the same class because the corresponding site-symmetry groups are conjugate subgroups of $I4mm$ but not of $P4mm$. Accordingly, the Wyckoff positions 4e and 4f in $P4mm$ coalesce to the single Wyckoff position 8d in $I4mm$.

The fundamental difference between crystallographic orbits and Wyckoff positions

Let us repeat: Wyckoff positions are classes of crystallographic orbits. In some cases, we can establish a 1:1 relationship between them; this essentially means that a class (Wyckoff position) contains only one crystallographic orbit. For that to be the case, the infinitely many positions belonging to a crystallographic orbit must contain no free parameters. In our example $P4mm$ this does not happen, because $P4mm$ is a pyroelectric space-group type, containing a polar axis (the c axis). Therefore, even the Wyckoff positions of lowest multiplicity correspond to infinitely many crystallographic orbits. For example, the Wyckoff position 1a is the class of crystallographic orbits generated by applying the infinitely many operations of $P4mm$ to the coordinates $0,0,z$. But for each value of z we have a different crystallographic orbit. The site-symmetry groups of each and every position in any of these crystallographic orbits are all of type $4mm$ and are all a conjugate subgroup of $P4mm$; therefore they all belong to the same class, *i.e.* to the same Wyckoff position.

The situation is different in space groups of type $P4/m$. All the infinitely many crystallographic orbits generated by applying the operations of $P4/m$ to a position with coordinates $0,0,z$ where $z \neq 0, \frac{1}{2}, 1$, *etc.* have a site-symmetry group of type $4..$ and all belong to the same class, *i.e.* the same Wyckoff position ($2g$). However, positions $0,0,0$, $0,0,\frac{1}{2}$, $0,0,1$, *etc.* have site-symmetry groups of type $4/m..$ and constitute a single crystallographic orbit corresponding to Wyckoff position 1a. In this case, there is a 1:1 correspondence between crystallographic orbits and Wyckoff positions.

Historical perspective

The classification of crystallographic orbits into classes was introduced in 1922 by Wyckoff³ and the results have since then been known as Wyckoff positions, whereas the term crystallographic orbit was introduced much later.² This is probably the reason why the more fundamental, simpler concept of orbit is unfamiliar to many crystallographers, whereas the more complex concept of classes of orbits, universally known as Wyckoff positions, is used on a daily basis.

¹ These are clearly not rigorous definitions, as justified by the purpose of this article, which is to draw the reader's attention to the different ways we classify crystals and their properties. The reader interested in a detailed analysis of these classifications can find many more details in our recent article in the *Journal of Applied Crystallography* [Nespolo, M., Aroyo, M. I. & Souvignier, B. (2018). *J. Appl. Cryst.* **51**, 1481-1491].

² The first time the term crystallographic orbit appeared in the literature dates back to 1979: Matsumoto, T. & Wondratschek, H. (1979). *Z. Kristallogr.* **150**, 181-198. For a detailed discussion with a historical perspective, see Nespolo, M. (2008). *Acta Cryst.* **A64**, 96-111.

³ Wyckoff, R. W. G. (1922). *The Analytical Expression of the Results of the Theory of Space Groups*. Carnegie Institution, Washington, pp. vii+180. The second edition, with three-dimensional drawings of the unit cells, was published again by the Carnegie Institution in 1930.

SPECIAL REPORT

IAEA activities on synchrotron radiation applications

Alessandro Migliorilan Swainson, Francois Foulon, Roman Padilla AlvarezDanas Ridikas



One example of IAEA's support in spreading information to the developing world. The IAEA assisted LAAAMP in the production of the Arabic version of its brochure for non-experts.

In the public's mind, the International Atomic Energy Agency (IAEA) is most closely associated with safeguards and nuclear energy. However, IAEA also carries out many activities supporting the development of research and applications of radiation and nuclear-related technologies, with an emphasis on assisting developing countries, as reflected in its motto *Atoms for Peace and Development*.

The IAEA, through its Physics Section and Technical Cooperation Program, has a long history of supporting research accelerator applications. The most common of these accelerators are ion-beam accelerators. However, in recent years, there has been a growth in the interest in large accelerators around the world, including synchrotrons.

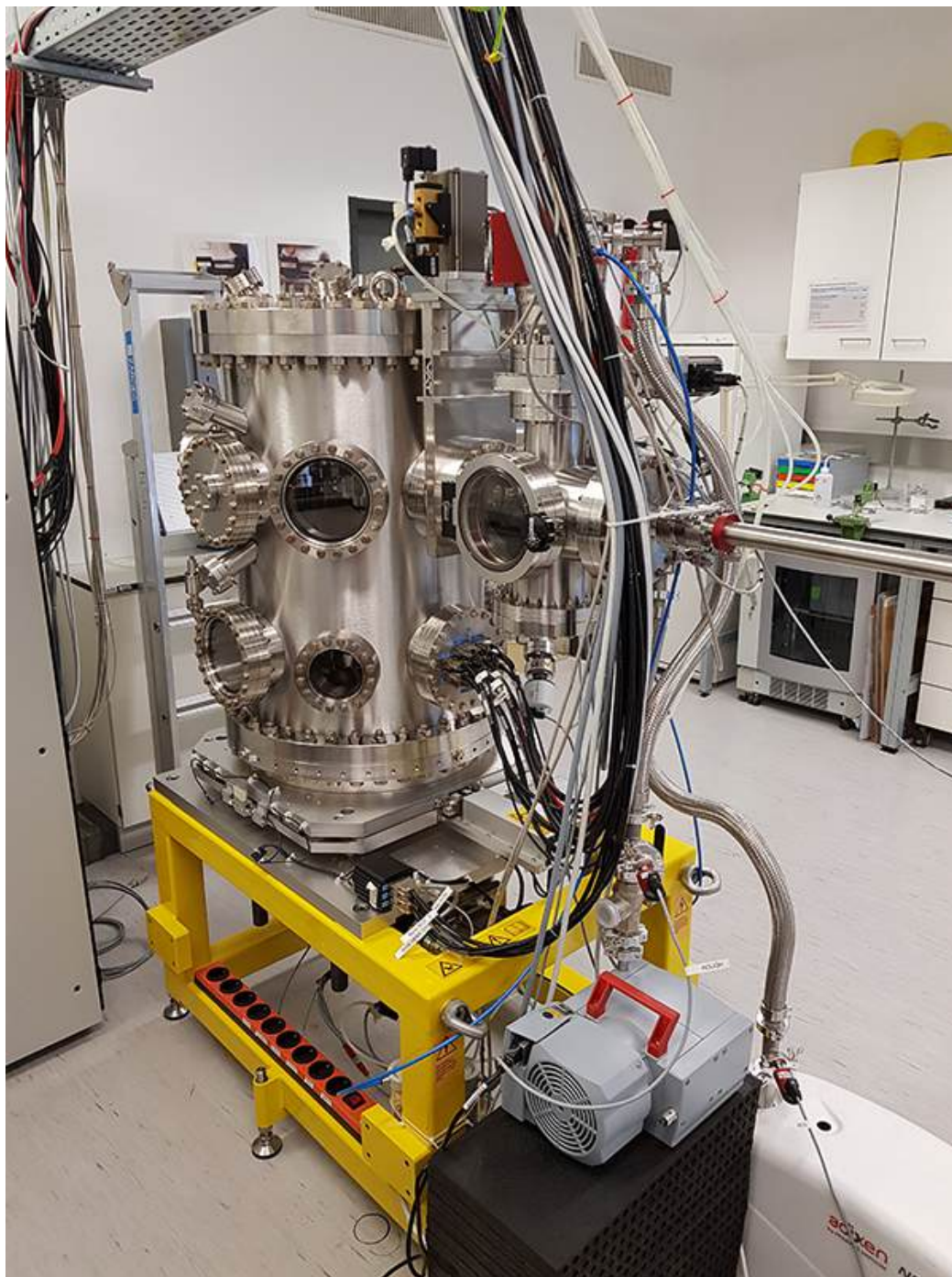
Partnership with Elettra Sincrotrone Trieste

The IAEA has several activities regarding synchrotron applications, especially concerning XRF. Since 2013, the main focus has been the beamline and endstation that were developed by Elettra Sincrotrone Trieste (EST; Italy) and IAEA. The endstation at the XRF beamline was provided by IAEA. This is equipped with a chamber that exhibits both standard and ultra-high-vacuum capabilities. It has an advanced seven-axis manipulator for the alignment and movement of the sample and a series of photodiodes that

allows different variants of XRF, such as 2D-XRF, GI-XRF, GE-XRF, XRR and TXRF, to be performed [A. G. Karydas *et al.* (2018). *J. Synchrotron Rad.* **25**, 189–203]. XAS can also be performed on the beamline. Thanks to a generous donation from Germany, a second identical endchamber resides at IAEA's laboratories just outside Vienna at Seibersdorf, Austria. This is used for the development and testing of new devices for the main chamber at Elettra, and IAEA uses it as part of its annual training in "classical" XRF, in the form of "group fellowships".

In the period 2015–2018, IAEA organised a Coordinated Research Project (CRP) involving 14 countries, entitled *Experiments with Synchrotron Radiation for Modern Environmental and Industrial Applications*. This CRP was chiefly focussed around the new XRF beamline and endstation at EST but also involved work carried out at eight other synchrotrons worldwide, resulting in 47 publications and 73 conference presentations, and involving 28 students and 10 post-doctoral fellows. During this time, the XRF endstation and its control electronics and software were substantially developed, and experiments performed by the participants used its flexibility for projects covering topics from air pollution, biomedicine, the broader environment, bioremediation, food, cultural heritage, energy materials and nanomaterials to X-ray fundamental parameters.

In 2019, the partnership between IAEA and EST was renewed, the IAEA providing funds for an annual training workshop, described below. In addition a limited budget was assigned to offset some of the costs of travel and subsistence for scientists coming from developing countries to perform experiments on the XRF beamline that have passed the appropriate review committee at EST. In 2019, EST made 23 beamtime allocations on the instrument.



The second, “mirror”, endchamber located at IAEA’s Seibersdorf Laboratories, identical to that installed at the XRF beamline at EST.

Training workshop at EST

The first joint IAEA-EST training workshop was held from 7 to 11 October 2019, and was attended by one Italian (as the hosting country) and one participant from each of six African states (Algeria, Benin, Egypt, Ghana, Kenya and South Africa). The intent is to make this an annual event limited to about 10 attendees. Our target audience is people from regions with limited access to such facilities and limited personal experience of such experiments. IAEA covers the full cost of attending, including transportation and subsistence.



Participants at the IAEA-Elettra Training Workshop held in October 2019.

The format of last year's event included lectures on the first day on general aspects of a synchrotron and the design of a beamline, and included a presentation on how to write proposals for beamline utilisation. The following three days were dedicated to hands-on training and covered advanced variants of XRF, XAS and XRD using three EST beamlines (XRF, XAFS and MCX, respectively). By the end of the training workshop, the attendees had written their own proposal, presented it and mutually reviewed each other's.

Advanced light sources in the developing world

The most recent advanced light source to be turned on in the world is SESAME (Synchrotron-light for Experimental Science and Applications in the Middle East), located just outside Amman, Jordan. SESAME started up under the auspices of another UN agency, UNESCO.

Institutionally modelled on CERN, SESAME is now an independent intergovernmental organisation comprising Cyprus, Egypt, Iran, Israel, Jordan, Pakistan, the Palestinian Authority and Turkey as Members. IAEA has worked alongside UNESCO in the support of SESAME for more than a decade, *e.g.* by providing training to staff, sending international experts to assist during design and commissioning, assisting the regulatory agency in developing appropriate regulations, providing fellowships to scientists from the region and procuring radiation monitoring and protection equipment to ensure the safety of the staff and visitors.



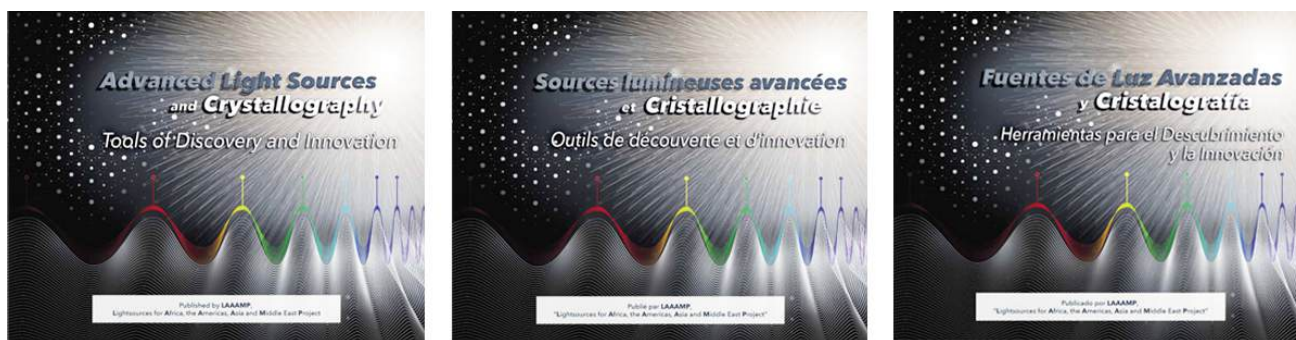
IAEA has procured approximately €190,000 of radiation protection and monitoring equipment in the last 2 years for SESAME as part of a Technical Cooperation project.

Among other projects, IAEA is currently working with the Synchrotron Light Research Institute in Thailand on the development of their final planned instrument on their current synchrotron (their XRF instrument), and the Agency has assisted the international initiative that, in the future, wants to bring a light source to Africa. Africa has the second largest population of any continent but does not host a single one of the ca 150 such facilities in the world. To date, the initiative has been spearheaded by the group known as the African Light Source (AfLS), and IAEA has supported their two significant events in Grenoble, France

(2015) and Accra, Ghana (2019). The latter was the occasion on which their efforts received their first official recognition by an African government. Ghana formally voiced its support for the project and promised to support it in the Economic Community of West African States (ECOWAS), the regional political and economic union, and the African Union.

Beyond direct work with or assistance to individual light sources, IAEA has also supported the promotion of information to the general public and officials that may help in the assistance of the development of future projects.

One example of such work is with *LAAAMP* (Lightsources for Africa, the Americas, Asia and Middle East Project), a project created by the IUCr in partnership with IUPAP under the 2016–2019 ICSU grants programme. One of the outputs of this project has been a brochure in plain language for non-experts and interested laypersons (students, visitors, funders) entitled *Crystallography and Advanced Light Sources: Tools of Discovery and Innovation*. This combination brings benefits to societies that host such facilities or have access to them. The Agency has supported *LAAAMP* in their endeavours to spread this information to the developing world: IAEA has six official languages and uses its professional translation services to assist in the creation of the Spanish and French versions. Together we recently created an Arabic version, a language in which very little information about such technologies is currently available.



In addition to the Arabic version, IAEA assisted *LAAAMP* in the production of Spanish and French versions of their brochure. These, together with the English version, are available for free download [here](#).

A second example was a side event at the 2019 IAEA General Conference. This annual event attracts delegates from more than 170 Member States of the IAEA, including government ministers, senior members of atomic energy commissions and their Permanent Missions to the UN. Recognising that synchrotrons are large-scale infrastructures and that in many developing countries the atomic energy commissions are often as, or more heavily, involved in their creation and operation than are the ministries of education and research, a side event, called *Light: the Way Forward!*, was held. This featured speakers from SESAME, the Synchrotron Light Research Institute (Thailand), EST and the AfLS, describing the experiences from quite different societies in different corners of the world. Beyond the research opportunities afforded to scientists in the region, and the involvement of industries in facility development and as users of the applications, it became clear that even a “national” facility is truly an international project and operation, as was firmly illustrated by the high utilisation of EST by neighbouring countries and the strong ASEAN links developed by the Thai synchrotron.

ICTP workshop

For more than 50 years, IAEA has had a close relationship with the International Centre for Theoretical Physics (ICTP), also based in Trieste. IAEA sponsors 10–12 workshops on nuclear and related themes each year. In April 2020, there will be a two-week [School on Advanced Light Sources: Principles, Methods, and Multidisciplinary Applications](#). This school, proposed in collaboration with EST and representatives from the ESRF, *LAAAMP*–IUPAP and the AfLS, is for people with more in-depth knowledge than the training workshop and will cover design elements of a light source, beamline optics and

applications. As with all ICTP activities, the main focus is on providing high-level courses and activities with access targeted to developing countries, and selected participants attend cost-free.

**The authors are at the Physics Section, Division of Physical and Chemical Sciences,
Department of Nuclear Sciences and Applications, IAEA, Vienna, Austria.**

MEETING REPORT (IUCR SUPPORTED)

17th International Summer School on Crystal Growth

Joan M. RedwingTom Kuech



ISSCG-17 participants at YMCA of the Rockies – Snow Mountain Ranch, Granby, CO, USA.

The 17th International Summer School on Crystal Growth (ISSCG-17) was held at the YMCA of the Rockies – Snow Mountain Ranch in Granby, CO, USA, from 21 to 26 July 2019. The event was organized and hosted by the American Association for Crystal Growth (AACG) under the auspices of the International Organization for Crystal Growth (IOCG). In keeping with tradition, the school was scheduled the week prior to the [19th International Conference on Crystal Growth and Epitaxy \(ICCGE-19\)](#), which was held in Keystone, CO, USA. The international meetings (ICCGE/ISSCG) represent a 60-year history of international cooperation in the field of crystal growth and epitaxy. The previous summer school (ISSCG-16) was held in 2016 at Lake Biwa, Shiga, Japan. The summer school was last held in the USA in Park City, UT, in 2007.

The technical program consisted of 14 lectures covering both theoretical and experimental aspects of growth and characterization of semiconducting, oxide, metallic, organic and biological crystals. Lectures were given by leading researchers in fundamentals of crystal growth and epitaxial processes, nanocrystallization and *in-situ* and *ex-situ* characterization. A textbook entitled *Fundamentals of Crystal Growth and Epitaxy* based on the lecture materials was provided to students in advance of the school to help them prepare for the immersive educational experience and to serve as a useful reference text for their future research.

The format of the school was designed to encourage interaction between lecturers and attendees. Informal discussions were facilitated *via* numerous social events including breakfasts and dinners, poster sessions with extended coffee breaks, a welcome reception and a barbeque. In addition, the YMCA venue had numerous activities providing additional opportunities for attendees to interact in a rustic and scenic outdoor setting.

The specific objectives of ISSCG-17 included the following:

- to provide an intellectual forum to promote and disseminate the science and practice of crystal growth and epitaxy to graduate students and early career researchers through lectures and activities given by internationally recognized experts who have significant experience in academic instruction
- to create networking activities for graduate students and early-career researchers to interact with the senior lecturers and each other to foster community development
- to develop a textbook on the fundamentals and practice of crystal growth for attendees and other interested parties that captures the intellectual content of the school and serves as a reference for research and graduate-level coursework in the field
- to facilitate interactions between participants from different countries and geographic regions to support and strengthen the international crystal growth community
- to promote and encourage participation by women and members of underrepresented groups in science and engineering in the field of crystal growth and epitaxy.

Participants

A total of 72 attendees participated in the school with ~50% being graduate students or postdoctoral scholars and the remainder consisting of both new and experienced researchers in the field of crystal growth. We were committed to achieving a diverse group of participants and specifically encouraged applications from women, members of underrepresented racial and ethnic groups in STEM in the US and a diverse range of geographic participants. A total of 14 internationally recognized scholars (21.4% women and minority) served as lecturers. The audience included 37 graduate students and postdoctoral scholars and 19 regular attendees, which included many early-career researchers from universities, national laboratories and industry. Among the school attendees, approximately 37% were female among both regular and student/postdoc attendees. The geographic distribution of regular and student/postdoc attendees included 66% from the US, 11% from Asia-Pacific and 23% from Europe.

IUCr awards

Five graduate students from Europe and Asia-Pacific were selected to receive the IUCr Young Researcher Awards, which were used to offset their transportation, housing and meal costs to attend ISSCG-17.

MEETING REPORT (IUCR SUPPORTED)

19th International Conference on Crystal Growth and Epitaxy

Vincent Fratello Jeffrey Derby Andrew Allerman



The American Association for Crystal Growth (AACG) would like to thank the IUCr for its support of the 19th International Conference on Crystal Growth and Epitaxy (ICCGE-19). The conference was held from 28 July to 2 August 2019 in beautiful Keystone, CO, USA, under the auspices of the International Organization for Crystal Growth (IOCG) jointly with the 19th US Biennial Workshop on Organometallic Vapor Phase Epitaxy (OMVPE-19) and, as is traditional, was preceded by the [17th International Summer School on Crystal Growth \(ISSCG-17\)](#), held at the YMCA of the Rockies in nearby Granby, CO.

ICCGE-19 was the latest in a sequence that started with ICCG-1 in Boston, MA, USA, in 1966, which marked the formation of the crystal growth community as an independent discipline. The key principle of the IOCG and the ICCGE conferences is that the theory and practice of crystal growth are common across a range of materials and applications and that we have much to share with one another. The year 2019 also marked the 50th anniversary of the founding of the American Association for Crystal Growth.

The OMVPE-19 workshop continued a tradition, started at Cornell University, Ithaca, NY, USA, in 1983, of bringing together specialists in the OMVPE field from industry, academia and government laboratories. The informal atmosphere and scenic surroundings of the workshop provided a venue for the presentation of new results and for newcomers to the field to familiarize themselves with OMVPE science and technology.

Overall there were 639 attendees from 31 countries, including 50% from the US, 20% from Europe, 19% from Japan and 4.5% from China.

A total of five IUCr Young Scientist subsistence and travel awards were made to graduate students. The recipients and the presentations they gave were as follows:

- Scott Dossa (University of Minnesota, Minneapolis, MN, USA) “Modeling the Optical Floating Zone Crystal Growth System for Materials Discovery” (Invited)
- Michael Liao (UCLA, USA) “Surface Evolution of Single Crystalline β -Ga₂O₃ with Chemical Mechanical Polishing and Annealing at an Elevated Temperature”
- Grace Pan (Harvard University, Cambridge, MA, USA) “Effective Dimensionality-Tuned Electronic States in Metastable Nickel Oxide Thin Films”
- John Roerig (University of Minnesota, Minneapolis, MN, USA) “Evaluation of an Inverted Vertical Gradient Freeze Method for Cadmium Zinc Telluride”
- Xiaotian Zhang (Penn State, PA, USA) “Epitaxial Growth of Layered β -In₂Se₃ Thin Films Via Metalorganic Chemical Vapor Deposition”

The IUCr also gave awards to two student poster presenters:

- Smail Amari (CEA LITEN LCO, Grenoble, France), “Crystal Growth and Characterization of CH₃NH₃PbBr₃ Single-Crystals for X-Ray Photodetection”
- Muad Saleh (Washington State University, Pullman, WA, USA) “Electrical and Optical Properties of Zr Doped β -Ga₂O₃ Single Crystals Grown by Czochralski Method”



Announced by Tom Kuech, Smail Amari (left) and Muad Saleh receive their IUCr poster prizes from Hanna Dabkowska (IUCr Vice-President) and Andrea Zappettini.

ICCGE/OMVPE-19 provided sessions for presentation and discussion of recent advances in research and development over all aspects of crystal growth, epitaxy, characterization and applications. A total of 473 oral presentations and 75 poster presentations were delivered. The conference was an excellent opportunity to advance the science and technology of crystal growth, epitaxy, characterization and applications, as well as to convene with old and new friends, colleagues and business associates.

The following plenary talks were given in the Monday, Tuesday and Thursday Plenary Sessions:

- Robert Feigelson (Stanford University, CA, USA) “A Retrospective on the American Association for Crystal Growth on Its 50th Anniversary”
- Thierry Duffar (SIMaP-EPM, Grenoble, France) “Defect Engineering in Bulk Crystal Growth”
- Darrell Schlom (Cornell University, Ithaca, NY, USA) and Reinhard Uecker [Leibniz Institute for Crystal Growth (IKZ), Berlin, Germany] “Development of New Perovskite Substrates Enabling Strain Engineering of Oxide Thin Films” (IOCG Frank Prize talk).
- Kazuo Nakajima (Tohoku University, Japan, emeritus) “Development of High-Quality Crystals for III-V Quaternary Optical Semiconductors and Solar Cell Silicon and Their Industrial Applications” (IOCG Laudise Prize talk).
- Anton Jesche (Center for Electronic Correlations and Magnetism, Institut für Physik, Universität Augsburg, Germany) “The Metal Flux as a Powerful Tool for the Solid State Physicist” (IOCG Schieber Prize talk).
- Kei May Lau (Hong Kong University of Science and Technology) “Advantages and Challenges of Growing III-V Photonic and Electronic Devices on Si”
- Jim De Yoreo (Pacific Northwest National Laboratory, Richland, WA, USA) “They Call It Free Energy So, Hey, Why Pay?”

General and Topical Sessions were held as listed below. There were two special sessions honoring pioneers in the field and three topical symposia that spanned the week.

- Fundamentals of Crystal Growth
- Modeling of Crystal Growth Processes
- Bulk Crystal Growth
- Nonlinear Optical and Laser Host Materials
- Biological and Biomimetic Materials
- Detector Materials: Scintillators and Semiconductors
- Industrial Crystal Growth Technology and Equipment
- Advanced Equipment and Growth Technology
- Surfaces and Interfaces
- *In situ* Observation and Characterization
- Characterization Techniques for Bulk and Epitaxial Crystals
- Thin Film Growth, Epitaxy, and Superlattices
- Nanocrystals, Quantum Dots, and Nanowires
- 2D Materials and Devices
- Materials for Photovoltaics and Other Energy Technology
- Selective and Patterned Epitaxial Growth
- III-V Epitaxial Growth for Devices
- III-V's on Silicon
- III-V Wide Bandgap Nitride Semiconductors and Devices
- Narrow Bandgap Semiconductors and Devices
- BN Epitaxial Growth and Characterization
- Silicon Carbide Materials and Devices
- Gallium Oxide Materials and Devices
- Special Session: Honoring Michael Schieber
- Special Session: Honoring George Gilmer
- Fourth Symposium on 2D and Other Low Dimensional Materials:
- Second Symposium on Complex Oxides
- Second Symposium on Ferroelectric Crystals and Textured Ceramics

Once again, we would like to express our appreciation for the IUCr's support. There is great synergy between our communities, and crystallographers were well represented. In particular we thank Hanna

Dabkowska and Andrea Zappettini for their support and attendance. Hanna is the new IOCG Vice President and Andrea will be the co-chair of ICCGE-20 in Naples, Italy, in 2022.

MEETING REPORT

77th Annual Pittsburgh Diffraction Conference

Leighton Coates Bryan Chakoumakos

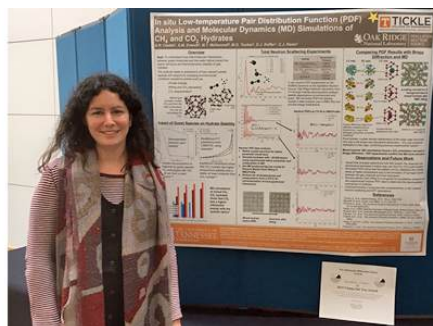


The 77th Annual Pittsburgh Diffraction Conference was held between 24 and 26 October 2019 at Oak Ridge National Laboratory (ORNL), Oak Ridge, TN, USA. The Pittsburgh Diffraction Society (PDS) is a not-for-profit organization that promotes fundamental and applied diffraction and crystallographic research and the exchange of ideas and information concerning such research. The PDS was founded by Surain S. Sidhu, who organized the first Pittsburgh Diffraction Conference in 1943. The conference was attended by 120 registered participants and featured 30 oral presentations and 36 poster presentations in sessions focused on all areas of diffraction, from powders to protein crystals. The conference was organized by a local committee of Kate Page, Bryan Chakoumakos, Xiaoping Wang and Matt Tucker, and was chaired by Leighton Coates.

The conference began with an opening reception on 24 October, followed by parallel scientific sessions on structural biology and “non-biological diffraction” on the 25th. The structural biology sessions started with a keynote address entitled “Electrons or photons, diffraction rules them all” by Fred Dyda (National Institute of Diabetes and Digestive Kidney Diseases, Bethesda, MD, USA) followed by talks from Robert Phillips (University of Georgia, Athens, GA, USA), Jahaun Azadmanesh (University of Nebraska, Omaha, NE, USA), Tim Mueser (University of Toledo, OH, USA), Chris Davies (Medical University of South Carolina, Charleston, SC, USA), Patricia Langan (Lentigen Technology, Gaithersburg, MD, USA), Martin Egli (Vanderbilt University, Nashville, TN, USA), Raktim Roy (The Scripps Research Institute, Jupiter, FL, USA), Jeremy Lohman (Purdue University, West Lafayette, IN, USA), Timothy Stachowski (Hauptman Woodward

Institute, Buffalo, NY, USA), David J. Schuller (Cornell University, Ithaca, NY, USA) and concluded with a talk by B.C. Wang (University of Georgia, Athens, GA, USA).

The non-biological sessions included powder diffraction, magnetic diffraction, total scattering and small-molecule crystallography. The powder diffraction session chaired by Cora Lind-Kovacs (University of Toledo, OH, USA) included talks by Bob Von Dreele (Argonne National Laboratory, Lemont, IL, USA) “Systematic determination of magnetic structures driven by space groups in GSAS-II”, Weiwei Xie (Louisiana State University, Baton Rouge, LA, USA) “Chemistry Perspectives to Novel Quantum Materials” and Corey Thompson (Purdue University, West Lafayette, IN, USA) “Probing magnetic interactions in Re-based double perovskites”. The magnetic diffraction session chaired by Ovi Garlea (ORNL) included talks by Joseph Kolis (Clemson University, SC, USA) “Magnetic behavior and neutron diffraction of transition metal vanadate single crystals”, Margarida Henriques (CAS Institute of Physics, Prague, Czech Republic) “Modulations in magnetic materials” and Haidong Zhou (University of Tennessee, Knoxville, TN, USA) “Structural and magnetic properties of $R_2\text{TiO}_5$ ($R = \text{Dy}$ and Yb)”. The total scattering session chaired by Kate Page (University of Tennessee, Knoxville, TN, USA) included talks by Emil Bozin (Brookhaven National Laboratory, Upton, NY, USA) “Local orbital degeneracy lifting as a precursor to an orbital-selective Peierls transition”, Ella Mara Schmidt (University of Oxford, UK) “A disordered superspace approach to understand highly structured diffuse scattering” and Robert Koch (Brookhaven National Laboratory, Upton, NY, USA) “Total scattering as a tool for quantifying local structure in iron-based superconductors”. The conference continued on 26 October, starting with a combined session on diffraction, which featured talks by Ken Herwig (ORNL), Leonard Mueller (University of California, Riverside, CA, USA) and Helen Playford (Rutherford Appleton Laboratory, Didcot, UK).



Jahaun Azadmanesh and Bernadette Cladek (right) each won a Chung Soo Yoo poster award for graduate students presenting the best posters at the conference. The poster prizes were presented by Leighton Coates (left), President of the PDS.

Following this session, the Chung Soo Yoo poster awards, established by the PDS in his honor, were presented to the graduate students presenting the best posters at the conference. These were presented to Jahaun Azadmanesh (University of Nebraska, Omaha, NE, USA) and Bernadette Cladek (University of Tennessee, Knoxville, TN, USA) during the business meeting of the PDS. The conference closed with a session on hybrid techniques featuring talks by Cora Lind-Kovacs (University of Toledo, OH, USA), Dustin Gilbert (University of Tennessee, Knoxville, TN, USA) and Rebecca McAuliffe (ORNL). We would like to thank all our sponsors and exhibitors: Art Robbins Instruments, Formulatrix, Molecular Dimensions, Rigaku, Thermo Scientific and TTP Labtech without whose support the conference would not have been possible. The 78th Annual Pittsburgh Diffraction Conference will be hosted at the Stanford Accelerator Research Center (SLAC), CA, USA, between 27 and 29 September 2020. Please see the [PDS website](#) for upcoming details.

MEETING REPORT (IUCR SUPPORTED)

11th School of the Argentinean Association of Crystallography



San Carlos de Bariloche XI AACr School (Year 2019).

The XV Annual Meeting of the Argentinean Association of Crystallography (AACr) was held from 12 to 15 November 2019 in the Centro Atómico Bariloche, San Carlos de Bariloche, Río Negro, Argentina. More than 160 crystallographers from different regions of Argentina and from several other Latin American countries participated in the event. Following the Meeting, the [XI School of the AACr](#) was held at the same location from 18 to 22 November. The School addressed two topics in parallel:

Course 1: “Structural and magnetic Rietveld refinement of X-ray and neutron diffraction data”, where 35 students had the opportunity to attend lectures by Juan Rodríguez Carvajal (ILL, Grenoble, France), financed by the Maldacena Program (from Instituto Balseiro, Bariloche, Argentina), and Oscar Fabello (ILL), financed by the IUCr Visiting Professorship scheme (replacing María Teresa Fernández Díaz). In addition, many local organizers (Federico Napolitano, Liliana Moggi, Gabirela Aurelio, Martín Saleta, Dina Tobia and Adriana Serquis) participated in the tutorials and special seminars.

Course 2: “Texture and strain in polycrystalline materials”, which was run by Raúl Bolmaro (IFIR Rosario, Argentina), Javier Santisteban (Argentinian Neutron Beams Laboratory) and Florencia Malamud (Centro Atómico Bariloche).



Students attending a Course 2 lecture.

The Local Organizing Committee obtained financial support from the IUCr, Analytical-Bruker, INBOX-Panalytical, Thermo Fisher and the local funding agency, Ministerio de Ciencia y Tecnología. The event brought together 58 participants (postdocs and PhD, MSc and undergraduate students) of different nationalities: Argentina (46), Colombia (5), Cuba (3), Venezuela (2) and Uruguay (2).

The School program consisted of a 40-hour schedule (20 hours of lectures, 18 hours for experimental and practical tutorial sessions and 2 hours for a final exam). A second practical exam was given to the students, to be completed at home and returned two weeks after the School had finished. This two-step exam was taken by 38 participants who will receive academic credit for their participation in the School.

The students were provided with an anonymous survey to obtain their opinions on different aspects of the School. The large majority of comments were positive, many of them including helpful remarks that will be of valuable help in the organization of future Schools.

MEETING REPORT

2019 Meeting of the Crystallographic Society of Japan

Kunihisa Sugimoto



Participants in the Young Crystallographer's Meeting 2019.

The 2019 Annual Meeting and General Assembly of the Crystallographic Society of Japan (CrSJ2019) was held during 19–20 November at Kanazawa Bunka Hall in Kanazawa, Japan, and about 250 crystallographers participated. The meeting featured 43 oral and 89 poster presentations covering all aspects of crystallography, three award lectures, three microsymposia, a special meeting promoting gender equality, a luncheon seminar and an industrial exhibition featuring 10 companies/organizations. The microsymposia were titled “Local structure study by pair distribution function analysis applied to crystalline materials”, “Crystal structures ranging from molecules to 3D networks based on metal elements and development of functional materials” and “Protein crystallography for progress in life science” and a total of 13 invited talks were given.

At the CrSJ Awards Ceremony, the S. Nishikawa Award was given to Kunio Takayanagi (Tokyo Institute of Technology) for his research “Pioneering study on the correlation between surface nanostructure and quantum conductivity by electron crystallography”, the Research Award went to Kunio Hirata (RIKEN) for his research “Development of a rapid automatic data collection system ZOO for structure determination from protein microcrystals” and the Young Crystallographer Award went to Wataru Shihoya (University of Tokyo) for his research “Crystal structures of the human endothelin ETB receptor providing the receptor activation mechanism”.



The recipients of the 2019 CrSJ Awards with the President of CrSJ. From left: K. Hirata, Y. Sugawara (President), K. Takayanagi and W. Shihoya.

Four young scientists were honored with Poster Awards: S. Takahashi (University of Tsukuba), H. Iida (Hyogo Prefecture University), K. Oda (University of Tokyo) and K. Hirose (Yokohama City University).



The Poster Awardees and the President of CrSJ. From left: K. Hirose, K. Oda, Y. Sugawara (President), H. Iida and S. Takahashi.

The Young Crystallographer's Meeting 2019, a satellite meeting of CrSJ2019, was held on 18 November at the satellite plaza of Kanazawa University close to the venue of CrSJ2019, and 38 researchers/students took part. This meeting is held every year to give an opportunity for young researchers and graduate students to communicate and network with each other, especially beyond the barriers between physics, chemistry and biology, in an informal environment. This year, four lectures by K. Hiroi (NIMS), Y. Terasawa (Waseda University), K. Ishida (Gakushuin University) and D. Urushihara (Nagoya Institute of Technology) were given and a banquet was held after the lectures.

The full program of the meeting can be viewed at the [CrSJ website](#). The chair of the organization committee was M. Okuno (Kanazawa University) and the program committee was chaired by H. Okudera (Kanazawa University).

The 70th anniversary annual meeting will be held during 27–29 November 2020 at the Tsukuba International Congress Center in Tsukuba, Japan. The chair of the organization committee is E. Nishibori (University Tsukuba) and the program committee is chaired by S. Adachi (KEK).

MEETING REPORT

Sixth joint Diamond-CCP4 MX workshop

David Waterman



Hazel Aitkenhead (right; University of Oxford, UK) receives her IUCr Journals Poster Prize from speaker Kevin Cowtan (centre) and David Waterman, the main organiser from CCP4.

The sixth joint Diamond-CCP4 Data Collection and Structure Solution Workshop was held from 1 to 8 December 2019 at Diamond Light Source, Oxfordshire, UK. As in previous years, there were more than four times as many applications as places on the workshop, demonstrating the high demand for a course of this type. Twenty graduate students, postdocs and other early career scientists had the opportunity to follow the full crystallographic structure solution pathway, guided by the expert scientists who design the experiments, run the beamlines and write the software. This year we were also joined by three additional students funded by the [INSTRUCT-ERIC OPEN SESAME](#) project. All participants had the opportunity to collect data on their own crystals at Diamond's MX beamlines. Progress was made on many projects, resulting in a few new structure solutions.

The diversity and strength of scientific projects brought to the workshop was clearly evident from the IUCr Journals Poster Competition. Choosing a winner of this competition is always a great challenge for the tutors! Discussion between the panel took place over multiple days until eventually Hazel Aitkenhead from the University of Oxford, UK, was chosen for her poster "Pestivirus membrane fusion – The Receptor".

The 2020 event will take place again in December at Diamond Light Source, with details to be confirmed [here](#) in due course.

MEETING REPORT

Joint Polish-German Crystallographic Meeting 2020

Maria Rutkiewicz



Rolf Hilgenfeld during his plenary talk on how crystallography can fight emerging viruses such as SARS, MERS and COVID-19.

The German Crystallographic Society (DGK) and the Polish Crystallographic Association (PCA) both have long-standing traditions of organizing annual events. However, this year a Joint Polish-German Crystallographic Meeting took place for the first time, gathering over 320 participants in Wrocław, Poland, from 24 to 27 February 2020. The conference chairs, Marek Główka (Łódź, Poland), Marek Wołczyr (Wrocław, Poland), Susan Schorr (Berlin, Germany) and Udo Heinemann (Berlin, Germany), filled a four-day program with great talks and posters. Multiple scientific sessions were interspersed with plenary talks, which – judging by the attendance – were found interesting by crystallographers from very different disciplines.



Conference chairs and organizing committee members during the welcome session of the Joint Polish-German Crystallographic Meeting 2020.

A measure of an extraordinary plenary talk is whether it can capture the undivided attention of an audience with varying backgrounds and interests. And this is exactly what happened when Rolf Hilgenfeld (Lübeck, Germany) presented his lecture on SARS and MERS viruses with a special focus on the recently emerged SARS-CoV-2 (formerly known as 2019-nCoV). The audience's interest was initially driven by the timeliness of the topic. However, the story of solving the crystal structures of the SARS-CoV-2 main protease and its potential inhibitors, already tested on cell lines, within just days of the publication of the virus genome was captivating in itself. [The Lübeck team's research has now been published in *Science*.]

Traditionally, the first day of the meeting was an opportunity for the DGK award winners to be announced. Dieter Fenske (Karlsruhe, Germany) received the Carl Hermann Medal to acknowledge his life-time achievements, especially for his research in inorganic chemistry and crystallography. The Will Kleber commemorative coin for outstanding scientific contributions went to Robert Dinnebier (Stuttgart, Germany) in recognition of his exceptional achievements in the field of powder diffraction, as well as in the training of young scientists and in the service to the research community.

This year, the Max von Laue Prize for outstanding achievements by young scientists was awarded twice for the third time in its history. Tobias Beck (Hamburg, Germany) was recognized for his work on the development and characterization of novel crystalline bio-hybrid materials, which are composed of combinations of protein container building blocks and inorganic nanoparticles. In addition, Matthias Zschornak (Dresden, Germany) was recognized for his methodical development of resonant X-ray scattering to make orbitals visible and his work on defect migration and separation in oxides under the influence of external fields. Finally, the Waltrude and Friedrich Liebau Prize promoting interdisciplinarity of crystallography was awarded to Julian Voss-Andreae (Portland, OR, USA) for his significant artistic and creative work.

The organizers provided an opportunity for 19 young crystallographers to present their work in the form of lightning talks. The jury decided to award three of them with Best Presentation Awards: Elżbieta Wątor (Kraków, Poland), Bipasha Debnatyh (Dresden, Germany) and Zhenyu Wang (Berlin, Germany).



The laureates of the Best Presentation Awards, Zhenyu Wang, Bipasha Debnatyh and Elżbieta Wątor, with the jury members.

After two sessions with 128 poster presentations, three scientists were honored with Poster Awards: Götz Schuck (Berlin, Germany), Katarzyna Kurpiewska (Kraków, Poland) and Yoshimichi Hagiwara (Kyoto, Japan).

The city of Wrocław, which is a traditional location for Polish crystallography meetings, was a beautiful venue for scientific discussions that lasted long into the night. The conference dinner, organized at the amazing Afrykarium of Wrocław's zoo, was a perfect ending to the third day of exciting talks and scientific discussions.



Polish and German participants sightseeing after the poster session.

The joint conference – an initiative bringing together crystallographers from two neighboring countries – created a very welcome platform for promoting current research and starting new collaborations.

OUTREACH

Winners of the 2019 IUCr Crystal Growing Competition



Crystalline versions of the Argentinian national flower, the ceibo (*Erythrina crista-galli*), grown by pupils from the Delfín Gallo School in Buenos Aires, impressed the judges of the 2019 IUCr Crystal growing competition.

Continuing the success of the crystal growing competition that launched during IYCr2014, we are delighted to announce the winners of the 2019 IUCr Crystal Growing Competition.

Schoolchildren were invited to convey their experiences of growing crystals through a video. The entries were judged on their creativity, aesthetic value, description of the working plan and experimental work, clarity of explanation, scientific background, and safety by an international panel. The winners, who will receive a certificate and medal, are as follows:

Category: 15-18 - GOLD: IES Sulayr, Pitres-La Taha, Granada (Spain); SILVER: Wellspring High School, Hanoi (Vietnam); BRONZE: Xuan Hoa High School, Vĩnh Phúc (Vietnam)

Category: 11-15 - GOLD: IES Antonio de Mendoza, Alcalá la Real, Jaén (Spain); SILVER: Colegio Diocesano de San Ildefonso, Almería (Spain); BRONZE: Wellspring Secondary School, Hanoi (Vietnam)

Category: under 11 - GOLD: Escuela nro. 1 DE 16 "Delfín Gallo", Buenos Aires (Argentina)

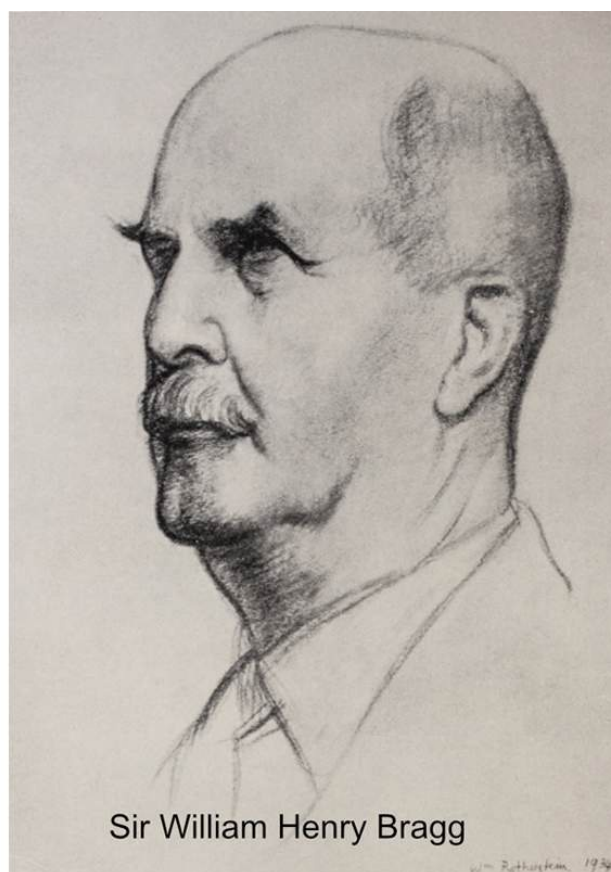
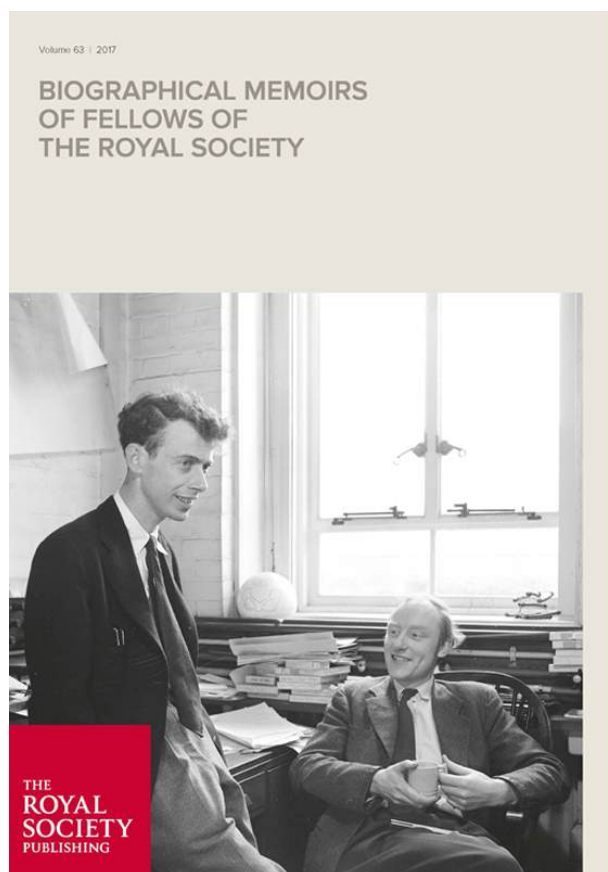
and the winning videos can be watched [here](#).



HISTORY OF CRYSTALLOGRAPHY

Royal Society Biographical Memoirs - now free to read online!

Mike GlazerHelen Eaton



The Royal Society is a learned society and the UK's national academy of sciences. Founded on 28 November 1660, it was granted a royal charter by King Charles II, and is the oldest national scientific institution in the world. The society [fulfills many roles](#): promoting science and its benefits, recognising excellence in science, supporting outstanding science, providing scientific advice for policy, fostering international and global co-operation, education and public engagement. It also performs these roles for the smaller countries of the [Commonwealth](#).

For many years the Royal Society has been publishing extended obituaries of its Fellows and Foreign Members. The *Biographical Memoirs* are recognised as definitive accounts of the life and work of these eminent scientists, providing a valuable resource for both scientists and historians of science. Until recently, some of these memoirs were available to subscribers only, but they are now all [free to read online](#).

Each memoir is carefully researched and creatively written, usually by a close colleague or research collaborator. The main focus is the science and scientific endeavour, but the memoirs also offer a fascinating insight into the character and personalities of the individuals involved. Readers can discover how the science was achieved within the historical context, and follow the development of specific scientific disciplines and fields of research.

Many of the great scientists of interest to crystallographers have been celebrated within the publication. Some crystallographers have been Presidents of the Royal Society; indeed, the current president, Venki Ramakrishnan, is well known to us.

Do visit the publication's website to search for other scientists or subject areas.

Below is a list of those whom we believe will be of interest to the crystallography community. We are sure that many will be instantaneously recognised, but others will probably be less well known to you.

[1] For those of you interested in the history of crystallography, the Royal Society's Biographical Memoirs collection is a rich and unique treasure trove.

 Ulrich Wolfgang Arndt 1924–2006	 Roger Arthur Cowley 1939–2015	 Henry Solomon Lipson 1910–1991
 Struther Arnott 1934–2013	 Ernest Gordon Cox 1906–1996	 Kathleen Lonsdale 1903–1971
 William Astbury 1898–1961	 Francis Harry Compton Crick 1916–2004	 Henry Alexander Miers 1858–1942
 Charles Glover Barkla 1877–1944	 Durward W. J. Cruickshank 1924–2007	 Frank Reginald Nunes Nabarro 1916–2006

William **Barlow**

1845–1934

Charles Galton
Darwin

1887–1962

Donald William **Pashley**

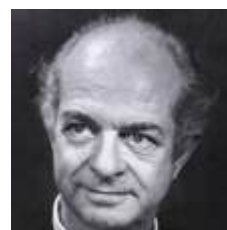
1927–2009

John Desmond **Bernal**

1901–1971

Guy **Dodson**

1937–2012

Linus Carl **Pauling**

1901–1994

Johannes Martin **Bijvoet**

1892–1980

Paul Peter **Ewald**

1888–1985

Max Ferdinand **Perutz**

1914–2002

David Mervyn **Blow**

1931–2004

John Thomas **Finch**

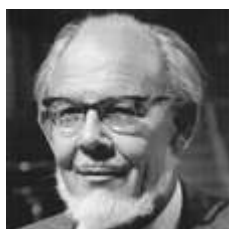
1930–2017

David Chilton **Phillips**

1924–1999

Albert James **Bradley**

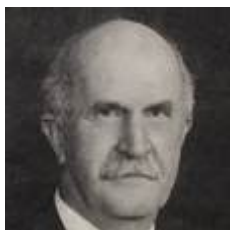
1899–1972

Frederick Charles
Frank

1911–1998

William Jackson **Pope**

1870–1939



William Henry
Bragg

1862–1942



Jacques **Friedel**

1921–2014



Herbert Marcus **Powell**

1906–1991



William Lawrence
Bragg

1890–1971



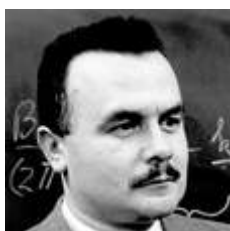
Victor Moritz
Goldschmidt

1888–1947



Gopalasamudram Narayana **Ramachandran**

1922–2001



Bertram Neville **Brockhouse**

1918–2003



Dorothy Mary Crowfoot **Hodgkin**

1910–1994



Chandrasekhara Venkata **Raman**

1888–1970



Maurice
de Broglie

1875–1960



William
Hume-Rothery

1899–1968



John Turton **Randall**

1905–1984



Charles William
Bunn

1905–1990



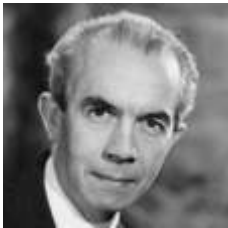
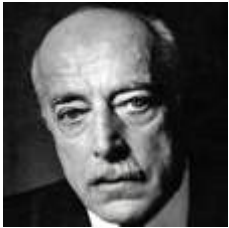
Reginald William
James

1891–1964



John Monteath **Robertson**

1900–1989

 Sivaramakrishna Chandrasekhar 1930–2004	 John Cowdery Kendrew 1917–1997	 Geoffrey Ingram Taylor 1886–1975
 John Wyrill Christian 1926–2001	 Aaron Klug 1926–2018	 George Paget Thomson 1892–1975
 William Cochran 1922–2003	 Andrew Richard Lang 1924–2008	 Maurice Hugh Frederick Wilkins 1916–2004
 John Maxwell Cowley 1923–2004	 Max Ferdinand von Laue 1879–1960	

[1] It is worth mentioning that the American Crystallographic Association has an excellent [website](#) dealing with crystallographers in history (including some who are still alive!). See also the IUCr's [Biographical notes on individual crystallographers](#).

Mike Glazer is Emeritus Professor of Physics at the University of Oxford, UK, and Editor of the IUCr Newsletter. Helen Eaton is Senior Commissioning Editor of the Royal Society's Biographical Memoirs, London, UK.

AWARDS AND PRIZES

Mitchell Guss honoured in Australia Day 2020 Honours List



We send our congratulations to Mitchell Guss, Emeritus Professor at the University of Sydney and former member of the IUCr Executive Committee (2011–2017), who has been made an Officer (AO) in the General Division of the Order of Australia *for distinguished service to education and scientific research in the field of molecular bioscience, and to professional organisations*. Professor Guss is a pioneer of protein crystallography in Australia and his research has focused on the way in which the versatile properties of metal ions are exploited by biological systems. He chaired the IUCr Commission on Biological Macromolecules from 1999 to 2005, during which time the Commission produced “[Guidelines for the deposition and release of macromolecular coordinate and experimental data](#).” To cater for the high-throughput requirements of the structural genomics community, IUCr Journals launched *Acta Cryst. F* in 2005, with Professor Guss as one of the founding Editors.

Retiring from the IUCr Commission on Journals in 2014 after a commendable 14 years' service, he has continued to prepare and promote publication guidelines, especially in connection with [structural modelling of small-angle scattering data from biomolecules in solution](#), and champions the preservation of raw data; for a full list of his articles in IUCr Journals, please see [here](#).

Other eminent positions held include the presidency of the Society of Crystallographers in Australia and New Zealand (1995–1998), receiving their Lawrence Bragg Medal in 2018, and of the Asian Crystallographic Association from 2008 to 2010 (see photo).

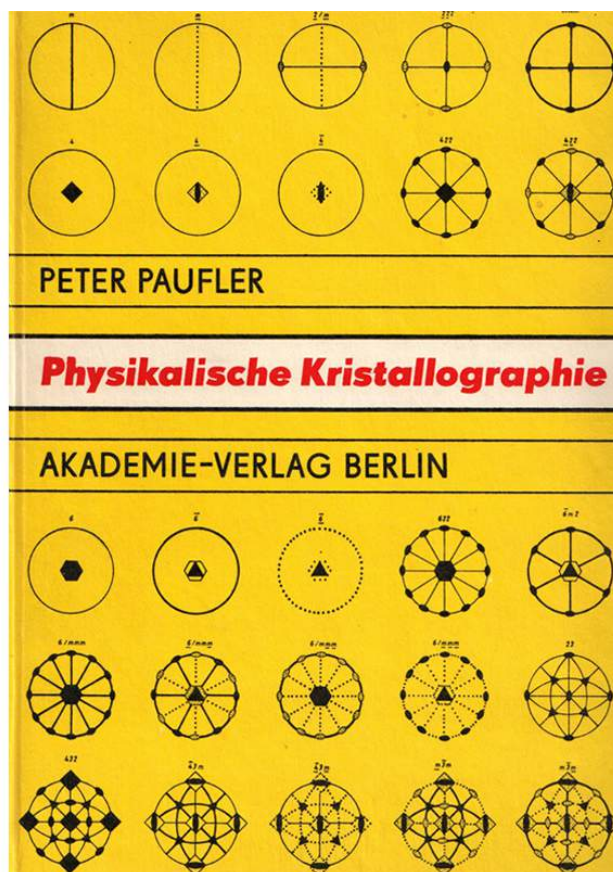
On hearing the news of his award, Professor Guss AO said: “I am truly grateful to be honoured in this way. I have been lucky and privileged to be able to undertake a career in science that has been more like

a hobby to me than a job. Any honours I receive should rightly be shared by the wonderful colleagues and students I have worked with in the past 50 some years.”

NOTICE

On the 80th birthday of Professor (retired) Dr Dr h.c. Peter Paufler

Dirk C. MeyerEkkehart Tillmanns



On the occasion of his 80th birthday on 18 February 2020, the authors would like to congratulate Professor (retired) Dr Dr h.c. Peter Paufler on behalf of numerous colleagues and students and wish him all the best for the future.

Peter Paufler was born in Dresden, where he began his studies of physics at the Technical University in 1958. In his diploma thesis in 1963 he dealt with the growth of single crystals of intermetallic compounds. His doctorate followed in 1967 and his habilitation in 1971. As a co-worker of Gustav E. R. Schulze he made fundamental contributions to our knowledge of the plastic behaviour of the Laves phase MgZn_2 . It was also his mentor and scientific teacher G. E. R. Schulze who shielded Peter Paufler from various politically motivated attacks and thus helped him to enter a university teaching career, which would probably not have been possible under the circumstances at that time.

From 1963 onwards Peter Paufler was a research assistant and senior assistant at the same institute. During this period he also dealt with questions on the systematics of crystal structures of intermediate phases in cooperation with Professor Schulze. At the same time, he worked temporarily at the Nuclear Research Centre in Grenoble and at the Institute of Nuclear Research in Dubna, as well as in 1971/1972 at the Faculty of Physics of Moscow State University. In 1970 Peter Paufler was appointed as a lecturer for experimental physics, and in 1973, after the retirement of Professor Schulze, he took over the leadership

of the teaching team for solid-state physics, which was responsible for the education of all physics students at the TU.

In 1968 Peter Paufler became a member of the Association for Crystallography of the GDR (VFK), in which he soon became actively involved. In 1972 he became chairman of the working group for lattice physics, from 1974 he was a member of the board of the VFK and the National Committee for Crystallography of the GDR, and from 1983 to 1985 he was chairman of the VFK. In 1978 Peter Paufler was appointed to the Chair of Crystallography at the University of Leipzig as successor of H. Neels. This position was connected with the management of the Institute of Crystallography, Mineralogy and Material Sciences. During his work he concentrated especially on the investigation of semiconducting III-V compounds. In 1992 he accepted a call to the Chair of Crystallography at the TU Dresden until he retired in 2006. Apart from his participation in university committees and national and international crystallographic organizations [for instance, he served on several IUCr Commissions: Crystallographic Computing (2002–2008), Crystallographic Teaching (1987–1993) and Mathematical and Theoretical Crystallography (2008–2011)], Peter Paufler has devoted special attention to teaching in both Leipzig and Dresden. His students and those who attended his lectures have always emphasized that he was very successful in this field.

His scientific work comprises more than 200 publications. Peter Paufler is the author of several textbooks, of which the *Physikalische Kristallographie* published in 1986 deserves special mention, a book which has familiarized generations of students with the basics of crystal physics and the physical properties of crystals. He was Book Review Editor for IUCr Journals from 2004 to 2014.

Peter Paufler is a member of a number of scientific associations. He was instrumental in the founding of the German Society for Crystallography (DGK), the successor organization of VFK and the Working Group for Crystallography (AGKr). He was a member of the board and chairman and was appointed honorary member in 2014. In 2006 Peter Paufler became an honorary member of the German Mineralogical Society (DMG), in which he also served as chairman. In the same year the University of Leipzig awarded him an honorary doctorate; in 1993 he was elected as member of the German Academy of Science Leopoldina, and since 1998 he has also been a member of the Saxon Academy of Sciences in Leipzig.

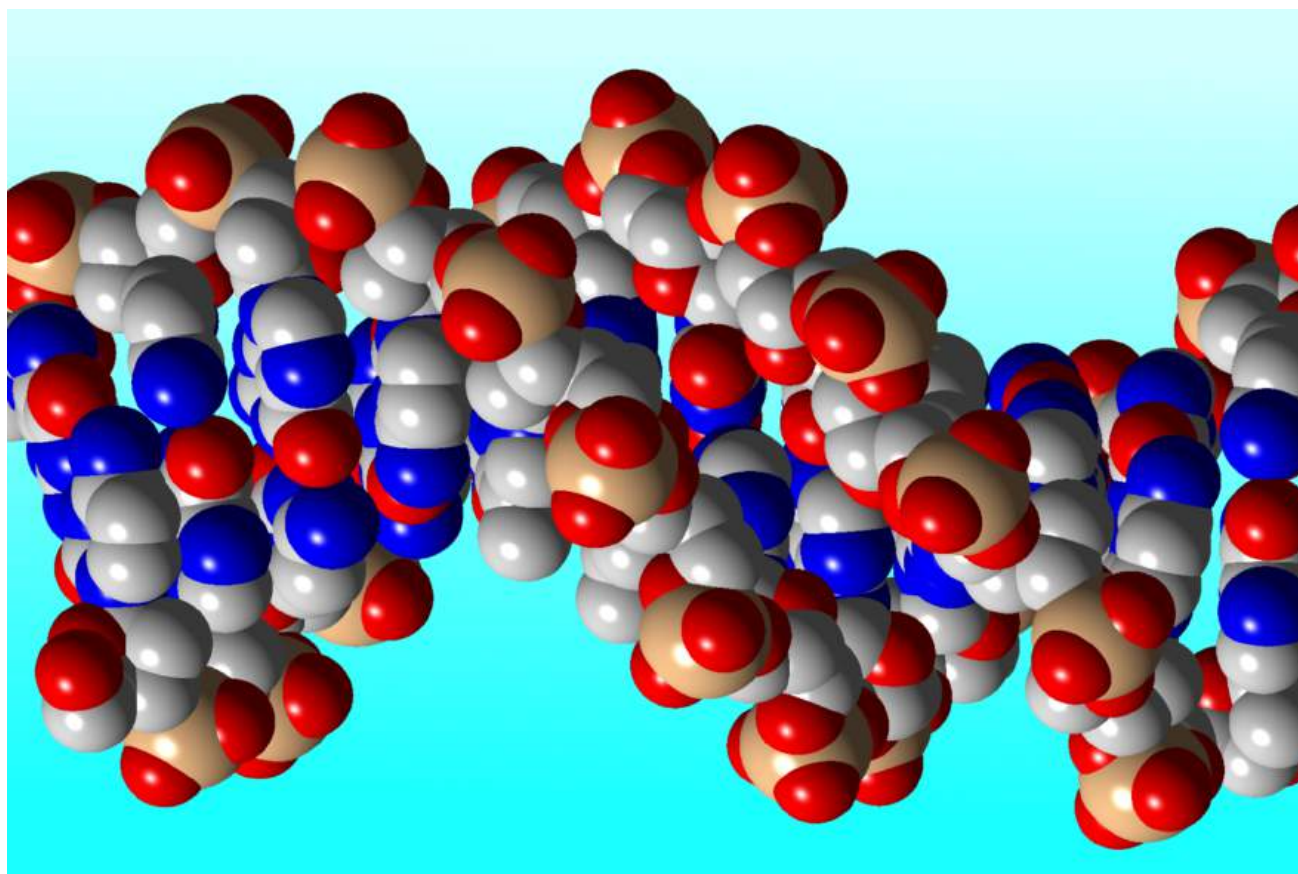
The authors wish Peter Paufler many more fulfilled years in life and science.

This article was originally published in *J. Appl. Cryst.* (2020). **53**, 306–307.

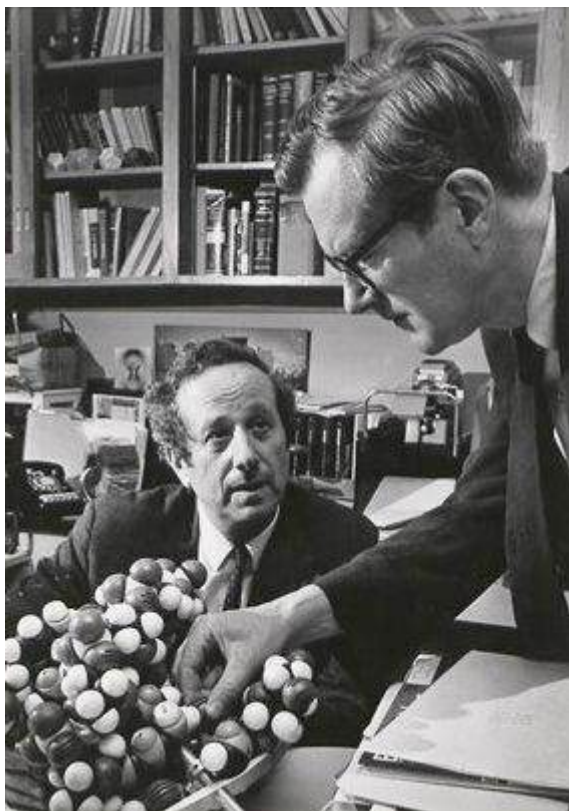
OBITUARY

Leonard Derwent Hamilton (1921–2019)

Watson Fuller



A few days before Christmas 2019, the media were gifted a great story: *The Mill, Pendlebury* by the Manchester artist L. S. Lowry, painted in 1943 but never before on public display, was to be auctioned in the New Year at Christie's with an estimated price of up to GBP 1 million (USD 1.3 million). To add to the interest, the owner had been a scientist involved in the determination of the structure of DNA, the most newsworthy of all biological macromolecules. The scientist was Leonard D. Hamilton, born in Manchester, UK, and educated at Manchester Grammar School and Balliol College, Oxford, UK, where he studied medicine. This iconic painting is said to have been given to him by his parents and was hung on the wall of his college room. He went on to obtain a PhD in Biochemistry at Trinity College, Cambridge, UK, moving to the United States in 1949 to work at the University of Utah, before joining the Sloan-Kettering Institute for Cancer Research in New York in 1950. In 1964 he moved to the Brookhaven National Laboratory as Head of the Division of Microbiology from where he retired in 1994. From 1968 he was also Professor of Medicine at Stony Brook University.

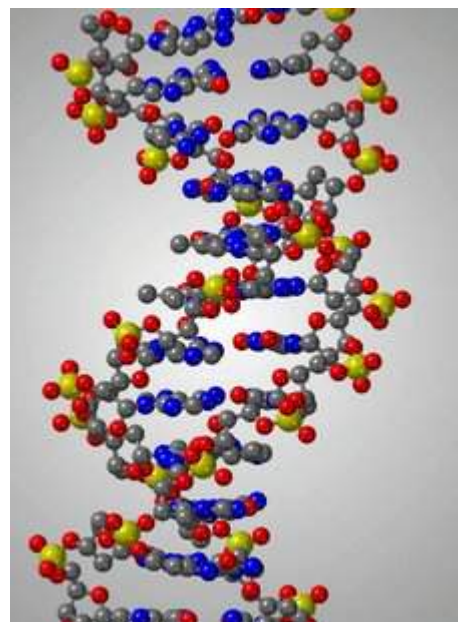


Leonard Hamilton (left) with Maurice Wilkins.

The proposal of the double helical structure for DNA, for which Francis Crick, Jim Watson and Maurice Wilkins shared the 1962 Nobel Prize in Physiology or Medicine, was derived from X-ray fibre diffraction studies at King's College London, UK, initially by Maurice Wilkins, Alec Stokes and Raymond Gosling and subsequently from much improved data by Rosalind Franklin and Raymond Gosling. The latter showed that the molecule was helical with a pitch of about 28 Å and a diameter of about 20 Å. The DNA used in these studies was primarily donated by the Swiss biochemist Rudolf Signer. The model proposed by Crick and Watson not only possessed a compelling beauty but crucially suggested how the genetic information might be coded in the sequence of the base pairs holding the two helical strands together, and how, following strand separation, it might be copied in DNA replication. Crick was a powerful voice in urging that in view of its central biological significance, a structure proposed on the basis of a largely qualitative analysis of the data should be refined to as high a degree as possible. A variety of alternative models had already been proposed, and during the subsequent decade more would follow: there had been proposals for three-stranded structures, for different pairing schemes between the planar heterocyclic bases that formed the steps in the DNA helical staircase, for "side-by-side models" in which the two polynucleotide strands could separate without unravelling the double helix and for left-handed helical variants of the right-handed Watson and Crick model. There had also been suggestions that unlike the B form found in semi-crystalline fibres, the A form in crystalline fibres might not be helical: a matter of some importance when the relevance to the biological environment of structures determined through X-ray crystallography was still being questioned.

If Maurice Wilkins' group was to continue to address these issues, they would require a reliable source of DNA. For this, a collaboration was established with Leonard Hamilton that over some 10 years would provide DNA from a wide range of organisms and tissues and with a variety of alkali metal counter ions. There were frustrations in a collaboration stretching over 3500 miles. Still, fortunately, DNA is a robust molecule that, following re-wetting after its journey by post from New York to London, could be drawn into well-ordered fibres. The quality of the specimens was high and with the development of higher-resolution camera data was routinely recorded to 3 Å and in the meridional direction to ~1 Å. The analysis of such greatly extended data followed the growth in digital computer power in the 1950s and

1960s from half an hour a week in an IBM centre behind Selfridges on the recently developed 360, with its mere 2000 words of memory located on a rotating drum, to long nights on the University of London Ferranti Mercury and finally to the joy of our own departmental Elliot 803. These facilities with the greatly improved characterisation of the material from the Sloan-Kettering laboratory resulted in definitive models for the A and B conformations together with the discovery of a new semi-crystalline C form. The definitive work on the B form rested on the discovery that, in contrast to the semi-crystalline form of the sodium salt of DNA, the lithium salt gave highly crystalline fibres in the presence of a critical amount of excess chloride.



At times, Leonard felt somewhat removed from the action once the DNA samples were in the post, and he was therefore greatly excited by the work of a number of us looking at the effect on diffraction patterns of the binding of various drugs to DNA. This area had been given high visibility by Crick and co-workers using proflavin, which intercalated between successive base pairs to disrupt the reading of the genetic information. From this, it was demonstrated that each amino acid was coded by three successive bases along one strand of the double helix. Leonard suggested that we look at the interaction of daunomycin, an antibiotic being used in the 1960s in cancer therapy. We were able to show that, as revealed by an increase in the average pitch of the helix, the planar group of this antibiotic intercalated into the DNA but – of particular interest – the pitch varied with the water content of the fibre indicating the importance of hydrophobic effects. Leonard also had international distinction on the human and environmental effects of radiation, contributing to the seminal report of the UN Scientific Committee on the Effects of Atomic Radiation and leading a group at Brookhaven on the health effects of various energy sources.

Leonard would have been greatly entertained by the media outpouring over his Lowry and seeing the sale price soar to £2.2 million – for a painting I recall him saying that was bought for £10. His art collection had grown over the years to include works by Picasso, Whistler, Matisse and Bonnard, and latterly was displayed in the modernist house looking out to Long Island Sound, designed for the Hamiltons by Barbara and Julian Neski. Leonard with his many interests and great enthusiasm for life was never blasé and his restless energy meant a visit to him or by him was never dull.

In 1945, Leonard married Ann Twynam Blake, a psychiatric social worker and a New Democratic Coalition activist. Ann died in 1997. He is survived by a daughter, Jane, sons Stephen and Robin and seven grandchildren.

Watson Fuller is Emeritus Professor of Physics at Keele University, UK; watson.fuller@gmail.com.

OBITUARY

Michael Woolfson (1927-2019)

Eleanor Dodson



Photo credit: W. L. Duax.

Professor Michael Woolfson, who died on 23 December 2019, contributed to the theory and practice of X-ray crystallography for over 60 years. He made major contributions towards a general solution of the phase problem for determining crystal structures and provided tools to use the theory of 'direct methods' to enable structures to be deduced directly from the diffraction data. Software developed in his laboratory, such as the computer package *MULTAN*, was responsible for about half of the structures determined around the world during the 1970s and 1980s.

Michael passed the 11+ exam in 1939, the only boy from his primary school to do so. When war was declared the school was evacuated from inner London, and Michael finally finished his secondary schooling in Wellingborough. He credited this opportunity with giving him an important skill – he learnt to play rugby – and claimed that some later opportunities were awarded to him, not for academic excellence, but because he would make 'a useful centre forward'. He won a place at Jesus College, Oxford, being awarded a degree in Physics in 1947. He then was conscripted for two years into the army and finished up serving in the Royal Engineers Survey Establishment. His interest in science was rekindled when thinking about mapping techniques, so when demobbed he applied to and was accepted as a research student by Henry Lipson at the University of Manchester Institute of Science and Technology (UMIST). His project was to build an optical instrument to simulate X-ray diffraction, and also to solve a crystal structure. The optical instrument was essentially a vast exercise in assembling Meccano parts, and proceeded slowly; the crystal structure intensity distribution was unusual, and Michael was quickly able to prove that this was due to the intrinsic inner symmetry of the molecule and

that the intensity distribution was *hypercentric*. He took the message to heart that it was much more satisfying to be a theoretician. Ideas about using sign relationships to solve the phase problem for centrosymmetric structures were circulating in the crystallographic journals and Michael was hooked. (Already in the 1952 volume of *Acta Crystallographica* there were three papers by Sayre, Cochran and Zachariasen showing that for strong intensities the product of the signs of 'reflection triples' **h**, **k**, **h + k** was probably 1.)

After completing his PhD, Michael went to work in Cambridge with Cochran, and continued exploring phase relationships whilst also solving structures. In 1955 he was invited to apply for a lectureship back with Lipson in UMIST, a position which gave him the long-term security needed to pursue his own research interests, and to find that he really enjoyed teaching. Cochran wrote sadly to say he was missed in Cambridge – 'Now I have no-one to talk to ...', and Michael replied, 'Me neither.' However, he rapidly recruited colleagues and students of his own.

The formalism for phase relationships was being unravelled, but applying the methods was a painfully slow task without the aid of computers. In 1960 Michael was fortunate to be allocated Peter Main as a research student, and together with Gabriel Germain they began to develop software to automate structure solution.

In 1965 Michael was appointed Professor of Theoretical Physics in the newly founded University of York, and that became his home until his retirement and beyond. He continued his crystallographic research, as well as taking responsibility for the astronomy teaching and research in the department, contributing numerous many stimulating ideas on the origin and dynamical evolution of the Solar System. He was able to appoint Peter Main to a lectureship in 1967 and established a world-class international research group. The software they developed was comprehensive, functional for all space groups and this made it relatively easy to solve larger and larger structures, providing atomic resolution data were available. It was also designed in a modular form, which meant it was relatively easy to modify the software to incorporate new ideas. New ways to assign the initial phase sets were explored, with a particular focus on finding optimal figures of merit to select the best solution from a given set of calculations.

From 1970 to 1980 Michael directed six direct-methods schools across Europe, attended by many young scientists who subsequently became leading crystallographic theoreticians. He also contributed to several volumes of the series *Computational Crystallography*, where papers given at International Summer Schools organized by the IUCr were published. It is interesting to look back at these; many complex ideas are described (often in extremely complex ways!), but Michael's contributions are models of clarity.

By the 1980s he was extending his interests to protein crystallography and working with scientists from the Institute of Physics, Chinese Academy of Sciences, Beijing. Synchrotrons were extending the available resolution for protein data sets, and there were techniques for estimating initial phase sets from heavy-atom positions or anomalous-scattering measurements, so methods combining experiment and theory were being developed.

After his retirement in 1994 Michael continued to teach, and to carry on with research. One particularly powerful idea he pursued is coded into the program *ACORN* (named for the proverb 'Mighty oaks from little acorns grow'). This uses the fact that if near-atomic-resolution data are available, and this data set is enhanced to 1 Å simply by adding *E* values with the expected mean intensity for that resolution range (viz, 1), then density modification of a map phased only from a correctly placed small fragment, such as a few heavy atoms, the sulfur sites or a helix, can quickly generate an extremely accurate set of phases for all reflections. *ACORN* incorporates a robust figure of merit, estimated using observations excluded from the modified maps.

Michael was awarded many honours: he was elected as a fellow of the Royal Society in 1984 and received the Royal Society Hughes Medal in 1986; he was given the Patterson Award from the American

Crystallographic Association in 1990 and was thrilled to receive the Gregori Aminoff Prize from the Royal Swedish Academy of Sciences in 1992. Michael was also awarded the Dorothy Hodgkin Prize by the British Crystallographic Association in 1997 and the Ewald Prize by the IUCr in 2002.

As well as being a prolific author, Michael was Book-Review Editor for *Acta Crystallographica* Sections A and B and the *Journal of Applied Crystallography* from 1968 to 1975, and a Co-editor for *Acta Crystallographica* Sections A and B from 1977 to 1980. He was a member of the IUCr Executive Committee from 1981 to 1984, during which he served as the first Convener of the IUCr Finance Committee. He was also a member of the IUCr Commission on Crystallographic Computing from 1972 to 1975.

He will be remembered for his innovative science, for the enthusiasm he brought to the subject, for the quality of his teaching, and for his interest in and kindness to his many colleagues and students.

This article was originally published in [Acta Cryst. \(2020\). A76, 263–264](#).

OBITUARY

Mark A. Beno (1951-2019)

Uta RuettLynda Soderholm



Mark Beno, an internationally recognized crystallographer and Deputy to the Division Director at the Advanced Photon Source, Argonne National Laboratory, USA, passed away suddenly on 24 August 2019 (<https://bit.ly/359iWmt>).

Mark received a BS in chemistry and mathematics from Marquette University in 1973 and a PhD in physical chemistry from Ohio State University in 1979. He was trained as a classical crystallographer with a deep understanding of the theory and practice of structure determination. He joined the Argonne National Laboratory as a postdoctoral fellow and was promoted to staff scientist in the Chemistry Division in 1982.

His crystallographic knowledge was put to work early in his tenure in the Chemistry Division, when he joined a group focused on the rational design of synthetic metal conductors, specifically donor-acceptor, molecular organic superconductors. His insight into organic metals was quickly tapped in 1987 when high-temperature superconductivity became an international research phenomenon. Within this intensely competitive environment, Mark was the first to solve the crystal structure of $\text{YBa}_2\text{Cu}_3\text{O}_7$, representative of the “123” superconductor series, publishing the results in a seminal paper in 1989. His findings were all-important to the understanding of the types of structures likely to form this unusual class of materials.

Mark’s extensive knowledge of both the theoretical and experimental aspects of X-ray scattering was tapped once again when in 1987, he was asked to move to the Synchrotron Radiation Science Group in

the Materials Science Division and join them in the design and construction of Basic Energy Sciences Synchrotron Radiation Center (BESSRC) beamlines at Sector 11 and 12 at the nascent Advanced Photon Source. He subsequently became the BESSRC Group Leader and moved in 2003, with his group, to what is now the X-ray Science Division (XSD). Mark was named the Deputy Division Director for the XSD in 2005, and held this and a number of other leadership roles within XSD, including serving twice as Interim Division Director. Most recently, he held the position as an Associate Division Director for beamline operations.

Mark's deep love for science kept him at the forefront of emerging fields in a broad area of research. He used his background as a chemist and a crystallographer, as well as his management position at a top-tier synchrotron facility, in an unceasing effort to educate those around him on opportunities available at the APS to advance their own projects. He always believed in the incredible opportunities provided by synchrotrons for scientists to attack grand challenges, and he used his multidisciplinary background to mediate tirelessly between scientists, especially chemists, wishing to use the beamlines, and the physicists developing them.

Mark loved people and always worked to make the staff feel recognized and appreciated. He mentored many early-career scientists to find their own way in science. He encouraged people to watch for opportunities and inspired them to use the resources available to create cutting-edge experiments.

In the last years, the APS upgrade was Mark's main focus. The success of this project guarantees the APS will remain a world-leading facility and the flagship for science in the US. He felt that he could prepare for retirement and leave the actual upgrade to the next generation of scientists, but he survived the project approval by only three weeks.

Mark's passing has left a big hole in the organization and he is dearly missed. At events, Mark preferred to stay in the background, happily smiling and taking pride in the staff. His humble kindness and loyalty created a highly respectful culture at work. We have lost a highly gifted scientist and great supporter of synchrotron-based science.

See a list of papers published by Mark in IUCr journals [here](#).

IUCr SUPPORTED EVENTS

IUCr supported meetings

During this period of meeting cancellations and postponements because of international health concerns, the IUCr will make best efforts to indicate the status of advertised meetings whenever we receive updates. Please refer in all cases to any website maintained by the meeting organizers for definitive information.

These forthcoming meetings are supported by grants from the IUCr Calendar Committee.



daba
11 – 16 October 2026

**11th Oct 2026 - 16th
Oct 2026**
Indaba10

Skukuza, Kruger National
Park, South Africa



5th Dec 2026 - 10th Dec 2026

African Materials Research Society (AMRS 2026)

Nairobi, Kenya

MEETINGS CALENDAR

Meetings calendar

During this period of meeting cancellations and postponements because of international health concerns, the IUCr will make best efforts to indicate the status of advertised meetings whenever we receive updates. Please refer in all cases to any website maintained by the meeting organizers for definitive information.

This is a list of meetings of interest to crystallographers occurring within the next twelve months, as recorded in the IUCr Calendar of Events. Please let us know of any that are missing by [completing this form](#) or sending an email to forthcoming.meetings@iucr.org.

	Indaba10 South Africa 11th Oct 2026 - 16th Oct 2026
	Synchrotron Radiation: Science, Knowledge, Innovations, Future (SKIF-2026) Russian Federation 18th Oct 2026 - 23rd Oct 2026
	CCP-EM/Diamond Icknield Workshop United Kingdom 9th Nov 2026 - 13th Nov 2026
	Instruct-ERIC hands-on workshop on Advanced cryo-FIB-SEM methods Germany 9th Nov 2026 - 13th Nov 2026
	Crystal36-AXAA 2026 Meeting Australia 26th Nov 2026 - 2nd Dec 2026
	African Materials Research Society (AMRS 2026) Kenya 5th Dec 2026 - 10th Dec 2026
	Joint Polish-German Crystallographic Meeting 2027 Germany 15th Feb 2027 - 18th Feb 2027