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LETTER FROM THE PRESIDENT

Slow recovery but still recovery



On a visit to the IUCr offices in Chester, UK, in May 2022.

Hanna Dąbkowska

I do not think that all our worries and problems are over but many things are moving in the right direction for the IUCr community. COVID-19 is not over yet – it probably never will be – but we are less terrified of it, and some of us are heading for in-person meetings. We have learned how to handle Zoom and hybrid meetings – we will probably have to live with them forever. The IUCr Regional Associates are all planning in-person meetings – all following local health rules and common sense. This summer I hope to meet some of you at ACA2022 in Portland, OR, USA, or at ECM33 in Versailles, France. Masks are still very much recommended even though many colleagues are double- and triple-vaccinated and some have already recovered from COVID-19.

CEO not ExecSec

In May 2022, I visited the Chester offices in person to help to appoint our new Chief Executive Officer (CEO). As many of you know, Dr Alex Ashcroft, who has served in the role of IUCr Executive Secretary (ExecSec) since the Hyderabad Congress, has decided to retire in August 2022. Alex has done an excellent job for the IUCr community and for the Chester Office. I respect his decision to leave and I wish him a happy and healthy retirement.

Now it is my great pleasure and honour to confirm that Dr Alex Stanley has accepted the position of the IUCr ExecSec/CEO, as offered to her by the General Secretary and Treasurer, Professor Bo Brummerstedt Iversen. She is assuming the role of CEO with slightly different duties than those of the ExecSec and she is starting on 18 July 2022.

Alex Stanley holds a PhD in Chemistry and Crystallography from Professor Orpen's Group at the University of Bristol, UK, and she may be known to some of you as, over the years, she has been an active member of the British Crystallographic Association (BCA). She has attended General Assemblies during many IUCr Congresses and now she is coming to the IUCr from her successful career at Rigaku.

Her skill base is broad as she has experience in managing people, projects and budgets, in product marketing, building a brand identity, and in governance at committee and corporate levels. She has a good understanding of many scientific fields related to structural science.

We are very pleased to have Alex join our ranks for the years to come. In the first month, Dr Ashcroft will be helping Dr Stanley to understand the nuances of the ExecSec position. Many of us will have a chance to welcome Alex Stanley and to say goodbye to Alex Ashcroft in person as they both will be present at the IUCr Executive and Finance Committee meetings in Versailles in August. I would like all of you to join

me in welcoming Dr Stanley to her new position.



Retiring Executive Secretary, Alex Ashcroft (left), and incoming CEO, Alex Stanley.

At the Chester Office, arrangements are now in place for hybrid working. Some of the staff are now working full time from Abbey Square and others continue to work from home.

Science in Exile

Tension around the world and especially the destruction happening in Ukraine have a lasting effect on many of our colleagues and friends. The political situation in Europe is not improving. Organizations like Science in Exile make a big effort to help displaced scientists from disadvantaged countries, but much more funding is needed to even temporarily resolve this problem. Science in Exile is an initiative led by The World Academy of Sciences for the advancement of science in developing countries (UNESCO-TWAS), the InterAcademy Partnership (IAP) and the International Science Council (ISC). The IUCr applauds many colleagues from different countries and institutions who provide temporary workplace and support to displaced scientists.

Melbourne Congress 22–29 August 2023

In preparation for the 26th IUCr Congress in Melbourne, the International Program Committee (IPC) meeting took place in two locations: Melbourne and Prague. This innovative but very challenging approach resulted in interesting proposals for the program where there is something appealing for everybody in the area of structural science. The members of the IPC are listed on the Congress website here, where the program will appear as soon as the invited speakers accept. We can expect a very exciting Congress with excellent science in Melbourne in 2023, which happily marks the 75th anniversary of the first IUCr General Assembly and the launch of *Acta Crystallographica*. Expect some celebratory events!



The IPC met online and in person in Melbourne (left) and Prague in April 2022.

Please make plans to attend the Congress in person. We hear that Melbourne is such a beautiful, inviting place. Obviously our plans have to be flexible as many decisions are being taken above our heads. Pandemics happen, countries close their borders, airlines cancel pre-booked flights, unwelcome wars derail our plans, universities and employers dramatically cut travel funding, forcing us to make tough and unpopular decisions. But with some effort we can plan to appear at the IUCr Congress – either in person or on screen – and to publish our newest, most interesting results in IUCr Journals. This will help our community to thrive and allows us all (chemists, physicists, biologists, engineers *etc*.) to be part of the large and important network of structural scientists.

IUCr Journals

Recently, I took part in a virtual Journal Management Board (JMB) meeting. Thanks to very effective work by the whole team, the journals are flourishing despite the pandemic, which is reflected in the increases in their impact factors. The JMB meeting also reported on the development of the IUCr's data-sharing policy, which requires the crystal structure data supporting the results in an article to be peer reviewed and archived either with the IUCr or in an appropriate public repository. More information can be found here.

Crystallography in Africa

The OpenLab in Brazzaville, Republic of Congo, using remote diffraction facilities, co-organized by Professor Claude Lecomte, was a real success. Thanks to efforts like this, even more African crystallographers will be trained soon.

Further outreach

This month sees the launch of two global initiatives of great interest to our community: the UNESCO-approved International Year of Basic Sciences for Sustainable Development 2022 (IYBSSD2022) and, under the IYBSSD's patronage, Mineralogy 2022. The IUCr is proud to be one of the Founding Unions, and the IUCr Executive Outreach Officer, Professor Michele Zema, is on the Steering Committee. Read more here.

IUCr Prizes

At this time, one year before the Congress, I encourage everybody to nominate colleagues and peers for IUCr Prizes. It is the most important recognition of personal scientific achievements and now we have tools to handle this recognition at different academic levels. These prizes signal to the world the importance of the work being done by the crystallographic community.

Ewald Prize

The thirteenth Ewald Prize for outstanding contributions to the science of crystallography will be presented at the Melbourne Congress. Nomination forms and more information is available here.

W. H. & W. L. Bragg Prize

The second W. H. & W. L. Bragg Prize, to be awarded to up to two promising early-career crystallographers, will also be presented in Melbourne. Nomination forms and more information can be found here.

Struchkov Prize

The Struchkov Prize will be awarded for the first time to the IUCr community at the Melbourne Congress. A call for nominations for this Prize, which is aimed at young researchers working in the field of small-molecule crystallography, is also open; more information can be found here.

My congratulations to the 2022 Guinier Prize winner, Professor Jill Trewhella (University of Sydney, Australia), in recognition of her outstanding contribution to the field of small-angle scattering (SAS).

One thing I learned recently is the value of being not only respectful of other people's decisions but also grateful. I am very grateful to Professor Mike Glazer and the *IUCr Newsletter* team for being very open-minded and for encouraging discussions. As long as we all agree to disagree we are safe. The discussions can be and are tough but at least they do not lead to destruction and open conflicts.

I wish you all a safe and relaxed summer. We all need a bit of a break from everyday routine and a slowing down of our busy lives. Please stay safe.

EDITORIAL

Editorial



Mike Glazer

Now that summer is in progress here in Europe, thoughts naturally turn to holidays and relaxing in the fine weather. But this summer is very different from before, notably because of the terrible war acting out in Ukraine as I write. Sometimes it isn't easy to think of normal life while that tragedy is happening not so far away. But Crystallography goes on.

So, in this issue, we have several interesting articles. I am always keen to publish accounts of important crystallographers who have largely been forgotten, and I think they deserve some recognition for their contributions to the science we love. For example, the Hargittais, with whom readers will undoubtedly have become familiar, have given us a contribution to remind us of Ernest Wollan, one of the early pioneers of neutron diffraction. Similarly, Larissa Zasurskaya has written a fascinating account of the life of Georg Wulff. Who you may ask? Well, he is someone largely forgotten but nonetheless was an important and talented crystallographer. Some of you may be aware of his famous net for use in stereographic projections. He was a contemporary of W. L. Bragg and even discovered the same law about the same time. Sometimes the law is even known as the Wulff–Bragg Law. Wulff was the first person to introduce X-ray diffraction into Russia.

In the last issue of the *IUCr Newsletter*, I wrote about a photograph of Victor Mordechai Goldschmidt that had come into my possession (probably from Helen Megaw) just before I moved from Cambridge to Oxford in 1976. The photograph referred to a Miss Maurice visiting Heidelberg. Now, when I looked into this, I discovered that an unusual person called Monica Maurice had been in Germany at the time of the photograph and was a frequent visitor. So I mistakenly assumed that the visitor to Heidelberg was Monica. I asked if anyone had any further information about this, and I have received a letter from Stefan Dietrich in Heidelberg, who is an expert on the life and work of Goldschmidt. It seems that the Miss Monica referred to in the photograph was more likely to be Margaret Maurice (no relation as far as I know), a visiting student with Goldschmidt at the time. Margaret was a student at Girton College Cambridge at the same time as Helen Megaw, and this makes the crystallographic connection. So I think we can assume that she was the person mentioned in the photograph. However, there are odd coincidences. First of all, both Maurices were travelling in Germany at the same time, and, furthermore, both Monica and Helen were on one of the secret BIOS (British Intelligence Objectives Sub-Committee) missions to German laboratories after the war. By the way, Goldschmidt was a fascinating person not just as a scientist but also because of his wide-ranging interest in the fine arts. The book by Stefan Dietrich is well worth a read and can be found at https://doi.org/10.17885/heiup.767
or https://doi.org/10.17885/heiup.767?lang=en

It is such a pleasure to see that Crystallography is booming in Africa, a continent for so many years overlooked. You can read about one of the latest OpenLab projects in Brazzaville, Congo. It looks to have

been highly successful, and the organizers are to be congratulated. A fine achievement. In addition, we have report on ePCCr: Pan-African Conference on Crystallography Online, whose theme was ‘Crystallography, an interdisciplinary science for the Africa of the future’.

We also have two contributions to this issue from China, an article about the Big Data Science Center at the Shanghai Synchrotron Radiation Facility and a report on the 2021 Conference of the Chinese Crystallographic Society. The latter was prepared by Professor Nan Zhang from Xi’an Jiao Tong University, and I am pleased to say that she has now agreed to join the regional editorial team for the *Newsletter*, so I hope we shall hear more about Crystallography in China. I recall how in 1978 during the IUCr Congress in Warsaw, China was admitted into the Union for the first time (I think our Union was the first to recognize Chinese scientists). This was not long after the end of the terrible Cultural Revolution and after the visit by Richard Nixon, China was beginning to open to the rest of the world. Several of us were invited by the Chinese delegation to meet outside the Congress Centre and then we were all bussed some distance into the city until we arrived at a huge grey building, the Chinese Embassy. But there was no-one in sight. Eventually, someone suggested that we should go in through the door. Again no-one to meet us. Strange. But then we saw a large staircase and decided maybe we should try climbing to the next floor. And there to greet us was this large contingent of Chinese crystallographers and Embassy personnel. After standing around for some time chatting with them, suddenly a huge wall behind us opened up to reveal a large hall with all kinds of Chinese food being laid out for us. This was truly memorable. There I met the leader of the Chinese delegation, Professor Tang Youqi, and after the Congress I was pleased to welcome him and his delegates to Oxford. China was clearly changing. They in turn invited us to a magnificent Chinese meal in London. You can tell that I love Chinese food!



Tang Youqi and his wife in 2014.

In another informative article, Stephen Neidle has written about the importance of water concerning the human genome. I personally hadn’t realised before that it was such an important component: pretty obvious, I guess in retrospect, as water is such a vital molecule in living organisms. I had never heard

about its role in DNA before, but then biological crystallography is not my field of study.

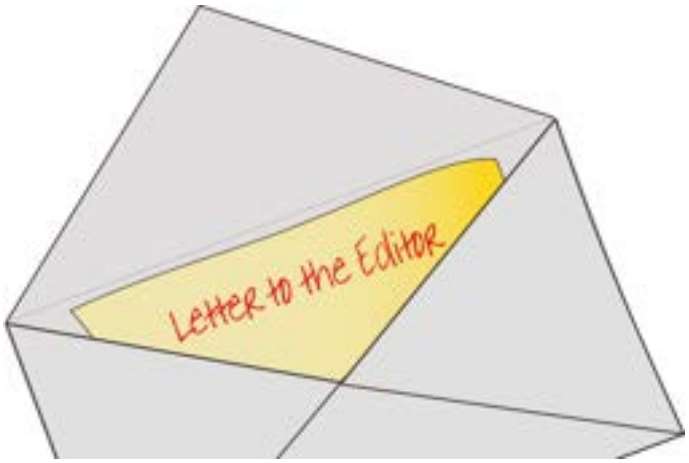
Maureen Julian has written about her time in the laboratory of Kathleen Lonsdale, mentioning most of the characters who were there in the late 1960s. Kathleen Lonsdale is well known to all crystallographers especially. She was one of the two first female Fellows of the Royal Society. As I (together with the late Howard Flack) was her last PhD student, I was personally interested in receiving this article from Maureen. They were exciting times indeed and, for me, reading this article is a trip down memory lane.

You may recall that a short time ago we ran an article on the definition of a crystal. You may be interested in one of the satellite meetings of this year's European Crystallographic Meeting, entitled: Mathematics and Computer Science for Materials Innovation: Crystal Lattice Classifications to be held in Liverpool, UK, from 5 to 9 September. This is organised by Vitaliy Kurlin and Mois Aroyo, see http://kurlin.org/ECM33MACSMIN2022crystal-lattice-classifications.html for more details.

Finally, I am sorry to say that some more of our colleagues have recently passed away. In this issue, there are obituaries for Lev Feigin (Russia), V. M. Vijayan (India) and Jim Ibers (USA).

LETTER TO THE EDITOR

Letter to the Editor



Stefan Dietrich

The other day, an acquaintance sent me your 2022 article on the "Lady of the Lamp", Monica Maurice. Two to three years ago, I had been in search of a Miss Maurice too, and I became all the more interested because your paper includes a photo of Victor Goldschmidt – he was the context for my search for a Miss Maurice. In 2020, my wife and I published a book on the occasion of the centenary of the von Portheim Foundation in Heidelberg, Germany. The Foundation was established and endowed by Victor Goldschmidt in 1919. The Foundation still maintains an Ethnographic Museum in Heidelberg.

At the end of the 1920s, Goldschmidt also set up a grant scheme of the Foundation to enable British post-graduates to carry out research in Heidelberg at whichever institute for one year. The candidates were chosen by the Universities Bureau of the British Empire, which generally administered the scheme. The first candidate for the academic year 1930/31 was Miss Maurice in the field of crystallography (she had the grant for one semester, and this was extended for another semester, the summer semester of 1931). The crystallographic Miss Maurice was, however, a Miss M. E. Maurice. I eventually spotted her in the "Girton College Register 1869–1946" (published in 1948) as Margaret Esther Maurice. Her entry mentions the stay at Heidelberg and that after marriage, she was M. E. Boyland (see below).

MAURICE, MARGARET ESTHER (Mrs BOYLAND): *b.* 14 Nov. 1906, at Colchester; *dau.* of Maj.-Gen. Sir Frederick Maurice, late Principal of Queen Mary Coll., London (see *Who's who*), and Margaret Helen Marsh; *yr. sister* of Joan Violet Maurice (Mrs Robinson) (*q.v.*); *m.* 24 Sept. 1931, Eric Boyland, D.Sc., Ph.D.; 2 *s.*, 1 *dau.* *Educ.* St Paul's Girls' Sch.; Girton 1926–30; Higgens Scholar; Nat. Sci. Trip., Pt I, Cl. II, 1928; Pt II, Cl. I (Mineralogy), 1929; Yarrow Research Student 1929–31; B.A. 1929; Portheim Studentship for Mineralogy, Heidelberg Univ. 1930; Finney Howel Fellowship for Cancer Research 1938–39. Research in Paramagnetism at Davy-Faraday Lab. 1931–32; biochem. research, Research Inst. of Roy. Cancer Hosp. 1932–40; farming 1940–42. *Publications:* (with E. Boyland) 'Studies in tissue metabolism' (*Biochem. J.* 1934–37). *Address:* 1 Woodlands Rd, S.W. 13.

MEGAW, HELEN DICK: See List of Staff 1946.

Concerning her research work after her stay in Heidelberg (1932ff.), she is briefly mentioned in the book *Fifty Years of X-ray Diffraction* (edited by P. P. Ewald, 1962), on p. 419, confirming crystallography as her field of research.

So the picture in your article was a souvenir given to Margaret Maurice when she departed from Heidelberg. A group picture which includes Margaret Maurice and which was taken during her stay in Heidelberg is published in the book on the von Portheim Foundation on p. 32 (reproduced below).



Front, second from right: Margaret E. Maurice. She was the first student to be awarded the von Portheim Foundation Grant for British students, 1930/31. A graduate in mineralogy (BA) from Girton College, Cambridge, she joined Goldschmidt's Crystallographic Institute. The photo was taken on the occasion of an excursion of the institute to Idar-Oberstein in March 1931. (https://heidicon.ub.uni-heidelberg.de/detail/1041998: Universitätsarchiv Heidelberg, Bildarchiv, inv.no. Alb 76 0077).

The same book, p. 31, includes all too brief remarks on the grant scheme for British students. On pp. 90–91 there is a very brief remark on Goldschmidt's contact with W. L. Bragg in the early 1930s. In case you are interested you can download the book (open access) at

https://doi.org/10.17885/heiup.767 or

https://doi.org/10.17885/heiup.767?lang=en

By the way, I am not a crystallographer or natural scientist, my field is ethnology – hence my occasional work at the Ethnographic Museum of the Foundation, and hence my acquaintance with the history of the Foundation (which in Miss Maurice's time still included a Crystallographic Institute).

FEATURE ARTICLE

The laboratory of Dame Kathleen Lonsdale at University College London from 1966 to 1968 and then fifty years later



Figure 1. Chemistry building on Gower Street where Professor Lonsdale's laboratory was located.

Maureen M. Julian

This article takes a snapshot of the laboratory of *Dame Kathleen Lonsdale* at University College London between 1966 and 1968, when I served as a postdoctoral fellow. I arrived, a freshly minted PhD from Cornell University, New York, thrilled to meet *Prof.* (as we then called *Dame Kathleen Lonsdale*), whom I had admired from afar for many years. I arrived with a background in mathematics and crystallography. *Prof.*, with her contribution to both mathematical crystallography (*e.g.* as an author of the *International Tables for X-ray Crystallography*, Lonsdale & Henry, 1952; Lonsdale & Kasper, 1959; Lonsdale *et al.,* 1962) and practical crystallography (*e.g.* proving the planarity of the benzene ring; Lonsdale, 1929), meant I was entering my dream job. Her involvement in the peace movement (Glazer, 2015; Lonsdale, 1942, 1949, 1957) and social justice (*e.g.* prison reform after having spent some time in prison as a pacifist) was equally appealing. Having a successful female role model and working in a research group with future stars, both male and female, was the icing on the cake. During my years as a postdoc in the group, I developed life-long friendships with the lab members. This article concludes with a summary of where these scholars from the late 1960s are fifty years later.

University College London 1966 to 1968: *Prof.*'s last two years before retirement

The physical laboratory and equipment

The laboratory was located in D1, the basement of the stately white-stone Chemistry building on Gower Street, University College London. See Fig. 1. Because of the need for lead shielding, high voltage, water and heavy equipment, crystallography labs were always found in the basement at that time. The X-ray ports had Weissenberg and precession cameras as well as Unicam 3-cm radius cameras for single-crystal rotation and oscillation work. These cameras used photographic film developed in a nearby darkroom. A Ferranti Pegasus Mark II computer contained hundreds of plug-in electronic modules consisting of two or three vacuum tubes each. A convenient supply of replacement vacuum tubes was on hand; it was not uncommon for a vacuum tube to fail, suddenly bringing all calculations to a halt. Binary data were entered by narrow 5-hole paper tape. Unusual for the time, this computer was for the exclusive use of *Prof.*'s lab.

In the corner of the lab was an 1800s electromagnet in a beautiful large wooden case. When we grumbled about our equipment, *Prof.* often pointed out to the group that when she returned to W. H. Bragg's lab after the birth of her third child, there was no diffraction equipment available, and yet she did very well with this abandoned electromagnet. In fact, as a result, she published experimental work on the diamagnetic anisotropy of aliphatic compounds (Lonsdale, 1937). Screwed to the wall was a signed original print of 'Day and Night' given to *Prof.* by M. C. Escher when he spoke on symmetry in his art at the 1960 IUCr Congress.

Faculty

The laboratory was headed by *Prof.*, age 66 at the time, who was born in Newbridge, County Kildare, Ireland, the daughter of a postmaster for the British army. She was raised in England and attended Bedford College, a higher-education college for women. W. H. Bragg was one of her oral examiners for her BSc degree in physics and was so impressed by her that he offered her a position in his laboratory at the Royal Institution, where later she received a DSc. In 1945 she founded her own crystallography group at University College London (Hodgkin, 1975). *H. Judith Milledge (née Grenville-Wells)*, age 39 at the time, experimentalist extraordinaire, was second in command. See Fig. 2. *Judith* was born in South Africa and, after earning a BSc and an MSc from Rhodes University, joined the De Beers Diamond Research Laboratory in Johannesburg. In 1949 she came to University College London as a PhD student to study diamonds with *Prof.* They became good friends and made a wonderful team, perfectly complementing and enhancing each other's research. *D. June Sutor*, age 37 at the time, was born in New Zealand. She earned a PhD from both the University of Auckland and Cambridge University and had done fundamental work in hydrogen bonding (Extance, 2019). She led the body stone group. *Marianne Ehrenberg*, had received her PhD from *Prof.* by solving the crystal structure of an organic molecule showing oestrogenic activity using two-dimensional Patterson and electron-density synthesis (Ehrenberg, 1963). She published the crystal structures of many organic crystals.



Figure 2. *Prof.*, left, and Judith Milledge.

Research

The research foci of the laboratory were divided into three sections: solid-state reactions, diamond inclusions and body stones.

Solid-state reactions

Prof. was interested in geometrical changes in single crystals during phase transitions caused by heat, pressure, time or irradiation. These phase transitions included isomerizations, dimerizations and mixed crystals or solid solutions. For example, she obtained X-ray patterns of crystals of anthracene peroxide, which after a few hours of radiation developed new reflections. After further irradiation, the original X-ray pattern completely disappeared, showing that the original crystal structure had transitioned into a new and different crystal structure (Lonsdale *et al.*, 1966).

After receiving a PhD in physical chemistry from Queen's University in 1966, *Terrance Rummery* studied colorless crystals that turned deep red when melted and then recrystallized into yellow crystals, again indicating a change in structure (Rummery, 1968). See Fig. 3. *Maureen O'Donnell Julian* observed the dimerization of anthracene: transparent crystals of anthracene turned yellow when exposed to UV radiation with wavelengths less than 3000 Å. Using the energy provided by UV, the crystal changed, in part, into new crystals of the dimer (O'Donnell, 1968; Julian, 1972, 1973). The structure of the anthracene dimer, di-*para*-anthracene, was solved by *Marianne Ehrenberg* (Ehrenberg, 1966). PhD student *Mike Glazer*, a Londoner and a graduate of St Andrew's University, Scotland, worked on diffuse scattering in mixed crystals of phenazine and *N*-oxyphenazine. Another PhD student, *Howard Flack,* a graduate of the University of Nottingham, made similar studies on anthrone and anthraquinone (Flack & Glazer, 1970). *Eugene Hoff* had an MD from Columbia University, NY, and worked on rubrene, a red organic semiconductor that is used as the yellow light source in light sticks. See Fig. 4. *Don Emerson* also worked on small organic crystals (Milledge & Harris, 1969; Milledge *et al.*, 1969) and *T. Richard Welberry* developed an interest in modeling cell growth disorder (Welberry & Galbraith, 1973). Another PhD student, *Josh Thomas,* came from Oxford University to UCL with an interest in low-temperature experimentation, and built and tested a helium temperature cryostat for direct measurements of zero-point vibrations in single crystals (Croft & Thomas, 1969). He also was the first to carry out powder Rietveld refinement using X-rays (Malmros & Thomas, 1977).



Figure 3. Terry Rummery.



Figure 4. Eugene Hoff.

Diamond inclusions

Diamonds are borne to Earth's surface in volcanic ultrabasic magmas, principally of kimberlite and much rarer of lamproite. These volcanoes are found in ancient cratonic regions of Earth, such as Africa, Canada and Russia. The inclusions inside diamonds can be used to gain information about the inaccessible mantle. PhD student *Jeff Harris*, a graduate of the University of Liverpool, measured the cell parameters of inclusions both inside the diamond and after their removal from diamond (Harris, 1968). PhD student *Richard Henriques*, from Jamaica, studied the orientations of olivine inclusions in the diamond crystal (Harris *et al.*, 1966).

Body stones

Prof.'s extensive body stone collection, begun with a gift from the urologist D. A. Anderson, contained the two-inch-long bladder stone of Napoleon III, who died in 1873 as a result of an operation to remove it (Lonsdale, 1968). Body stones are found in the urinary tract, the gallbladder and in the salivary glands, and can vary in size from a few millimetres to tens of centimetres. *June Sutor* identified the constituents by grinding them into a powder and using X-rays to identify the compounds (Lonsdale *et al.*, 1968). PhD student *Robin Shirley,* who was born in Harrow (North London) and had a BSc in chemistry from UCL, wrote computer programs for X-ray analysis of these powders (Shirley & Sutor, 1968).

A few stories

Howard Flack would put a mop over his head to make a convincing caricature of *Prof.* and scare the daylights out of us. He did an excellent imitation of her voice. *Judith Milledge* used to treat us individually to a fancy restaurant. I remember her having the chef concoct a special dessert that contained hazelnuts. One year, the group lab outing was a ride on the *Bluebell Railway* steam trains, an 11-mile heritage line in West Sussex. *Maureen O'Donnell Julian* was married at the University of London Chaplaincy at 111 Gower Street on a Wednesday, which *Prof.* declared to be a holiday. *June Sutor* was the bridesmaid, and *Jeff Harris* and *Eric Nave* took the pictures that illustrate this article. See Figs. 5 to 8. Immediately afterwards, as part of *Prof.'s* long-term dedication to advocacy, she visited a women's prison.



Figure 5. Group on left: Mike Glazer, Josh Thomas, Howard Flack and (second row) Richard Henriques. Group on right: My mother, Elizabeth O'Donnell, *Prof.*, *Prof.*'s husband, Tom Lonsdale, my father, John O'Donnell, June Sutor, Carl Julian and Maureen Julian.



Figure 6. David Puxley, Don Emerson, Richard Welberry and Richard Henriques in front of the University of London Chaplaincy.



Figure 7. Mike Glazer, Maureen Julian, organist, Howard Flack and Richard Welberry.



Figure 8. Josh Thomas.

Fifty years later

Five have died: *Prof., June Sutor, Robin Shirley, Howard Flack* and *Judith Milledge*, in that order. *Dame Kathleen Lonsdale* (1903–1971) retired in September 1968; just before she died, she permanently stored references to help her students get positions. *June Sutor* (1929–1990) continued at University College London until her death by cancer. She left a considerable fortune to the Moorfields Eye Hospital. *Robin Shirley* (1941–2005) was in the Department of Chemistry and then in Psychology at the University of Surrey, where he continued his interest in X-ray powder indexing and was a popular teacher of statistics. In 1972, *Howard Flack* (1943–2017) located to the Laboratoire de Cristallographie at the University of Geneva, Switzerland, where he worked to improve the collection of experimental diffraction data. The well known Flack parameter is used to estimate the absolute configuration of a crystal structure. He was also interested in theoretical symmetry, particularly the partially ordered sets, for example, in the relations among the Bravais lattices. After *Prof.*'s death, the Geology Department took over the building. To maintain the same physical lab, *Judith Milledge* (1927–2021) transferred the crystallography group to the Geology Department, where she continued the diamond research. Particular areas of focus were the formation of diamonds, isotopic compositions, inclusions and defects. She had a huge collection of diamonds which, after retirement, she split between South Africa and the United Kingdom (Wood *et al.*, 2021).

Members of this laboratory have spread out all over the world. Here is a short summary in alphabetical order. *Marianne Ehrenberg* taught at Sir John Cass College, London, and later at the City of London Polytechnic. *Don Emerson* is a geophysical consultant in Australia specializing in the physics of minerals and rocks. He studied the relationship between iron content and paramagnetic susceptibility in rocks (Clark & Emerson, 1991). Don also has an interest in ancient and medieval geoscience. *Mike Glazer* is now an emeritus Professor of Physics at Jesus College, Oxford University. He invented a classification system for tilted octahedra in perovskites (Glazer, 1972, 2021; Glazer & Burns, 2013). Mike has been

active in the IUCr and loves the history of crystallography. *Jeff Harris* is now an emeritus senior research fellow at the University of Glasgow and holds honorary professorships at the Universities of Frankfurt and Alberta. He continues to conduct research into mineral inclusions in diamonds (https://cms.eas.ualberta.ca/team-diamond/contact-us/members/jeff-harris/; Harris *et al*., 2022). *Eugene Hoff* returned to medicine, where he practised psychiatry, first in New York City, and later, at San Quentin Prison, California. *Maureen O'Donnell Julian* teaches crystallography at Virginia Tech in Blacksburg, now by Zoom. She wrote a widely used textbook (Julian, 2015). Currently, she is studying relationships between crystallographic groups and their related abstract groups with emphasis on partially ordered sets. *David Puxley* went into industrial X-ray diffraction in the Analytical Division of the British Gas Cooperation, London. *Terrance Rummery* became President of Atomic Energy Canada (Research), where he focused on the safe disposal of used nuclear fuel. Corrosion was also an important aspect of his work (Rummery & MacDonald, 1975). *Josh Thomas* is an emeritus Professor of Solid-State Electrochemistry at Uppsala University, Sweden. In 2009, he founded the Ångström Advanced Battery Centre. He developed a furnace for lithium battery studies (Eriksson *et al.*, 2001). In 1975, *Richard Welberry* moved to Australia and is now an emeritus Professor at the Institute of Advanced Studies at the Australian National University. His field is the analysis of diffuse X-ray scattering (Welberry, 2004).

Finale

The Kathleen Lonsdale Building at University College London officially opened in 2018, and houses the Department of Earth Sciences.

Mike Glazer wrote (Glazer, 2005):

"A few years ago, I was fortunate to be invited to the Science Museum to see the unveiling of a Ferranti Pegasus Mark II computer that Robin and the Society had managed to restore and get working. This was the very same machine that I, Howard Flack and Robin used during our graduate research, and I believe that it is now the only working valve computer in the world. Robin and I felt quite nostalgic when it was switched on to play 'Teddy Bear's Picnic' and 'Rocking through the Rye' (we used to play this during long hours at night working on this machine)."

We conclude with a poem written by Robin Shirley for the IUCr Congress in Glasgow (Shirley, 1999):

The Zen of Scale

Launched on a journey
across the void of dimensions
from the intricate tapestry of tissues
to the rhythms encoded in atoms
arriving at last
to orbit electron-gas giants.

Thanks to Mike Glazer for suggesting I write an article on University College London and to Jeff Harris, Josh Thomas and Mike for their memories. Carla Slebodnick and Kathleen Julian helped with editing.

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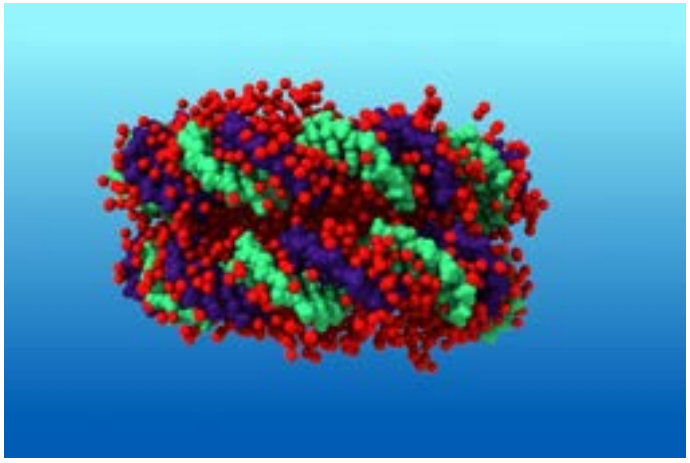
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FEATURE ARTICLE

Water, water everywhere: the maintenance of the genome’s structural integrity



Stephen Neidle

All forms of life on planet Earth require water for survival and growth, from the most primitive of organisms through to *Homo sapiens*. They reproduce, develop and evolve through the expression of genetic information encoded and stored in DNA (in some viruses, RNA instead) for all the proteins and enzymes needed by the organism. Mammalian DNA is tightly packaged into chromosomes with specialised proteins; as described below, the DNA is still directly exposed to the aqueous environment. Water plays a crucial role in maintaining the structural integrity of DNA and undoubtedly also plays a role in protecting the genetic message from external invaders.

The history of DNA structural studies in the early 1950s is almost universally well known, even if some aspects still excite controversy. What is not controversial is that the model-building of Watson and Crick, together with the experimental X-ray fibre diffraction data of Franklin, Wilkins and Gosling, led directly to the double-helical model of DNA. The experimental data were crucial for the work of the modellers. However, the diffraction patterns, demonstrating helicity and base stacking, would themselves not have been obtainable without the realisation and subsequent demonstration by Rosalind Franklin and her student Raymond Gosling that controlled hydration of the fibres was essential for obtaining high-quality fibre diffraction data of the different polymorphs of DNA. The landmark paper describing this work was submitted to *Acta Crystallographica* in March 1953 and published in September of that year (Franklin & Gosling, 1953). The subsequent three back-to-back papers in *Nature* describing the double helix model and the experimental work on which it was based were published in April of the same year. Thus, the essential role of water, and even its likely localisation around DNA structural groups, notably the phosphates, was recognised in this work, although any structural detail was lacking. In an almost throw-away remark towards the end of their *Acta* paper, Franklin and Gosling mentioned the single-crystal structures of adenine and guanine published in 1948 and 1951, also in *Acta Crystallographica*, by June Broomhead (Obituary, *IUCr Newsletter*, March 2022), and the potential significance of her results for determining how the bases in DNA might be organised. Franklin and Gosling did not remark on June Broomhead’s observation of water molecules in the guanine hydrochloride crystal structure (Broomfield, 1951) being hydrogen-bonded directly to the bases; this was the first indication that DNA bases themselves could be involved in DNA hydration.

X-ray fibre-diffraction studies after 1953 enabled determination of electron density in individual polymorphs (of which the B-form is the most significant in biological terms) using the standard Fourier map methodology of single-crystal methods. Fibre diffraction methods have been unable to define

discrete water molecules around individual sequences in the double helix. By contrast, neutron diffraction fibre methods using deuterated specimens and high-intensity neutron beams have been successful in revealing the presence of some localised water molecules around phosphate groups and in narrow grooves. An inherent limitation of fibre diffraction, in that DNA sequences are averaged, has precluded any more detailed water structure analysis by this, the classic approach to determining DNA structure.

The Watson–Crick model for the B-form of the DNA double helix is supported by extensive genetic and biochemical evidence and is universally accepted as representing genomic DNA. However, a formal proof of the anti-parallel double-helical structure without any preconceived model only became available in 1980 with the *ab initio* single-crystal structure determination of a short dodecanucleotide (12-mer) duplex in 1980 by Dickerson, Drew and their colleagues. This was followed in 1984 by the (then) remarkable feat of the determination of the crystal structure of the basic repeat unit of the chromosome, the nucleosome. This contains a B-DNA chain 146 base pairs in length, wound around a core of eight histone proteins. Many subsequent X-ray crystallographic studies on nucleosome sequence variants have confirmed that the B-DNA duplex is the major biologically important form of genetic material. One such study, at a remarkable 1.9 Å resolution (Davey *et al.*, 2002), revealed many details of the hydration around the particle, involving 3130 water molecules (Fig. 1, left). The crystals have over 50% solvent content, so these 3130 water atoms, out of a total of over 13,000 protein and nucleic acid atoms, represent only a small fraction of the total water content in these crystals. The rest were too mobile to be captured by the X-ray technology of the day.

The 12-mer structure, which has been re-determined several times at increasingly higher resolution, has also provided the bonus of revealing a detailed atomic level view of first- and second-shell water arrangements around a DNA molecule. The double helix, by its nature, has two continuous indentations, grooves, between the chains, and these are filled with water molecules, especially in narrow regions of this groove (Fig. 1, middle). This continuous two-dimensional ribbon of connected water molecules, known as the `spine of hydration' (Drew & Dickerson, 1981) uses hydrogen bonds to link to the nucleobase edges in the groove, especially to those of adenine–thymine base pairs (Fig. 1, right). This highly structured water spine is not unique to double-helical DNA. Closely similar arrangements have also been observed in quadruplex DNA (Li *et al.*, 2021), the four-stranded arrangements that can form in regions of repetitive guanine-rich genomic sequences and which have analogous grooves (but four rather than two).

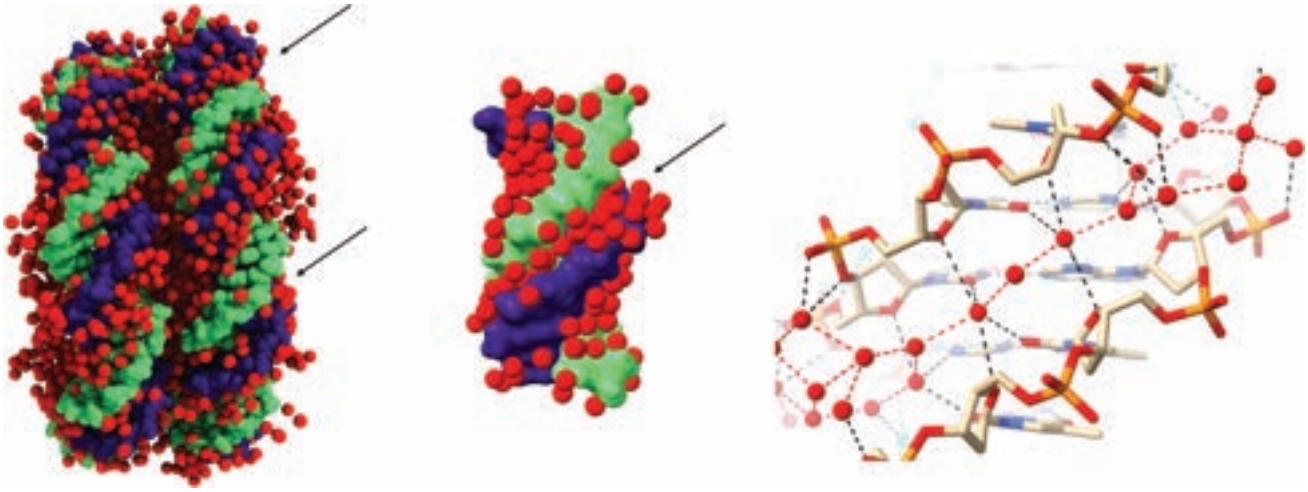


Figure 1. Water molecules around and embedded in DNA. (Left) The waters (red spheres) in the nucleosome crystal structure (PDB entry 1KX5). The two strands of DNA are coloured blue and green. Protein has been omitted for clarity. Several DNA minor grooves are indicated by the arrows. (Middle) The waters in a 12-mer DNA duplex structure (Minasov *et al.*, 1999; PDB entry 4C64). (Right) Detailed view of the spine of hydration in the 12-mer, with waters indicated by red spheres and hydrogen bonds by dotted lines. Drawings created with the *ChimeraX* program (Pettersen *et al.*, 2021).

Water is not just an insulating and stabilising agent for nucleic acids. There are many examples where individual water molecules embedded in DNA play crucial roles in mediation with bound proteins,

enzymes and drug molecules.

It is 69 years since the discovery of the structure of the double helix, and it has catalysed much of modern science to the extent that could not have been envisaged by those early pioneers. They would surely have been gratified that the B-DNA structural concept has not only stood the test of time, but that we have been able to visualise the richness of its aqueous environment.

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FEATURE ARTICLE

The Big Data Science Center at the Shanghai Synchrotron Radiation Facility: the dawn of the scientific superfacilities



Users at the Shanghai Synchrotron Radiation Facility (SSRF) can seamlessly access the scientific and technological architecture of the Big Data Science Center (BDSC) through thin terminals to take full advantage of all the services offered by the BDSC in real time while running their experiments at the SSRF beamlines.

Alessandro Sepe

The massive amount of raw data produced nowadays at large scientific facilities creates not only enormous new opportunities but also tremendous challenges. Already, only a small fraction of this multidisciplinary and scientifically complex Big Data are fully analysed and, ultimately, used in scientific publications, and it is predicted that in a few years, conventional data-analysis approaches will be overwhelmed, preventing the users from producing meaningful science from their large-scale experiments. This is a problem for all synchrotron and neutron facilities, as well as for X-ray free-electron laser facilities, where tens of petabytes are produced annually. Beamtime is expensive and the lack of automated data-analysis pipelines reduces beamtime efficiency. This “data deluge” effect [1] has implications for all large scientific facilities worldwide in that it affects fast data collection and storage and curation of the data, including data movement and deposition in a database.

We are witnessing the dawn of artificial intelligence (AI), machine learning (ML) and robotic automation within the field of large scientific facilities, generating profound changes in how petabytes of interdisciplinary datasets are intelligently processed, managed, analysed and visualised. The consequent evolution of large scientific facilities into superfacilities enables multimodal user science to confront the Big Data challenges, fundamental for the entire scientific community. The Big Data Science Center (BDSC) at the Shanghai Synchrotron Radiation Facility (SSRF), Shanghai Advanced Research Institute (SARI), Chinese Academy of Sciences, Zhangjiang Laboratory, is the first scientific superfacility in China, and one of the first worldwide [2]. With its state-of-the-art developments it aims to dramatically accelerate and automate the multidisciplinary research of all the users at large national scientific facilities, thus effectively increasing the rate of their scientific discoveries and the resulting technological advancements, with a clear societal impact. Therefore, the BDSC Big Data Science platform targets the research projects that several national and international universities, academies, research institutes and industries are pursuing at SSRF, where massive support in terms of scientific computation is required to enable the most complete knowledge transfer from scientific research to industrial developments, while elastically interfacing them with the top Chinese National Supercomputer Centers.

Video documentary showing the BDSC activities, international potential and impact on the user's science at the SSRF, and more in general on the large national scientific facilities worldwide. Here, the BDSC state-of-the-art superfacility capabilities are demonstrated using the Biological Macromolecular Crystallography Beamline (MX Beamline) as a case study, including users performing a real experiment in real time. The entire BDSC workflow is shown, from the preparation of the user experiment at the beamline, through the setting-up of the beamline, to the real-time Big Data analysis and results visualisation, including the user's data being processed and monitored through the BDSC superclusters and Control Room, as well as the users interacting with the BDSC Platform. If you experience problems playing the video, please click here.

The BDSC is one of the latest SSRF upgrades resulting from the SSRF Phase II project. The BDSC aims to support all the SSRF beamlines, as well as their national and international users, through its state-of-the-art scientific computational infrastructure, including high-performance computing (HPC), latest-generation storage systems and advanced software platforms. Users and beamlines at the SSRF are thus benefiting, seamlessly, from the most advanced Big Data processing, movement, analysis, results interpretation and visualisation capabilities offered by the BDSC. The BDSC aims, in fact, to support all the SSRF users to produce high-impact science and technology, matching the highest international quality standards, thus further enabling the users to publish their results in top-notch internationally renowned peer-reviewed journals.

Professor Alessandro Sepe, Director of the BDSC, has designed and architected this novel Big Data Science platform at SSRF, which allows all its users to fully exploit the scientific and technological potential of the BDSC for their research. The entire BDSC staff supported Professor Sepe's efforts in developing, deploying and then constantly upgrading the BDSC infrastructure, which is now fully operational. Here, state-of-the-art Big Data science and technologies, AI, internet of things (IoT), real-time unstaffed and remotely controlled experiments, robotic automation, HPC, cloud/fog supercomputing and massive parallelisation are converging on the SSRF through the BDSC's fully centralised platform, accelerating the user multidisciplinary science performed at SSRF by an outstanding factor of 60, and effectively creating the first-ever world-class user-friendly Chinese superfacility. This is greatly augmenting the interpretation of all the scientific data generated by all the experiments at SSRF. By developing, deploying and upgrading the Big Data Science platform, the BDSC is fostering full robotic automation at the SSRF beamlines, aiming at real-time unstaffed and remotely controlled experiments, while sharing its successful experience with all the other multidisciplinary large facilities worldwide.

The Big Data Science platform developed by the BDSC is, in fact, collecting, tagging and tracking large volumes of metadata from all the experiments at SSRF to fully automate the entire large-facility lifecycle. Hundreds of petabytes of scientific data are thus tagged, to be then ingested by neural networks for ML. This remarkable scientific and technological achievement also allows non-expert users at SSRF to obtain scientifically meaningful results in real time, instead of spending months, after returning to their home institutions, processing unstructured raw data. The BDSC is thus effectively extending the use of large national facilities to the largest number of scientific and industrial disciplines ever, dramatically increasing the scientific and technological productivity of large scientific facilities like SSRF, shifting the focus of their users from data science to pure science, thus enabling a true user-science-centric and multimodal infrastructure.

The BDSC is also engaged in further expanding the SSRF scientific computational capabilities, directly interfacing, through the BDSC framework, the SSRF with the supercomputers in China, including the Shanghai Supercomputer Center (SSC), with extremely low-latency networks.

The BDSC further aims at seeding the Chinese National Scientific Grid based on the platform model being developed at the BDSC.

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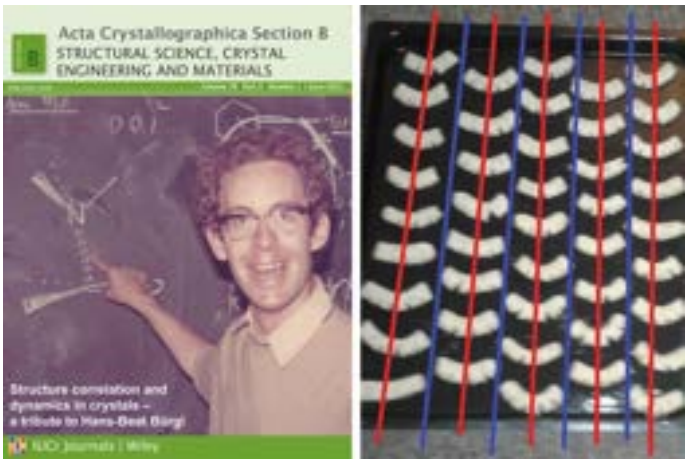
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Mach. Learn.: Sci. Technol. **2**, 035003.

EDITORIAL

Structure correlation and dynamics in crystals – a tribute to Hans-Beat Bürgi



Left: the cover of the special issue showing Hans-Beat Bürgi teaching in the early 1970s. On the blackboard is a sketch of the nucleophilic attack at carbonyls. Right: Christmas biscuits used as a teaching aid.

Simon Grabowsky, Mark A. Spackman

Crystal structures are not static; they contain a wealth of information about dynamics and reactivity of molecules and extended solid materials. On the one hand, dynamic properties are related to the atomic and lattice vibrations in crystals, so that an accurate description of both atomic displacement parameters and diffuse scattering are important for crystallographic experiments. Disorder in crystals may be also related to dynamic behaviour, and the description of dynamic behaviour renders the determination of thermodynamic properties of crystals possible. On the other hand, series of related crystal structures can be understood as snapshots along a pseudo-reaction pathway, which relates ‘static’ crystallography to chemical reactivity.

These aspects of structural science have been major themes in Hans-Beat Bürgi’s scientific career spanning nearly 60 years. Structure correlation, disorder, and the dynamics of atoms and molecules in crystals remain important but sometimes still underrated fields of structural chemistry today. For those reasons, we have taken the opportunity to highlight these topics in this thematic special issue dedicated to Hans-Beat Bürgi on the occasion of his 80th birthday this year.

Hans-Beat Bürgi’s university education in the Chemistry Department of the Federal Institute of Technology (ETH) Zürich culminated in the completion of his PhD research in 1969 under the supervision of Jack Dunitz. After a two-year postdoctoral stint with Lawrence (Larry) Bartell at the University of Michigan, focusing on structure determination in the gas phase by electron diffraction, Hans-Beat returned to single-crystal X-ray diffraction and to the laboratory of inorganic chemistry at the ETH for his habilitation work leading a small independent research group. In this period between 1971 and 1979, many of the ground-breaking studies that determined Hans-Beat’s career were undertaken, such as the development of the structure correlation idea (Bürgi, 1973) of which the highly cited concept of the Bürgi–Dunitz angle of nucleophilic attack at carbonyls is part (Bürgi *et al*., 1974). The cover illustration of this special issue [see above] shows Hans-Beat at the blackboard in Zürich in 1974, explaining the concept to a guest researcher from Japan.

His substantive position for nearly 30 years was Professor and Director of the Laboratory of Chemical and Mineralogical Crystallography at the University of Bern (Fig. 1). During this time, Hans-Beat established

himself as one of the leading international authorities of X-ray crystallography. Many of his achievements are described and reflected in this special issue, and he has described them in substantial detail in a recent essay in the *Israel Journal of Chemistry* (Bürgi, 2017), so we have not attempted to list or summarize them here. In his post-retirement roles as an Emeritus Professor in Bern and a permanent academic guest at the University of Zürich, Hans-Beat continues as an active researcher who does not miss a single day in the office.



Figure 1. Hans-Beat Bürgi in his office at the University of Bern in 1981.

Throughout his distinguished career, Hans-Beat has held visiting appointments at Princeton University, the Australian National University in Canberra, Technion in Haifa, Caltech in Pasadena, Tokyo Institute of Technology, University of California Berkeley and San Diego, and the University of Western Australia. With the end of the Covid pandemic, he plans to revive some of these collaborations during his annual trips around the world from winter in Switzerland to summer in Australia, then on to New Zealand, California, and back to Switzerland just when winter has passed in the northern hemisphere.

In preparation for this special issue, we had naturally contacted Jack Dunitz who, in May last year, astonished Mark by a phone call at his home in Perth, and Simon in his office in Bern. Jack was genuinely disappointed that he could not contribute, as with macular degeneration he was no longer able to write. But he did very vividly paint for us a picture of Hans-Beat as a young scholar. Jack said that Hans-Beat had always stood out from others for his insatiable interest in (chemical) science and discussions around it. If necessary, he could stand his ground with much older and more established colleagues in heated discussions from early on in his career. Hans-Beat’s perseverance and accuracy when finishing a study for publication was also something that Jack remembered. There were supposedly hour-long discussions between the two of them around the formulation of a single paragraph in a manuscript draft. In the end, the quality of the published work speaks for itself. In this context, it is worth reading the obituary that Hans-Beat wrote for Jack Dunitz, published in the previous issue of *Acta Crystallographica B* (Bürgi, 2022).

Hans-Beat has not lost any of the characteristics that Jack Dunitz attributed to the young scholar. He is well known in the crystallographic community as the critical mind who always has a question at the end of a scientific talk, and who enjoys to passionately discuss. Along the same lines, Hans-Beat's dedication to teaching and mentoring young crystallographers and his excellent didactic skills have been widely recognized and appreciated for some time. Among his many other achievements, Hans-Beat has been director of an Erice School of Crystallography in 1991 and, with Tony Linden, he continues to be an organizer and director of the Zürich School of Crystallography that he co-founded upon retirement in 2007 (Fig. 2). It will see its 10th edition this year, and the 2024 School is already in the planning.



Figure 2. The first Zürich School of Crystallography in 2007.

We also compiled a list of Hans-Beat's scientific colleagues and collaborators, and in early 2021 invited them to contribute in the form of personal reminiscences, research papers or review articles. Given the disruption caused by the global pandemic, we were delighted that most invitees were both keen and willing to contribute to this festschrift volume. We were especially pleased to receive personal contributions from Dieter Schwarzenbach, Roald Hoffmann, Jean-Marie Lehn and Hans-Beat himself, and these are included as Opinion pieces at the beginning of this special issue. Two Topical Reviews follow that are fundamentally different in content and reflect the considerable breadth of Hans-Beat's interests: a review of bone hierarchical structure by Henrik Birkedal *et al.*, as well as a review of X-ray constrained wavefunction fitting by Dylan Jayatilaka *et al.* The research articles in this special issue are dedicated to numerous aspects of dynamics in crystals, disorder, structure correlation and even quantum crystallography and, on the latter field, Hans-Beat has contributed an additional critical commentary. Finally, we are indebted to the many scientific referees whose willingness to support this special issue was remarkable, reflecting Hans-Beat's standing among, and respect from, the international community of crystallographers and structural chemists.

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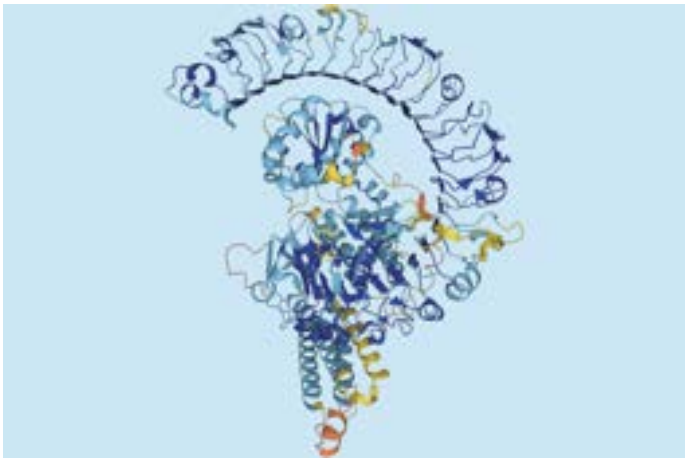
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EDITORIAL

Whither structural biologists?



Gerard J. Kleywegt, Sameer Velankar

Between December 2020 and July 2021, several spectacular developments in the field of protein-structure prediction changed structural biology profoundly, and they are expected to have an impact on much of modern (molecular) biology, medicine, biochemistry and biotechnology. The unprecedented accuracy of blind protein-structure predictions produced by DeepMind's *AlphaFold2* was revealed at the CASP 14 meeting in December 2020. In July 2021, this was followed by publication of the method and release of the code (Jumper *et al.*, 2021). Simultaneously, a prediction method from the Baker lab that achieved similar accuracy was published (Baek *et al.*, 2021). A week later, an additional publication described proteome-scale application of protein-structure prediction using *AlphaFold2*. This coincided with the launch of a new resource in which DeepMind and EMBL–EBI collaborate to make hundreds of thousands (and, eventually, hundreds of millions) of high-quality predicted protein structures openly available: the AlphaFold Protein Structure Database, or AlphaFold DB (Tunyasuvunakool *et al.*, 2021; Varadi *et al.*, 2022). These developments stunned not only the protein-structure prediction community, but most of the structural biology, bioinformatics, and machine-learning communities and beyond. It was truly an '*annus mirabilis*'.

Much has been said and written about these developments in the scientific, technical and popular press (not to mention on social media) and the journal *Nature Methods* selected protein-structure prediction as its *Method of the Year* (Editorial, 2022). The potential impact of the developments has also been the subject of much prognostication and speculation. When AlphaFold DB was first announced, a group of senior EMBL structural biologists released a white paper discussing its possible impact (https://www.embl.org/news/science/alphafold-potential-impacts/). At the time of writing (late May 2022), using the term 'alphafold' to search the literature at Europe PMC (https://europepmc.org/search?query=alphafold), there are over 700 published research papers, over 150 unpublished preprints and, somewhat astonishingly, almost 300 review papers.

The structural community was impressively quick to embrace the new resources (both the software and the database) and a flurry of preprints appeared in bioRxiv within weeks, both analysing the holdings of the database (*e.g.* correlating the estimated reliability of models and the location of intrinsically disordered regions in proteins) and putting the software through its paces (*e.g.* various 'hacks' to make *AlphaFold2* predict structures of multimeric complexes). Experimental structural biologists were also quick to react and within days of AlphaFold DB being announced and the AlphaFold code being released, there were many reports of structures (X-ray and cryo-EM mostly) that had previously resisted solution but that suddenly could be cracked using one or more (parts of) AlphaFold (DB) models. Methods and software developers were also quick to adapt their procedures and programs to make use of the astonishing new opportunities offered by high-quality protein-structure predictions. Over time, scientists

outside the structural field are expected to embrace and discover the power of the new methods and the fact that soon a structure model will be available (or easily obtainable) for just about any protein sequence, be it natural or designed. Moreover, developments in the structure-prediction field will continue, *e.g.* concerning structure prediction for macromolecular and ligand complexes, RNA, post-translational modifications, and the impact of point mutations. The full scientific impact will take many years to be realized, and by then the new tools (methods and models) will be textbook material for undergraduate curricula.

While this is all fantastic news for bioscience, medicine and biotechnology, there are also concerns, not least in the field of structural biology, regarding the impact of these developments on funding and careers. We understand that when the announcement of AlphaFold DB was made via the CCP4 mailing list, there was a crystallography course ongoing, and the news made some of the students on the course question their study or career choices. We wouldn't be surprised if, upon first hearing the announcements, more than a few structural biologists went through several of the five stages of grief (denial, anger, bargaining, depression and acceptance) and have asked themselves questions such as: '*Is there even a need for experimental structure determination anymore?*', '*Is there a future career in structural biology for me?*', '*Will it be possible to get tenure in this field?*', '*Will funding agencies now think that structure determination is not necessary and stop funding my projects/methods development efforts?*', '*Will expensive infrastructure (synchrotrons, microscopes, spectrometers) still get funded?*'.

It is almost unavoidable that there will be changes in terms of funding and career opportunities. However, as the adage (attributed to Einstein) goes: '*In the midst of every crisis, lies great opportunity.*' Perhaps this time of profound change in our field is merely a transition to a new golden era for structural biology. For one thing, structure prediction still has limitations and is anyway not a panacea; also, predictions will need validation. More importantly, there will be entirely new opportunities leading to a 'new normal' in structural biology which will, in turn, support a new normal in biology. This means that there will continue to be a need for experimental structure determination (and the requisite people and infrastructure). However, it is likely that structures can generally be determined much more quickly than before and hence that we can focus more on the real objective of structural biology: to understand or influence function, mechanism, activity, binding interactions, *etc.* using structural information (as well as other biophysical and computational methods). Moreover, in the wider field of biology, medicine, biotechnology, agriculture *etc.*, there will be a huge need for scientists (and teachers) who know and understand structures, who can assess which parts of a structure, be it experimental or predicted, are likely to be reliable enough to (help) answer the biological question that is posed, who can assess which prior biological data about function *etc.* can be explained in light of a structure, who can compare multiple related structures and draw sensible conclusions, and who can help design follow-up experiments based on the insights gained from structures. In summary, we are optimistic that, far from witnessing the end of structural biology, we are part of an exciting revolution in biology where structure will play a much more prominent role than in the past, at least on a par with the role that protein sequences are playing today.

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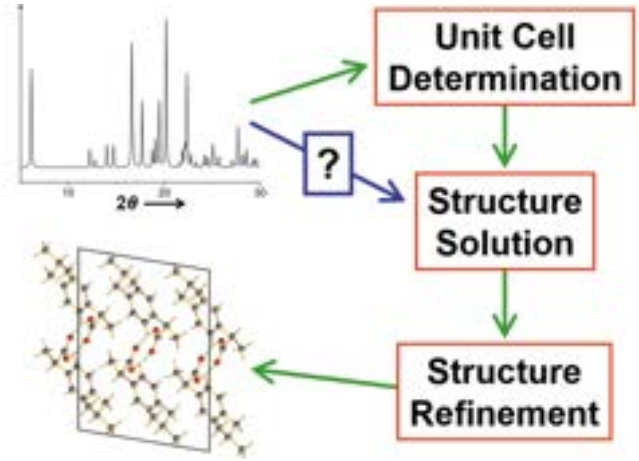
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COMMENTARY

Circumventing a challenging aspect of crystal structure determination from powder diffraction data



Kenneth D. M. Harris

Although recent decades have seen significant progress in the opportunities for carrying out crystal structure determination directly from powder X-ray diffraction (XRD) data (McCusker, 1991; Harris *et al.*, 2001; David & Shankland, 2008; Etter & Dinnebier, 2014; Černý, 2017), this task is still considerably less routine than structure determination from single-crystal XRD data. In some cases, the structure determination process fails at the first stage, namely unit-cell determination (often referred to as 'indexing' the powder XRD data), such that the process cannot progress to the subsequent structure solution and structure refinement stages. For reasons highlighted below, unit-cell determination from powder XRD data can encounter challenges under certain circumstances, which may represent an insurmountable hurdle in the structure determination process. Specific challenges arise in cases of poor-quality powder XRD data (for example, when severe peak broadening arises due to poor crystallinity of the sample) and/or poor-quality samples (for example, containing an unknown impurity crystalline phase).

In light of the challenges that can arise in unit-cell determination, the report of a strategy by Schmidt and co-workers (Habermehl *et al*., 2022) that essentially circumvents the indexing step in structure determination from powder XRD data will be welcomed with interest by practitioners in the field. However, to place this development in context, it is relevant first to explain briefly the methodology for indexing powder XRD data, and the reasons that challenges may be encountered in this stage of the structure determination process.

Unit-cell determination involves analysis of the 'positions' of Bragg reflections in the powder XRD data, where 'position' refers to the diffraction angle 2θ or the corresponding d-spacing. The aim is to find a set of unit-cell parameters (a, b, c, α, β, γ), together with the corresponding set of Miller indices (h, k, l) for each Bragg reflection, that successfully account for the positions of all peaks in the powder XRD data. Typically, the positions of about 20 peaks are used in the indexing process, measured from the low-2θ region of the experimental powder XRD data, where the extent of peak overlap is less than in the high-2θ region. It is important to note that the aim of the indexing stage is to establish a good initial approximation to the correct unit cell, which is then improved in subsequent profile-fitting calculations [typically carried out using the algorithms of Pawley (1981) or Le Bail (1988)] to generate a more accurate set of unit-cell parameters for use in subsequent structure solution and refinement calculations.

In the early days, when powder XRD data were measured on photographic film and data analysis relied on mathematical insight rather than computational power, the challenges associated with unit-cell determination were already well recognized (Henry *et al*., 1961), and a number of approaches were pioneered for indexing such data (Hesse, 1948; Vand, 1948; Lipson, 1949; Ito, 1949; de Wolff, 1957). Subsequently, the emergence of computer technology stimulated the development of a wide range of new strategies for unit-cell determination from powder XRD data (Taupin, 1968; Visser, 1969; Kohlbeck & Hörl, 1976; Werner *et al*., 1985; Boultif & Louër, 1991; Paszkowicz, 1996; Kariuki *et al*., 1999; Coelho, 2003; Neumann, 2003; Habershon *et al.*, 2004; Le Bail, 2004; Altomare *et al*., 2009; Oishi-Tomiyasu, 2014; Louër & Boultif, 2014), many of which form the basis of indexing programs that are widely used today.

With this large array of computational approaches at our disposal, unit-cell determination from powder XRD data often proceeds straightforwardly and successfully. However, a number of factors can limit the chances of success in specific circumstances. Two particularly serious situations are: (i) severe peak overlap in the powder XRD data (which may limit the ability to extract accurate values of peak positions, as required for indexing calculations, and can even obscure the existence of certain peaks that may be crucial for successful indexing), and (ii) the presence of a crystalline impurity phase in the powder sample (such that the set of peak positions used for attempted unit-cell determination actually arise from two different unit cells).

Problems of severe peak overlap arise in the case of samples that give broad peaks in the powder XRD pattern, for example due to poor crystallinity and/or particle size effects. In such cases, it may be challenging to extract a sufficient number of reliable peak positions from the powder XRD data to allow successful indexing. Furthermore, even for samples of good crystallinity, significant peak overlap can still arise in cases with large unit cells and/or low symmetry due to a high density of peaks in the powder XRD pattern, even though the individual peaks are relatively narrow. This situation often arises for organic molecular materials. In such cases, measuring the powder XRD data using a synchrotron radiation source may help to alleviate the extent of peak overlap by minimizing the instrumental contribution to peak widths, and hence may be conducive to successful indexing.

To assess whether failure to index a powder XRD pattern is due to the presence of an impurity crystalline phase in the powder sample, independent evidence may be established by other experimental techniques (particularly solid-state NMR) or simply by checking whether the powder XRD pattern contains peaks due to plausible impurity phases of known crystal structure (such as known polymorphs of the material of interest). In favourable cases, the impurity phase may be identified as a material of known structure, in which case the peaks due to the impurity phase can be readily excluded from the indexing calculation, allowing successful unit-cell determination using only the peaks for the phase of interest (for example: Al Rahal *et al.*, 2019). In some cases, the existence of two crystalline phases in a powder sample may be identified by observing that the powder XRD data contain two sets of peaks with significantly different peak widths (for example: Dinnebier *et al*., 1997).

Of course, when attempts to index a powder XRD pattern prove to be problematic, other experimental techniques may be exploited to determine the unit-cell parameters for the material of interest, such as electron diffraction (for example: Gorelik *et al*., 2009; Smalley *et al*., 2022) or measurement of XRD data for magnetically oriented micro-crystalline arrays (Kimura & Kimura, 2018). Another approach to generate possible unit cells independently of the powder XRD data is to take advantage of crystal structure prediction algorithms (Price, 2014; Woodley *et al.*, 2020), which generate a set of energetically feasible crystal structures for the molecule of interest; in principle, one of the crystal structures may be found to give an acceptable match to an unindexed experimental powder XRD pattern (for example: Paulus *et al.*, 2007).

Although the approaches discussed above provide a wide range of opportunities to overcome problems encountered in indexing powder XRD data, the development of structure determination protocols that effectively circumvent the indexing stage may be seen as an attractive alternative. The strategy reported

by Schmidt and co-workers (Habermehl *et al.*, 2022), entitled '*Structure determination from unindexed powder data from scratch by a global optimization approach using pattern comparison based on cross-correlation functions*', achieves this objective by combining unit-cell determination and structure solution within a single process, rather than handling them as sequential stages on the structure determination pathway. The methodology, which is embodied within the program *FIDEL-GO*, requires only an experimental powder XRD pattern and knowledge of the molecular structure as input information. The strategy starts from a set of trial crystal structures generated by random selection of both the unit-cell parameters and the arrangement of molecules within the asymmetric unit, with the space groups of the trial structures selected from a set of 'common' space groups. The strategy then applies a multi-step procedure for global optimization, with the ultimate aim of generating structural models that give an acceptable quality of fit to the experimental powder XRD data by optimization of *both* the unit-cell parameters *and* the set of structural variables that define the atomic positions within the asymmetric unit. In common with standard practice in direct-space structure solution of molecular materials from powder XRD data (Harris *et al.*, 2001; David & Shankland, 2008), the structural models used in *FIDEL-GO* are typically based on the assumption of standard bond lengths and bond angles (although one example discussed by Schmidt and co-workers includes relaxation of certain bond lengths), and the set of structural variables comprises, for each crystallographically independent molecule, the position of the molecule in the unit cell, the orientation of the molecule with respect to the unit-cell axes and a set of variable torsion angles that define the molecular conformation.

Importantly, a critical aspect underlying the *FIDEL-GO* approach is the selection of an appropriate method to assess the quality of agreement between the powder XRD pattern calculated for each trial structural model and the experimental powder XRD pattern, as the similarity measure used for this assessment must be able to give a meaningful indication of the quality of agreement even when the unit cell for the trial structural model deviates significantly from the correct unit cell. This task is achieved using a similarity measure based on a weighted cross-correlation function, specifically the generalized similarity measure S_{12} defined previously by de Gelder *et al*. (2001).

Following the optimization process, promising structural models generated by *FIDEL-GO* are subjected to an automated Rietveld fitting procedure to provide a more reliable assessment of the quality of fit to the experimental powder XRD data. The authors recommend that this stage is then followed by manual Rietveld refinement calculations as the final step of the process, to allow a robust ranking of the best candidate structures to be determined.

The authors note that their approach has a tendency to generate several different structures that give a similar quality of fit to the experimental powder XRD data. Clearly, under such circumstances, it is essential to further scrutinize the validity of the candidate structures using information from other computational (for example, DFT-D) and experimental (for example, solid-state NMR) techniques, in order to provide robust and conclusive evidence that the correct structure has actually been assigned on the basis of the final Rietveld refinement calculations. The application of methods for structure determination from PDF data (Billinge, 2019) for the same material may also be valuable in this regard (for example: Schlesinger *et al*., 2021).

It is important to note that the concept of carrying out unit-cell determination and structure solution from powder XRD data synchronously within the same protocol has been reported in previous studies (Hofmann & Kuleshova, 2006; de Gelder *et al.*, 2008; Padgett *et al.*, 2007; Rapallo, 2009; Guguta *et al.*, 2019), which are based on a variety of different strategies for carrying out optimization of the fit to the experimental powder XRD data, in some cases incorporating information from calculations of the energetic properties of trial crystal structures. The workflow of the *FIDEL-GO* algorithm also includes the opportunity to carry out DFT-D calculations, particularly in the context of validation of promising candidate structures in the final stages of the structure determination process.

The successful application of the *FIDEL-GO* strategy is demonstrated by structure determination of four materials from powder XRD data of poor quality, described by the authors as 'un-indexable', typically

containing only a relatively small number broad peaks. In comparison to current capabilities in the application of direct-space structure solution using high-quality ('indexable') powder XRD data, the selected materials represent structural problems of relatively low complexity, defined by a small number of structural variables (two of the four examples are rigid molecules, for which no variable torsion angles are required to define the intramolecular geometry, while the other two examples involve only a few variables to define the intramolecular geometry). Nevertheless, the successful structure determinations reported by Schmidt and co-workers clearly represent promising progress in demonstrating the feasibility of the *FIDEL-GO* strategy, and the success is particularly notable given the poor quality of the experimental powder XRD data used in some of their applications. Certainly, the results of future applications of *FIDEL-GO* to tackle structural problems of greater complexity, defined by a higher number of structural variables, will be eagerly awaited.

By demonstrating the prospect of achieving successful crystal structure determination from poor-quality powder XRD data, the *FIDEL-GO* strategy certainly offers a promising opportunity that may prove to be particularly beneficial when challenges are encountered in applying the conventional approaches for indexing powder XRD data.

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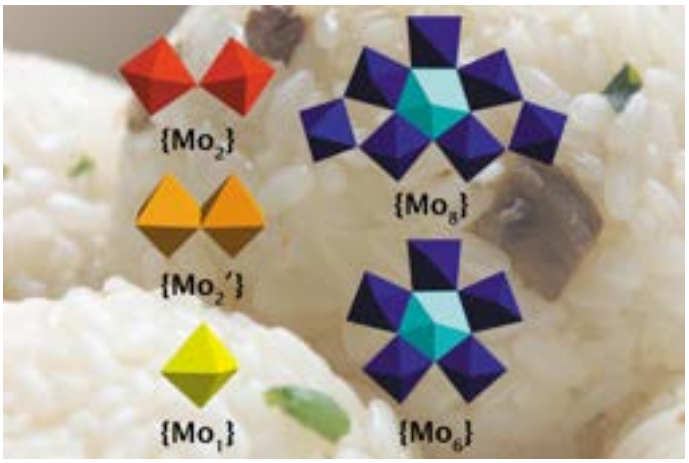
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COMMENTARY

Comment on molybdenum polyoxo clusters: from the 'Blues' to the 'Reds'



Bernt Krebs

Molybdenum 'Blues', 'Browns' and the recently discovered 'Reds' are unquestionably some of the most impressive nanoscale architectures in polyoxometalate (POM) chemistry (Müller & Gouzerh, 2012; Lin *et al.*, 2020). These three polyoxomolybdate classes, with the general formula $[X_aY_bH_cMo^{VI}{}_xMo^{V}{}_yO_z(H_2O)_v]^{n-}$ (*a* and *b* = number of organic ligands and heteroelements, respectively; *c* = degree of protonation; *x* and *y* = number of Mo^{VI} and reduced Mo^{V} centres, respectively) are classified as gigantic mixed-valence ($Mo^{V/VI}$) polyoxomolybdate clusters with various topologies ranging from 'wheels' to 'balls', 'cages' and the 'blue lemon', known to be the largest inorganic molecule to date (Lin *et al.*, 2020; Müller & Gouzerh, 2012). While the subclasses of molybdenum 'Blues', 'Browns' and 'Reds' have the reduction of an acidified solution containing orthomolybdate ($[MoO_4]^{2-}$) or heptamolybdate ($H_x[Mo_7O_{24}]^{(6-x)-}$) in common, they can be distinguished by three characteristic features. Firstly, 'Blues', 'Browns' and 'Reds' exhibit distinct degrees of reduction, with the 'Blues' being the least (~18%) and the 'Reds' the most (up to ~81%) reduced representatives (Ribó *et al.*, 2022).

Secondly, in terms of molybdenum building blocks, wheel-shaped molybdenum 'Blues' are composed of $\{MoO_6\}$ ($\{Mo_1\}$), corner-sharing $\{Mo_2O_{11}\}$ ($\{Mo_2\}$) and the essential building block $\{Mo_8O_{35}\}$ ($\{Mo_8\}$), while the ball-shaped 'Browns' consist of $\{Mo_1\}$, edge-sharing $\{Mo_2O_{10}\}$ ($\{Mo_2'\}$) and $\{Mo_6O_{27}\}$ ($\{Mo_6\}$) units (Fig. 1) (Müller & Gouzerh, 2012).

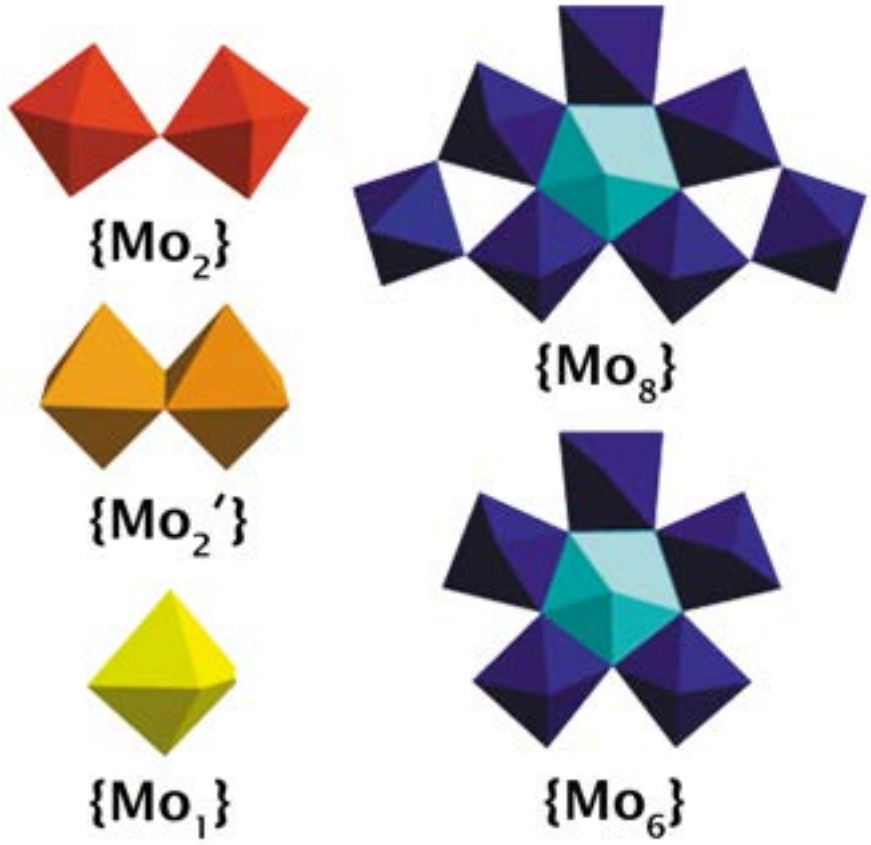


Figure 1. Polyhedral representation of the building blocks in mixed-valence polyoxomolybdates. Colour code: $\{MoO_6\}$ ($\{Mo_1\}$), yellow; corner-sharing $\{Mo_2O_{11}\}$ ($\{Mo_2\}$), red; edge-sharing $\{Mo_2O_{10}\}$ ($\{Mo_2'\}$, ochre; $\{Mo_6O_{27}\}$ ($\{Mo_6\}$) and $\{Mo_8O_{35}\}$ ($\{Mo_8\}$), blue, with the central $\{MoO_7\}$ unit in cyan.

Remarkably, molybdenum 'Reds' exclusively comprise $\{Mo_1\}$ and $\{Mo_2'\}$ units (Fig. 1) (Lin *et al.*, 2020), hence requiring a comparably trickier synthetic approach than their blue counterparts, which necessitates the use of additional cluster-stabilizing coordinated ions (*e.g.* SO_3^{2-}, 3*d*- and 4*f*-metal ions) (Lin *et al*., 2020; Ribó *et al*., 2022) to aggregate the $\{Mo_1\}$ and dumbbell-shaped $\{Mo_2'\}$ into larger clusters, consequently promoting molecular growth (Ribó *et al*., 2022).

Contrary to molybdenum 'Reds', additional ions (*e.g.* 3*d*- and 4*f*-metal ions) react differently in 'Blue' systems as they are utilized here for fine-tuning the molecular shape (Garrido Ribó *et al*., 2020) and alternating the molecule's physical characteristics rather than spanning the cluster framework.

For instance, large electrophilic 'open-shell' metal centres, such as 4*f*-metal ions, alter the overall charge, molecular shape and size, and always cause a symmetry reduction of molybdenum 'Blues' when introduced into their frameworks (Al-Sayed & Rompel, 2022). In 'Blue' systems, however, 3*d*-metal ions seem to behave in a significantly opposite manner from their 4*f* counterparts considering their relatively small size which leaves them without any impact on the assembly of the $\{Mo_1\}$, $\{Mo_2\}$ and $\{Mo_8\}$ building blocks (Fig. 1).

Thirdly, in terms of functionalizability, the 'Blues' and 'Browns' can be organically functionalized, resulting in reaction vessels for studying confined molecules. Organic hybridization is typically accomplished by attaching carboxyl groups to the cluster and nitrogen-containing pendant groups in carboxylic acids. Such constructs are capable of promoting molecular growth and triggering the formation of various guest@host architectures (Xuan *et al.*, 2017, 2019; Imai *et al*., 2009). In stark contrast to their 'Blue' counterparts, nothing is yet known about the organic hybridizability of molybdenum 'Reds'. In the event of a molybdenum 'Red' hybridization, the dumbbell-shaped and edge-sharing $\{Mo_2'\}$ (Fig. 1) building blocks would have to be functionalized organically.

In this context, Al-Sayed *et al.* (2022) recently made a significant contribution, reporting a new binding mode of an organic hybridization reagent in mixed-valence polyoxomolybdates (Al-Sayed *et al*., 2022).

The organic functionalization of a dumbbell-shaped and edge-sharing {Mo_2'} building block occurred through chelation of a pyridine derivative, as shown by the isolation of the $[Mo_2O_2(OH)_4(C_6H_4NO_2)_2]^{2+}$ unit (Fig. 2) acting as a charge-balancing dication for the molybdenum 'Blue' cluster $Na_4[Mo_2O_2(OH)_4(C_6H_4NO_2)_2]_2[Mo_{120}Ce_6O_{366}H_{12}(OH)_2(H_2O)_{76}]$~$200H_2O$. As 'Reds' comprise a multitude of {Mo_2'} units, the outstanding work of Al-Sayed *et al.* (2022) represents a major synthetic step forward, since it showcases the first example of the *N*,*O*-chelation of {Mo_2'} units (Fig. 2).

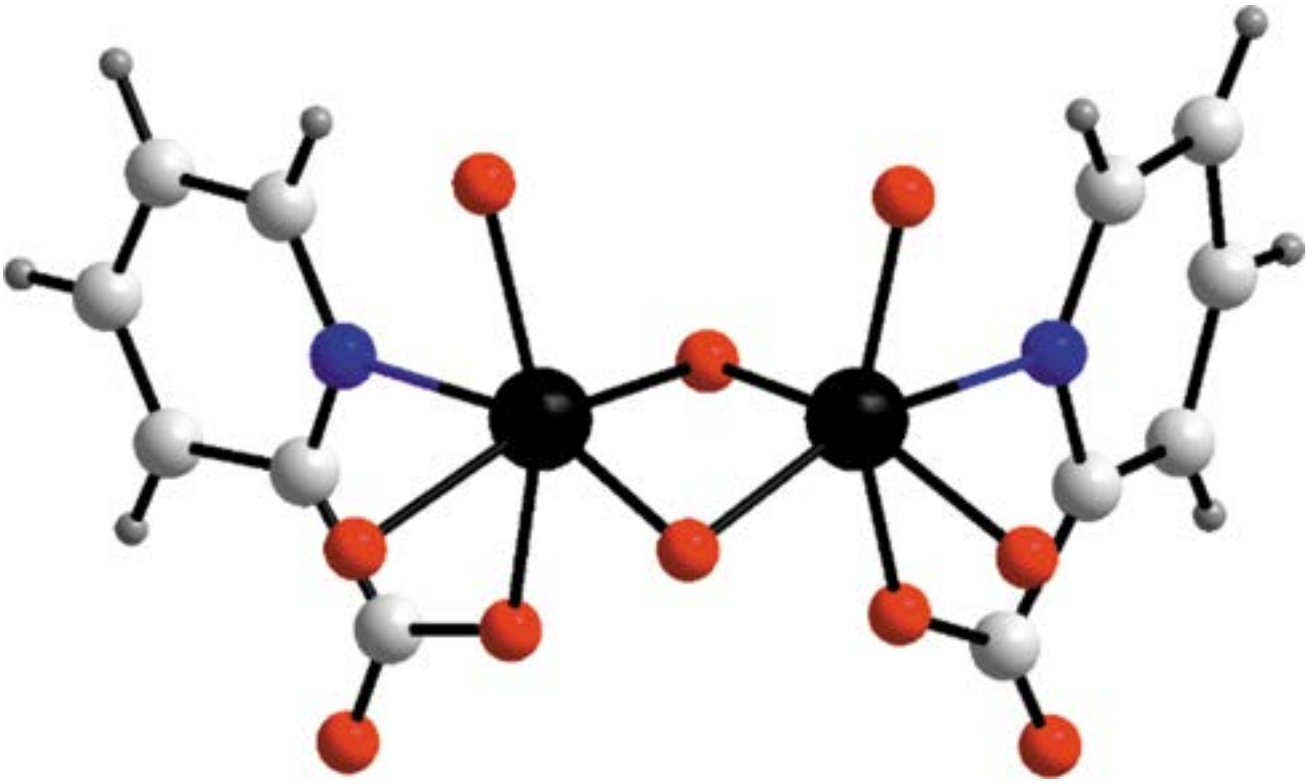


Figure 2. Ball-and-stick representation of the organofunctionalized $[Mo_2O_2(OH)_4(C_6H_4NO_2)_2]^{2+}$ unit acting as a charge-balancing dication for the Japanese rice-ball-shaped Molybdenum Blue $Na_4[Mo_2O_2(OH)_4(C_6H_4NO_2)_2]_2[Mo_{120}Ce_6O_{366}H_{12}(OH)_2(H_2O)_{76}]$~$200H_2O$. Colour code: Mo black, O red, C grey, N blue and H white.

To understand the fundamental principles of a multicomponent system driving the assembly of these aesthetically pleasing constructions, as well as the prospects for forming novel ones, is a major undertaking. Identifying novel steps in the formation process of mixed-valence polyoxomolybdates, such as an effect of a structure-directing ligand or implementing a unique functionalization of building blocks, will enable the construction of POM clusters with unprecedented sizes and topologies, thereby shedding light upon the mostly elusive self-assembly mechanisms, ultimately perhaps paving the way towards novel POM architectures with nuclearities surpassing the famous $Na_{48}[H_xMo_{368}O_{1032}(H_2O)_{240}(SO_4)_{48}]$~$1000H_2O$ (Mo_{368}) 'blue lemon' (Müller *et al*., 2002).

More than two decades separate the discovery of the 'Blues' and the 'Reds'. Is that the end of the molybdenum classes, or will the synthetic chemists have yet another new colour to enjoy?

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COMMENTARY

Water and 2,2,6,6-tetramethylpiperidine: an odd couple make a solid match

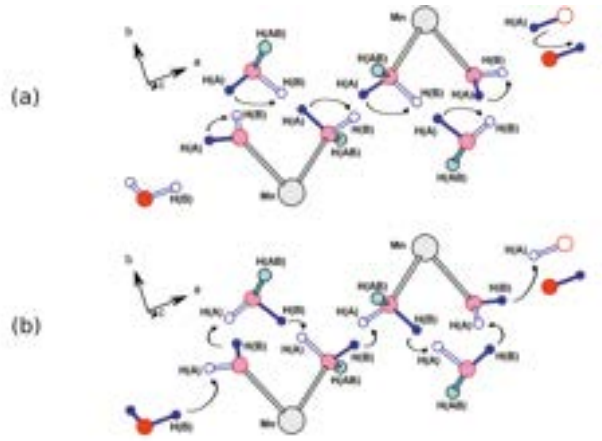


Figure 1. The two stages of the Grotthuss mechanism for proton transport. Atomic coordinates are from the neutron diffraction analysis of Capelli *et al.* (2013). (*a*) Reorientation of water molecules. (*b*) Proton hopping. See the text for a detailed description.

Larry R. Falvello

It is fair to state that in chemical research the least expected results can be the most interesting. In this issue of **IUCrJ**, Butler *et al*. (2022) report the discovery of a new molecular solid – a two-component crystal of water and 2,2,6,6-tetramethylpiperidine (TMP) – formed as a by-product during the course of a study on a different subject, namely the deprotonation of ferrocene derivatives using TMP as base. The new substance, which has a simple composition of TMP·2H_2O and is stable in a vacuum manifold but is degraded when exposed to air at room temperature, has a number of features that would not have been easy to predict.

The most prominent structural feature is the formation of what Butler *et al*. describe as a water pipe. The structure, which was analyzed by single-crystal X-ray and neutron diffraction at 100 K and by neutron diffraction at 10 K, has a central channel with TMP molecules at its periphery and with water molecules forming an inner lining of the channel walls. (Such a lining can be called a bushing in mechanical jargon, but that term has not often been applied to water-based channel linings in molecular crystals.)

The overall layout of the structure, as viewed in a projection along the channel axis, can be described as a rhombic distortion of a square-prismatic proto-structure. The water aggregate has a distorted cubic motif, which is stacked along the channel axis. The presence of latent symmetry, in which the aggregate structure has more or at least different symmetry than do the individual components, dovetails with the fact that the new solid forms only for a very specific proportion of the components – the H_2O:TMP molar ratio must be 2:1. When this odd couple of two quite different molecules do form the solid, though, they apparently do so readily, yielding centimetres-long rod-shaped crystals.

Further interest lies in the fact that both of the pure components are liquids at room temperature. This ‘liquid–liquid co-crystallization’ has been observed previously, but to my knowledge, it does not abound (Peloquin *et al*., 2020).

I might refer to the new solid as a co-crystal to reflect the observation that H_2O is a full structural partner and is not merely present as a dispensable solvate. However, that would risk venturing into the definition of a co-crystal, a complex subject that has been treated more fully by others (Bond, 2007; Parkin *et al*., 2008; Aitipamula *et al.*, 2012).

Beyond the existence of the channel lined by water is the suggestive fact that there is disorder among the sites of the water hydrogen atoms. This is where the use of single-crystal neutron diffraction is critical, since with this technique even half-occupied proton sites can be characterized accurately. The TMP2H2O structure has segments of hydrogen-bonded chains, which may be identified as proton wire fragments. A proton wire was the key structural element in a previous discovery by Capelli *et al*. (2013), in which it was proved by a deuterium substitution experiment that the protons along a disordered hydrogen-bonded chain of water molecules were mobile in crystals of a manganese coordination polymer.

The concept of a proton wire merits a brief description, because such a construct may exist in more crystals than those in which it has been explicitly identified as such. As described by Nagle & Morowitz (1978), a simple proton wire consists of a chain of hydrogen bonds along which proton transport can occur. The Grotthuss mechanism, a two-step "bucket-brigade" model, is invoked to explain the proton movement along the chain (Ädelroth, 2006; Cukierman, 2006).

The example reported by Capelli *et al.* (2013) has an unbounded proton wire and is an ideal example for discussion, since, as mentioned, that system was shown by experiment to transport protons. In that study, a neutron structure analysis revealed disorder between two stages of a putative Grotthuss proton transport mechanism. The disorder is shown in Fig. 1, which is based on the atomic coordinates from the neutron diffraction analysis by Capelli *et al*. (2013). The H(A) and H(B) sites are half-occupied and H(AB) are fully occupied. In Fig. 1(*a*) congener (A) is present, indicated by solid blue bonds and solid blue H atoms. The arrows indicate the concerted rotations that move protons from the H(A) to the H(B) sites in one stage of the Grotthuss mechanism. In Fig. 1(*b*), in which the H(B) sites are now occupied (solid blue bonds and atoms), the arrows represent proton hopping to nearby empty H(A) sites. This stage of the mechanism restores the original configuration shown in Fig. 1(*a*), except that each of the H(A) protons has moved to the next oxygen carrier atom along the chain.

The structured, extended water aggregate in TMP·2H_2O, supported by its walled TMP enclosure, may be capable of at least short-range proton transport. In addition to its structural features and possible dynamics, there is a lesson in the fact that the TMP·2H_2O crystal forms at all. This new and interesting solid was not the target of the research that produced it. It seems that nature has not exhausted its store of surprises, and we cannot know when our next unexpected observation will turn out to be an opening to a new avenue of exploration.

Acknowledgements

The author thanks Dr Milagros Tomás for incisive observations on the subject matter of this commentary.

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IUCR ACTIVITIES

Alex Stanley appointed IUCr Chief Executive Officer



As reported by Dr Hanna Dabkowska in her Letter from the President in this issue of the *IUCr Newsletter*, the IUCr has named Dr Alex Stanley as its first Chief Executive Officer. Dr Alex Stanley will succeed Dr Alex Ashcroft, IUCr Executive Secretary, who retires at the end of August 2022.

Alex Stanley holds a PhD in Chemical Crystallography from the University of Bristol, UK, and has a good understanding of many scientific fields related to structural science. She brings experience not only from a society level, having acted as the Bursaries Co-ordinator and Secretary for the British Crystallographic Association and as a UK delegate at the IUCr General Assembly, but also from an industrial level, having worked for Agilent Technologies, Oxford Diffraction and Rigaku. Her skill base is broad as she has experience in managing people, projects and budgets, in product marketing, building a brand identity, and in governance at committee and corporate levels.

The long handover period will help in the transfer of this complex leadership role and will mean that both Alexes will attend the IUCr Executive and Finance Committee meetings in Versailles, France, in August, overlapping with ECM33.

Dr Dabkowska commented: "*I am thrilled that Dr Stanley is joining our ranks. I sincerely believe that this international organisation will achieve new heights under her leadership. We are a strong team of people very proud of the job they are doing. We are happy to have Alex in control and I trust she will guide us towards many new future achievements.”*

Dr Stanley commented: "*I am excited to join the IUCr whose core values of community, integrity and passion for promoting high-quality structural science are so very evident. I look forward to building on the strong foundations, put in place by Alex Ashcroft and his predecessor Mike Dacombe, to support this well-regarded organisation.*"

Dr Ashcroft commented: "*I am delighted that Alex has been appointed, and that the IUCr is in safe hands.*"

Dr Stanley can be contacted at ceo@iucr.org.

IUCR CONGRESS

Bragg Your Pattern



Stuart Batten

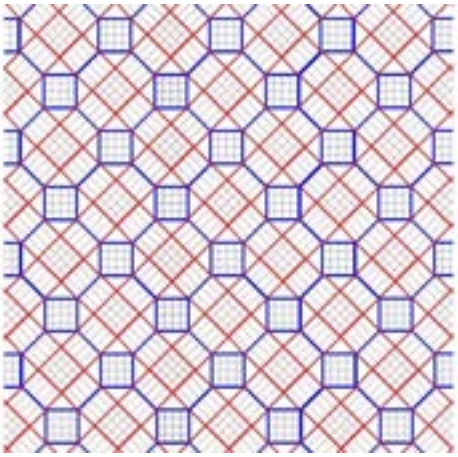
In the lead-up to the 2023 IUCr Congress, to be held in Melbourne, Australia (22–29 August 2023), the local organising committee will be embarking on a series of public outreach activities. The first of these is a pattern competition for kids of ALL ages.

The competition will open at the start of Australia's National Science Week (13–21 August 2022), and there will be two age brackets (10 and under; 11 and over). In addition, there will also be two competition categories.

The first category is "I See a Pattern". Patterns are all around us. See a cool or unusual pattern? Take a photo and enter it with a description. The second category is "I Made a Pattern". Make your own pattern – a 2D drawing, a 3D model or artwork, whatever – and send in a photo and description of it. Simple! Great prizes are on offer, and the top 100 images will also be exhibited at the IUCr2023 Congress itself.

You can also contribute by following along and contributing on social media (Twitter, Instagram) using the tag #BraggYourPattern, voting in the People's Choice awards when the competition opens, and following Everyday Crystallography (@Crystal_a_day) on Twitter for more Bragg Your Pattern goodness. Full competition details are on our website (braggyourpattern.com), which is also the place to go for hints and eventually full details of other exciting activities coming up in 2023!





IUCR CONGRESS

IUCr 2023: meet the team this summer (in the northern hemisphere)



The IUCr 2023 team will be hosting a booth at both ACA2022 in Portland, OR, USA, from 29 July to 3 August 2022 and ECM33 in Versailles, France, from 23 to 27 August 2022. Find out about the arrangements for the Congress and about Melbourne and the Melbourne Convention and Exhibition Centre; you may even win a prize!

Other news

The IUCr 2023 International Program Committee has announced the list of Microsymposia that will be included in the Congress program.

Sponsorship opportunities are now available.

Key dates

Call to action

Date

Call for abstracts open

23 August 2022

Bursary applications open

23 August 2022

Early bird registration open

23 August 2022

Call for abstracts close

21 November 2022

Bursary applications close

21 November 2022

Notification of abstract acceptance

1 February 2023

Early bird registration deadline

15 February 2023

Deadline for presenters to register

15 February 2023

Accommodation booking deadline

22 July 2023

IUCr 2023 pre-Congress workshops

21 & 22 August 2023

IUCr 2023 Congress

23–29 August 2023

To sign up for IUCr 2023 notifications, please go here. IUCr 2023 posters for printing or electronic circulation to your colleagues are available here. Thank you!



IUCR ACTIVITIES

Call for nominations for the thirteenth Ewald Prize



The IUCr invites your nominations for the Ewald Prize for outstanding contributions to the science of crystallography. The Prize is named after Paul P. Ewald, in recognition of his significant contributions to the foundations of crystallography and to the founding of the IUCr. Professor Ewald was the President of the Provisional International Crystallographic Committee from 1946 to 1948, the first editor of the IUCr journal *Acta Crystallographica* from 1948 to 1959 and the President of the IUCr from 1960 to 1963.

The Prize is presented once every three years during the triennial International Congresses of Crystallography and consists of a medal, a certificate and a financial award. The recipients to date are as follows:

Year

Place

Recipient(s)

1987

Perth, Australia

Professor J. M. Cowley and Dr A. F. Moodie

1990

Bordeaux, France

Professor B. K. Vainshtein

1993

Beijing, China

Professor N. Kato

1996

Seattle, WA, USA

Professor M. G. Rossmann

1999

Glasgow, UK

Professor G. N. Ramachandran

2002

Geneva, Switzerland

Professor M. M. Woolfson

2005

Florence, Italy

Professor P. Coppens

2008

Osaka, Japan

Dr D. Sayre

2011

Madrid, Spain

Professor E. Dodson, Professor C. Giacovazzo and Professor G. M. Sheldrick

2014

Montreal, Canada

Professor A. Janner and Professor T. W. J. M. Janssen

2017

Hyderabad, India

Professor T. L. Blundell

2021

Prague, Czech Republic

Dr Olga Kennard

The thirteenth Prize, for which nominations are now being invited, will be presented at the Melbourne Congress in August 2023.

Scientists who have made contributions of exceptional distinction to the science of crystallography are eligible for the Prize, irrespective of gender, nationality, age or experience. The Selection Committee will give careful attention to the nominations of outstanding scientists who have not yet won a Nobel Prize. Either an exceptionally distinguished scientific career or a major scientific accomplishment may be recognised. Current members of the Selection Committee and the President of the IUCr are not eligible. No restrictions are placed on the time or the means of publication of the nominee's contributions. The prize may be shared by more than one contributor, but no more than three, to the same scientific achievement.

Nominations for the thirteenth Ewald Prize should be submitted electronically using the Ewald Prize Nomination Form to the IUCr Chief Executive Officer at ceo@iucr.org. The closing date for nominations is **30 September 2022.**

IUCR ACTIVITIES

Call for nominations for the second W. H. & W. L. Bragg Prize



James Fraser (left) and Jean-Philippe Julien, winners of the inaugural W. H. and W. L. Bragg Prize for outstanding early-career crystallographers.

The IUCr is pleased to invite nominations for the second W. H. & W. L. Bragg Prize for outstanding contributions to the science of crystallography. The Prize is named after Professors W. H. and W. L. Bragg, in recognition of their significant contributions to the foundations of crystallography. Professors William Henry and William Lawrence Bragg pioneered the use of X-rays as an instrument for the analysis of crystal structure, and were the joint recipients of the 1915 Nobel Prize for Physics.

The Prize consists of a certificate and a financial award, and is presented once every three years during the triennial International Congresses of Crystallography. The inaugural Prize was awarded in Prague, Czech Republic, in 2021 to Professor James Fraser (USA) and Dr Jean-Philippe Julien (Canada). The second edition of this Prize, for which nominations are now being invited, will be presented at the Melbourne Congress in August 2023.

Unlike the Ewald Prize, the W. H. & W. L. Bragg Prize is awarded to a scientist in the early stages of their career, irrespective of gender, nationality or age. Nominees will be expected to have registered in the World Directory of Crystallographers.

Nominations for the second W. H. & W. L. Bragg Prize should be submitted electronically using the Bragg Prize Nomination Form to the IUCr Chief Executive Officer at ceo@iucr.org. The closing date for nominations is **30 September 2022**.

IUCR ACTIVITIES

Call for nominations for the 2023 Struchkov Prize



Yuri T. Struchkov (photo contributed to the IUCr gallery by Leonid Aslanov).

The IUCr is pleased to invite nominations for the 2023 Struchkov Prize. Professor Yuri T. Struchkov (1926–1995) was an outstanding Russian crystallographer who made substantial contributions to the structural chemistry of organic and organometallic compounds. To commemorate Professor Struchkov's achievements, his friends and former colleagues established the Struchkov Prize in 1997. Between 1997 and 2020, the Prize was awarded annually to a young (<35 years) scientist from the Former Soviet Union (FSU) for the best research work in the field of X-ray crystallography.

IUCr support and selection

In 2020, the Struchkov Prize Association asked the IUCr to administer the Prize to ensure its longevity. From now on, the Prize will be bestowed by the IUCr every triennium at the IUCr Congress and General Assembly beginning in Melbourne in 2023. The Association has endowed a Prize Fund. Any interest earned on the Fund, along with any donations received during the triennium, will be split equally between the winners. The principal of the Fund can never be used to supplement the Prize. The IUCr will select a committee that will consider nominations and determine the winners.

Eligibility and application process

The Struchkov Prize will be awarded to up to three young scientists (under 35 years of age at the time of the submission deadline) at each Congress for outstanding achievements in small-molecule X-ray diffraction methods application in the fields of chemistry, crystal chemistry or materials science.

Applicants for the 2023 competition may be nominated or self-nominate. The following information should be submitted:

Participant’s information:
(a) Full name
(b) Title of the submitted research work
(c) Date of birth
(d) Education: college name, year of graduation
(e) Affiliation: name of the institution
(f) Academic degree, current position
(g) Address for correspondence by mail
(h) Phone number

(i) Email address

Summary of the work up to three pages in length (Times New Roman, 12 points, spacing 1.5). The summary should emphasise scientific impact and personal contribution of the applicant.

A list of the applicant’s publications on the topic of the presented research.

Full texts of all publications listed in item 3 in the form of PDF files.

30 September 2022.

ceo@iucr.org. The closing date for nominations is

One or two supporting letters (no more than two). Each letter may not exceed two pages in length.

By submitting an application, the applicants agree to provide the submitted content to the organising committee and members of the jury. Applications should be submitted electronically to the IUCr Chief Executive Officer at

CRYSTALLOGRAPHY IN AFRICA

First OpenLab in Brazzaville, Republic of Congo: Utilization of diffractometers in remote mode for the first time



Loufoulakari Falls, a Congo River tributary.

Ferland Ngoro-Elenga, Patrice Kenfack Tsobnang, Joseph-Marie Saint-Bastia Moutou, Claude Lecomte

The first OpenLab on Crystallography using remote diffraction facilities was held in Brazzaville, Republic of Congo, from 11 to 22 May 2022 at the Marien Ngouabi University under the auspices of the IUCr Africa Initiative and the African Crystallographic Association (AfCA). This OpenLab was organized by the Congolese Association of Crystallography (ACCr) in partnership with Marien Ngouabi University, the Geological and Mining Research Center (CRGM) of Brazzaville, the CRM2 laboratory (CNRS/Université de Lorraine, France) with financial support from the IUCr, the European Crystallographic Association (ECA) and the National Petroleum Society of Congo (SNPC). The organizing committee of this event was chaired by Professor Joseph-Marie Saint-Bastia Moutou and Dr Ferland Ngoro-Elenga, President and General Secretary of the Congolese Association of Crystallography (ACCr), respectively.

This event was attended by 28 young academics and PhD students in Chemistry, Geology and Physics from Republic of Congo, Democratic Republic of Congo and Gabon. The unique aspect of this OpenLab was the realization, for the first time, of remote X-ray diffraction measurements thanks to a partnership with the Plateforme de mesures de diffraction et de diffusion des rayons X (PMD2X) of the Université de Lorraine. Three African lecturers, Drs Patrice Kenfack Tsobnang (University of Dschang, Cameroon), Adam Bouraima (Université des Sciences et Techniques of Masuku, Gabon) and Yvon Bibila Mayaya Bisseyou (University Houphouët Boigny, Ivory Coast, and Marien Ngouabi University, Congo), taught on site with Professor Claude Lecomte (Université de Lorraine). Drs Florence Porcher, Emmanuel Wenger, El-Eulmi Bendeif and Pierrick Durand, all from the Université de Lorraine, connected remotely.



Participants in the first OpenLab in Brazzaville.

The OpenLab began with an inaugural lecture given by Professor Lecomte on the theme "Crystallography: an essential science at the crossroads of Physics, Chemistry, Geology and Life Sciences". The lecture took place in the auditorium of the main Library of the Marien Ngouabi University in the presence of the authorities of the Marien Ngouabi University, several professors of the Faculties of the University and of the Denis Sassou-N'Guesso University of Kintélé, and all the participants of the OpenLab. This activity was covered by the national media and ended with exchanges between Professor Lecomte and the audience on the one hand and on the other hand between Professors Moutou and Lecomte (respectively, president of the organizing committee and the scientific committee of this OpenLab) with the written, oral press and TV. The event was the subject of an article in the daily newspaper of the Congolese Information Agency of the Ministry of Communication and Media of the Republic of Congo.



Participants listening to the inaugural lecture given by Professor Claude Lecomte during the first crystallography OpenLab in the Republic of Congo.

After this opening ceremony, theoretical courses and tutorial sessions were carried out. These courses focused on crystal lattices, crystallographic calculations, point groups, space groups, the *International Tables of Crystallography*, Fourier series and transforms, X-ray scattering by atoms and molecules and

diffraction by crystals, introduction to single-crystal and powder diffraction, and resolution and refinements of crystal structures from single-crystal and powder diffraction data.



Attendees listening to the course given by Claude Lecomte.

After 3.5 days of lectures and tutorials, practical activities were carried out for 5 more days. These activities focused on the remote use in Brazzaville of the CRM2 Bruker D8 Venture single-crystal and Panalytical X'pert PRO powder diffractometers. During these hands-on activities, the attendees discussed and proposed data collection strategies; then, data reduction was critically made. Data from the single-crystal diffraction measurements were used to determine crystal structures.

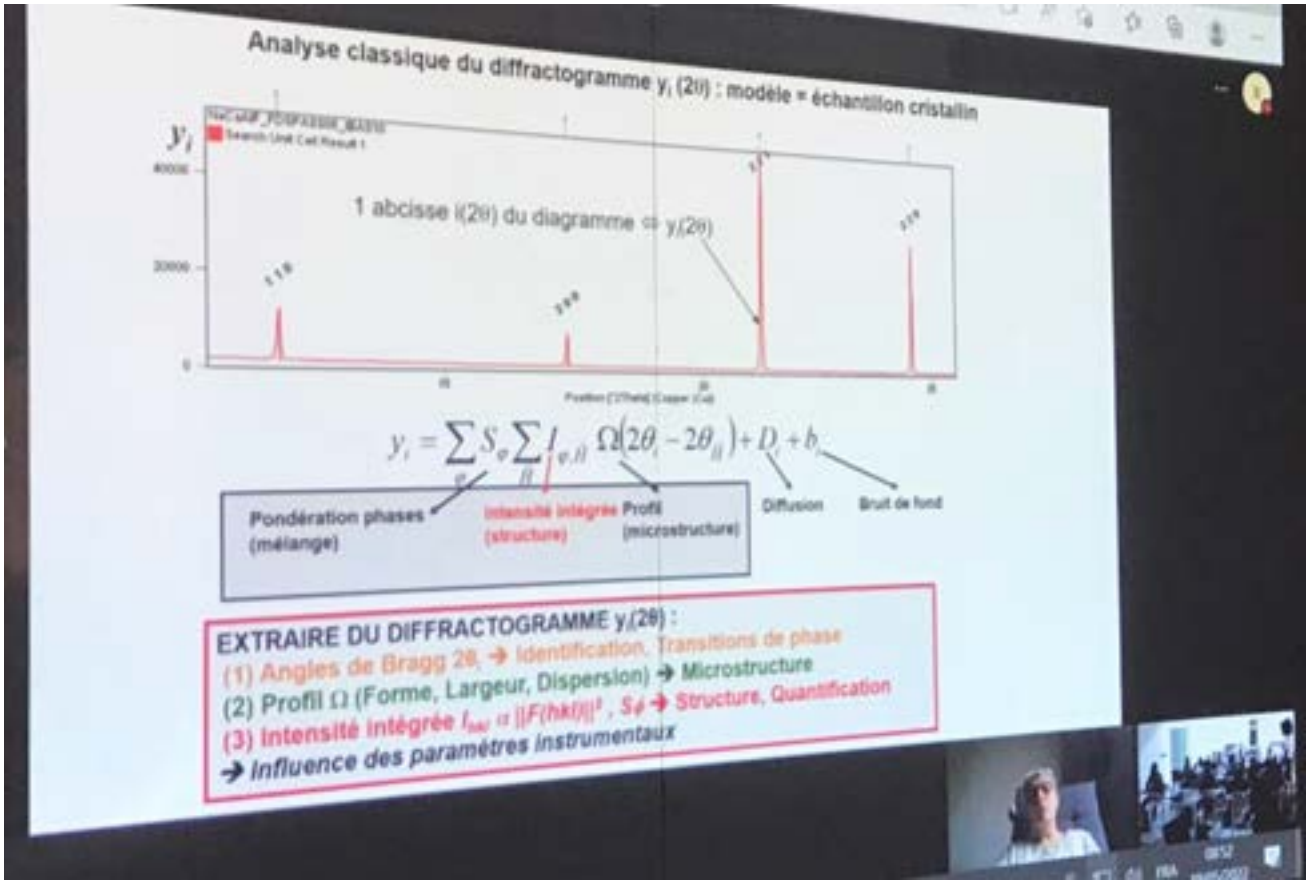
Crystals of phases I and II of paracetamol were used for application. Thanks to the cameras installed in the CRM2 diffractometer rooms in Nancy, Dr Wenger, in remote mode, showed the participants how to select and mount a crystal, put it on the goniometer head and center the crystal on the diffractometer. Then, Dr Kenfack Tsobnang, on-site in Brazzaville, controlled the diffractometer and showed the attendees how to perform the measurements.



E. Wenger centering a crystal of one phase of paracetamol at CRM2 and Kenfack Tsobnang controlling the diffractometer from the Brazzaville teaching room.

After diffraction data collection and processing, the participants solved the crystal structures of the two paracetamol phases under the supervision of the teachers present on site. *WINGX* was used for this purpose with a special emphasis on *SHELXS*, *SHELXL*, *ORTEP* and *Mercury* software. The output files were thoroughly discussed and explained to the participants and the CIFs were reviewed. All participants solved and refined the structures of the monoclinic and orthorhombic phases of paracetamol.

For powder diffraction, the activities focused on the measurements of samples of pure paracetamol and of the drug 'doliprane for children'. The analysis was performed using *Qualx* software and the Crystallographic Open Database (COD). A Rietveld refinement was done on 'doliprane for children' with *Fullprof* software under the remote supervision of Dr Porcher, assisted by the teachers present on site.



Dr Florence Porcher remotely teaching the course on processing powder X-ray patterns.

After these hands-on activities on the determination of the crystal structures of phases I and II of paracetamol (with single-crystal measurements) and the phase analysis of the pure paracetamol followed by the quantification of phases in 'doliprane for children', lectures on crystallographic databases, in particular the Cambridge Structural Database (CSD), were given. Practical activities with the database were carried out and the Frank H. Allen International Research and Education (FAIRE) Program was presented.

At the end of the training, the attendees participated in a general discussion on using crystal structures to answer basic questions in research. Drs Kenfack Tsobnang, Bibila Mayaya Bisseyou and Adam Bouraima, and Professor Lecomte, used some of their research works to teach this course. The course ended with a critical reading of some papers published in *Acta Crystallographica* reporting crystal structures.



Some participants with their certificates.

On Sunday during the OpenLab, the organizing committee offered participants an opportunity to relax at the Loufoulakari Falls on a tributary of the Congo River.



Sunday relaxing of participants at the Loufoulakari Falls.

This OpenLab shows that measurements in remote mode can be carried out successfully and easily anywhere. These remote measurements, in the unanimous opinion of the attendees, have undeniably contributed to the success of this Brazzaville OpenLab and opened the way for the future.

REGIONAL NEWS

ACA celebrates science diversity with a brand new logo

Thomas Proffen

The American Crystallographic Association (ACA) has a long history of being a home to all structural science disciplines. In 2021 the ACA council voted to add “The Structural Science Society” as a tag line and a committee was formed to create a new logo replacing the original one created by Helen Berman over 35 years ago. After a lot of hard work by the logo committee, the new logo was unveiled in December 2021 celebrating the breadth of science the ACA represents.

Personally I fully agree with this Cora Lind-Kovacs quote: “*I am excited to add “The Structural Science Society” to our logo. Crystallography has played, and continues to play, a very important role in structure determination. However, other methods have advanced significantly in recent years, and techniques like NMR or cryo-EM also have made many important contributions to structure determination. As we continue to push for new frontiers, combining data obtained by such methods with traditional crystallographic diffraction or other scattering techniques holds tremendous potential, and the addition of “The Structural Science Society” shows that the ACA is committed to providing a place that brings together all scientists that are pursuing atomic level structural analysis.*”

Learn more about the new logo here.

MEETING REPORT (IUCR SUPPORTED)

ePCCr: Pan-African Conference on Crystallography Online



Gift Mehlana, Susan Bourne, Dickson Andala, David Dodoo-Arhin, Delia Haynes, Patrice Kenfack Tsobnang

The first Pan-African Conference on Crystallography, PCCr1, was held in Dschang, Cameroon, in 2016. This highly successful meeting attracted 192 participants from 32 countries, including 20 African countries. PCCr2 followed in 2019, with over 200 participants from 35 countries. This was a joint meeting with the African Light Source (AfLS), and was hosted in Accra, Ghana. PCCr3, which will take place in Nairobi, Kenya, was scheduled for 2021. Unfortunately, due to the pandemic, this meeting had to be postponed. The Steering Committee of the African Crystallographic Association, along with the Local Organizing Committee of PCCr3, decided to hold an online meeting in order to continue the momentum and sense of community generated during the first two PCCr meetings. ePCCr was therefore organized.

The theme of ePCCr was '*Crystallography, an interdisciplinary science for the Africa of the future*'. Submissions were invited in the following thematic areas:

Crystallography Databases

Inorganic Materials and Industrial Minerals

Crystal Engineering and Structural Chemistry

Crystallography and Life Sciences

Large Facilities for Emerging Countries

Diffraction Physics and Phase Transitions.

The meeting was held entirely online *via Zoom*. The conference ran from 15 to 19 November, 10:00–17:00 Central African Time.

ePCCr organising committee

Gift Mehlana (Chair), Midlands State University, Zimbabwe
Susan Bourne (Secretary), University of Cape Town, South Africa
Dickson Andala, Multimedia University of Kenya, Kenya
David Dodoo-Arhin, University of Ghana, Ghana
Delia Haynes, Stellenbosch University, South Africa
Patrice Kenfack Tsobnang, University of Dschang, Cameroon

Joint meeting

The ePCCr was organized as a joint meeting together with the AfLS and the African Physical Society (AfPS). There is significant overlap of interest between these three groups, as well as overlap of communities. There was also a group of ten co-convenors, also with overlap of interests (Fig. 1).

The joint event was hosted by X-Tech Lab in Benin. Each day had two plenary sessions that were common to all delegates. The Opening and Closing Ceremonies were also organized jointly by ePCCr, AfIS and AfPS.

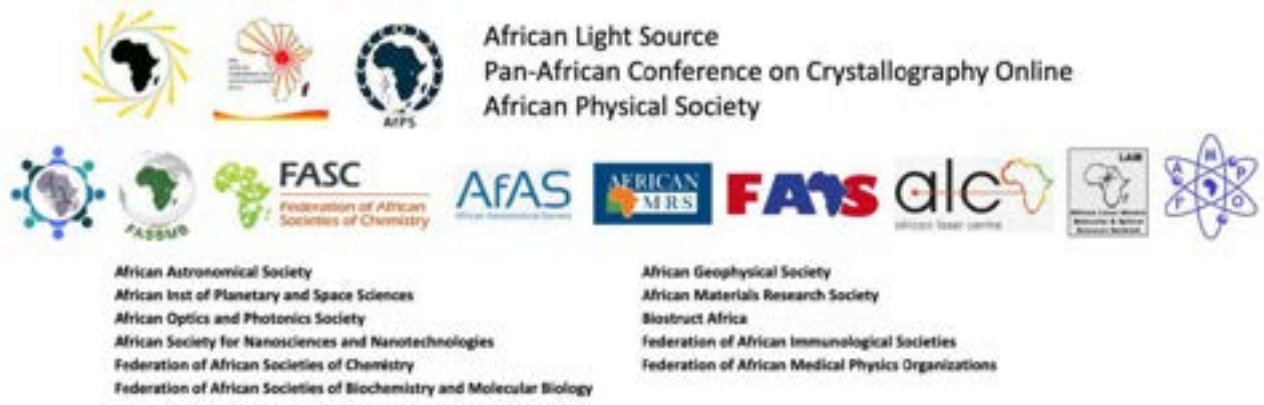


Figure 1. The companion Pan-African Conferences and the co-convenor organisations that worked together for the joint event.

Delegates

The joint meeting had a total number of 554 registered delegates. Of these, 208 delegates were specifically registered for the ePCCr meeting.

A total of 42 nationalities were represented at ePCCr, with delegates affiliated with institutions in 39 countries. The largest numbers of delegates were affiliated with institutes in South Africa (41), Cameroon (26), Nigeria (13), the UK (13) and France (11). The registered delegates were 59% male, 36% female and the remaining 5% did not specify a gender. One hundred and six delegates were academics, 81 were students, 15 from industry and 6 registered as observers.

Scientific sessions

The ePCCr was characterized by great talks from plenary lectures to keynote addresses. These talks covered aspects of crystal engineering, inorganic materials and structural chemistry and biology where crystallographic tools have been applied in the characterization of different materials that are used in our everyday lives. Plenary lectures and keynote addresses were presented by scientists from Africa and other parts of the world.

Polymorphism was a key feature in some of the talks that were presented by renowned and emerging scientists. Professor Susan Reutzel-Edens (CCDC, Cambridge, UK) had an inspiring plenary talk on *Inspiring Medicines Design Through Solid State Chemistry* (Fig. 2). In her presentation she highlighted methods for identifying drug polymorphs using computational and experimental methods. This was a well-timed presentation given the fact that the world is facing pandemics that need to be eradicated through developing new drugs and vaccines. Professor Bill Jones (University of Cambridge, UK) gave a presentation on *Crystallization methods and disappearing polymorphs*. This further reinforced the importance of understanding the occurrence of polymorphic materials. This knowledge is very useful especially in pharmaceutical industries involved in the manufacture of drugs. In a keynote address Dr Eunice Nyawade (Jomo Kenyatta University of Agriculture and Technology, Kenya) stressed the importance of single-crystal X-ray diffraction in characterization of polymorphic ruthenium complexes. Dr Tetteh Samuel (University of Cape Coast, Ghana) showed how polymorphism could be predicted using the Cambridge Structural Database (CSD). His presentation acted as a forerunner to the CSD workshop, for which 58 delegates registered, that looked at structural visualization with *Mercury* and searching the

CSD using *ConQuest*. This workshop was attended by students and early-career researchers.

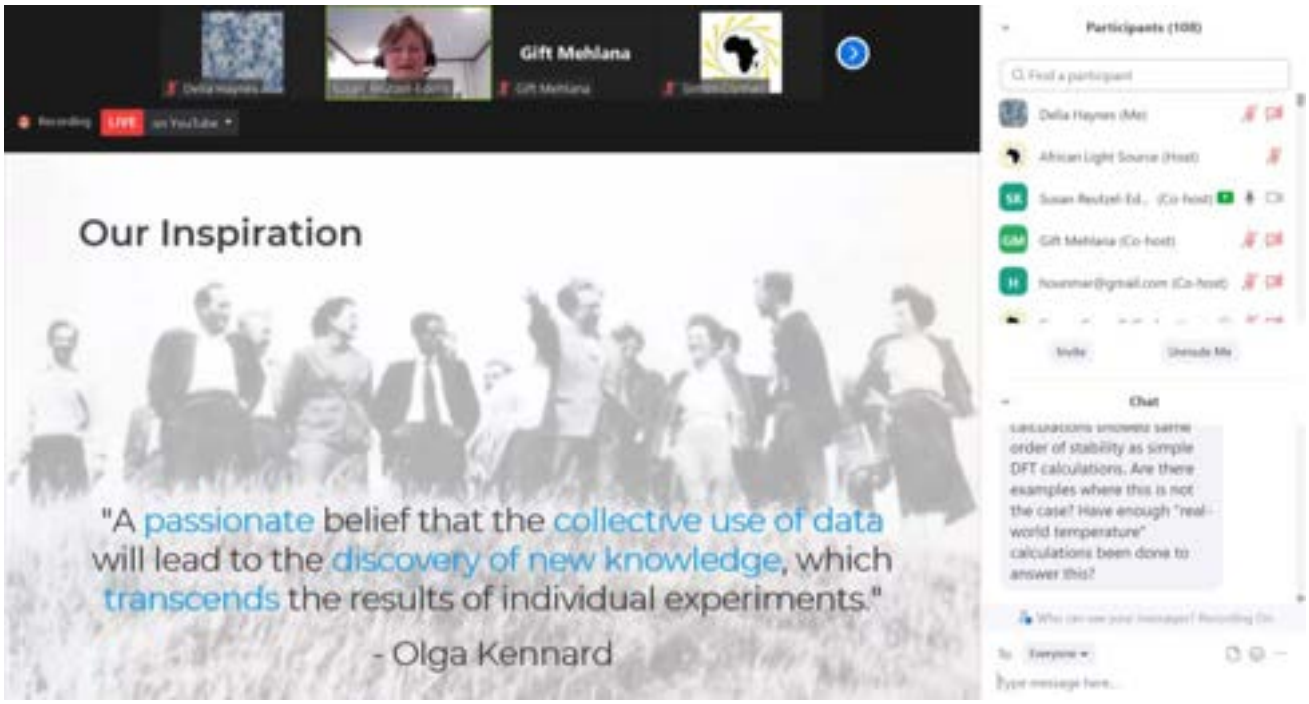


Figure 2. A screenshot from Susan Reutzel-Edens' presentation.

A plenary lecture on protein crystallography was presented by Professor Richard Garratt (University of São Paulo, Brazil). He spoke about unravelling the complexities of septin filaments using crystallography. He also spoke about the importance of infrastructure development in advancing science in Brazil, particularly in terms of the construction of the first synchrotron in Latin America, and how this has changed science in the region. Another excellent plenary lecture was given by Professor Seham Abdel-Aal (Cairo University, Egypt) who presented her work on hybrid perovskite materials and their properties.

Metal–organic frameworks (MOFs) are a topical issue and as such their research is moving fast in directions as diverse as indoor climate and biotechnology. This was evidenced by keynote addresses by Professor Neil Champness (University of Birmingham, UK) and Dr Tendai Gadzikwa (Kansas State University, USA). The importance of these new materials in gas storage to catalysis was also highlighted. Dr Gadzikwa outlined how these materials can be used as scaffolds for enzyme-inspired catalysis paving the way for their potential application in biotechnology. In a related presentation, Professor Lars Öhrström (Chalmers University of Technology, Stockholm, Sweden) explained how MOFs can be made by design and how their structures could be reduced to simple nets, which we read about everyday in general chemistry textbooks. He further explained how MOF research has completely changed the way we look at the solid state, what we can do with it, how we can investigate it and how it pushes the limits of experimental techniques. Cyrielle Dazem Fogou (University of Yaoundé, Cameroon) briefly presented topological analysis in metal oxalates. Both Dazem Fogou and Öhrström explained the importance of topological analysis in the context of solving structures from powder X-ray diffraction data, and how success has been achieved with covalent organic frameworks. Other presentations in the MOF area included structural transformations mediated by solvents. These talks were presented by Dr Christophe Ndamyabera and Alan Eaby of the University of Cape Town and Stellenbosch University, respectively.

Large facilities and presentations from our sponsors

During the conference, several instrument manufacturers had the opportunity to showcase their products. Vernon Smith of Bruker, James Gordon of Rigaku and Marcus Mueller of Dectris presented on some of the instruments useful in material characterization, with a focus on crystallographic-oriented research. Dr Tom Blanton of ICDD presented on material characterization using powder diffraction techniques and the Powder Diffraction File (PDF). By using the PDF, it is possible to identify all phases that are present in a material making the PDF an invaluable tool in mining and pharmaceutical industries.

ePCCr had a total of nine invited lectures (three plenary lectures and six keynote lectures). Five of the invited speakers were African, with three currently based in Africa. Four of the nine invited speakers were female. There were an additional 20 talks selected from submitted abstracts, of which nine were given by students or young researchers, and six by female delegates. An additional five presentations were given by sponsors.

ePCCr invited speakers

Dr Susan Reutzel Edens, CCDC, UK
Professor Seham Abdel-Aal, Cairo University, Egypt
Professor Neil Champness, University of Birmingham, UK
Professor Bill Jones, University of Cambridge, UK
Dr Emile Engel, KTH Royal Institute of Technology, Sweden
Dr Tendai Gadzikwa, Kansas State University, USA
Professor Richard Garratt, University of São Paulo, Brazil
Dr Eunice Nyawade, Jomo Kenyatta University of Agriculture and Technology, Kenya
Professor Bernard Omondi Owaga, University of KwaZulu-Natal, South Africa

The ePCCr also involved several lively poster sessions, which were held online in *Gather Town*. A total of 26 posters were accepted.

Thanks to generous sponsorship, six poster prizes and three speaker prizes were awarded. The winners of poster and speaker prizes, selected from participants in the ePCCr conference by a panel of judges, were announced at the conference closing ceremony. All prize winners received a certificate and a USD200 prize.

Launch of AfCA

A special session was organized on the Wednesday morning to constitute the African Crystallographic Association. The session involved presentations on the history leading up to the formation of an African Crystallographic Association, as well as addresses by representatives from the IUCr, the European Crystallographic Association, the International Science Council and UNESCO. This session was very well attended by delegates as well as observers. See the session report here.

Summary

The ePCCr successfully brought together the crystallography community in Africa, showcasing African research in crystallography and enabling networking with the worldwide crystallography community. The launch of AfCA was a highlight of the meeting and could easily be attended by people from many countries due to the online format. Working together with AfLS and AfPS was of great value – our communities have much in common and all benefitted from the interaction between the various groups. The PCCr meetings are now well established as a conference series for the African scientific community.

MEETING REPORT

CCrS: Conference of the Chinese Crystallographic Society 2021



View of the Tengwang Pavilion and the modern skyscrapers in Nanchang city by night.

Nan Zhang

The 8th Annual Academic Conference of the Chinese Crystallographic Society (CCrS) and the 7th National Members' Meeting took place last year (21–23 October 2021) in Nanchang, Jiangxi Province. Nanchang is located in southeastern China, which is around 727 km north of Hongkong and about the same distance southwest of Shanghai. Nanchang has a history dating back over 2000 years and became an administrative capital of the local area in 201 BC. The famous Tengwang Pavilion (滕王阁) is located in the city, which is well known nationally because of the classic poem *Tengwang Ge Xu* (滕王阁序). More than 700 participants were able to attend the conference on-site, and more than 100 domestic and international participants attended the conference online because of pandemic travel restrictions.



Tengwang Pavilion in Nanchang.

The conference was jointly chaired by Professor Rao Zihe, academic of the Chinese Academy of Sciences and Professor of Nankai University and Tsinghua University; and Professor Gao Song, academic of the Chinese Academy of Sciences, member of the Academy of Sciences for Developing Countries and Professor of Peking University and South China University of Technology. The theme of the conference was 'Prosperity with Crystals'.



The audience during the opening ceremony of the 8th Annual Academic Conference of the Chinese Crystallographic Society.

There were 11 conference keynote speeches and 12 sessions, which included Macromolecular Crystallography, Functional Molecular Crystal Length and Characterization (sessions 1 and 2), the 8th National Symposium on Crystalline Drug Development Technology, Small Angle Scattering, Polycrystal (Powder) Diffraction, Crystalline Materials under Extreme Conditions, Functional Crystal Growth and Characterization and Crystal Applications in Industries, PDF-Neutron Scattering Technology and

Applications, Electron Microscopy, Crystallography Education Forum and Youth Committee Topics. The special committees discussed the latest advances in related fields extensively at home and abroad, and the Youth Committee made a preliminary selection of the latest batch of Youth Talents. More than 100 posters were presented at the conference, and 15 awards for the best posters were selected. These awards were sponsored by the Royal Society of Chemistry *Dalton Transactions*.



Keynote lectures delivered during the conference.

The 7th National Members' Meeting of the Chinese Crystallographic Society was held after the opening ceremony of the conference. Secretary General Sun Junliang presided over the meeting. The Chair of the Society, Su Xiaodong, gave a report on the work of the Sixth Council. Professor Mai Zhenhong gave a report on the First Supervisory Board of the Chinese Crystallographic Society. Then, a new Council of 98 members and a new Supervisory Board of 7 members were elected. At the same time, the revised constitution of the Chinese Crystallographic Society and a new membership fee were considered and adopted. The first meeting of the new Council was held on 22 October 2021. Academic of the Chinese Academy of Sciences Hong Maochun was elected Chair of the 7th Council of the Chinese Crystallographic Society, and Guo Guocong, Su Chengyong, Hu Zhanggui, Liao Libing, Du Guanhua, Sun Junliang, Lai Luhua and Jin Changqing were elected Vice Chairs. Chen Jianzhong was elected Chair of the Second Supervisory Board, and Sun Junliang was appointed Secretary General of the Society.

To provide some history of the organization of crystallographic sciences in China, the foregoing organization of the Chinese Crystallographic Society (CCrS) was the Chinese Committee of the IUCr, which joined the IUCr in 1978. The Chinese crystallographer Tang Youqi was one of the first members of the Chinese Committee who conducted early international communication with the world. He was elected a member of the 13th Executive Committee of the IUCr in 1984 and was elected as the 14th Vice President of the IUCr in 1987. The Society organized the 16th Congress and General Assembly of the IUCr in Beijing in 1993. The establishment of the current Chinese Crystallographic Society was approved by the State Science and Technology Commission on 30 March 1993. The 1st Members' Meeting was held on 31 May 1994, and the Society was formally established and affiliated with Beijing University. The last conference was held in September 2018 in Tianjin.

MEETING REPORT (IUCR SUPPORTED)

17th European Powder Diffraction Conference – EPDIC17



Participants of EPDIC17.

Jasminka Popović, Aleksandar Višnjevac, Ana Šantić

The 17th European Powder Diffraction Conference (EPDIC17) organized by the Croatian Association of Crystallographers was held as an in-person conference from 31 May to 3 June 2022 in the Convention Centre Šibenik, Croatia. The Conference was generously supported by a number of sponsors: Bruker AXS and Malvern Panalytical (*Diamond sponsors*); Anton Paar, ICDD and Rigaku (*Platinum sponsors*); STOE and PROTO X-ray diffraction (*Gold sponsors*), as well as many other sponsors and supporters: Huber, ThermoFisher, Dectris, X Spectrum, Eldico Scientific, Oxford Cryosystems, Excelsus, Crystal Impact, IUCr, European Crystallographic Association (ECA), Croatian Academy of Science and Arts (HAZU), Fidelta, Faculty of Science, University of Zagreb and Ruđer Bošković Institute. Prior to EPDIC17, two satellite events took place: TOPAS Intensive Course from 29 to 31 May and ICDD Workshop on 31 May 2022.

EPDIC17 gathered 303 participants from 28 countries. Thirty-four young participants (PhD students and postdoctoral fellows) received support to attend EPDIC17: 25 were awarded IUCr Young Scientist Awards and 5 received ECA bursaries. All of them, along with another four young participants, received additional support funded by the Croatian Association of Crystallographers.

The scientific programme contained 2 award lectures, 6 plenary lectures, 26 keynote lectures, 40 oral presentations and 147 poster presentations organised into 12 thematic microsymposia. In the Exhibition area, 13 companies presented their products and services.

Programme

The Conference was opened on Tuesday, 31 May 2022 with a welcoming word from Dr Jasminka Popović (Chair of EPDIC17), Professor Paolo Scardi (Chair of the EPDIC Committee), Dr Aleksandar Višnjevac (President of the Croatian Association of Crystallographers), Dr Marko Jelić (Governor of the Šibenik and Knin County) and Mr Stjepan Prugovečki (representative of Malvern Panalytical). In between the remarks of the speakers, the audience enjoyed traditional Croatian *a cappella* singing performed by a local *klapa* "Sebenico“. The opening session ended with an excellent talk on “*Symmetry assisted insights into functional materials*” given by EPDIC17 Young Scientist Awardee Dr Mark Senn from the University of Warwick, UK. After the formal opening, participants were invited to continue the evening in an informal get-together with delicious Dalmatian prosciutto and cheese, a selection of Croatian wines and a programme of traditional music.



Figure 2. EPDIC17 Young Scientist Awardee Dr Mark Senn (left) and Stjepan Prugovečki from Malvern Panalytical.

The programme on Wednesday morning started with a plenary lecture "*Electrical Battery Development with the Help of Powder Diffraction*" given by Professor R. Černý (University of Geneva, Switzerland) who presented the use of metal hydrides in battery technology. After the lecture, parallel microsymposia were held during the day; *Total scattering studies and disorder* and *Structural biology and pharmaceutical materials* in the forenoon and *Rational design of materials for energy conversion and storage* and *Line profile analysis* in the afternoon. All the sessions were filled with interesting keynote talks and oral contributions interlaced with vivid discussions. The final part of Wednesday's programme was devoted to the poster session, discussion with the exhibitors in the Exhibition area and the online plenary lecture "*Tuning Chemical Short Range Order in the Solid State*" given by Professor K. Page (University of Tennessee, SAD), who presented current challenges and future opportunities in investigations of short-range order in various functional materials. The second day of the Conference ended with a student mixer where young participants mingled, networked and, simply, had lots of fun. This event was the organizer's present to the young members of the crystallographic community.



Figure 3. Plenary lecturer Professor Radovan Černý.

The next day commenced with the plenary lecture "*Exploring layered solids over broad length scales*" in which Professor S. T. Misture (Alfred University, USA) presented the most recent approaches and current developments in using total scattering and pair distribution functions in ambient and *in-situ* study of materials and devices. After the lecture, parallel microsymposia were held; *Functional materials: structure and properties* and *Stress-strain, texture and thin film analysis* in the forenoon and *Non ambient in-situ and in-operando studies* and *Progress in structure solution and refinement* in the afternoon. Again, they were packed with cutting-edge keynote talks and interesting oral contributions, as well as vivid discussions. In the afternoon, the poster session continued to gather participants for the presentation of the research results and discussions, after which followed the outstanding plenary talk "*The nanostructure inverse problem in the time of artificial intelligence*" given by EPDIC17 Distinguished Powder Diffractionist Awardee Professor S. J. L. Billinge (Columbia University and Brookhaven National Laboratory, USA). The evening of the conference's second day was reserved for a trip to Šibenik city centre and the enchanting soirée musicale by choir "*Kolo*", one of the oldest and most successful amateur cultural associations in Croatia, who presented a potpourri of Croatian composers' opera, folklore and classic pieces.



Figure 4. The EPDIC17 Exhibition area (left) and discussion at the poster session.

The last day of the Conference started with a plenary lecture on the "*The history, development, highlights and future possibilities for synchrotron X-ray powder diffraction*" delivered by Professor R. Cernik (University of Manchester, UK) after which the parallel microsymposia took place: *Nanomaterials: Structural, microstructural and surface aspects* and *Magnetic structures and neutron scattering* in the forenoon and *Utilization of complementary techniques for structural studies and imaging* and *Advances in powder diffraction instrumentation at large facilities* in the afternoon. The scientific programme ended with two plenary talks: "*Multivariate control, diffraction and imaging of crystalline matter*" by Professor P. Radaelli (University of Oxford, UK) and "*Linking Powder Diffraction to Art and Conservation: A synergy story*" by Professor R. E. Dinnebier (University of Stuttgart, Germany). After the last plenary lecture, the EPDIC17 was officially closed with an entertaining presentation "*EPDIC17 in all kinds of numbers*" given by Dr Zoran Štefanić (MyEPDIC online registration system creator and developer); the distribution of the nine best poster awards, which was charmingly chaired by Dr Ana Šantić and Dr Martina Vrankić; and with closing remarks by Professor Paolo Scardi who thanked the organizers and participants for such a successful conference and announced the next EPDIC edition, which will be held in 2024 in Padua, Italy. The intense day ended with an elegant yet relaxed gala dinner.

Best poster awards

The poster session contained 147 presentations which were displayed during the entire duration of the Conference and the strict juries had a difficult task to select best posters in not less than nine categories.

The best poster award by Anton Paar for non-ambient powder/thin film XRD (high/low temperature, pressure, humidity) research was given to Gustav Ek for his work on "*In-situ investigation into the hydrogen sorption pathways of high entropy alloy based metal hydrides*".

The best poster award by Bruker AXS for the best work on microstructure analysis/structure determination/structure refinement from laboratory data was given to Paolo P. Mazzeo for his presentation entitled "*Changing the game of time resolved X-ray diffraction on the mechanochemistry playground by downsizing*".

The best poster award by the Croatian Association of Crystallographers for the most creative concept, ingenious, innovative, never before seen visualization of research results was given to Fanny Costa for her research on "*Challenges in Pair Distribution Function generation from synchrotron-XRPD data of pharmaceuticals*".

The "Pushing the Limits of XRPD Best Poster Award" by Excelsus for research related to pharmaceutical or organic compound investigations was given to Lucas B. M. Pinheiro for his contribution entitled "*Co-milling of ritonavir and lopinavir reveals different polymorphic behaviour*".

The best poster award by ICDD for XRD-related research with the emphasis on phase identification was given to Bojana Simović for her study of "*Structural transformation from titania nanoparticles to sodium titanate nanosheet exhibiting sensing properties*".

The “Paving the way towards sustainable future Award” by Malvern Panalytical for the best poster on energy storage and battery materials was awarded to Morten Johansen for his investigation of “*Electrochemical driven disordering of monoclinic distorted VO_2 as cathode in Li-ion batteries*”.

The best poster award by PROTO for the most innovative use of XRPD instrumentation in research was given to Michela La Bella for her “*In-situ investigations of phase transition processes in the calcium sulphate system*”.

The best poster award by Rigaku for the new method to analyse advanced materials by use of XRD diffraction was given to Marta Ross for her study entitled “*SDPD-SX: a new methodology for data collection for crystal structure determination from powders*”.

The poster award for junior scientists by STOE for the most innovative research results using laboratory powder diffraction as main analysis method was received by Alena Shlyaykher for her research on the “*Synthesis of NH_4SeCN and study of its thermal behaviour by VT-PXRD*”.



Figure 5. Best poster awardees and representatives: Fanny Costa and Aleksandar Višnjevac from the Croatian Association of Crystallographers (left) and Bojana Simović and Tom Blanton from ICDD (right).

Overall, EPDIC17 was a very successful conference with highly motivated participants, enthusiastic lecturers, a vibrant atmosphere and, as was expressed in much of the feedback received from exhibitors and participants, brilliant organization.

NOTICE

News from CCDC



CSD data and software updates

The Cambridge Structural Database, CSD, was updated with 15,450 new experimentally determined structures in June 2022. This brings the total number of entries in the CSD to almost 1.2 million.

The Cambridge Crystallographic Data Centre, CCDC, which manages the database, is grateful to the crystallography community who continue to publish data into this collaborative resource, which goes on to aid scientific research around the world.

As well as new data, new software features have been released over 2022, including a new 3D visualizer in the browser-based *WebCSD*. This brings improved performance — particularly for larger structures. It also allows hit highlighting; when you search for a substructure, the matching part of the structure is now highlighted in the viewer.

See the latest updates and learn how to install them on the CCDC website here.

2022 CSD Statistics available now

Each year the CCDC publishes annual statistics on the contents of the CSD. These include records of authors cited in 500 or more CSD entries, space group number ordering and frequency, crystal system statistics, *R*-factor statistics, space group, publication year and journal statistics.

The 2022 CSD statistics are available on the CCDC website here.

Online learning: how to use the CSD Python API

The CSD Python API allows for programmatic access to the CSD, for search and analysis. The CCDC has just published a short, free, online learning course to learn how to use it.

The course covers how CSD entries are represented in the CSD Python APi, how to access entries, how to read different file formats and how to run a search and output results.

Find out more and access the free online course here.

Meet the CCDC team

ACA annual meeting, 29 July to 3 August 2022

CCDC is very excited to exhibit and present at ACA this year. You will meet our team at the following sessions:

Computer-Aided Materials Design & Development

Speaker: Susan Reutzel-Edens, Head of Science at CCDC

Date: Sunday, 31 July

Time: 2:00 pm - 5:00 pm PDT (10:00 pm - 1:00 am BST)

The Future Of Past Data For Single xtal, Cryo-EM, Powder & Micro-ED

Speaker: Lee Daniels, Business Development Executive at CCDC

Date: Sunday, 31 July

Time: 2:00 pm - 5:00 pm PDT (10:00 pm - 1:00 am BST)

Open Exchanges In Crystallographic Education

Speaker: Yinka Olatunji-Ojo, User Support Scientist at CCDC

Date: Monday, 1 August

Time: 8:30 am - 11:30 am PDT (4:30 pm - 7:30 pm BST)

Learn more and register here

33rd European Crystallographic Meeting, 23–27 August 2022

CCDC is presenting a workshop at ECM33 for researchers to learn how to better leverage the CSD in their research.

Title: Cambridge Structural Database for structural chemists and biologists

Date: Tuesday, 23 August

Time: 09:00–16:00 CEST (08:00–15:00 BST)

Learn more and register here

We will also be exhibiting on stand 19.

HISTORY OF CRYSTALLOGRAPHY

Ernest O. Wollan: an unsung hero of crystallography



Ernest O. Wollan (kneeling) and Clifford G. Shull at the double-crystal neutron spectrometer installed at the Oak Ridge graphite reactor in 1949 (courtesy of Oak Ridge National Laboratory).

Istvan Hargittai, Balazs Hargittai

Ernest O. Wollan (1902–1984) was a native of Glenwood, MN, USA. He earned his PhD under Arthur H. Compton from the University of Chicago in 1929. Compton discovered the effect named after him, the Compton effect, for which he received the Nobel Prize in 1927. In his Nobel lecture, Compton stressed that X-rays produce phenomena that had already been known for light. He also predicted the discovery of further phenomena connected with radiation. His prediction proved prophetic in view of the subsequent discoveries of electron diffraction and neutron diffraction. Following his PhD, Wollan spent the next dozen years teaching and doing research at North Dakota State College; Washington University in St. Louis; Zürich, Switzerland; and the Chicago Tumor Institute.

In 1942, Compton and Enrico Fermi invited Wollan to the Metallurgical Laboratory at the University of Chicago, which was part of the American war efforts. His task was measuring radiation exposure, and he developed the film-badge dosimeter. He was present when the Chicago Pile (nuclear reactor) went operational on 2 December 1942. Soon after, he moved to the Clinton Laboratories in Oak Ridge (now the Oak Ridge National Laboratory) and was present when the Clinton Pile went operational on 4 November 1943. Wollan started neutron scattering experiments in 1944 using the Clinton Pile, later called the Graphite Reactor. He added a spectrometer to the Graphite Reactor for measuring coherent neutron scattering of various atoms. Soon, he and chemist Lyle Benjamin Borst recorded the neutron diffraction patterns of some crystal samples.



Ernest Wollan (left) with Arthur Compton in the late 1930s at the University of Chicago (courtesy of the US Department of Energy).

When in 1946, another physicist, Clifford G. Shull (1915–2001), visited the Clinton Laboratories, Wollan showed him the latest neutron scattering data that he and his colleague, R. B. Sawyer, had collected. Thus, Wollan and Shull's interactions began. Shull soon moved to Clinton Laboratories, where they jointly analyzed the intricacies of neutron scattering and made pioneering observations. Their cooperation led to the invention of neutron crystallography, whose main advantage over X-ray crystallography is the possibility of accurately determining the positions of light atoms, such as hydrogen, in crystal and molecular structures. Its main limitation is that it necessitates having access to a neutron source.

At the time of their initial work, Eugene P. Wigner was the director of research and development of Clinton Laboratories. When the two innovators ran into difficulties, they turned to Wigner, who encouraged them to continue the work. They later remembered Wigner's words, "Maybe there is something new here". Wigner was not yet a Nobel laureate, but he was already 'Wigner', a tremendous authority in science. He was appointed director in 1946 and left the position in 1947 to return to Princeton, but he continued as a consultant at Clinton for many years.



Eugene Wigner and Ernest Wollan (right) in Wigner's office at the Clinton Laboratories (courtesy of Oak Ridge National Laboratory).

In one of their first joint publications, Wollan and Shull declared that "The new field of neutron diffraction, which has been made possible by chain-reacting piles, promises to be a useful partner to the above fields (X-ray diffraction and electron diffraction) in molecular and crystal structure studies." [1] It is worth referring to a few more ground-breaking publications from the Wollan–Shull joint work [2–4]. Their cooperation lasted for a decade until, in 1955, Shull moved to academia and became a professor at the Massachusetts Institute of Technology. Wollan continued as a researcher at Oak Ridge and retired in 1967, consulting though for another ten years at the Oak Ridge National Laboratory. He spent his last years back in Minnesota, and he died there, but is buried in the Oak Ridge Memorial Park. As for his honors and awards, Wollan Island in Antarctica is named after him in recognition of his work on the structure of ice using neutron diffraction. Concordia College, where he earned his bachelor's degree in 1923, awarded him an honorary doctorate in 1965, and in 1967 he received the John Price Wetherill Medal of the Franklin Institute.

Clifford Shull went on to have a brilliant career and enhanced the knowledge of and applicability of neutron scattering. He did much to disseminate the information about neutron crystallography and lectured about it extensively. In the mid-1970s the reception of this technique was still rather mixed. It was a most uplifting episode when, in one of his talks at the time, Shull showed the first Laue pattern using neutron diffraction, and Max von Laue was present among the audience. Upon the presentation, "von Laue's response was simply to come up to Cliff and give him a big hug."[5,6] (this was recorded many years later by Shull's fellow MIT professor Laszlo Tisza in his congratulatory letter to Shull in October 1994).

Shull was awarded the prestigious Gregori Aminoff Prize of the Royal Swedish Academy of Sciences in 1993 for the development of the neutron diffraction technique. In 1994, he received the Nobel Prize in Physics "for the development of the neutron diffraction technique." It was half of the prize, and the other

half went to Bertram N. Brockhouse “for the discovery of neutron spectroscopy.” Both of Shull’s prizes came when Wollan had been dead for many years, and posthumous recognition being precluded, his name could not figure as an awardee, even symbolically. In the introductory presentation at the award-giving ceremony, the Swedish academician did not mention Wollan’s contribution to the prize-winning achievements. However, Shull eloquently remembered it both in his lecture and in his autobiography in the volume of the Nobel Prize materials. He expressed regret that Wollan’s demise ten years before precluded him from sharing the prize, which he would have amply deserved.

The Nobel Prize is a watershed in that the recipients are remembered conspicuously, whereas those who might have contributed to the prize-winning work in equal proportion but do not appear in the Nobel roster may disappear into oblivion. Ernest O. Wollan, just as Clifford G. Shull, deserves to be remembered among the pioneers of modern crystallography and the science of structures.

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HISTORY OF CRYSTALLOGRAPHY

Georg (Yuri) Viktorovich Wulff



Figure 1. Georg (Yuri) Viktorovich Wulff. Photo from the "Scientific Heritage of Russia" Electronic Library.

Larissa Zasurskaya

Science, like art, requires creativity,
but creativity without love is impossible.

– G. V. Wulff

In 2023, it will be 160 years since the birth of Georg (Yuri) Viktorovich Wulff (Вульф in Russian, also known as Vulf and Wulff) (Fig. 1). He was born on 22 June 1863 to a large family in the city of Chernigov (according to other sources, in the city of Nizhyn, Chernigov province) of the Russian Empire (now Ukraine). His mother, Lydia Egorovna, was a daughter of a famous teacher, E. V. Gudim, who came from the nobility of the Poltava province. His father, a teacher of literature, Viktor Konstantinovich Vulf, served as the director of the Chernigov, later of Poltava and 6th Warsaw, Gymnasiums. Georg spent his childhood and youth in Warsaw, where in 1880 he graduated from the 6th Warsaw Gymnasium. From childhood, he showed interest in exact disciplines.

After graduating from the gymnasium, Georg entered the Natural Department of the Faculty of Physics and Mathematics of the Imperial Warsaw University (Poland was then part of the Russian Empire). From the second year, he began to engage in scientific work under the guidance of a mineralogist and crystallographer, Professor A. E. Lagorio, and physicists Professor N. G. Egorov and Professor P. A. Zilov. Professor Egorov took the mineralogist student into the world of physics, showing that there are properties of crystals that are reflected in their external forms. The main focus of Wulff's studies was on physics and crystallography, which was then considered a branch of mineralogy. Being just a third-year student, he was awarded the gold medal of the Faculty of Physics and Mathematics for his work “Experimental Study of the Electrical Properties of Quartz”, the topic of which was proposed by Professor Zilov. In this experimental work, this talented student managed to show that the electrification of quartz crystals is associated with a piezoelectric effect. The work was published in the periodical *Варшавские университетские известия* (*Warsaw University News*) in 1886. In his fourth year, Wulff was already assisting in lectures on physics by Professor Zilov, who taught physics at the highest methodological level.

After graduating from the University in 1885, on the recommendation of Professor Lagorio, Wulff remained at the University at the Department of Mineralogy to prepare for professorship and, at the same time, was enrolled as an assistant at the Department of Physics. With Professor Lagorio, he studied the heat capacity of minerals, and under the guidance of Professor Zilov, he performed several studies on the dependence of the optical properties of crystals on their crystallographic characteristics. The results

of the study of crystals that rotate the plane of polarization were summarized in his 1888 paper "On the Theory of Rotatory Polarization". The study of the physical, primarily optical, properties of crystals led him to create his own direction in science, which was later called "crystal physics". In many cases, experimental studies by G. V. Wulff can rightfully be considered to be pioneering. His contribution to the development of solid-state physics can be assessed from the book by Academician V. D. Kuznetsov, *Solid State Physics* (1937).

After the master's exam in 1888, Wulff did his best to be sent to St. Petersburg University, where he worked for six months in the Mineralogical Cabinet. At that time, an outstanding, now world-famous Russian crystallographer E. S. Fedorov, author of the monograph "The Foundation of the Teachings of Figures", in which he laid the foundations of theoretical crystallography and derived all possible 230 space groups of symmetry of crystals, also worked there. Wulff highly appreciated E. S. Fedorov's achievements and later, with Professor P. von Groth, contributed to the popularization and publication of his works. For example, Wulff published abstracts of Fedorov's main works in German in *Zeitschrift für Krystallographie und Mineralogie*, of which von Groth was the founder and an editor, trying to establish the priority of the Russian scientist in the field of theoretical crystallography. Communication with Fedorov influenced his formation as a crystallographer. There he also met V. I. Vernadsky, whom he later found to be a like-minded person, in all respects, not just in science and teaching, but also in political and public issues.

At the beginning of 1889, Wulff was sent abroad, first to Munich, to von Groth, a famous crystallographer and representative of the German crystallographic school, in whose laboratory he laid the foundations of his master's thesis. In addition to working in von Groth's laboratory, Wulff listened to his lectures on crystal physics and attended Professor Zonke's seminars in physics at the Polytechnic School. Studying crystals of salts, he concluded that it is possible to reveal the internal structure of a crystal by correlating its external form and symmetry with physical, primarily optical, properties. In crystals of beryllium sulfate, he discovered a composition of two pseudosymmetric plates, and in a double salt, lithium potassium sulfate, he revealed the rotation of the polarization plane.

Then, in 1890 and 1891, Georg Viktorovich worked in Paris at the laboratory of the famous French physicist Professor A. Cornu, where he studied physical methods for studying crystals, in particular, the elasticity of solid bodies, primarily the properties of glass. Wulff discovered that the Poisson ratio of these materials was not 0.25, as was found by Cornu, but 0.23, which means that glass does not have the properties of an isotropic body. While they tried to find out the cause, V. Foygt also obtained the value of 0.23, which meant that Wulff could not publish his numerous experimental data. Wulf published the results of his work in Paris later, only in 1893–1894, in *Warsaw University News*. In Paris, Wulff married Vera Vasilyevna Yakunchikova (she took his family name), and together they lived a long life.

Returning to Warsaw, Georg Wulff defended his master's thesis "The Properties of Some Pseudosymmetric Crystals in Connection with the Theory of the Crystalline Structure of the Substance" (1892). Wulff was one of the first crystallographers who, when trying to solve the problem of the structure of crystals, began to use their physical properties, such as optical activity and piezoelectric and elastic properties. Having received the title of Privatdozent of Warsaw University (1893), Wulff began to give lectures on mineralogy and crystallography.

At the same time, Wulff turned to the experimental side of crystallography. For two years, he was engaged in experiments on the growth and dissolution of crystals. He measured relative growth rates of various crystalline faces, building up layers of one crystal upon the layers of another, composed of an isomorphic substance but of different color. Wulff also studied the effect of concentration flows on the morphology of growing crystals. To eliminate irregularities in the growth of crystals, he invented a rotating crystallizer, used thermostats with temperature controllers, and developed a method for obtaining crystals of regular habit. Critical and interesting studies of the growth rate and dissolution of the faces of crystals were carried out by Wulff on the example of Mohr's salt. He showed that the rate of dissolution of various faces of crystals of this salt is almost the same, while the growth rates of these

faces significantly differ from each other.

Studying the processes of crystal growth, Wulff modified Curie's principle of the minimum of the surface energy of crystals growing from saturated solutions in equilibrium conditions. It was later called the "Gibbs–Curie–Wulff principle" (or "Wulff Theorem"), according to which the growth rate of the faces of the crystal is proportional to specific surface energies of these faces. The essence of the Wulff theorem can be determined by the following expression: $n_1:n_2:n_3:... = k_1:k_2:k_3:..$, where n_i are the lengths of perpendiculars (Wulff's vectors), directed through the faces from the central point taken inside the crystal (Wulff's point), and k_i is the specific surface energy of the face (capillary constants of faces). Thus, normals to the faces are proportional to the capillary constant and express the growth rate of the crystal's faces under constant concentration conditions. At each moment of such stationary growth, the surface energy of the crystal has the lowest value corresponding to the volume that the crystal has at this moment. That is, during the growth of the crystal, provided that constant concentrations are maintained, its faces move away from the growth point of the crystal (Wulff's point) with speeds equal to the Wulff's vectors, which are proportional to the capillary constants.

Wulff published the results of his two-year-long studies in 1895 in *Warsaw University News* in the work “On the Issue of Growth and Dissolution of Crystalline Faces”, which he presented at the Physics and Mathematics Faculty as a dissertation for the degree of Doctor of Mineralogy and Geognosy. In this work, the author monitored the correlation of external forms and optical properties of crystals with their internal structure and with crystallization conditions. Wulff's modern biographers call it one of the most significant works on crystallography performed at the University of Warsaw. However, at the University itself, this non-traditional pioneer work was not appreciated and was rejected under the pretext of its insufficient volume. Georg Viktorovich was able to defend a dissertation only a year later at the Imperial Novorossiya University in Odessa (1896). After his article appeared in *Zeitschrift für Kristallographie und Mineralogie*, the theorem on the proportionality of the growth rate of capillary constants of faces was recognized and received the name of the author.

With the advent of two-circle or theodolite goniometers (E. S. Fedorov, 1889), it became easier to measure the angles between crystal faces. E. S. Fedorov was the first to use stereographic projections to present the results of crystal measurements. However, Wulff alone succeeded in developing in 1902 a simple graphical method for processing the results using a stereographic grid. His method turned out to be very convenient; it quickly gained popularity and spread among crystallographers world-wide, and was named after the author – "Wulff net" (Fig. 2).



Figure 2. The Wulff net in the laboratory of crystal chemistry at the Faculty of Chemistry of Moscow State University (photo by the author).

He also proposed the "Wulff ruler" designed by him. Using the Wulff net, you can graphically calculate the symbols of all crystal faces, as well as the crystal constants – axial angles. The Wulff net is still used in crystallography today. In 1904, Wulff published his *Guide to Crystallography*, which is in many ways a synthesis of his work in this area. In geometric crystallography, one of the most important works of Wulff was devoted to the analysis of errors in measuring crystals with a theodolite goniometer.

An interesting feature of teaching at the University of Warsaw was that teachers could declare optional courses that were related to their personal interests. Wulff took advantage of this and, in the 1896/1897 academic year, declared an optional course on the symmetry of crystals for second- and third-year students. In this course, he presented crystallographic symmetry in an original way, using successive reflection only in planes (textbooks by G. V. Wulff *Fundamentals of Crystallography*, 1923, and *Crystals, Their Formation, Type and Structure*, 1926) (Fig. 3).

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Figure 3. Textbooks by G. V. Wulff: *Fundamentals of Crystallography* and *Crystals, Their Formation, Type and Structure* (photo by the author).

Wulff considered planes of symmetry to be the main "independent elements of symmetry", as reflection cannot be reproduced by either rotation or inversion taken separately. He deduced all 32 types of symmetry of crystals, which today we call crystallographic point groups, and came up with his own nomenclature and ways of designating it (Fig. 4).

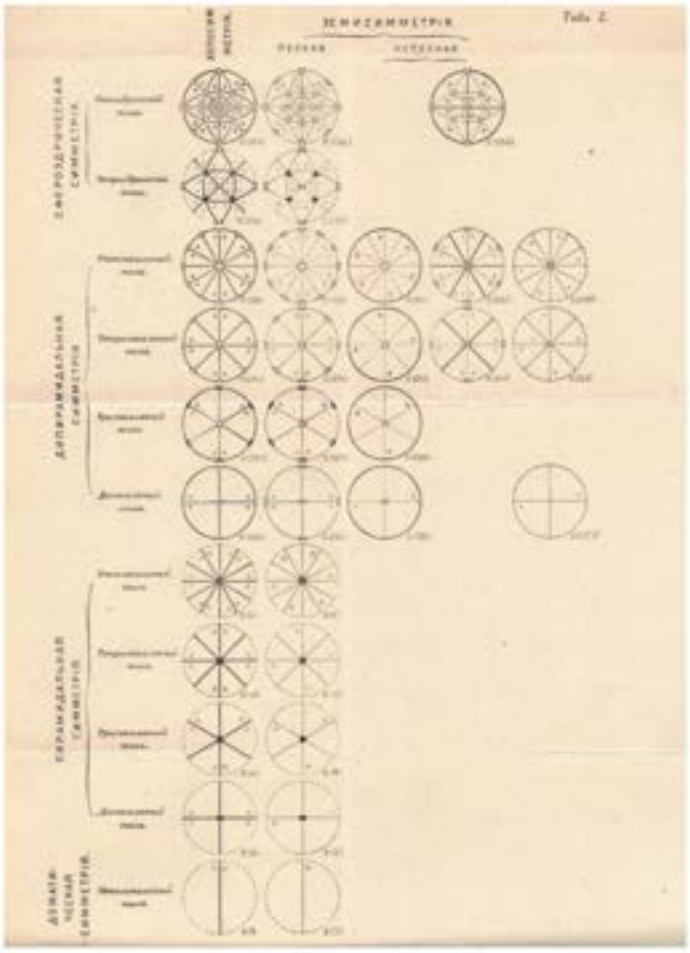


Figure 4. All 32 types of crystal symmetry. Table from *Fundamentals of Crystallography* by G. V. Wulff (1923).

Wulff wrote about the wholeness of the concept of symmetry, that symmetry is a consequence of reflection in one or more mirror planes acting either separately, or in pairs (axes are obtained), or in threes (inversion center is obtained). Contemporaries did not accept Wulff's proposals and continued to use the familiar symbols of Schoenflies, and later the international symbols (Hermann–Maugin).

In 1897, Wulff accepted an offer from the Ministry of Education and left to work as an extraordinary professor at the Department of Mineralogy and Crystallography at the Imperial Kazan University. However, after A. E. Lagorio was appointed as director of the Warsaw Polytechnic Institute, Wulff returned to Warsaw in 1899 and held the position of ordinary professor at the Department of Mineralogy.

The revolutionary situation in Russia in 1905 was accompanied by student unrest. In particular, students demanded that teaching at Warsaw University should not be conducted in Russian, but in Polish, the native language of most of the students. Wulff considered these requirements fair and publicly supported the students, which caused the discontent of colleagues and the leadership of the faculty. In connection with student unrest in 1906, the classes were suspended and resumed only in 1908. In this situation, Wulff worked only until he had gained 20 years of experience, which gave him the right to resign the professorship. He decided to finally leave Warsaw University and go to Geneva, where his family then lived. Saying goodbye to students of Warsaw University, Wulff called on them to be selflessly devoted to science: "... *The desire for truth will only be fruitful when you are imbued with love for the truth. Science, like art, requires creativity, and creativity without love is impossible. ... Love the truth and be, above all, humans, in the noble sense of the word* ... ".

In 1907, Wulff moved to Moscow at the invitation of V. I. Vernadsky. In the same year, he was approved as a Privatdozent of the Department of Mineralogy of the Physics and Mathematics Faculty of Moscow University. Since 1908, Wulff also read the crystallography course at Moscow University and at the A.L. Shanyavsky Moscow City People's University. In 1909 and 1910, at Moscow University, Wulff lectured

on crystallography and crystal optics, using his crystallographic net in practical classes. Thanks to the support of Vernadsky, who was the head of the Department, Wulff got the opportunity to found his crystallographic laboratory, which was equipped, at his own expense, in the premises of the Mineralogical Institute. A. V. Shubnikov, who later became an outstanding Russian crystallographer, worked there as a student. At Moscow University, Wulff closely communicated with the famous physicist P. N. Lebedev, and actively participated in the work of colloquia at his physical laboratory, on the basis of which the Moscow Physical Society was organized in 1911. Lebedev was elected the first chairman of the Society (Wulff was elected the chairman of the Society in 1921). At the same time, he taught a course in crystallography at Moscow University and at the A.L. Shanyavsky Moscow City People's University.

At the University, Wulff continued to pursue studies in geometrical crystallography and started a new topic – the study of liquid crystals, about which he made reports at the general meeting of the Russian Physicochemical Society (1908), and at the XII Congress of Russian Natural Scientists and Physicians, in the Physics section (1910).

However, shortly after a strike at Moscow University in 1911, Wulff, because of his independent nature and beliefs, left the University together with a group of progressive professors in protest against the reactionary policy of the Minister of Education, L. A. Casso. Moscow University lost its best professors, including P. N. Lebedev, V. I. Vernadsky, N. A. Umov, N. D. Zelinsky and K. A. Timiryazev.

Wulff went to work at the A.L. Shanyavsky Moscow City People's University, in which he had already given a course of crystallography lectures starting from its founding in 1908. Wulff’s crystallographic laboratory was also transferred there, for which he himself purchased equipment and materials, spending all his university earnings. There, Wulff carried out his experimental work. At the same time he gave lectures on crystallography and mineralogy at Moscow Higher Women's Courses.

In 2022, it will have been 110 years since the fundamental discovery of the diffraction of X-rays by crystals made by Max von Laue, a Privatdozent at the University of Munich, and his colleagues (article in *Nature* in 1912, the 1914 Nobel Prize). Wulff immediately appreciated the value of this discovery. He founded the first X-ray laboratory in Russia and actively took up the study and deciphering of X-ray diffraction by crystals. Based on the experiments of Laue and independently from W. L. Bragg, Wulff theoretically deduced the condition for the interference of X-rays reflected from crystals, known in Russian-speaking literature as the “Wulff–Bragg” or “Bragg-Wulff formula”. In the same 1913, a month earlier, W. L. Bragg published the well-known equation called "Bragg's law":

$2d\sin\theta = n\lambda$

(where d is the interplanar spacing, θ is the glancing angle (or Bragg corner), n is the diffraction order and λ the wavelength).

Studying the history of the development of X-ray diffraction analysis, the Russian historian of chemistry, Doctor of Science A. M. Smolegovsky, noted that the question of the priority of deriving the Bragg–Wulff equation remains unclear. It is known that both scientists made their reports on this topic as early as the end of 1912, and published their work at the beginning of 1913. Brief information on the content of Bragg's report was published in *Nature* in December 1912. In an article dedicated to the 75th anniversary of the discovery of X-ray diffraction, an outstanding scientist in the field of solid-state physics and X-ray diffraction analysis, G. S. Zhdanov, wrote that the formula that was empirically found by Bragg and theoretically established by Wulff can rightly be considered the “Wulff–Bragg formula”.

Professor A. K. Boldyrev (a disciple of E. S. Fedorov) wrote in the textbook on his lectures *Fundamentals of Crystallography* (1926) and in the book *Crystallography* (1934) that “this is an empirical law, derived even earlier by Professor G.V. Wulff on the basis of Laue's experimental data." The theoretical substantiation of this law was derived in different ways – Bragg substantiated it more simply and elegantly than others. Smolegovsky writes that Bragg's student H. Lipson later opposed the use of the expression "Wulff–Bragg Law" in scientific and educational literature. Wulff himself, in the book *Crystals, Their Formation, Form and Structure* (1926, second edition), gives his original derivation of the formula

with different notation and also notes that "this equation, fundamental to the whole phenomenon, was independently derived by W. L. Bragg and the author of this book". Considering that Bragg and Wulff were working independently in this field at the same time, it is fair to say that both scientists deserve to have this law named after them – the "Bragg–Wulff Law". The English version of Wikipedia formulated it as follows: "Bragg's law", "Wulff–Bragg's condition" or "Laue–Bragg interference".

In 1913–1914, father and son Bragg, inspired by Laue's discovery, improved the mathematical apparatus and technique (ionization spectrometer) and devised a standard procedure for determining the structures of crystals. They determined the structures of a number of crystalline substances, such as NaCl, KCl, KBr, ZnS (sphalerite) and diamond. The work was extremely successful and already in 1915 the first monograph by W. H. Bragg and W. L. Bragg was published, in which they described the structures of 33 substances. In the same year, father and son Bragg received the Nobel Prize, but it was awarded only in 1919, after the end of World War I. Lawrence Bragg is still the youngest Nobel laureate to receive this prestigious award for natural science research.

Apparently, because of this success, the name "Bragg's law" is widely used in the world scientific literature for the well-known expression, which allows one to determine the crystal structure from the diffraction pattern, while the name of Professor Wulff remained in the shadows. When translating the Braggs' book *X-rays and the Structure of Crystals* (1916), Wulff highly appreciated the work of his English colleagues. He wrote that, despite the simultaneous derivation of this law by the author of the book and the translator, "this coincidence in no way detracts from the significance of the results obtained by the Braggs, and it is in these results that the interest of the research of these scientists lies."

This is how X-ray diffraction analysis – the main reliable method for determining crystal structures – was born. In 2023, the famous Bragg–Wulff formula, which marked the beginning of a new scientific discipline, X-ray crystallography, will be 110 years old. The accumulation and analysis of data on the structure of crystals led to the creation of the first tables of atomic radii (W. L. Bragg, W. Goldschmidt, L. Pauling), which laid the foundations of a new science – crystal chemistry. The significance of these pioneering works can hardly be overestimated, since subsequently, extensive structural databases were created, which are necessary for the fruitful and efficient work of scientists from various fields.

Professor A. K. Boldyrev wrote about the significance of Laue's discovery, which also applies to the Bragg–Wulff law: "From now on, more than ever before, Crystallography is most intimately connected with Physics and Chemistry, and the further development of this science goes before our eyes under the sign of this connection. And we clearly realize that only in this connection lies the guarantee of its future achievements". Paying tribute to the work of Professor Wulff in the field of properties of crystals, he begins the section "Physical crystallography" with a photograph of Yuri Viktorovich Wulff and the dates of his life (Fig. 5).



Figure 5. A page from the book *Crystallography* by A. K. Boldyrev (1934).

Unfortunately, the successful work of Wulff, initiated in 1913, was interrupted by World War I, and then by the Russian October Revolution. Active scientific activity in Russia was suspended. At the behest of the times, during World War I (1914–1918), Wulff and his staff developed a new method of making X-ray screens for medical purposes. In Wulff's X-ray research laboratory, almost all of the instruments and apparatus were made in the laboratory's workshop, and in 1912–1913 some of the equipment for working with X-rays was made by Wulff himself.

More than once Wulff was forced to abruptly change his difficult life of a Russian scientist in the midst of successful scientific work because of his high civic position. For example, soon after he had equipped a room for his scientific studies at Shanyavsky University, he again had to create everything anew within the walls of Moscow University after the October Revolution. In 1917, Wulff was reinstated as Privatdozent of the Department of Mineralogy at Moscow University, where he transferred his X-ray laboratory. In 1917, Wulff was elected a member of the Council of the Moscow P.N. Lebedev Physical Society, and since 1921 he was its chairman. In 1918 he became Professor of the Department of Mineralogy, Professor of the Department of Physics (1919–1925) and Professor of the Department of Crystallography (1922–1925) of the Faculty of Physics and Mathematics of Moscow University. In 1919, at the initiative of Wulff, the Institute for Physical and Chemical Research of Solid Matter was established at the Supreme Economic Council, in which he became director and head of the Department of Crystal Physics. In 1921, Wulff was elected a corresponding member of the Russian Academy of Science, and as a member of the Russian Academy of Artistic Sciences. In 1922, the Institute of Physics and Crystallography (later the Research Institute of Physics) was established at Moscow University, which was rightfully headed by Wulff.

During years of his creative studies in science, Wulff published more than 150 scientific and popular science works in Russia and abroad. Here are some of his main works: *Crystals, Their Formation, Type and Structure* (1917, 1926), *Life of Crystals* (1918), *Fundamentals of Crystallography* (1923), *Guide to*

Crystallography (1904), *Symmetry and its Manifestation in Nature. Lectures* (1907), P*ractical Course in Geometric Crystallography with a Stereographic Grid* (co-authored with A. V. Shubnikov, 1924).

It should be noted that Wulff, who was a man with broad scientific views, did a lot of pedagogical and educational work. He was the chairman of the Society for the Care of Students in the Tverskoy District of Moscow and the chairman of the Literacy Society in Tarusa, Kaluga province, as well as the chairman of the N.A. Umov Society for the Propagation of Physical Sciences. Wulff's work testifies to his great interest in the history of science. Possessing literary talent and the gift as a popularizer of science, in his books and lectures Wulff spoke about the history and successes of science. His reports were always accompanied by spectacular demonstrations. He contributed to the history of science with works on the work of such scientists as R. J. Haüy (1922), P. N. Lebedev (1912) and Nobel laureates W. H. Bragg and W. L. Bragg (1916).

In lectures on crystallography, Wulff used microprojections, which made it possible to immediately reproduce and visually demonstrate the phenomena that occur during the growth and development of crystals. He taught various courses: "On the Phenomena of Crystallization", "General Course of Crystallography for Mathematicians and Physicists", "The Theory of the External Shape of Crystals", "X-Ray Methods for the Study of Crystals", "Special Issues of Crystallography", "Introduction to Crystallography", "Crystal Physics", "Thermal Analysis as Applied to Mineralogy".

Wulff educated worthy disciples, who followed his trends and traditions in science: most close to him were A. V. Shubnikov (piezoelectricity and crystal optics), E. E. Flint (geometric crystallography) and S. A. Veiberg (crystal growth). Wulff's desire to convince crystallographers that this science (crystallography) is a part of solid-state physics was later confirmed and continued in the work of Academician A. V. Shubnikov. After Wulff's death, he developed his crystal physics direction and the direction associated with the symmetry of crystals. In 1943, the Laboratory of Crystallography, created by A. V. Shubnikov, was transformed into the Institute of Crystallography of the USSR Academy of Sciences, which he headed from the moment of its creation until 1962. And it is no coincidence that the institute now bears the name of Academician A. V. Shubnikov.

It is also known that, in addition to science, Wulff was fond of music and theater, vocals and photography. In the last years of his life, since 1914, Wulff spent every summer with his creative family in Tarusa at the rented suburban dacha "Pesochnoe". It was the dacha where the Tsvetaev family used to live, and where the artist V. E. Borisov-Musatov also lived and worked. This house became not only the study of the scientist but also the center of the cultural life of Tarusa. The Wulffs organized "Musical Subbotniks" at their dacha, giving free concerts of classical and folk music. Wulff himself also took part in these evenings, performing romances to the accompaniment of his wife, Vera (Fig. 6).



Figure 6. Vera Vasilyevna Wulff (Yakunchikov) (1871–1923). Photo, taken in Moscow in the late 1880s, from the *Tretiakov Gallery Magazine*. © Manuscript Department of the Tretyakov Gallery.

The scientist was very musical and had a pleasant baritone, which allowed him to participate in operas that were staged in Polenovo and in the People's House in Tarusa. Thanks to the efforts of the Wulff family, autumn exhibitions of artists were organized. In memory of these amazing and talented people, in November 2016, the Tarusa Museum of Local Lore hosted an express exhibition dedicated to the 145th anniversary of the birth of the artist and musician Vera Wulff and her famous husband Georg Wulff. Vera had instilled in her children a love of the arts. Their elder son, Vladimir, became a professional pianist, and the younger, Boris, an artist. Boris's talents came to fruition when he was about 30, when he found himself abroad after World War I and the October Revolution.

It should be noted that the picturesque town of Tarusa on the banks of the beautiful Oka river has always attracted talented creative people – writers, poets, artists and musicians (K. G. Paustovsky, M. I. Tsvetaeva, V. D. Polenov, S. T. Richter and others).

It is interesting that Professor Wulff himself preferred the name of Georg, Georg (Prof. G.V. Wulff) as the author of his books, but both names of Professor Wulff are used in the literature - Georg and Yuri. According to his will, he was buried in the town of Tarusa above the Oka River (Kaluga Region) next to his wife, Vera (1871–1923). The monument over his final resting place bears the Cyrillic inscription (which reads as): “Fizik-kristallograf, chlen-korrespondent AN SSSR Yurii Viktorovich Vulf (1863–1925)" [Physicist-crystallographer, Corresponding Member of the Academy of Sciences of the USSR, Yuri Viktorovich Wulff (1863–1925)]. Later, their son Vladimir was also buried there (Fig. 7).



Figure 7. Grave of the Wulff family in the cemetery in Tarusa
https://f.otzyv.ru/photoalbum.php?id=14835:33143&p=200#a-3.

A handsome and noble man with a broad outlook, who was in love with science and art, will remain in history with achievements bearing his name and with the good memories of his disciples and followers (Fig. 8).



Figure 8. Ukrainian envelope commemorating the 150th anniversary of G. V. Wulff's birth (photo from Wikipedia).

A lot has been written about Georg Viktorovich Wulff, an outstanding Russian physicist, one of the greatest crystallographers (see references). Most fully and interestingly Wulff's creative life is presented in the book by A. S. Sonin. Nevertheless, there remains an impression that his contribution to the development of science is underestimated. His life coincided with revolutionary events in the world and in science, which changed his life and scientific destiny more than once. Georg Wulff turned out to be one of the outstanding scientists in the constellation of world-renowned scientists such as Fedorov, Groth, Laue and the Braggs. They communicated closely and jointly moved science, making great discoveries for the benefit of science and humankind. In 1924, shortly before his death, Wulff traveled to Germany, England and France, where he spoke with the leading crystallographers M. Laue, W. L. and W. H. Bragg, M. Myers and others, and in Paris he made a report on his work in the field of crystal physics.

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AWARDS AND PRIZES

Jill Trewhella wins 2022 Guinier Prize



The IUCr Guinier Prize, honouring the great scientist and "father" of small-angle scattering, André Guinier, is awarded in recognition of lifetime achievement, a major breakthrough or an outstanding contribution to the field of small-angle scattering (SAS).

Nominations for this year's prize were considered jointly by representatives of the Organizing Committee of the XVIII International Small Angle Scattering Conference (SAS2022) and the IUCr Commission on Small-Angle Scattering, and we are delighted to announce that Jill Trewhella (University of Sydney, Australia) is the recipient of the 2022 Guinier Prize.

In the prize citation, Professor Trewhella's many contributions to this thriving field were recognised as follows:

Prof. Trewhella is a pioneer and acknowledged leader in the application of SAS to problems in structural biology as well as a tireless promoter of the technique in general, and to the structural biology community in particular. During her career, she has published over 180 original research papers, reviews, book chapters, essays, pedagogical articles, and reports. She has served on the editorial boards of the Biophysical Journal, Biochemistry, Biology and Medicine and others. Her leadership in the development of and advocacy for well-defined standards for assessing data quality, model validation, publication and data archiving appropriate for the biosciences field culminated in the publication and wide acceptance of guidelines used by the bioSAS community today. Throughout her stellar career, she has also mentored generations of biomolecular SAS researchers. Prof. Trewhella's achievements, positive influence and strong contributions clearly make her a most deserving recipient of the Guinier Prize.

The award will be formally announced during the opening of SAS2022 in Campinas, SP, Brazil, on 11 September 2022, and Professor Trewhella will present the Guinier Lecture on 15 September.

Professor Trewhella has served on the IUCr Commission on Small-Angle Scattering (2005–2014; Chair 2014–2017) and is a Co-editor of **IUCrJ**.

Previous winners of the Guinier Prize

Year

Recipient

2018

Dmitri Svergun (EMBL, Hamburg, Germany)

2015

Sow-Hsin Chen (MIT, Cambridge, MA, USA)

2012

Otto Glatter (University of Graz, Austria)

2009

Vittorio Luzzati (Centre de Génétique Moléculaire, CNRS, Gif-sur-Yvette, France)

2006

Heinrich B. Stuhrmann (GKSS Forschungszentrum Geesthacht, Germany)

2002

Michael Agamalian (ORNL, Oak Ridge, TN, USA)

AWARDS AND PRIZES

IUCr Poster Prizes at BCA 2022



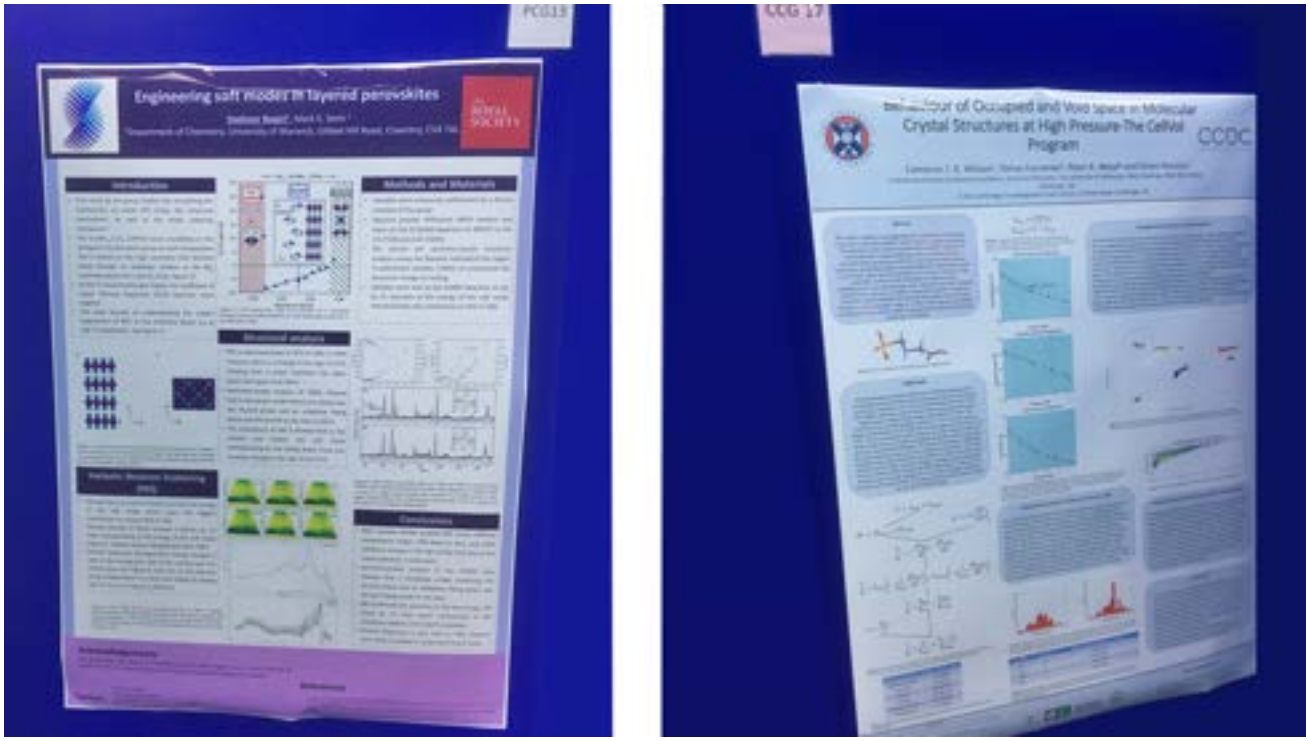
Dashnor Beqiri (left) and Cameron Wilson, winners of the IUCr Poster Prizes at BCA 2022.

John R. Helliwell

There were two poster prizes awarded at the annual conference of the British Crystallographic Association, held at the University of Leeds (UK) just before Easter 2022. The criterion was a “prize to be awarded for the best two posters chosen across any or all branches of crystallography.” The Judging Panel was Professor John Helliwell (University of Manchester, UK), Dr Petra Bombicz (Research Centre for Natural Sciences, Hungarian Academy of Sciences, Budapest, Hungary) and Professor Xiaodong Zou (Stockholm University, Sweden). All the posters were of a very high standard. The winners were as follows:

Dashnor Beqiri of the University of Warwick (Coventry, UK) for his poster with Mark Senn entitled “*Engineering soft modes in layered perovskites*”

Cameron Wilson of the University of Edinburgh (UK) for his poster with Tomas Cervenka, Peter Wood (CCDC) and Simon Parsons entitled “*Behaviour of occupied and void space in molecular crystal structures at high pressure - The CellVol program*”.



The winning posters from Dashnor (left) and Cameron.

OBITUARY

Lev Feigin (1928–2022)



Dmitri Svergun, Jill Trewhella

We are extremely sad to report that Lev Abramovich Feigin, a pioneer in the use of X-ray scattering for the analysis of macromolecular systems, passed away on 15 May 2022 in Moscow at the age of 94.

Lev earned his masters degree, graduating with distinction, from the Physics Faculty of Lomonosov Moscow State University in 1950. He went on to defend his PhD thesis on X-ray diffraction from fine dispersions in 1959 at the Institute of Physical Chemistry of the USSR Academy of Sciences. That same year Lev joined the Laboratory of Protein Structure established by the Academician B. K. Vainshtein at the Institute of Crystallography in Moscow. Here, Lev suggested conducting research on proteins in solution that would complement crystallographic studies by providing structural information relevant to native conditions. This idea led to small-angle X-ray scattering (SAXS) developing at the Institute and it became a main focus for Lev's research activities.

Possessing excellent organizational skills, Lev gathered a bright team of scientists and engineers to work on theoretical and experimental aspects of SAXS as a tool for structural diagnostics of nanosystems. Under his guidance, the first automatic SAXS laboratory diffractometer was built in 1972. In 1975 he was awarded an advanced degree defending his thesis entitled *Small-Angle X-ray Studies of the Structure of Biopolymers in Solution*. He conducted pioneering research to elucidate solution structures of numerous proteins and macromolecular assemblies, including the ribosome and bacterial viruses. He developed and successfully applied heavy-atom labeling with SAXS, analogous to the method used in X-ray crystallography. Lev's group at the Institute of Crystallography was eventually transformed into a SAXS laboratory, which he led for 20 years.

Beyond biomolecular SAXS, Lev oversaw the establishment of a group focused on molecular design based on the Langmuir–Blodgett (LB) method. In 1985, he organized a seminar series 'Langmuir Films and Ensembles of Amphiphilic Molecules' with the participation of prominent international scientists. Lev was a member of the Editorial Board of the journals *Kristallografiya* (*Crystallography Reports*) and *Molecular Materials*, an honorary member of the Hungarian Biophysical Society, and a foreign member of the Regional Academy of Sciences and Arts (Marche region, Italy). He was awarded an International Prize in Nanotechnology (RUSNANOPRIZE) in 2010, the Fedorov Prize of the Russian Academy of Sciences in 2012, and the title 'Honored Scientist of the Russian Federation' for his fruitful scientific activity.

During his career, Lev supervised over 20 PhD theses and published more than 140 peer-reviewed scientific papers. One of the co-authors of this obituary (DS) had the privilege to be a diploma student and later a member of Lev's group in Moscow. Lev was a fantastic supervisor and highly intelligent scientist. He was always open to communication and discussions, especially with younger group members. He considered all questions in depth and provided wise and helpful counsel in the face of

problems of all kinds (scientific, organizational, personal). In 1986, he and DS co-authored a monograph *Structure Analysis by Small-Angle X-ray and Neutron Scattering*, which became an important textbook for the field.

Lev Feigin's passing is a great loss for our community, but we are grateful that his ideas and accomplishments will continue to inspire new generations of scientists working in X-ray scattering and diffraction.

A complete tribute to Lev, in Russian and English, will be published in *Crystallography Reports*.

This obituary was originally published in *Acta Cryst.* (2022). A**78**, 384–385.

OBITUARY

James A. Ibers (1930–2021)



Jim Ibers lecturing at the 20th IUCr Congress in Florence, Italy, in 2005.

Carol Brock, Jim Kaduk

James Arthur Ibers, renowned chemist and crystallographer, died on 14 December 2021 at the age of 91. He was an active scientist to the end; during November, he was still collaborating on scientific papers.

Ibers is an author of more than 870 substantive research publications, 115 of them in IUCr journals. His papers have been cited nearly 46,000 times; his *h* index is 102. His name is linked to 640 CSD refcodes and 723 ICSD entries. He was elected to the U. S. National Academy of Sciences. He received both the American Chemical Society's Award in Inorganic Chemistry and its Award for Distinguished Service in the Advancement of Inorganic Chemistry. He also received the Linus Pauling Medal, the California Institute of Technology Distinguished Alumni Award and the American Crystallographic Association's Martin J. Buerger Award. He trained scores of graduate students and postdocs – more than 175 as of 2002, when many returned for a weekend celebration of his career. His last student graduated in 2014.

Jim, as he insisted on being called by even his most junior graduate students, was born and raised in Los Angeles. He earned his BS in 1951 and his PhD in 1954 from the nearby California Institute of Technology. After a postdoc (1954–1955) with John Cowley in Australia, he worked at the Shell Development Company in Emeryville, CA, for six years (1955–1961), then spent three years at Brookhaven National Lab before moving in 1965 to Northwestern University in Evanston, IL. There he was a professor (later an emeritus professor) for more than 55 years.

As an undergraduate at Caltech, Jim first did research with Norman Davidson. When Davidson's funding became tight, he advised Jim to talk with crystallographer David Shoemaker, whose name he said but did not spell. By chance, Jim first saw the door of Verner Schomaker, who, like Shoemaker, was a former student of Linus Pauling and then his protégé. Jim later considered the mistake to have been lucky; last September he described Schomaker as the most perceptive scientist he had ever known. Jim's first papers concerned electron diffraction: a study of OF_2 with Schomaker and calculations of electron scattering amplitudes with postdoc Jean Hoerni. Jim also calculated X-ray form factors for C, N and O atoms with Hoerni and later for Be, B and Hg while at Shell. In Volume III of the "red series" of *International Tables* (published in 1962) Jim was responsible for Table 3.3.1 (scattering factors for X-rays) and, with Boris Vainshtein, for Table 3.3.3 (scattering amplitudes for electrons).

Jim's first report of the study of a crystal was a sole-author paper published in *Acta Cryst.* in 1953 while he was still a graduate student; the title was *The unit cell and space group of tetrapyridinecopper (II) fluoborate*. The last sentence of that short report is "Because of the size and the complexity of the unit cell, no further work is contemplated on this structure." In 2013 two groups did finally publish the

structure of the $P2_12_12_1$, Z'=1 unit cell Jim had found (refcode family ZZZSWE; 35 non-H atoms).

During his postdoc with John Cowley, Jim used a combination of electron diffraction and powder diffraction to study graphite intercalated with $FeCl_3$. While at Shell, he published work based on vibrational spectroscopy and solid-state NMR studies, and reported a new calculation of the barrier to the inversion of ammonia (325 citations so far).

When corporate changes made conditions at Shell Development difficult, Jim moved to Brookhaven to work with Walter Hamilton, a close friend from graduate school, and with Sam La Placa. An early project was the structure of XeF_4. In his memoir for the ACA's History Portal, Jim wrote:

Shortly after my arrival, at a seminar there was excitement over the report of xenon tetrafluoride, the first stable rare-gas compound. Someone in the back of the seminar room said he had repeated the synthesis following the announcement of its discovery. [One can prepare XeF4 from Xe, F2, and sunlight]. ... The sample of XeF4 started the La Placa, Hamilton, and Ibers adventures in crystallography.

With Walter and/or Sam, or by himself, Jim published more than 20 structures determined while he was at Brookhaven; the three scientists demonstrated that crystal structures of moderate size could be determined, at least by experts, in a semi-routine way. Most data were collected on film but later there was a GE quarter-circle diffractometer that put out numbers but was still far from automatic. Jim, Walter and Sam had to develop methods and procedures as they went along. Jim was later recognized as having compiled an excellent collection of crystallographic software. Most of the large programs (*ORFLS*, *ORFFE*, *FORDAP* and *ORTEP*) had been written by others, but Jim added features (*e.g.* correction for dispersion, group refinement), and integrated the documented routines into a simple, but very effective and reliable, system that he and many others used for decades and that became the basis of two commercial packages.

Neutron data could be collected at Brookhaven so Jim and Walter determined the structures of some simple H-bonded systems. That work led to their 1968 book *Hydrogen Bonding in Solids*. Later they worked together to edit Volume IV of the "red series" of *International Tables*, which was published in 1974.

When Jim moved to Northwestern, he was able to purchase an "automated" Picker four-circle diffractometer. Determination of the matrix relating the crystal and diffractometer coordinate systems still required lots of manual work, but once the orientation matrix was known, data collection was automatic, at least as long as the punched cards generated at the campus computer center, carried across campus and loaded into an IBM keypunch connected to the diffractometer didn't jam.

Jim had the fourth Picker diffractometer manufactured, and it was no black box. He and two of his first postdocs (Peter Corfield and Bob Doedens) had to devise procedures and write programs. The description of data collection in their 1967 paper in *Inorg. Chem.* (**2**, 197–204) was so thorough and useful that it was referenced often enough to be designated in 1981 as a "*Citation Classic*". It is notable that the reported methodology, like that for least-squares group refinement [*Acta Cryst*. (1965). **18**, 511–519], is somewhat buried within a paper that focusses on the discussion of the structure of a chemically important compound.

Jim's longstanding interests in coordination chemistry and organometallic chemistry fit well with those of Professors Fred Basolo and Ralph Pearson (and later Tobin Marks) at Northwestern, and during Jim's early years at NU the field of inorganic chemistry, especially structural inorganic chemistry, was "hot". Jim made important contributions in many areas: metal hydrides; five-coordinate compounds; coordination complexes of N_2, O_2 and NO ligands; *M*-S (especially Fe-S) clusters; porphyrins, phthalocyanines and related molecules; organometallics.

Jim's stature as an inorganic chemist meant that journals wanted him on their editorial boards. During his long service (1965–1978) on the editorial board of *Inorg. Chem*. he played a major role in setting the standards for the publication of crystallographic work. Other boards included those of *Inorg. Chim. Acta*,

J. Organomet. Chem., *Organometallics*, *J. Am. Chem. Soc.*, and later *J. Solid State Chem.*, *Eur. J. Solid State Inorg. Chem*. and *J. Porph. Phthal*. He was also a longtime editor of the series *Structure and Bonding*.

After a sabbatical year at Caltech (1981–1982) Jim shifted his focus to the synthesis and characterization of chalcogenides (compounds containing S, Se or Te), and chemical syntheses became a much larger part of his research program. Earlier projects continued, especially in collaboration with other Northwestern faculty, but the chalcogenide project became central.

While crystallography continued to be the backbone of Jim's research, he would not start data collection unless the new solid-state compound, which had almost always been synthesized in his lab, had been characterized by complementary methods. Reactions of solids were carried out at high temperature (>600°C) and under vacuum to avoid oxide formation. Physical characterization included elemental analysis, resistivity, optical absorption and magnetic measurements. Powder X-ray diffraction was used to evaluate purity. Single-crystal X-ray diffraction then gave the details of the structure.



The work progressed from binary to ternary to quaternary chalcogenides. In the last decades Jim became known for his studies of actinide chalcogenides. The pace was such that there were many results to write up even after 2015, when he no longer had laboratory space for active synthetic work. So he wrote. As always, the language in his papers was laconic, direct and data-driven. He invariably changed "due to" to "owing to" and "compared with" to "compared to".

Late in his career Jim worked with complex crystal structures that required non-standard crystallographic techniques, such as multi-dimensional crystallography, for their refinement. Many of the compounds Jim's group synthesized were found to be modulated with either commensurate or incommensurate supercells as a result of positional or occupational periodic distortions.

Among crystallographers Jim sometimes referred to himself as the Practicing Crystallographic Idiot, which he most certainly was not. In his ACA memoir he claimed that in that role and at a 1962 meeting of a

small group planning the space-group volume of *International Tables*, he had saved the **b**-unique setting of monoclinic unit cells from being left out by physicists who preferred **c**. He also claimed to have insisted that the information necessary to define the unique part of a unit cell be included with the space-group diagrams. Including that information was intended to prevent the measuring of half the data twice back in the days when collecting even just the unique reflections could take weeks, but sometimes half the data got measured twice anyway, even at Northwestern.

Classes taught by Jim were never dull because of his dry wit and somewhat quirky sense of humor. He once gave a class of non-science majors averages for the length, width and depth of Lake Michigan, the volume of a fish, the number of fish per cubic yard of lake and the densities of lake water and of the fish, and then asked the students to calculate the weight of Lake Michigan with and without the fish. That assignment kept the teaching assistants for the class quite busy.

Jim had a long and happy marriage with Joyce (*née* Joyce Audrey Henderson), whom he had known since their elementary school days. Their two children brought them great joy, and they welcomed a succession of well-loved Persian cats into their homes during their 69 years together. Cultural events, especially concerts, opera and the theatre, were an important part of their lives. They also cooked seriously and entertained with pleasure; members of Jim’s research group and special visitors were treated to memorable dinners and parties at the Ibers’ home.

More information is available in the memoir Jim wrote for the ACA’s History Portal and in the more informal remembrances of 19 former students, postdocs and visitors that appeared in the Spring 2022 issue of the ACA’s newsletter *RefleXions* (pp. 34–46). We thank all whose memories we have used and are particularly grateful to Christos D. Malliakas for information about Jim’s most recent research.

OBITUARY

Mamannamana Vijayan (1941–2022)



S. Mande, R. Sankaranarayanan, D. M. Salunke, T. P. Singh, B. Gopal

Professor Mamannamana Vijayan, born in 1941, passed away on 24 April 2022 after a prolonged degenerative illness. Vijayan was one of India's finest biophysicists who used crystallography to address fundamental problems in molecular biology. Examples of this approach included studies on the supramolecular self-assembly of small molecules in early evolution, the role of water molecules in protein structures, understanding of carbohydrate recognition by plant lectins and protein–DNA interactions. During these research endeavors, he established a vibrant structural biology laboratory at the Indian Institute of Science (IISc), Bangalore, the first in India. Vijayan had previously received his training at this Institute as a PhD student, which was followed by a remarkable stint in the laboratory of Dorothy Hodgkin at the University of Oxford before returning to India.

Vijayan was born to an orthodox family in the southern state of Kerala in India. He was, however, deeply influenced by Marxism during his student days. After his undergraduate education, he briefly explored Marxist political activism. This phase, where he spent some time in Delhi, led to a chance encounter with Achutha Menon, a prominent political leader from Kerala at that time. At the insistence of Menon, he went on to pursue higher education, eventually obtaining a Masters in physics from the famed Allahabad University. His academic journey began with this transformation and led him to complete his PhD at IISc, Bangalore, followed by post-doctoral training under the wings of Dorothy Hodgkin at the University of Oxford. These early influences profoundly affected his career in science and aspects of life that Vijayan himself readily acknowledged in his memoir *A Life Among Men, Women and Molecules*.

In 1971 when Vijayan returned to India and joined the Department of Physics at the Indian Institute of Science, Bangalore, there was no infrastructure to collect or process X-ray crystallographic data on protein crystals. Obtaining proteins in their pure form was also considered challenging. Moreover, the public support system for funding Science was only just being established. Indeed, by his own admission, the establishment of the Department of Science and Technology (DST) under the Ministry of Science and Technology in 1971 was a watershed moment for receiving support for such facilities. Also, as a result of the Bangladesh liberation war, funding remained elusive, and positions were frozen for a few years. Using existing facilities in the Physics department, he initiated a program on structure and function studies of anti-inflammatory, analgesic and antibacterial molecules as well as on amino acid complexes. His group made highly significant contributions in these areas of small-molecule crystallography. Vijayan moved to the newly formed Molecular Biophysics Unit in 1974, where he began to build his macromolecular crystallography laboratory from scratch. His joint research projects with Avadhesha Surolia, a lifelong collaboration, ensured that purified plant lectins were readily available for crystal structure determination. With this, he could pursue his dream of examining protein structures in atomic detail. Yet, collecting crystallographic data on photographic plates, scanning photographs, assessing the intensity of

diffraction spots, computational processing of the data and solving structures remained a huge challenge. He was soon joined by a younger colleague, M. R. N. Murthy. Photographs were scanned at the National Aerospace Laboratories, Bangalore, in Kalyani Vijayan's laboratory, his life-long partner. With Murthy's help, many computer programs were developed to start structure determination activities in true earnest. The first structure determined was that of Peanut lectin, which showed a more remarkable deviation in its quaternary structure than anticipated. Indeed, it formed one of the first examples of an 'open quaternary structure' in the literature, where the homo-tetrameric protein was expected to exhibit the anticipated 222 or D_2 symmetry rather than the association of two dimers in an 'open' manner. Soon, many plant lectin structures were determined. Much of the protein–carbohydrate recognition events began to be understood through a series of these studies on various plant lectin categories.

A chance observation in Vijayan's laboratory demonstrated that protein crystals showed significantly altered diffraction when dehydrated under controlled conditions. This had earlier been observed in studies on haemoglobin, but a systematic study with controlled dehydration was needed. Vijayan and his group undertook studies on many protein crystals under different environment humidity conditions. One startling observation was that of a dramatic improvement in the diffraction of certain crystals, and this occurred alongside abrupt changes in crystal dimensions. This water-mediated transformation was exciting, especially when viewed in the context of crystal structures. These studies led to an appreciation of the critical roles invariant water molecules play in protein structure and function.



While building up a protein crystallography laboratory, Vijayan was also struck by the problem of spontaneous assembly of small molecules in early evolution. This would form an important thread in the transition from the chemical to the biological world. Vijayan hypothesized that the self-assembly of amino acids was an intrinsic property that would propel amide bonds and, consequently facilitate the formation of linear polypeptides. A similar self-assembly in mixed L- and D-peptides would not be as favorable. A series of peptide–peptide complex structures hinted that this might be a natural phenomenon. Only amino acids of the same chirality would associate with each other and condense to form an amide bond; amino acids of opposing chirality would not.

In the late 1990s and the early part of this century, Vijayan started to take an interest in neglected tropical diseases, and he joined the newly established worldwide consortium of 'Tuberculosis Structural Genomics'. In collaboration with many talented young faculty members in the IISc, several structures were rolled out of Vijayan's laboratory in this phase from *Mycobacterium tuberculosis* proteins or its

cousin *Mycobacterium smegmatis*. Inhibitor design of these proteins through structural studies also started as a natural consequence.

Vijayan was engaged with the entire scientific community, having been part of scientific/academic advisory boards of many institutions and committees of funding bodies. He was extremely generous, particularly to those from less endowed organizations. In true spirit, he was an academic father figure to his students and associates, and other younger colleagues who worked closely with him. Over the years, he mentored generations of structural biologists and was a guiding force for biophysics research in India.

Vijayan was a humanitarian *par excellence*. He took a deep interest in the welfare of whosoever came into his sphere of influence. It was not uncommon to find Vijayan in the corridors of the Institute, spending time enquiring about the difficulties of research staff and students. He solved the problems of many people around him, even going beyond those within his own institution, leaving deep impressions on all. The scale did not quite matter, whether it was that students were not receiving fellowships after finishing their tenure or those who had family difficulties, Vijayan would work to solve those difficulties; it required raising money from multiple sources, helping needy families or simply support in the form of solid mentorship. It is often said that good human values percolate in different schools of people, and Dorothy Hodgkin was a prime example. In the true sense, Vijayan had imbibed all the values taught by Dorothy in this respect and had even bettered those by bringing his experiences from childhood or those in his student days. Vijayan will be sorely missed.

An obituary for Professor Vijayan by Ted Baker has recently appeared in *Acta Cryst. D*.

A list of papers published by Professor Vijayan in IUCr journals can be seen here, and a collection of photos from the IUCr gallery here.

APPLICATION NOTE

Optimization of protein crystals obtained with the FUSION screen



Fabrice Gorrec, François-Xavier Robert, Claire Seddon, Simon Tanley

FUSION formulation

The most efficient components of the three MORPHEUS crystallization screens have been combined into a single 96-condition screen called FUSION. FUSION integrates 17 previous MORPHEUS mixes of additives ("LiNaK", "alkalis", "polyamines", *etc.*) and two new mixes ("cryopolyols" and "NDSBs"). Mixes of additives were paired to form 96 unique combinations (see some examples in Table 1). FUSION also integrates the four mixes of precipitants and three buffer systems of the original MORPHEUS screen. All components were systematically sampled to formulate an incomplete factorial. The efficiency of FUSION was demonstrated by producing useful crystallization hits with test samples in multiple FUSION conditions (Fig. 1).

For more details about the FUSION screen, please refer to the original publication: Gorrec & Bellini (2022).

Table 1. Examples of crystallization conditions in the FUSION screen. The eight conditions listed are the ones that produced the crystallization hits shown in Fig. 1. In FUSION, there are 81 conditions prepared with two mixes of additives each, such as C6, D6, D8, F11, G5 and G8 shown here. The 15 other FUSION conditions are prepared with only one mix of additives (*e.g.* H11 and H12), whose concentration is doubled compared with when they are paired with another one. For example, the concentration of the mix “polyamines” is 10 m*M* in F11 whereas it is 20 m*M* in H11.

Well

Mix of precipitants

Buffer system

Mix of additives (set 1)

Mix of additives (set 2)

C6

12.5% *w*/*v* PEG 1000, 12.5% *w*/*v* PEG 3350, 12.5% *v*/*v* MPD

0.1 *M* bicine/Trizma base pH 8.5

30 m*M* of each LiNaK

20 m*M* of each monosaccharide 1

D6

10% *w*/*v* PEG 20 000, 20% *v*/*v* PEG MME 500

0.1 *M* bicine/Trizma base pH 8.5

30 m*M* of each LiNaK

20 m*M* of each monosaccharide 2

D8

10% *w*/*v* PEG 4000, 20% *v*/*v* glycerol

0.1 *M* bicine/Trizma base pH 8.5

1 m*M* of each alkali

20 m*M* of each monosaccharide 2

F11

10% *w*/*v* PEG 4000, 20% *v*/*v* glycerol

0.1 *M* MOPS/HEPES-Na pH 7.5

10 m*M* of each polyamine

0.5% *w*/*v* of each cryopolyol

G5

10% *w*/*v* PEG 8000, 20% *v*/*v* ethylene glycol

0.1 *M* bicine/Trizma base pH 8.5

20 m*M* of each amino acid

0.3% *w*/*v* of each NDSB

G8

10% *w*/*v* PEG 20 000, 20% *v*/*v* PEG MME 500

0.1 *M* MES/imidazole pH 6.5

1 m*M* of each alkali

0.3% *w*/*v* of each NDSB

H11

10% *w*/*v* PEG 20 000, 20% *v*/*v* PEG MME 500

0.1 *M* MES/imidazole pH 6.5

20 m*M* of each polyamine

None

H12

10% *w*/*v* PEG 8000, 20% *v*/*v* ethylene glycol

0.1 *M* MES/imidazole pH 6.5

0.4% *w*/*v* of each anaesthetic alkaloid

None

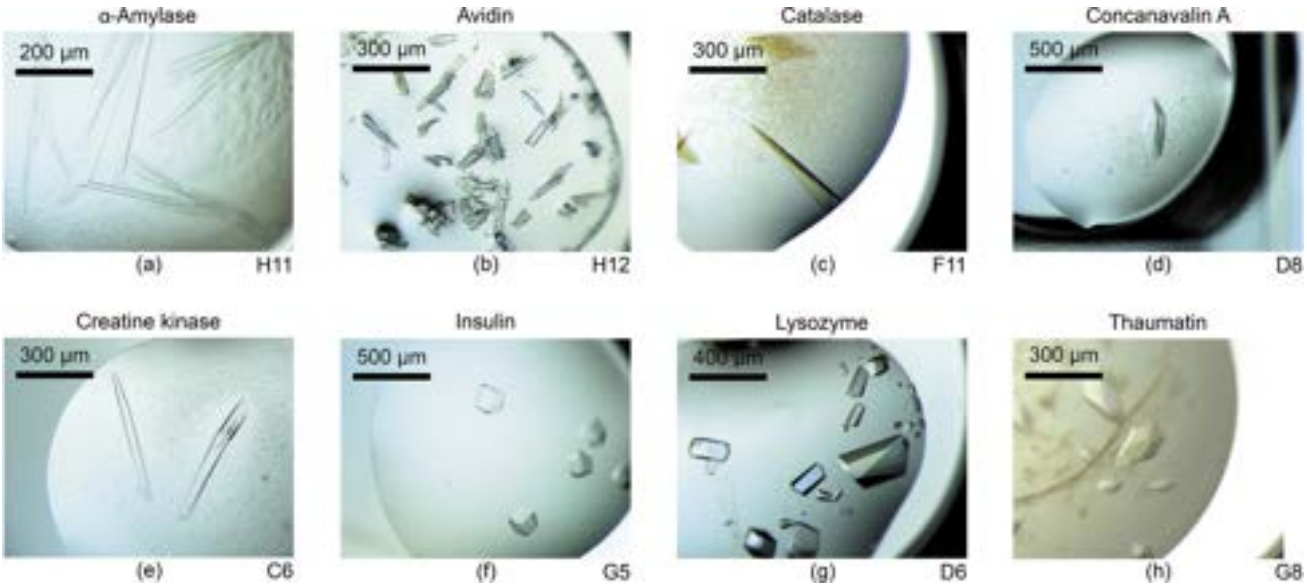


Figure 1. Examples of initial crystallization hits obtained with FUSION. Diffraction-quality crystals were obtained against eight test samples and are shown here: α-amylase (*a*), avidin (*b*), catalase (*c*), concanavalin A (*d*), creatine kinase (*e*), insulin (*f*), lysozyme (*g*) and thaumatin (*h*). The FUSION conditions used are indicated in the bottom right corner of each micrograph.

Optimizing the concentration of the main components

Despite the success of FUSION with test samples, where many diffraction quality crystals could be obtained without further optimization (Fig. 1), in most cases it will be required to optimize crystal growth by setting up modified experiments based on the initial FUSION hits. For this, it is normally required to investigate the impact of varying the concentrations of the main components of the condition that produced that hit, and to observe effects on crystal nucleation and growth.

As with the original MORPHEUS formulation, the simplest way to approach optimization with FUSION is to consider each mix of reagents as a single component. When there are two mixes of additives in a condition, the corresponding stock solutions can also be combined to reduce the number of components and parameters to test and investigate. Because of the systematic nature of the FUSION formulation, the starting ratios between components is always 0.5 mix of precipitants + 0.2 mix of additives from set 1 and/or 2 + 0.1 buffer system + 0.2 water.

At the Laboratory of Molecular Biology, we recommend using the "4-corner solutions" method to produce 2D concentration gradient screens (Fig. 2). The initial step is to prepare 4-corner premixes "A, B, C and D" manually. This is to handle difficult solutions with a relatively large volume range and bypass the liquid-classes programming with automated liquid handling technology. Based on the fixed ratios of stock solutions in FUSION, the formulation of the required 4-corner premixes "A, B, C and D" to vary the concentration of precipitants and additives will typically be as follows:

Solution A: 0.35 mix of precipitants + 0.05 mix of additives from set 1 and 2 + 0.1 buffer system + 0.5 water

Solution B: 0.55 mix of precipitants + 0.05 mix of additives from set 1 and 2 + 0.1 buffer system + 0.3 water

Solution C: 0.35 mix of precipitants + 0.25 mix of additives from set 1 and 2 + 0.1 buffer system + 0.3 water

Solution D: 0.55 mix of precipitants + 0.25 mix of additives from set 1 and 2 + 0.1 buffer system + 0.1 water.

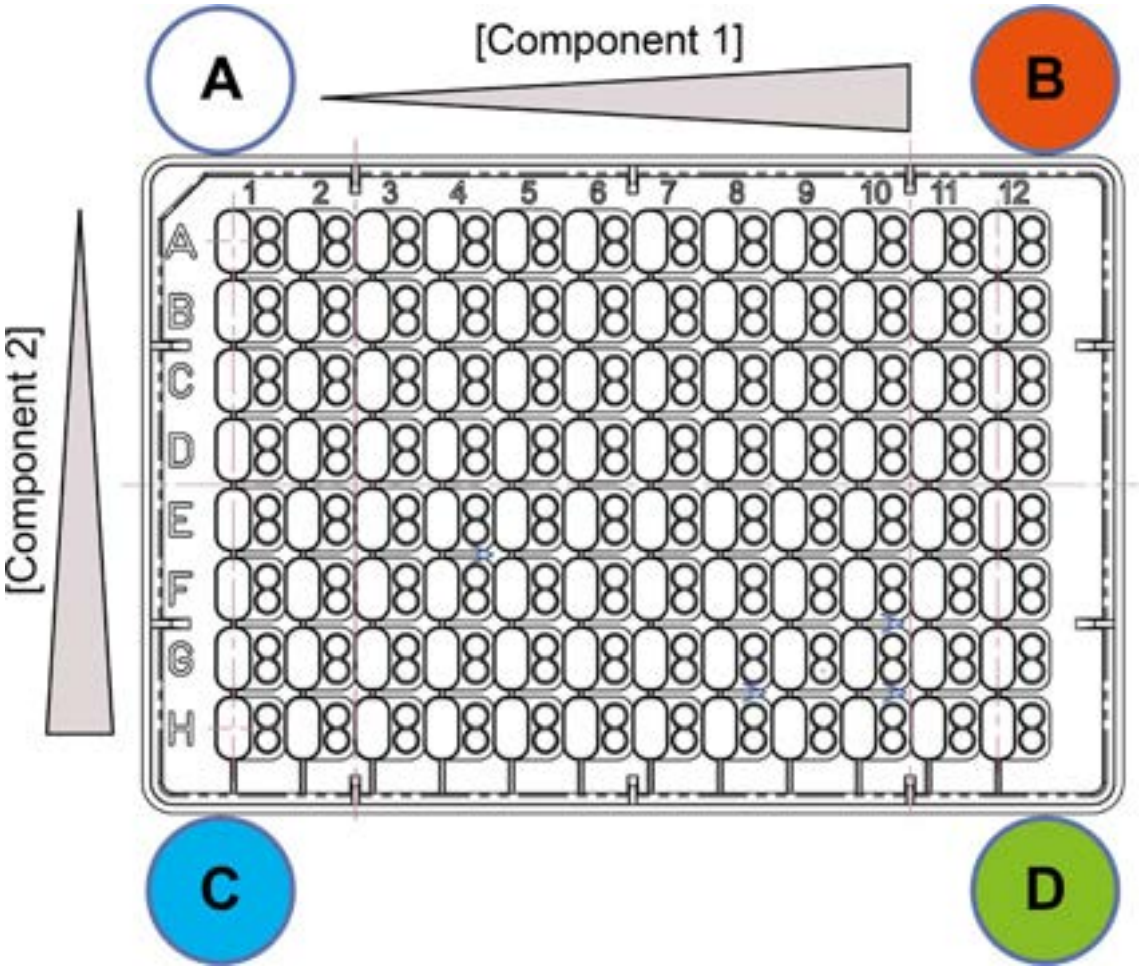


Figure 2. Representation of the 2D linear gradients of concentrations across the standard 96-well plate layout that result from the "4-corner solutions" method. Two linear gradients of concentrations for the two components to be varied are automatically generated by a liquid handler, which dispenses the four solutions "A, B, C and D" at different ratios into the reservoirs of a crystallization plate. Figure adapted from Gorrec & Löwe (2018).

Optimizing the pH

Apart from varying the concentration screen components, the pH is often an important variable that is typically investigated in parallel with the concentrations of precipitants and additives. This can be done by preparing separate 2D concentration gradient screens (Fig. 2), each containing a buffer adjusted to different pH values. Conveniently, the preparation of buffers adjusted to different pH values is made straightforward with the MORPHEUS buffer systems (Table 2).

For more details about the MORPHEUS buffer systems, please refer to the original publication: Gorrec (2009).

Table 2. Recipes for preparing the three MORPHEUS buffer systems as used in the FUSION screen. Buffers are pH-adjusted by mixing 1 *M* stock solutions of acidic and basic buffer molecules. The table lists volumes of the stock solutions needed to yield 50 ml of buffer mix. The buffer mix preparations should be checked with a pH meter. *Please note that a pH meter equipped with a solid-state probe will avoid inaccuracies with Tris-containing buffers, unless explicit corrections are applied when using other probes.*

pH

1 *M* MES (ml)

1 *M* imidazole (ml)

6.1

36.0

14.0

6.3

33.5

16.5

6.5

30.6

19.4

6.7

27.5

22.5

6.9

25

25

pH

1 *M* MOPS (ml)

1 *M* HEPES-Na (ml)

7.1

34.5

15.5

7.3

30

20

7.5

25.9

24.1

7.7

22.1

37.9

7.9

17.7

32.3

pH

1 *M* bicine (ml)

1 *M* TRIS-base (ml)

8.1

35.6

14.4

8.3

31.7

18.3

8.5

26.7

23.3

8.7

21.2

28.8

8.9

15

35

"Biasing experiments" to increase chances during optimization

When an additive mix is revealed as being essential during the optimization of an initial hit condition, we recommend adding the additive mix to the sample and then to screen against other crystallization screens. This "biasing" of the sample often increases the yield of useful crystals and can be particularly helpful when a better crystal form is needed through re-screening [Fig. 3(*a*)]. Conversely, when a precipitant mix is revealed as essential in the initial screen or optimization, a custom-made FUSION screen formulated with a single precipitant mix (instead of the normally used four different ones) can be an efficient approach to additive screening as FUSION combines so many different additives [Fig. 3(*b*)]. All mixes needed to proceed with optimization work are available commercially as ready-made stock solutions, making this task even more straightforward.

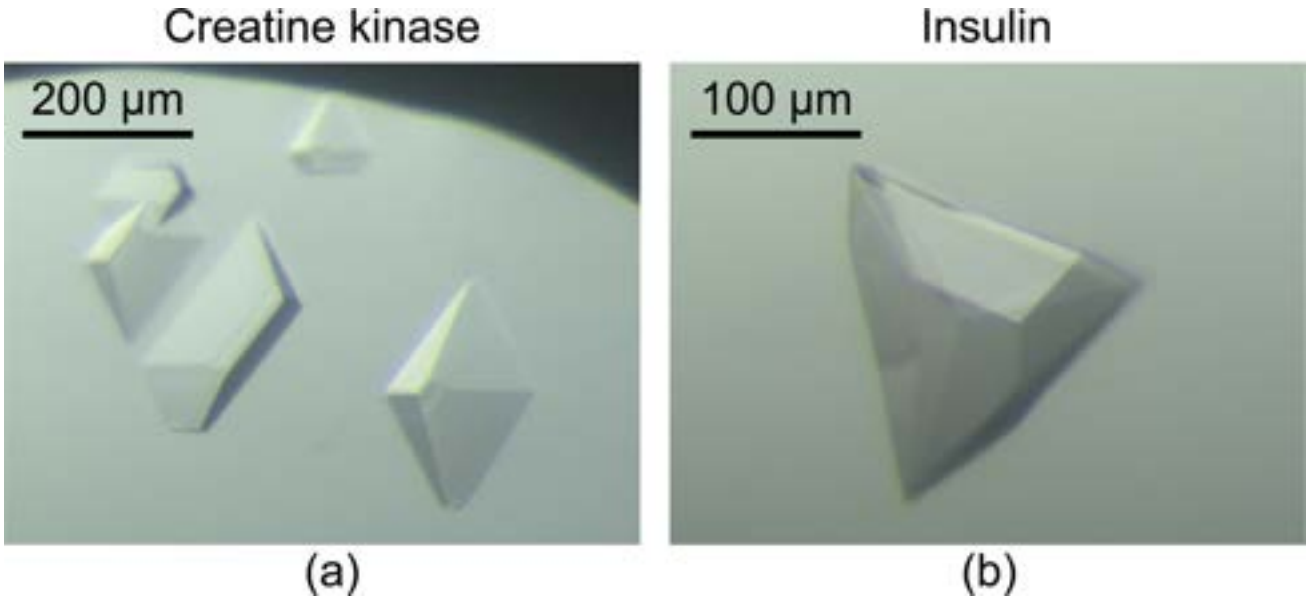


Figure 3. (*a*) Creatine kinase crystals obtained after "biasing" the sample with 10% of the mix of additives "LiNaK". (*b*) Insulin crystal resulting from a custom-made FUSION screen prepared with a single MORPHEUS precipitant mix ("precipitant mix 3" integrating PEG 4000 and glycerol).

This work was supported by the Medical Research Council (MRC), as part of United Kingdom Research and Innovation. FUSION is sold by Molecular Dimensions Ltd as "MORPHEUS® FUSION" under an exclusive licence from the MRC and the medical research charity LifeArc.

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IUCR SUPPORTED EVENTS

IUCr supported meetings

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



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

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